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RESEARCH IN THE AREA OF PREPARING ACTIVATED ALUMINA. PART 3. EFFICIENCY OF USING RUSSIAN ADDITIVE-INTENSIFIERS DURING MILLING HIGH-ALUMINA RAW MATERIALS

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Results of studying the influence of Russian commercial milling additives of various chemical nature on the process of dry milling calcined alumina are presented. It is shown that use of the additives studied makes it possible not only to reduce the time of alumina raw material fine milling of by a factor of 1.5 – 2, but also to increase the submicron particle fraction (<1 μm) yield within finished activated product.

Keywords: reactive alumina, particle size distribution, milling aid additives, fine powders, ball mill.

INTRODUCTION

Currently there are a number of theories describing solid breakdown during their refinement. The most well-known are the Rittinger surface theory and the Kick-Kirpichev volumetric theory establishing a correlation between energy expended and the size of particles during failure [1, 2]. According to Rittinger failure energy is proportional to the newly formed surface. The Kick-Kirpichev rule confirms that deformation energy is proportional to the deformed body volume and makes it possible to evaluate the limiting value of elastic deformation energy above which elastic deformation is transferred into irreversible deformation, i.e., failure. General milling theory has been proposed by academician Rebinder. According to this theory work for milling A is described by an equation

$$A = A_v + A_s = k\Delta V + \alpha\Delta S,$$

where A_v is work for material volumetric deformation; A_s is work for new surface formation; ΔV is the deformed part of volume; ΔS is the increase in material surface; k and α are proportionality coefficients.

A common disadvantage of these theories is the limitation of their practical application in view of the fact that they do not consider actual process conditions and cannot be used for quantitative calculations.

Dispersion of solid materials and in ball mills may conditionally separated into two processes: crushing predominantly in the initial stage and fine reduction (milling). The crushing process is implemented mainly by an impact scheme. Failure by impact is possible when elastic deformation energy accumulated by a body is converted into surface energy for new surface formation [3]. Cleavage of coarse mineral grains with a dense uniform structure proceeds through an area of defects that are separated into linear (vacancies within a crystal lattice) and linear points (dislocations). Plastic deformation precedes brittle failure caused by dislocation movement within crystals. Under the effect of an external load (impact) dislocations move readily and emerge at a surface, facilitating formation of generated cracks that then develop into failure cracks [1]. Therefore. contemporary theory for crack generation within a crystal is based on a dislocation model.

For such materials as corundum, the contribution of plastic deformation in the failure process is insignificant. In fact, corundum has high bond energy and dislocation movement activation [4]. Therefore, in the case of applying critical stresses to corundum grains deformation energy is mainly consumed by brittle failure.

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During fine milling an important role is played by a wear mechanism (friction failure). This is implemented due to accumulation of failure tangential stresses within milled grain surface layers, which leads to accumulation of ultrafine particles of critical size [1, 3]. As a consequence a feature of powder materials prepared by dry milling in ball mills is agglomeration with achievement of a certain level of dispersion ($D_{50} < 3$ μm) [5 – 7]. This agglomeration of finely dispersed particles is accompanied by sticking to a drum liming and body wall, formed as a so-called “pseudoviscous milling medium”. Therefore the proportion of useful energy expended in surface failure for mineral particles and new surface creation decreases sharply. Therefore, powder agglomeration processes play a decisive factor governing the rate of mineral material fine milling.

It is suggested that fine particle consolidation within an agglomerate proceeds as a result of occurrence of local compaction pressure with contact of impacting spheres. Approach of these particles to a critical distance leads to maximum development of Van-der-Vaals forces [1].

Van-der-Vaals forces providing adhesion of particles are electrostatic in nature and arise as a result of non-equilibrium distribution of surface charges [8, 9]. In essence they depend upon the size of crystals and distance between surfaces (see Table 1).

In recent decades the problem of deagglomeration of finely dispersed particles with achievement of a critical size has been successfully resolved by introducing into a system a certain amount of special surface active substance (SAS), called traditionally milling intensifiers or according to similar technology additives improving milling (grinding aid additives) [5, 10, 11].

Action of similar additions as a rule has been explained by absorption reduction of solid strength theory composed by A. P. Rebinder [1, 5, 12, 13]. According to this theory the weakening mechanism of SAS action is considered as occurrence of the their molecules within cracks and subsequent creation of wedging pressure. However, this is only valid for cracks of significant size and a slow solid loading rate. In the majority of cases the magnitude of crack cross section within material is less than the SAS molecule size, and therefore they cannot penetrate into milled material [7]. It has also been established [14] that cleavage crack propagation rate during solid refinement is essentially higher than the rate of additive molecule absorption at a newly formed surface. Therefore, an added SAS molecule is incapable of penetrating sufficiently rapidly into a forming crack with a subsequent wedging effect. A later SAS action mechanism has been based on the fact [5] that absorption of molecules only proceeds within narrow cracks preventing merging. In this case SAS molecules are absorbed at a surface by its own polar groups directed towards a solid.

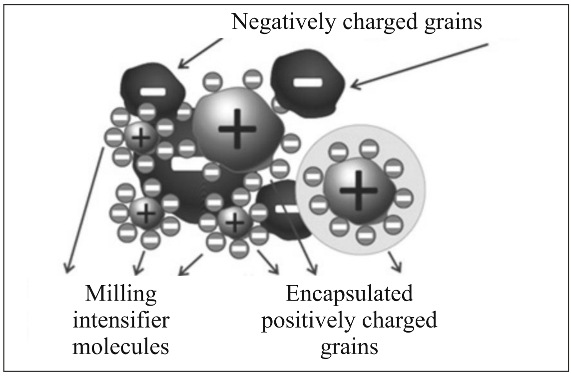

Fig. 1. Encapsulating effect of additive — milling intensifier.

It has been demonstrated [15] that addition of SAS that is previously added before milling facilitates improvement of reduction and therefore prevents repeated agglomeration of finely dispersed particles. Probably there is a so-called “encapsulating effect” of a substance-intensifier (Fig. 1) that improves formation of an adsorbed layer of its molecules of milled material particles. In this case there is a reduction in surface energy.

Currently action of additives is explained mainly by a reduction in adhesive forces between particles that in turn leads to a reduction in agglomeration processes within powders [16]. Due to this there is a reduction in significant loss of energy in overcoming “pseudoviscous milling of a medium” and microprocesses are improved for development of failure stresses within a milled aggregate. This is especially important for ball mills within which particles are subjected to layer-by-layer loading between milling bodies [17].

It has been known for a long time that effective milling intensifiers are additions based on glycols and amino-alcohols, mainly ethanol amine. In turn ethanol amines are separated into three independent products: monoethanol amine (MEA), diethanol amine (DEA), and triethanol amine (TEA) [1, 18, 19]. Both glycols and ethanol amines not only reduce a tendency towards milled particle agglomeration, but also overcome “healing” of microcracks formed during milling. However, a disadvantage of substances based upon amines is related to the fact that they are potential sources of ammonia separation. At the same time commercial ethanol amines used under normal conditions are stable and exhibit good stability within an alkaline medium [20].

Currently Russian and overseas companies produce a broad range of commercial products, i.e., milling intensifiers [19, 21]. Large overseas producers of chemical additives are the companies Sika AG (Switzeraland) and W. R. Grace (Italy). Within Russia there is also active development of the

TABLE 1. Effect of Particle Size on Inclination Towards Agglomeration

Particle size, μm	F_{vdw}/F_G	Degree of adhesion
10 – 100	1 – 100	Weak
1 – 10	100 – 104	Strong
0.01 – 1	104 – 108	Very strong

market for milling intensifiers. The most well-known domestic producers are OOO Sintez OKA – Building Chemistry, within the composition of GK Sintez OKA and OOO Poliplast Novomoskovsk. Characteristics of some commercially available milling intensifiers are given below produced by overseas and domestic companies.

Trade name of intensifier / producer	*Chemical composition*
HEA2 61 / WR Grace	Multicomponent mixture based on water, including ethylene glycol diethylene glycol and triethylene glycol
SikaGrind-700 / Sika	Milling intensifier based on tri-isopropanol amine, bright yellow liquid
Litemill / OOO «Eurosintez»	Composition chemical base — hydrocarbons, azocompounds alcohols; liquid from yellow to dark brown color
Lithoplast I / OOO «Poliplast Novomoskovsk»	Synthetic additive based on modified polymethylenenaphthalene sulfonates with different degree of polymerization (*n* = 7 – 25) with end radicals
ImCem grades FL / GK «Sintez OKA»	Aqueous composite of triethanolamine with different functional additions, transparent liquid from dark brown to black color ImCem RAW grade M / GK
ImCem RAW grades FL / GK «Sintez OKA»	Aqueous solution of glycols, being a transparent liquid from colorless to dark brown

In spite of numerous fields of application, the effect of intensifiers on powder mechanical properties, in particular predominance of physicochemical effects, has not been studied adequately. Selection and application of these additives is primarily based upon on empirical knowledge.

The aim of the present work is evaluation of the possibility and efficiency of using Russian specialized additives for intensifying dry milling of high alumina raw material in an α-modification.

RESEARCH PROCEDURE

In order to prepare finely dispersed activated α-alumina the model starting material used was calcined alumina containing not less than 98% α-Al_2O_3 crystalline phase. Integral curves and a histogram of alumina particle size distribution are provided in Fig. 2.

Results of studying the grain size composition of the original calcined alumina showed that the nature of particle distribution within powder is bimodal. However, the maximum of the first mode is weakly expressed and is recorded in the range 8 – 9 μm. The second mode is more intense and corresponds to the interval 70 – 80 μm. The median particle size D_{50} is 68 μm, and there is no sub-micron faction (<1 μm).

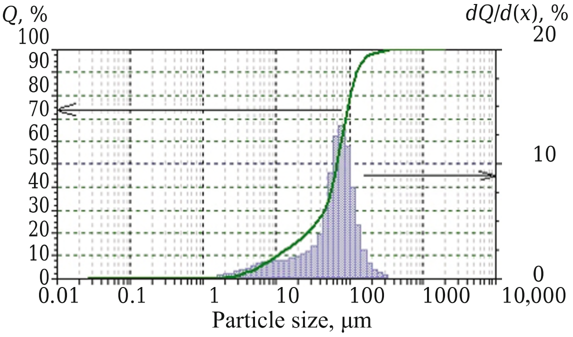

Fig. 2. Integral curve and histogram of alumina raw material particle distribution.

The starting raw material has less specific surface (0.37 m^2/g) that is a consequence not only of the small size of its grains, but also inadequate dense particle packing (see Fig. 2).

The commercial products use as milling intensifiers were provided by GK Sintez OKA. In order to preserve commercial information about intensifier composition they were given the following indices: KD-1, KD-2, KD-3, KD-4, and KD-5. Additive KD-1 is a dark brown liquid with a weak smell, additive KD-2 is a colorless liquid with a specifically sharp smell. The rest of the additives KD-3, KD-4, and KD-5 are colorless or weakly colored liquids with a weak amine smell. The concentration of the additive-intensifiers introduced an all milling experiments was 0.1 wt.% (above argillaceous raw material weight).

In order to mill an alumina specimen an ML-1 ball mill was used with a milling rum made of high-alumina porcelain with a working volume of 2 dm^3. Optimum production parameters for dry milling of alumina in α-form have been established in previous studies [22].

Dry milling duration of high-alumina raw material was 14 h (without additive) and 10 h (with addition of milling intensifier). Kinetic dependence of the milling process has been studied by analyzing the dispersed condition of powder through each 2 h of milling with the production parameters prescribed. In each stage of the study integral curves and particle distribution diagrams were obtained using a Micfrotrac S3500 analyzer (Microtrac Inc., USA). Distilled water was used as a dispersion medium. Powder specific surface was determined by low-temperature absorption and thermal absorption of nitrogen by means of a Triustar II 3020 unit (Micrometrics USA).

Morphological studies of materials were conducted in Quanta 200 3D and Quanta 600 FEG (FEI, Netherlands) electron microscopes. Images were obtained using a

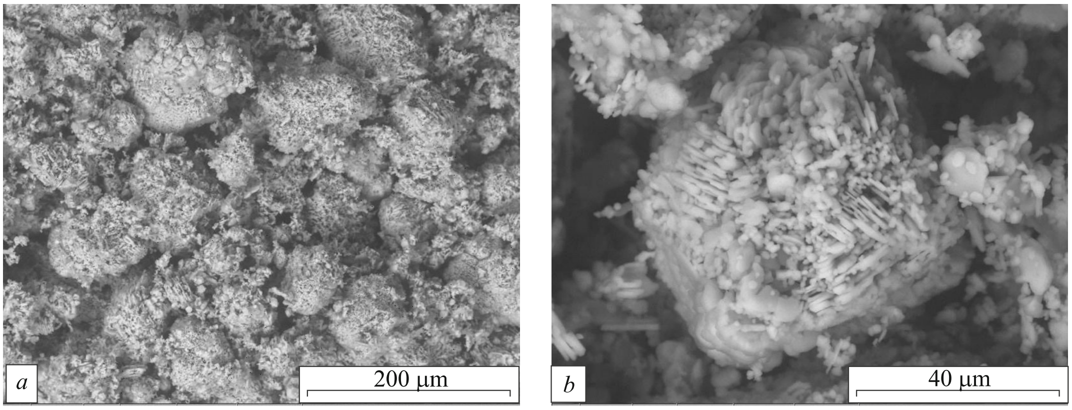

Fig. 3. High-alumina raw material SEM-microphotographs.

back-scattered electron detector in a low vacuum regime. Pressure within the chamber 80 Pa, accelerating voltage 30 kV. For the study in a scanning regime specimens were glued to a copper substrate by means of conducting carbon adhesive.

RESULTS AND DISCUSSION

Grains used in the present work of model raw material as an analog were commercial alumina (Fig. 3) whose morphology does not have a dense monolithic microstructure and is spherulitic agglomerates (from 40 to 80 μm). Spherulites are primary crystallites of lamellar shape that are joined locally by phase contacts within sticking zones. Slotted pores are clearly differentiated forms due to stacked primary crystal growths. Agglomerates are surrounded by finely dispersed particles of rounded shape with sizes of the order of 5 – 6 μm.

Dispersion of this material may be represented by the scheme shown in Fig. 4.

In the first stage there is crushing with predominance of an impact mechanism leading to spreading of coarse spherulitic grains (*I*) into fragments of agglomerates (*II*). In the second stage of impact energy the rest of agglomerated particles (*II*) are subjected to impact action that leads to final splitting into primary crystals (*III*). Then the wear mechanism starts to predominate with formation of rounded particles (*IV*).

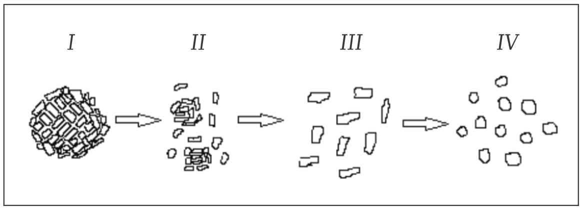

Fig. 4. Scheme of main stages (*I* – *IV*) of calcined alumina refinement.

During determination of the grain size composition some difficulties were observed in the course of analyzing specimens obtained using additives KD-2 and KD-4, in particular, an increased tendency was observed towards aggregation of powder within an aqueous medium that required preliminary repeated ultrasonic dispersion in order to obtain the correct results. During use of the rest of the additives primary products in an aqueous medium did not aggregate. Kinetic curves are shown in Fig. 5 for materials dry refinement.

Analysis of Fig. 5*a* showed that in the case of dry milling of calcined alumina without additive a considerable refinement rate is observed in the first 4 h that is indicated by the maximum slope of the kinetic curves for all control fine fractions. In the interval 4 – 8 h the process rate slows down. Sections of curves corresponding to fractions D_{20} and D_{50} are flat in nature and demonstrate a tendency towards emergence into a plateau. At the same time, beyond this time interval a curve corresponding to D_{90} retains a slope. This may complicate the crushing process for residual aggregates both as a result of occurrence of "pseudo-viscous milling of a medium" due to repeated agglomeration and significant sticking of argillaceous particles to the drum liming and milling bodies. This in turn leads to retardation of the transfer process into a finely milled phase. During subsequent refinement (after 10 – 14 h) particle size comprising controlled dispersion functions, does not change considerably, which points to prevalence of a wear mechanism in the time range in question.

Unloading of finished product in this case was difficult since significant sticking of the finely dispersed material to the milling bodies and lining of the milling drum was observed (Fig. 6*a*).

On the whole milling kinetic curves of the model alumina raw material in the presence of test additive GK OAK Sintez have a complex nature similar to the curve shown in Fig. 5. Analysis of curves showed that the fastest milling arte for calcined alumina n the presence of intensifiers was observed within the first 2 h, which corresponded to the stage of spherulite crushing. Preliminary introduction of SAS in all cases makes it possible to accelerate alumina grain cleavage,

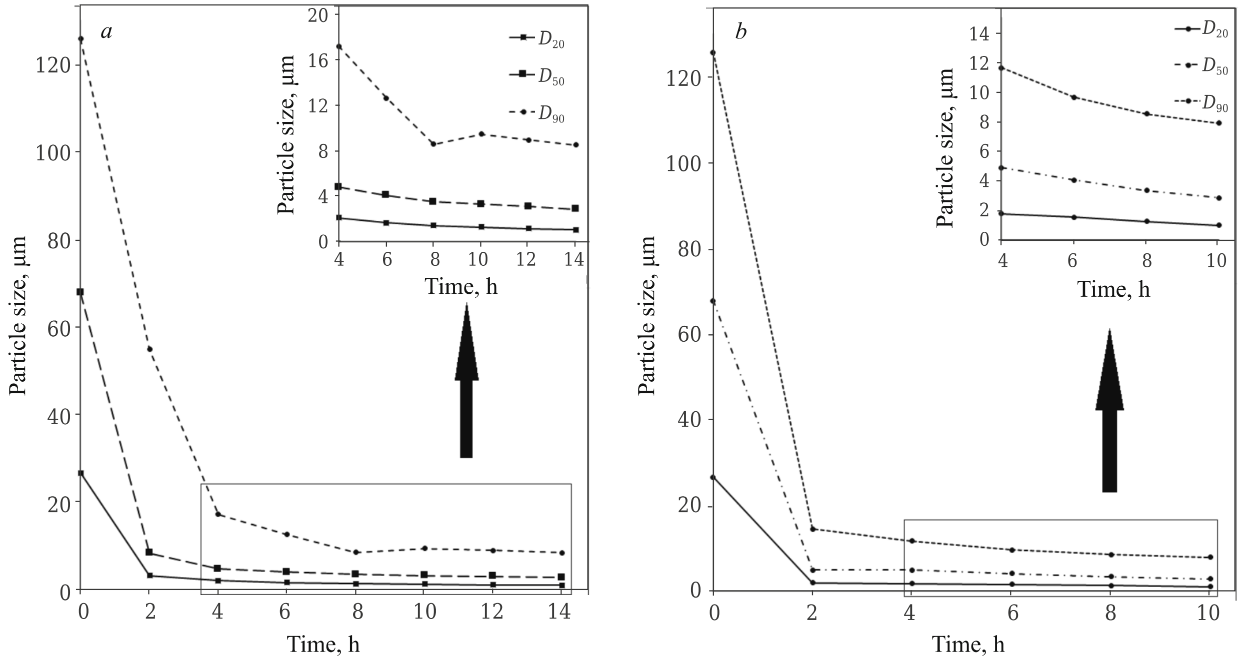

Fig. 5. Calcined alumina refinement kinetic curves: *a*) without additions (control); *b*) with introduction of KD-1 additive.

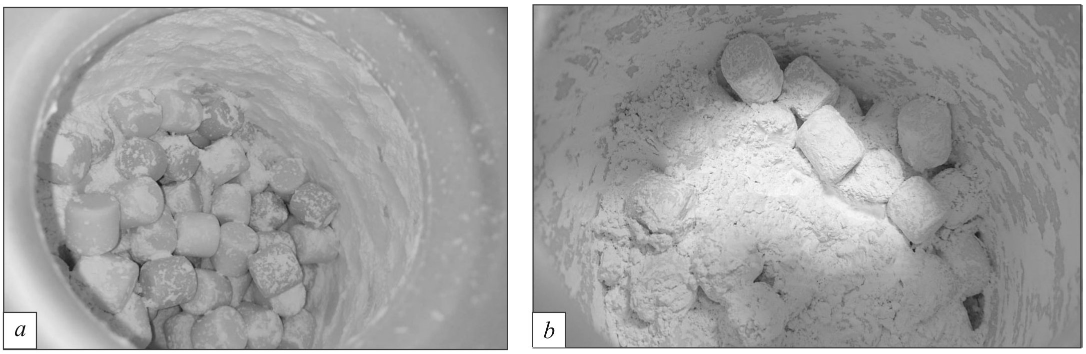

Fig. 6. Alumina refinement products in ball mill after 10 h: *a*) without addition (control); *b*) with introduction of KD-1 additive.

not only due to a reduction in strength of phase contact between primary crystallites, but also due to avoiding repeated agglomeration of the finely dispersed particles formed. This in turn makes it possible to reduce by a factor of 2 – 4 the time for transfer of the process from a crushing phase into a wear phase. Sticking of finely dispersed particles to the milling body lining was insignificant (Fig. 6*b*), and unloading products after 10 h of milling high-alumina raw material was accompanied by considerable dust formation.

Results of comparative tests for milling intensifiers are shown in Fig. 7. Additive introduction makes it possible not only to reduce milling duration, but also the reduce the average particle size in all control dispersed fractions. Even in the first 2 h of refinement with introduction of additive-intensifiers (see Fig. 7*a*) particle size corresponding to D_{20}, D_{50}, and D_{90} decreases on average by a factor of 1.5 – 2.0. The best milling capacity for α-alumina for this time period was demonstrated by additive KD-1, KD-4, and KD-5 for which the value of *D*50 was 25 – 35 μm. In this case the least median particle size was recorded in products after 2 h of milling in the presence of KD-1 and it was 4.83 μm.

After 10 h of milling using all types of additive (see Fig. 7*b*) a comparable aprticle size was recorded for D_{20}, D_{50}, and D_{90}, i.e., on average 1.0, 2.8, and 8.0 μm respectively. This points to the identical nature of the action of intensifiers and in addition points directly to achievement of the limiting primary crystal size constituting the original calcined alumina. With 10 h of milling alumina without additives the particle size of all control fractions on average exceeds by a factor of 1 – 2 the value for the same fractions for powders obtained in the presence of milling intensifiers. Without adding SAS the powder comparable granular com-

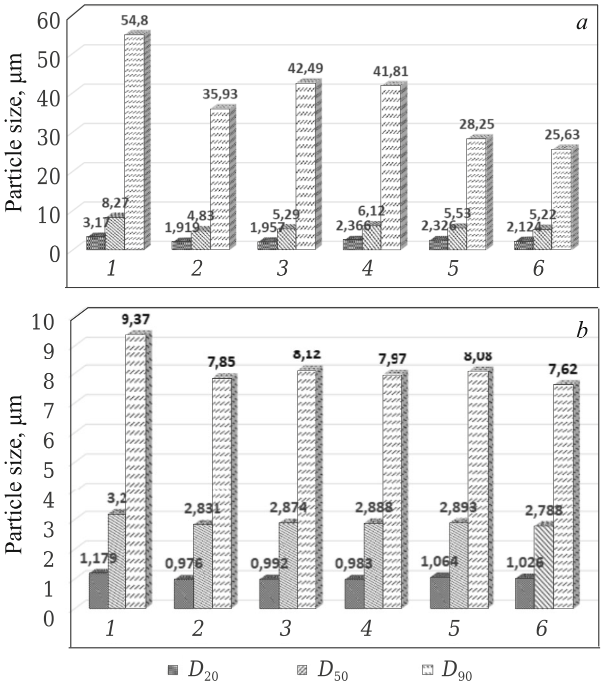

Fig. 7. Particle distribution for three dispersed fractions after 2-h (*a*) and 10-h milling (*b*): *1*) without addition; *2*) with addition of KD-1; *3*) KD-2; *3*) KD-4; *5*) KD-4; *6*) KD-5.

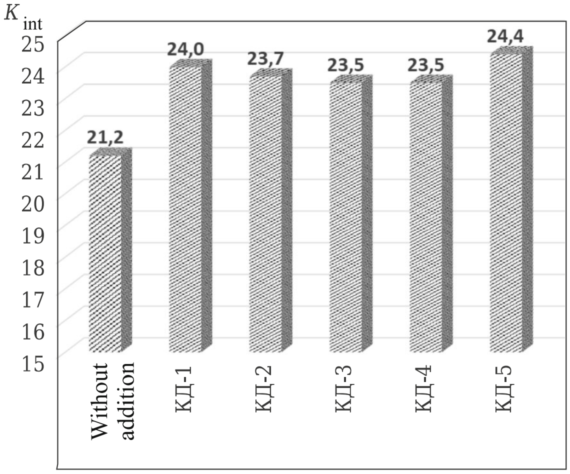

Fig. 8. Value of calcined alumina refinement effective coefficient.

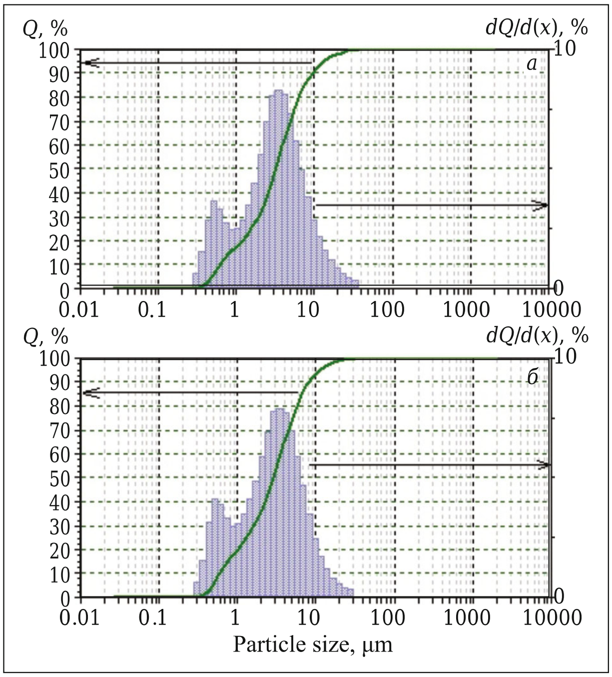

Fig. 9. Integral curves and particle distribution histograms after 10-h refinement: *a*) without addition; *b*) alumina + KD-1.

position was only achieved after 14 h of high-alumina raw material milling.

For comparative evaluation of the effect of experimental additives on milling intensity the milling efficiency coefficient K_{int} was calculated (Fig. 8) by an equation

$$K_{\text{int}} = \frac{D_{50\text{orig}}}{D'_{50}},$$

where $D_{50\text{orig}}$ is the median size of original alumina particles; D'_{50} is product median particle size after 10 h of diminution.

Analysis of Fig. 8 showed that use of all of the additives studied provides a marked favorable effect during dry milling of high-alumina raw material. The best results are obtained with use of intensifiers KD-1 and KD-5. Other additives studied (KD-2, KD-3, and KD-4) have comparable values of K_{int} and are only surpassed insignificantly by additives KD-1 and KD-5.

The content of submicron fraction <1 µm) in finished powder without adding SAS is 17.2%. in powder of α-Al_2O_3 prepared within the presence of KD-2, KD-3, KD-4, and KD-5 the proportion of particles with size less than 1 µm is within the limits 18.5 – 19.5%. The best approach to a sub-micron fraction is achieved with milling high-alumina raw material with KD-1 addition (22%).

In addition, the change in specific surface S_{sp} of milling products with introduction of different types of SAS was evaluated. Activated alumina powder prepared in the presence of intensifiers KD-1, KD-2, KD-3, KD-4, and KD-5 has S_{sp} in the range 0.75 – 1 m^2/ton, whereas S_{sp} of product during milling without additive is only 0.02 m^2/ton. This fact is explained by the comparatively low proportion of particle sub-micron in the material.

In studying grain size composition features presence has been established of a bimodal nature of distribution for all of the powders obtained of finely dispersed α-Al_2O_3 (Fig. 9). In all specimens presence is recorded of two modes in the ranges s 0.5 – 0.6 and 4.5 – 5.5 µm respectively. The results obtained confirm the proposal advanced previously that the nature of particle size distribution is governed to a consider-

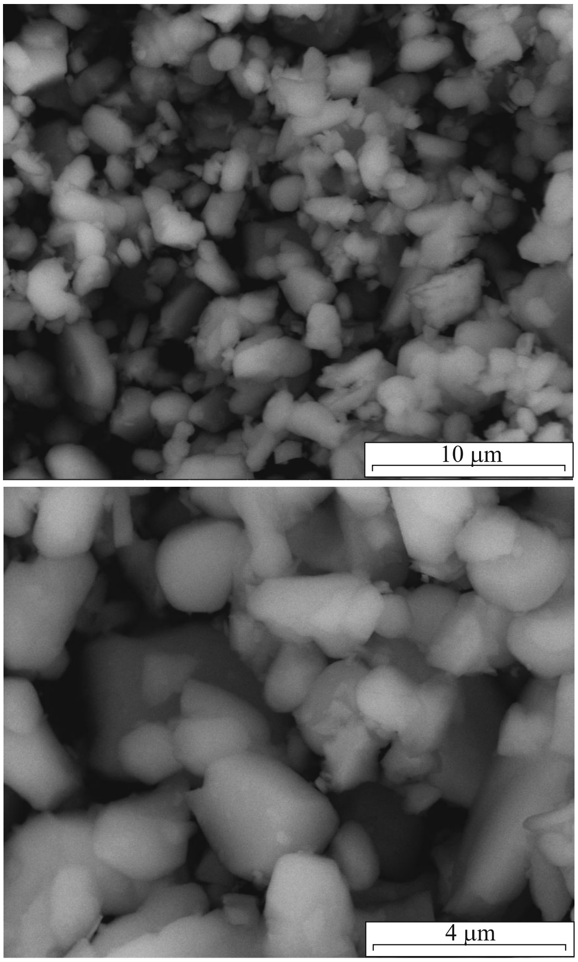

Fig. 10. SEM-microphotographs of calcined alumina refined product in the presence of KD-1 at different magnification.

able extent by the selected milling production parameters and depends little on the nature of the added SAS [22, 23]. In this case with an increase in milling duration the proportion of sub-micro fractions (<1 μm) increases, and the proportion of coarser fractions, comprising the second mode, conversely decreases uniformly. In all cases the volumetric ratio is retained between modal phases equal to 1:4.

SEM photographs are shown in Fig. 10 for α-Al_2O_3 powder after 10 h of milling alumina raw material in the presence of KD-1. Two types of crystals are clearly identified, differing not only in morphology, but also in size, which is in good agreement with data about the grain size composition (see Fig. 9*b*). The first type is represented by volume of particles of isometric shape with a size of 2 – 5 μm, and thickness of the order of 1 μm. For this type of crystals there is typically absence of sharp corners and shears. The second type of particles forms a highly dispersed faction and is rounded platelets with a size from fractions to 1 μm. For the microstructure of powders there is typically absence of spherulitic grains typical for the original raw material (see Fig. 3).

The authors of publication [24] have proposed that the maximum reduction in adhesive forces is achieved under conditions of creation of an adsorbed monomolecular layer formed by SAS molecules at the surface of particles. The optimum SAS concentration required for formation of this monomolecular layer is of scientific and practical interest and will be the subject of our subsequent research.

CONCLUSIONS

– Kinetics of the dry milling process for α-alumina in the presence of Russian commercially available additive-intensifiers of a different chemical nature have been studied. It is confirmed that powder agglomeration is a decisive factor governing the rate of fine milling of mineral materials. This is fine particle agglomeration, accompanied by presence of sticking to a drum wall and milling bodies, forming both as a so-called “pseudo-bonded milling medium”. It has been established that introduction of test additives effectively avoids adhesive attachment of fine particles and their agglomeration. Taking account of features of grain morphology a scheme has been proposed illustrating the features of the main stages of milling calcined alumina.

– It has been demonstrated that introduction of additives makes it possible to reduce moiling duration by a factor of 1.5 to 2, to obtain finished product with a median particle size of about 2.8 μm, and a yield of sub-micron fraction (more than 22%).

– It has been established that during milling alumina in the α-modification a bimodal nature of grain distribution is retained that is due to structural and morphological features of the raw material and prescribed milling production parameters.

(*To be continued*)

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