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RESEARCH IN THE AREA OF PREPARING ACTIVATED ALUMINA. PART 1. METHODS FOR PREPARING REACTIVE ALUMINA

M. A. Trubitsyn,[1,3] **N. A. Volovicheva,**[1,4] **L. B. Furda,**[1]
V. I. Kuzin,[2] **and R. V. Zubashchenko**[2]

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Properties, production methods, and fields of application for finely dispersed activated α-aluminum oxide are considered. Information about international and Russian manufacturers of reactive alumina used in the production of engineering ceramics and low-cement refractory cast mixes is presented. Characteristics of reactive alumina produced by different companies are provided.

Keywords: reactive alumina, dispersion, particle size distribution, median particle size D_{50}, low-cement refractory cast mixes.

INTRODUCTION

Aluminum oxide may be found in various crystalline modifications, and the most thermodynamically stable is aluminum α-oxide. This Al_2O_3 modification is encountered in the form of mineral crystals of corundum in trigonal syngony. The corundum crystal lattice consists of trivalent aluminum ions and bivalent oxygen ions. Oxygen ions form an almost hexagonal densely packed structure with aluminum ions filling two thirds of the octahedral spaces [1].

The main properties of α-Al_2O_3: molecular weight 101.96 g/mole; density 3.99 g/cm^3; melting temperature 2054°C; ultimate strength in bending 282 MPa; in compression 2550 – 3100 MPa; elasticity modulus 365 – 393 gPa; crack resistance 3.3 – 5.0 MPa; hardness on Moh’s scale 9; LTEC 7.1 – 8.3 ppm/K; thermal conductivity 36 – 39 W/(m·K); specific volumetric electrical resistance 1012 – 1018 Ω·cm; relative dielectric permittivity 8 – 11.1 [2 – 4].

Due to its exceptional physicomechanical properties and biocompatibility α-Al_2O_3 is used for preparing implants, artificial joints and micro-scalpels, and also high quality optical ceramics, single crystal aluminate garnet, and leucosapphire. The latter serves as a starting material for producing light diodes, microcircuit substrates, and laser diodes. A key product in the α-alumina market is high purity alumina. Its main importance is the production of single-crystal corundum that in turn is used in many fields of civil and military engineering. In particular, it is used in the production of wear-resistant glasses for aircraft and rocket construction, and for chemical and thermally stable structural components [5, 6].

An important application field for α-alumina has been and remains production of engineering and special corundum ceramic. It has been demonstrated [7 – 9] that a reduction in sintering temperature during preparation of dense corundum ceramic may be achieved due to increasing the starting powder particle fineness, increasing crystal lattice energy, and also with introduction of modifying additives. With an increase in fineness there is an increase in the particle contact area and the overall surface energy increases. The maximum particle size capable of with a capacity for active sintering should not be more than 5 μm [10], and preferred size is less than 1 μm. According to [9] only with particles size less than 1 μm is surface energy is sufficient for material maximum compaction with a firing temperature of 1650 – 1750°C. The following specifications have been formulated for powders employed for preparing high quality corundum ceramic [8 – 11]: particle fineness range 0.5 – 2.2 μm, narrow particle size distribution, particle spherical shape, and grain size composition stability.

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[1] Belgorod State National University, Belgorod, Russia.
[2] PKF NK Staryi Oskol, Russia.
[3] trubitsin@bsu.edu.ru
[4] volovicheva@bsu.edu.ru

The abovementioned has become a basis for development, organization of production, and extensive application of very fine activated α-alumina powder. In generally accepted anglicized terminology the name of this product is reactive alumina. In reactive alumina material is dispersed with respect to primary crystal size (05 – 2.0 μm) and a significant proportion of particles is sub-micron in size, i.e., less than 1 μm (20 – 90%). These powders are reactive in the sense that they sinter readily to maximum density at temperatures 200°C lower than for coarsely dispersed powder. Structure and surface activity of reactive alumina is determined by the defective nature of the crystal structure, particle size, and shape that give rise to a reduction in sintering activation energy [12, 13].

Among leading overseas companies producing corundum ceramic and also reactive alumina the following should be noted: Martinswerk GmbH (Germany), KT Refractories and Alberox/Morgan Advanced Ceramics (USA), CTI — Ceramica Technica Industria (France), Sumitomo Chemical (Japan), etc. Among Russian producers of similar product it is possible to separate NPP Ékon concerned with preparation of fine powders and output of vacuum-dense corundum ceramic based upon them, and AO Polikor produces α-alumina powder ($D_{90} < 3$ μm) for preparing structural ceramics [14 – 16]. Some properties of reactive alumina for production of corundum ceramic are provided in Table 1.

As is seen from table 1, commercial reactive alumina for corundum ceramic is distinguished by a high content of sub-micron particles (1 μm). Sub-micron activated alumina powder provides the required collection of production and physicomechanical properties for various forms of dense corundum ceramic. Corundum ceramic specimens have been prepared [17] within which as reactive alumina sub-micron powder grade MARTOXID MR70 (Martinswerk GmbH) is used successfully with particle size of 0.2 – 0.8 μm. Objects have been prepared by slip casting in a gypsum mold followed by casting drying and firing at 1580°C. Slip relative viscosity is important and is 20, 25, 30% and 1.8, 1.9, and 3.0°E. Objects obtained have almost zero open porosity P_{open} and apparent density ρ_{app} close to theoretical (3.9 g/cm^3).

Us of ultrafine alumina grades A16SG and A1000SG (Almatis GmBH) combined with addition of deflocculant based on carbonic acid after firing at 1580°C has made it possible to prepare specially dense corundum ceramic with $\rho_{\text{app}} \approx 3.9$ g/cm^3 and $\Pi_{\text{open}} = 0.8\%$ [18]. On the basis of reactive alumina the same company has prepared corundum complexly shaped objects by casting under pressure [19]. After firing in an electric furnace at 1600°C specimens prepared on the basis of alumina powder grade ST 11200 SG had the following properties: apparent density 3.83 g/cm^3, porosity 3.82%, ultimate strength in compression (1030 ± 25) MPa.

According to [21 – 23] reactive alumina powder also appeared to be very effective in low-cement refractory technology new generation of castables. The first stage for creating the refractory composites was development in the 1970 – 80s of low-cement castables (LCC) in which a significant proportion of calcium aluminate cement was replaced by a fine matrix (particle size < 45 μm) which also included sub-micron particles (0.5 – 1 μm). These fine products filled cavities between coarser grains that provided a reduction in the amount of water for production with retention of the required mobility. The main design of these components of similar low-cement mixes was given in an Alcoa patent (Almatis) DE 4125511C2 Feuerfeste Gieâmasse in 1991 [24].

In the initial stages of low-cement castable technology development micro-silica was used as an ultrafine matrix component. However results of recent studies have demonstrated that addition of micro-silica into matrix systems in a number of cases has an unfavorable effect on thermomechanical properties of heat engineering composite materials [25, 26]. This is especially important in preparing linings for critical designation subjected to action of erosion and mechanical loads at high temperature.

In order to surmount this disadvantage within the composition of low-cement castables instead of micro-silica fine α-alumina has been introduced. Initially there was use of medium-crystalline calcined aluminum oxide refined to a

TABLE 1. Reactive Alumina for Preparing Corundum Ceramic Supplied by Different Producers

Grade / producer	Weight fraction, %, not more than (for calcined substance)			Particle size distribution, μm		Specific surface, m^2/g, not more than
	SiO_2	Fe_2O_3	Na_2O	D_{50}	D_{90}	
Martoxide MR-70 / Martinswerk GmbH	0.08	0.02	0.10	Not more than 0.8	Not more than 3.0	6.0
Martoxide MZS-1 / Martinswerk GmbH	0.06	0.03	0.10	Not more than 1.9	Not more than 5.0	2.5
B1L-4D / KT Refractories	0.18	0.01	0.05	3.0	—	—
B3M-12D / KT Refractories	0.20	0.20	0.30	Not more than 1.5	—	—
KC 501 / Sumitomo Chemical	0.02	0.01	0.45	Not more than 1.5	—	2.0
Grade K / AO Polikor	0.30	0.05	0.20	—	3.0	5.5-7.5
Grade K1 / AO Polikor	0.30	0.04	0.20	—	3.0	5.5-7.5

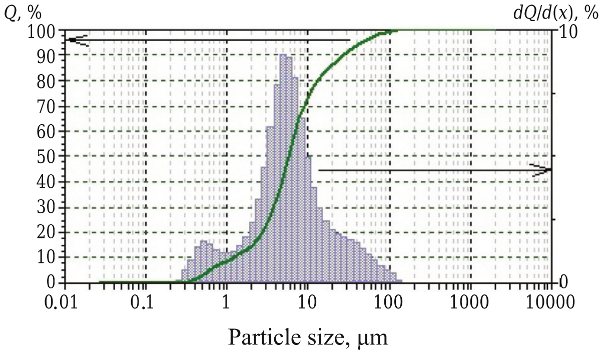

Fig. 1. Integral curve and particle size distribution histogram for calcined alumina CT 9 FC.

particle size of less than 45 μm (325 mesh). The authors of the present article have studied some physicomechanical properties of calcined alumina grade CT 9 FC (Almatis Company, Germany). An integral curve ad a particle size distribution histogram are given in Fig. 1 for a specimens of this alumina. A specimen has a bimodal particle distribution with maximum in regions 0.4 and 4 μm. The median particle size D_{50} is 5.73 μm, and the size of D_{90} particles reaches 27 μm. As a consequence particles of this material have a low specific surface equal to 0.34 m²/g.

SEM pictures are shown in Fig. 2 of the calcined alumina. It is seen that within calcined alumina CT and FC both individual particles and also porous agglomerates are present, there are primary Al_2O_3 crystals with size from 0.5 to 6 μm. Presence of excess porosity within calcined alumina leads to an increase in the proportion of increase in the proportion of setting water that in turn worsens cast composition fluidity. In addition, in low-cement cast refractory technology of a new generation an important task is provision of the required rheotechnological properties with minimum water content.

In contemporary production solutions within the composition of low-cement cast composites today there is use of very fine activated α-Al_2O_3 with a predominant particle size of 1 – 2 μm containing up to 30% sub-micron fraction.

Currently the leading world producers of finely dispersed activated aluminum oxide for low-cement castables are transnational companies Almatis and Nabaltec. They produce a number of reactive aluminas with a different type of mineral particle distribution [27, 28]. Among Russian large tonnage producers of reactive alumina is the Borovich Refractory Combine (BKO) [29]. Comparative properties are provide in Table 2 for reactive alumina from various producers.

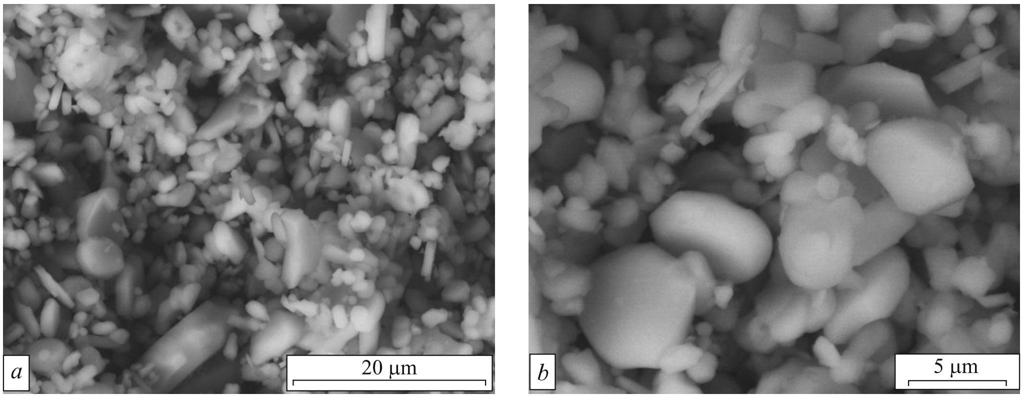

Fig. 2. Calcined alumina CT 9 FC SEM-pictures.

TABLE 2. Properties of Some Grades of Reactive Alumina Manufactured by Different Producers

Grade / producer	Weight fraction, %, not more than (for calcined substance)			Particle size distribution, μm		Specific surface, m²/g, not less than
	SiO_2	Fe_2O_3	Na_2O	D_{50}	D_{90}	
GRT for TU 14-194-280-07 with change No. 1 / AO BKO	0.05	0.1	0.35	2.5	7.5	0.85
CTC-20 (monomodal) / Almatis	0.03	0.03	0.12	1.8	4.5	2.1
CTC-22 (bimodal) / Almatis	0.03	0.03	0.12	1.9	5.8	2.7
NO-625 10 (monomodal) / Nabaltec	0.03	0.03	0.12	2.5	7.5	1.6
NO-615 30 (bimodal) / Nabaltec	0.05	0.03	0.2	2.0	4.6	5.0

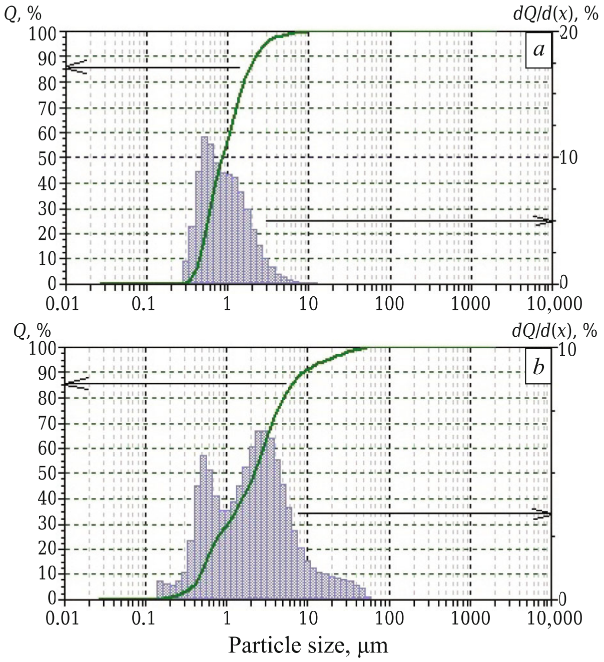

Fig. 3. Integral curve and particle size distribution for reactive alumina GRT (*a*) and CTC-22 (*b*).

The product presented by domestic and overseas producers has D_{50} less than 3.0 µm.

In accordance with grain size composition reactive alumina may be separated into two groups characterized by monomodal or multimodal particle size distribution. By multimodal we understand a property having more than one maximum in a particle size distribution histogram. As an example integral curves and distribution histograms for particle size distribution for two reactive alumina specimens from different producers obtained by the authors are shown in Fig. 3. For a specimens of reactive alumina grade GRT (Fig. 3*a*) there is a typical monomodal particle distribution with a maximum of 0.4 µm. The material has D_{50} equal to 2.5 µm. The submicron fraction content (≤1 µm) is of the order to 21%, but particle material specific surface is 1.1 m^2/g. the grain size distribution within a specimen of reactive alumina grade CTC-22 (see Fig. 3*b*) has a clearly defined bimodal nature with maxima 0.4 and 3.0 µm respectively; $D_{50} = 2.2$ µm and the sub-micron fraction content reaches 30%. Quite a high proportion of sub-micron fractions determines the developed specific surface of material particles that is 2.3 m^2/g.

Shown in Fig. 4 are SEM pictures obtained by the authors for specimens of reactive alumina CTC-22. Within them individual crystallites with sizes from fractions to 3 – 4 µm are clearly seen, forming the bimodal nature of a specimen grain size composition. The shape of dispersed α-Al_2O_3 particles should be noted comprising a finished product after grinding. Crystallites of reactive aluminum oxide have a rounded isometric shape; typically there is absence of shears and cracks.

Significantly improved grain size distribution may be achieved by optimizing particle packing due to use of bimodal reactive aluminum oxide. This facilitates a reduction in water requirement and improvement in cast properties that is demonstrated predominantly by bimodal compared with monomodal aluminum oxide. Self-spreading castables require a more significant amount of very fine particles within a matrix system than vibration cast material. This is achieved by increasing the proportion of reactive alumina. Finely ground (<20 µm) tabular aluminum oxide with an average particle size of about 3.5 µm and polyfraction distribution may be used successively in combination with bi- or multimodel reactive aluminum oxide for subsequent optimization of fine particle packing within a matrix.

The role of matrix component grain size composition in low-cement castables has been considered in an article [30] whose authors have studied the effect of matrix grain size composition including a combination of fine calcined and reactive alumina on cast composite rheological properties. It has been demonstrated that in order to regulate rheological properties of these mixes not only D_{50} should be considered, but also the overall particle fraction distribution. Bimodal reactive alumina has been prepared by combined dry grinding

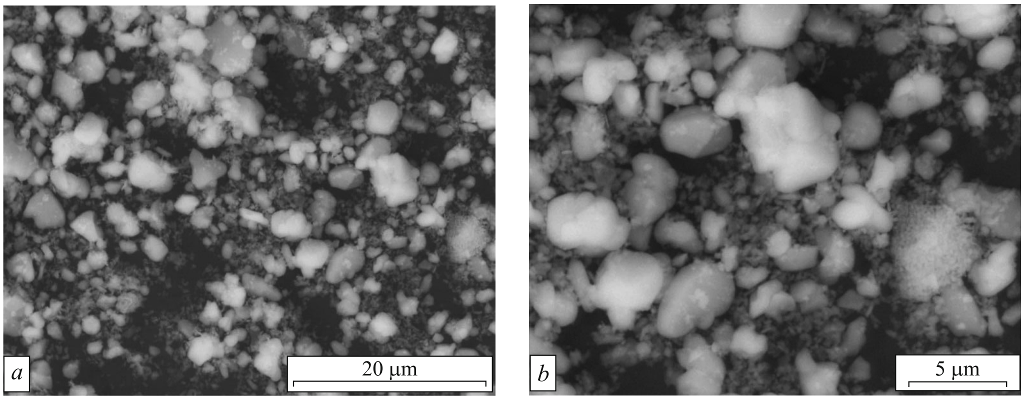

Fig. 4. Reactive alumina CTC-22 SEM-pictures.

in a periodically operating ball of two aluminum oxides with single crystal sizes of 2 and 0.5 μm respectively. It has been revealed that use of bimodal reactive aluminum oxide within a matrix system composition makes it possible to increase fluidity, to reduce mixing duration, and on the whole to improve production properties of low cement castables.

However, according to [31] comparable fluidity for single mode reactive alumina grade CTC-20 has been obtained for a suspension with a moisture content of 15%, and for multimodal alumina grade CTC-30 with a suspension moisture content of 17 – 18%. The author of [31] explains this fact by significantly greater fineness of multimodal product.

A marked improvement of rheological properties of low-cement castables based on fine matrices is achieved with introduction of deflocculants. As a result matrix systems of different composition, including multimodal reactive alumina combined with contemporary deflocculants and correspondingly low-cement castables, acquire optimum production properties.

Rheological behavior of low-cement composites including two versions of bimodal finely dispersed α-Al_2O_3, have been considered in [32]. In the first version a matrix was prepared by separate addition of two monomodal reactive aluminas with D_{50} 2.5 and 0.8 μm respectively. In the second version there was use of a previously homogenized mixture of the same reactive aluminas. The effect of a bimodal matrix, obtained by the first and second versions on viscosity of castables has been evaluated by measuring the required mixer power. It has been established that there is a reduction in power required (and directly viscosity) for mixes prepared using previously homogenized original reactive aluminas.

Currently depending on the specific application fields there is use of various methods for preparing very fine reactive aluminum oxide: hydrolysis, sol-gel technology, hydrothermal, and electrolytic methods, and mechanochemical refinement in high-energy milling units [21, 33, 34]. During pyrolysis [33] atomization of aluminum chloride solution has been accomplished in furnaces with temperature of the order of 1000°C:

$$4AlCl_3 + 6H_2 + 3O_2 = 2Al_2O_3 + 12HCl.$$

Very fine α-alumina prepared by hydrolysis is used as an abrasive material and also in the production of structural ceramic, vacuum lamps, etc.

Sol-gel technology includes the following operations: aqueous sol preparation, water removal, gel preparation, and gel heat treatment in order to prepare oxide [33]. This method assumes formation of an amorphous gel from precursor solution, which are normally aluminum alkoxides. A feature of aluminum oxide prepared in this fashion is the submicron crystal size (1 μm). These nano-powders are in demand for producing catalysts, porous ceramics, and membranes, biocompatible materials, and objects for medicinal purposes. In the majority of cases in preparing very fine aluminum oxide as starting materials there is use of aluminum hydroxide. However, the abovementioned methods are hardly suitable for multi tonnage production of reactive aluminum oxide.

For mass production of finely dispersed α-alumina currently as a rule there us use of calcined alumina. This alumina is prepared by heat treatment (calcining) of Bayer aluminum hydroxide from 1300 to 2000°C [21]. The aluminum α-oxide crystals size formed is affected by both temperature and calcination rate. It is apparent that the faster the heating rate the smaller is the primary crystal grain size. A dependence is provided in Table 3 for the primary crystal size and α-Al_2O_3 content on calcination temperature.

It is seen from Table 3 that the higher the firing temperature, the larger is the dense primary crystal size and Al_2O_3 content in α-form. This in turn has a considerable effect on grinding capacity for material and D_{50} of reactive material.

During mass production of finely dispersed activated aluminum oxide there is use of a dry or wet grinding method for alumina raw material to a size of primary crystals within various types of grinding unit: in vibration, jet, and vortex mills.

The effect of production parameters (amount and size of milling bodies, vibration frequency) on wet grinding process for two forms of alumina in a vibration mill has been described in [35]. In the authors' view with use of the optimum production parameters it was possible to obtain aluminum oxide powder with D_{50} of 3 μm and D_{90} of the order of 10 μm with grinding duration of 60 min. However, the wet milling process has marked disadvantages: the powder obtained requires subsequent drying after grinding.

Mechanochemical aspects of dry grinding for three types of alumina powders (with an average particle size of 0.6, 3.9, and 21.8 μm) in a vibration ball mil for 300 h has been described in [36]. It has been established that a reduction in primary crystal size leads to an increase in crystal lattice deformation. In particular, uniform lattice distortion (for example elongation over axis *a*) in α-Al_2O_3 is observed in the attrition process.

In addition some of the most economic and widespread grinding units are drum ball mills. The authors of the present article have conducted a set of scientific and experimental studies concerning optimization of the process for preparing

TABLE 3. Some Physicochemical Properties of calcined Alumina Prepared at a Different Temperature

Calcining temperature, °C	Average primary crystal size, μm	Specific surface (BET) m^2/g	Product α-Al_2O_3 content, wt.%
1250-1350	0.02	60 – 80	<50
1400 – 1550	0.2	6 – 12	70 – 90
1550 – 1700	2.0	0.6 – 0.9	95 – 98
1700 – 1800	3.0	0.4 – 0.7	>98

activated aluminum α-oxide by dry grinding in a ball mill. As a result of this specimens of very fine alumina have been prepared that with respect to properties are no worse than commercial analogs of the leading domestic and overseas producers: $D_{50} = 2.2$ μm, $D_{90} = 7.1$ μm, S_{sp} (BET) = 0.86 m^2/g, density in a dry condition 0.62 g/cm^3, in a compacted condition 0.88 g/cm^3, natural repose angle 73 degrees, moisture content 0.15 wt.%, Δm_{cal} 0.32 wt.%. Special attention has been devoted to forming sub-micron fractions (≤1 μm) whose content within material was 20 – 25%. More detailed results of experimental studies with be provided in subsequent publications.

CONCLUSION

Very fine α-Al_2O_3 is currently used extensively in the production of biocompatible materials, light diodes, microcircuit substrates, laser diodes, etc. The main application field for α-alumina powder is production of engineering and special corundum ceramics. Currently reactive alumina is used extensively in a new generation of low-cement castable technology. Introduction into their composition of finely dispersed activated α-alumina makes it possible to reduce considerably the proportion of high-alumina cement and the amount of setting water, and also to improve mix rheological properties. However, in products intended for low-cement castables inadequate attention has been devoted to the submicron fraction content (<1 μm) that has a considerable effect on production and thermal properties of unmolded refractories. Domestic multi-tonnage production for similar purposes is almost absent within the Russian market. Therefore scientific research aimed at developing and creating manufacturing technology for preparing finely dispersed activated aluminum α-oxide is very important.

(*To be continued*)

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