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Technogenic Raw Materials in High-Alumina Chamotte Production

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Abstract. The paper presents studies on the production of a high-alumina aggregate with an Al_2O_3 content of 75% based on fine-grained corundum dust generated by grinding fused corundum. Finely dispersed corundum powder is a by-product and due to the low Al_2O_3 content (93–95%) is not used in further production. It is proposed to obtain high-alumina aggregate without joint grinding of the components. Aggregate was obtained by pressing through a “false grain” from a mixture of kaolin and corundum dust in a certain ratio. The samples firing was carried out at a temperature of 1700°C. The phase composition of the obtained aggregate is represented by 46–51% corundum, 45–49% mullite. The water absorption of such chamotte is 14–16%, open porosity is 33–35%, apparent density is 2230–2290 $\text{kg}\cdot\text{m}^{-3}$. Now, it was not possible to obtain a high-density filler grade ZML and ZMK that meets the requirements of GOST 23037–78. The production flow chart of high-alumina chamotte based on kaolin and corundum dust should include a joint fine grinding of the components, or a separate grinding of corundum dust until it passes completely through the 0.063 mm mesh.

1. Introduction

A promising direction in the development of the production of aluminosilicate refractories is the production of materials with increased operational characteristics [1]. Despite the very wide spread of chamotte in metallurgy, its use is being reduced. It is replaced by new refractory materials with higher service properties, the use of which is due to the continuous intensification of the thermal service of furnaces [2–7]. The growth of their application is limited by a relatively high cost.

Aluminosilicate materials have aluminum and silicon oxides as the refractory base. The structure, properties and application field of these materials depend on the content of Al_2O_3 and SiO_2 . On this basis, they are divided into semi-acidic (15–30% Al_2O_3), chamotte (30–46% Al_2O_3) and high-alumina (over 46% Al_2O_3). In turn, high-alumina refractories are divided into mullite-siliceous (45–62% Al_2O_3), mullite (62–72% Al_2O_3), mullite-corundum (72–90% Al_2O_3) and corundum refractories (over 90% Al_2O_3) [8–10].

Mullite refractories include materials with 62–72% Al_2O_3 content. The phase composition of mullite refractories is mainly represented by mullite, also corundum and glass phase (6–12 wt.%). Mullite refractories are usually obtained in two stages: first, mullite synthesis, and then producing products from it. As starting materials for the mullite synthesis, mainly technical alumina, pure refractory clays, kaolins and quartz are used [11–13]. To transfer the silica formed during the



silicates firing into the mullite, technical alumina is introduced in the mass composition. Mullite formed during the interaction of silica with technical alumina is called secondary. Production technology in most cases is determined by the properties of raw materials. The main troubles in the production of high-alumina products are associated with a large shrinkage of technical alumina during firing as a result of the conversion of γ -alumina to α -alumina, with low sintering ability of α -alumina and with the difficulty of obtaining a specified structure. To reduce shrinkage during the firing of products, a chamotte flow chart is used, i.e. part of the material is preliminarily fired on chamotte. The main difference between high-alumina refractory technology and multi-chamotte technology is the production of high-alumina aggregate, which is prepared in various ways. Upon receipt of the briquette from technical alumina and refractory clay, alumina is preliminary ground to a particle size of 3–5 μm . Briquettes are prepared by plastic or semi-dry molding and fired at 1700°C. Chamotte with water absorption of 1–2% is obtained by firing the briquette in a rotary kiln. Then a granular product is prepared by crushing, coarse grinding and fine grinding.

When fused alumina is crushed, dust is formed [14]. It is captured by electrostatic precipitators and stored in the enterprise [15]. This by-product is substandard material to produce corundum refractories, as there is an insufficient content of Al_2O_3 (93–95%). In addition, electrocorundum is used as an abrasive material [16–17].

To produce chamotte refractories, kaolins with a content of 30–39% Al_2O_3 are used. Due to the supplanting of chamotte refractories by high-alumina refractory materials, it is important to produce materials in demand with high content of Al_2O_3 based on kaolins. Thus, the aim of this project is to develop a technology for the production of high-alumina chamotte based on enriched kaolin and corundum dust generated during crushing of electrocorundum.

2. Experimental procedure

Phase composition was determined by an X-ray phase method in a Miniflex 600 (Cu $K\alpha$ -radiation, $\lambda = 1.541862 \text{ \AA}$, recording interval 3.00 – 90.00 deg, scanning step 0.02 deg), Rigaku, Carl Zeiss, Japan, with MiniFlex Guidance and PDXL Basic data treatment package program control and data collection. Identification of diffraction maxima was carried out using a JSPDS data bank. The chemical composition of quartz sand was determined by emission spectral analysis with inductively coupled plasma in an Optima 4300 DV (Perkin Elmer, USA) optical emission spectrometer.

The water absorption, open and total porosity and apparent density were determined in accordance with [18]. The grain size composition of finely milled powders was determined by sedimentation analysis in a SLAD 2201 (Shimadzu Corp.) particle laser diffraction analyzer according to [19]. Sieve analysis was carried out in accordance with [20]. The plasticity number of clay raw materials was determined according to [21]. The real density of the materials was determined by the pycnometric method according to [22]. The specific surface of the materials was determined according to [23].

3. Results and discussion

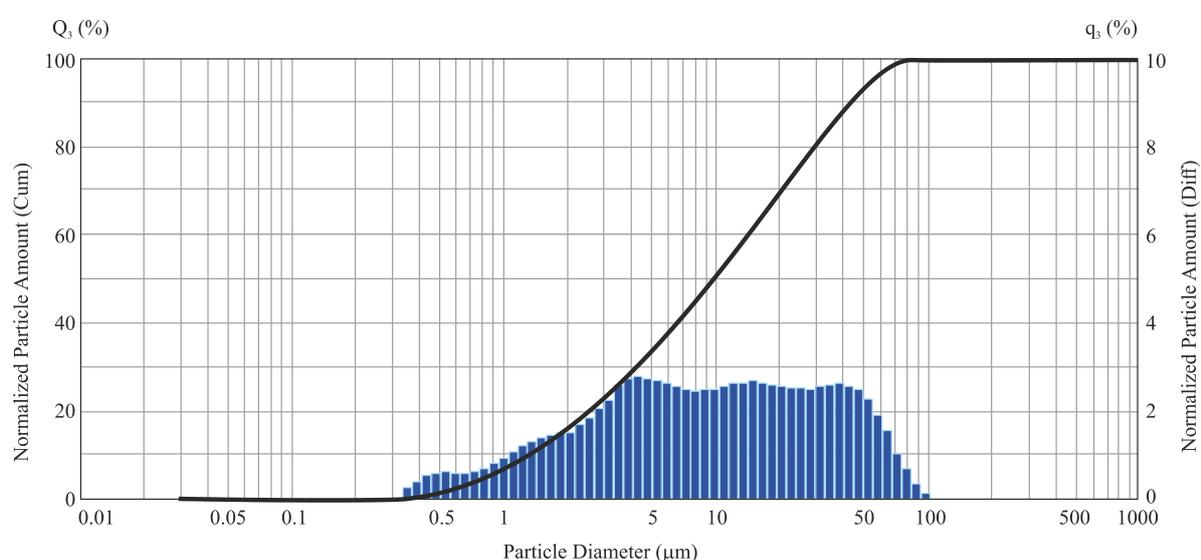
To solve the problem of obtaining high alumina material in this project, the introduction of finely dispersed corundum dust in the process of enrichment of kaolins without joint grinding of raw materials is considered. Thus, the modernization of the existing production of chamotte will consist in the installation of a thermal unit providing a firing temperature of chamotte 1700°C.

Enriched kaolin of Kyshtymkoye deposit and finely dispersed corundum dust were used as raw materials. The chemical composition is presented in Table 1. Kaolin is the basic raw material. The mineral composition of kaolin is as follows: muscovite (up to 5 wt.%), quartz (up to 2 wt.%), kaolinite (95–98 wt.%). The content of Al_2O_3 in corundum dust is 93–95 wt.%. According to the XRD analysis corundum content is 90%. As impurities, thialyte and rutile are present in an insignificant amount, 1.2 and 0.16 wt.% respectively. According to the results of chemical and X-ray phase analysis, it is possible to determine the content of the glass phase in corundum dust, it is about 10%.

Table 1. The chemical composition of materials.

Material	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	MgO	R ₂ O	TiO ₂	LOI
Kaolin (%)	43.66	40.59	0.16	0.98	0.16	0.56	0.62	13.28
Fine substandard corundum powder (%)	0.05-0.3	93.0-95.0	0.2-0.24	0.13-0.5	0.44-0.62	0.3-0.55	2.2-5.0	–

The particle size distribution of kaolin is shown in Figure 1. Particles with a size of less than 2 μm in kaolin is 8–15%. Particles smaller than 63 μm in size more than 95%. Particles 2–55 μm in size are distributed almost uniformly and make up about 85%.

**Figure 1.** The dispersed composition of kaolin.

The granulometric composition of Kyshtymskoe kaolin, determined in accordance with [21], is given in Table 2. According to the particle content of less than 1 μm (43.2%), kaolin in accordance with [24] is a medium dispersed raw material.

Table 2. The granulometric composition of Kyshtymskoe kaolin.

Particle size (mm)	> 0.06	0.06–0.01	0.01–0.005	0.005–0.001	< 0.001
Fraction content (%)	3.6	16.9	17.5	31.7	30.3

The specific surface area of kaolin in the delivery state is 16035 cm²·g⁻¹, real density is 2.65 g·cm⁻³. The plasticity number of kaolin is 9.1, kaolin in accordance with [24] refers to moderately soft raw materials.

The particle size distribution of finely dispersed corundum is presented in Table 3. The main fractions of the powder are particles 0.16–0.063 mm in size and less than 0.063 mm.

Table 3. The distribution of fine fractions in corundum.

Particle size (mm)	> 0.5	0.5–0.16	0.16–0.063	< 0.063
Fraction content (%)	3.07	4.52	51.45	40.96

The composition of the mixture to obtain high-alumina chamotte was calculated in view of 75% Al₂O₃ content in chamotte. Plastic cakes of kaolin with a moisture content of 20–25%, obtained after the filter press, were carefully mixed with corundum dust. The plastic mass was dried at 105°C. It was

ground to a grain size of less than 3 mm. To reduce dust during the chamotte firing, the briquette must have a transport strength of at least 4 MPa. To determine the briquette forming parameters (humidity and pressing pressure) in order to obtain a solid raw briquette, the obtained powder was moistened with a technical lignosulfonate solution of density $1.05 \text{ g}\cdot\text{cm}^{-3}$ to a moisture content of 5, 7, 9%. Then, cylinder samples of 20×20 mm size were molded at a specific pressure of 5, 10, 15, 20, 25 MPa. After drying at a temperature of 105°C , the compressive strength was determined. Figure 2 shows the dependences of the briquette strength on humidity and pressing pressure.

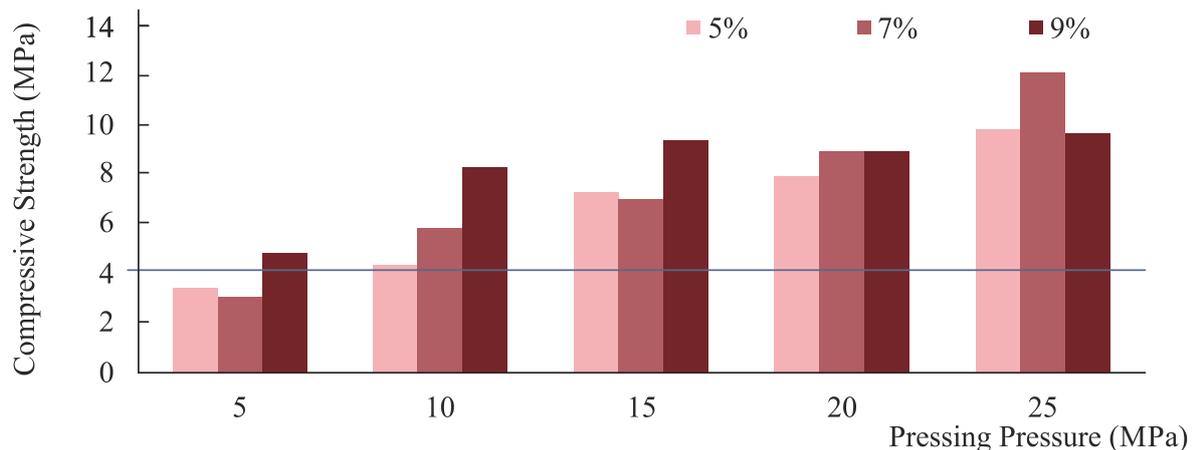


Figure 2. Dependence of compressive strength of unfired briquette on specific pressing pressure and relative humidity.

Strength of 4 MPa is possessed by samples formed at a moisture content of 9% at a pressure of 5 to 25 MPa [26–27]. Samples formed at lower humidity at a compression pressure of 5 MPa will not be strong enough and will crumble during firing, increasing dust extraction. At a pressing pressure from 10 to 25 MPa with molding humidity of 5, 7, 9%, briquettes with a strength of more than 4 MPa were obtained. The most favorable conditions for briquette molding are the lowest humidity and pressing pressure. Redundant moisture causes the formation of increased porosity after firing. The use of high values of molding pressure suggests the presence of expensive equipment, and the use of low ones - insufficient contact between the particles of corundum and kaolin. Thus, samples formed at a humidity of 5 and 7% at a molding pressure of 10 MPa were fired at a temperature of 1700°C . The water absorption, porosity, apparent density, and strength of fired samples were determined. The results are presented in Table 4.

Table 4. Post-firing properties of the samples.

Molding humidity, %	Total shrinkage (%)	Water absorption (%)	Open porosity (%)	Apparent density ($\text{kg}\cdot\text{m}^{-3}$)	Compressive strength (MPa)
5	1.0	15.6 ± 0.7	34.8 ± 1.4	2230 ± 30	30.5 ± 5
7	1.1	14.5 ± 0.8	33.2 ± 1.3	2290 ± 50	39.3 ± 6

Complete shrinkage is 1% at molding humidity of 5%. With an increase in molding humidity to 7%, it leads to an increase in shrinkage of 1.1%. Water absorption and porosity of samples molded at a humidity of 7% is less than that of samples molded at a humidity of 5%. This effect can be explained by the fact that a more moistened mass provides better molding conditions and a denser contact between the particles of kaolin and corundum. In addition, the density and strength of samples molded at 7% are greater than those molded at 5%.

The XRD results are shown in Figure 3. The following phases were found in the samples of high-alumina chamotte are corundum, mullite, thialite, rutile, quartz. The presence of quartz can be explained

by rubbing it in the process of sample preparation on the XRD. Samples were ground in an agate mortar. The high hardness of corundum causes grinding of quartz from a mortar and pestle. The absence of cristobalite indicates its complete interaction with corundum with the formation of secondary mullite.

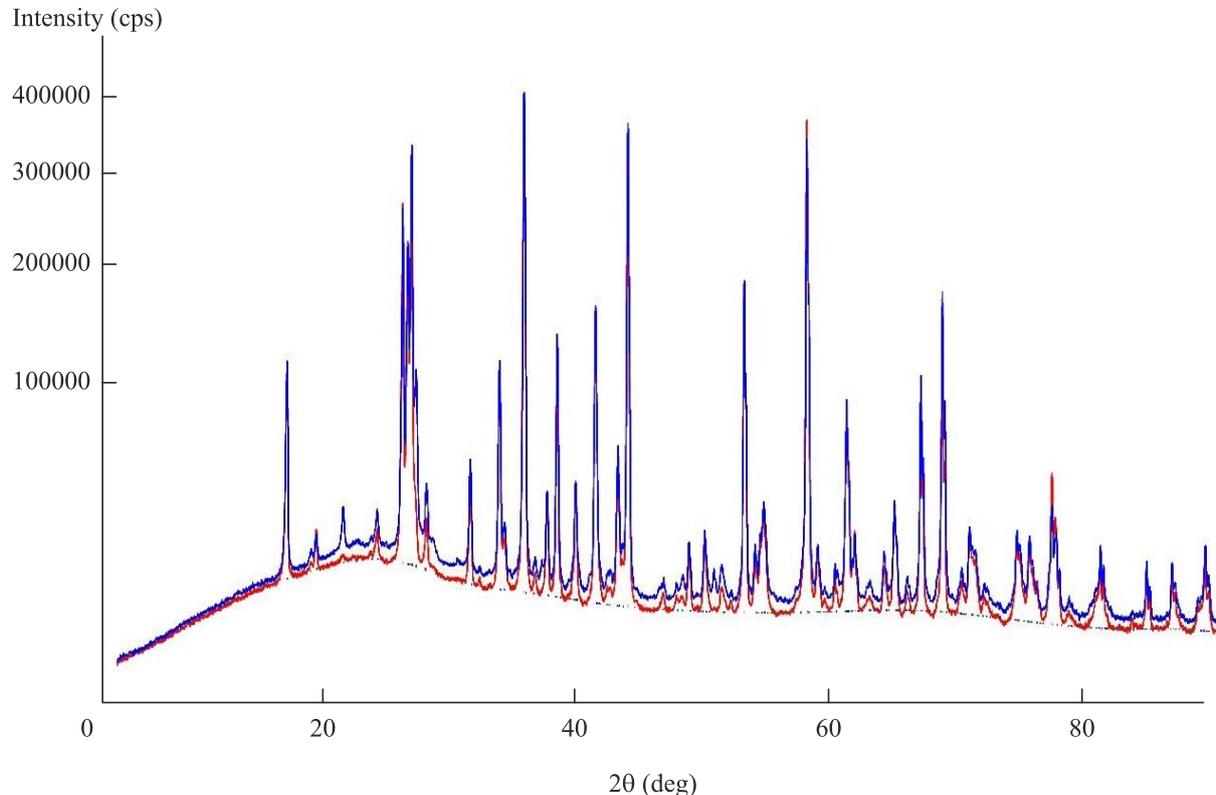


Figure 3. XRD results of high-alumina chamotte.

The results of X-ray phase analysis are presented in Table 5.

Table 5. Phase composition of high-alumina chamotte.

Molding humidity, %	Corundum	Mullite	Thialite	Rutile
5	46	49	3	2
7	51	45	3	1

The results of a semi-quantitative analysis according to the results of XRD are within the error of the method. The composition of high-alumina chamotte is as follows: 46–51% corundum, 45–49% mullite, rutile ~ 1%, thialite ~ 3%.

4. Conclusion

As a result of the research, samples of high-alumina aggregate with an Al_2O_3 content of 75% were obtained on the basis of finely dispersed corundum dust generated by grinding fused corundum. The phase composition of chamotte is represented by 46–51% corundum, 45–49% mullite. The water absorption of such chamotte is 14–16%, open porosity 33–35%, apparent density 2230–2290 $\text{kg}\cdot\text{m}^{-3}$. In accordance with [25], aluminosilicate aggregates of the ZML and ZMK brands should have a water absorption of not more than 3%. Thus, as a result of the studies, a high-density aggregate of the ZML and ZMK brands was not obtained in accordance with [25]. Further research should be aimed at reducing water absorption, for example, by using a joint grinding of kaolin and corundum, or by replacing kaolin with refractory clay. The flow chart for the production of high-alumina chamotte should include joint

fine grinding of corundum dust with a plastic component, or separate grinding of corundum dust until it passes completely through a 0.063 mm mesh, followed by mixing with a plastic component.

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