Structure and properties of alumina bubble obtained by electrodispersion of AD0E grade electrical aluminum waste in distilled water

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The paper solves the problem of alumina bubble production. This problem is proposed to be solved by electrodispersion of AD0E grade electrical aluminum waste in distilled water. To develop technologies for reuse of the obtained alumina bubble and to assess the efficiency of its utilization requires complex theoretical and experimental studies. The purpose of the work was to study the structure and properties of alumina bubble obtained by electrodispersion of AD0E grade electrical aluminum waste in distilled water.

The tasks set in this work, aimed at studying the structure and properties of alumina bubble obtained by electrodispersing AD0E grade electrical aluminum waste in distilled water, were solved using modern equipment and complementary methods of physical materials science: the shape and surface morphology of fused alumina particles were investigated on an "QUANTA 600 FEG" electron-ion scanning microscope with field emission of electrons (Netherlands); the elongation coefficient and particle size distribution of powder particles were investigated on a "Analysette 22 NanoTec" laser particle size analyzer (Germany); *X*-ray spectral microanalysis of powder particles was performed on the "EDAX" energy dispersive *X*-ray analyzer (Netherlands), integrated into the "QUANTA 200 3D" scanning electron microscope (Netherlands); phase analysis of powder particles was performed on the "Rigaku Ultima IV" *X*-ray diffractometer (Japan).

It was found experimentally that spherocorundum has the following characteristics: shape — spherical, elliptical and agglomerates; volume-average particle diameter is 42.6 μm; particle surface is oxygen clad; phase composition consists of Al_2O_3 and $Al(OH)_3$.

Key words: electrical aluminum, electrodispersion, alumina bubble, properties.

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Introduction

Currently, synthetic corundum powders are widely used in industry as an abrasive material for grinding, polishing, sharpening and cutting products [1–4]. The most common methods of obtaining α-Al_2O_3 (corundum) powder are methods in which aluminum hydroxide (hydrargillite) or aluminum oxyhydroxide (boehmite) is sintered in air at temperatures above 1200 °C and calcination of aluminum hydroxide at 500–1500 °C in an atmosphere of hydrogen halide in the presence of seed crystals. The disadvantages of these methods are high energy consumption, the size of the obtained corundum crystals cannot be regulated, as well as environmental problems [5–8].

One of the most promising ways to produce metal oxides with low production costs, low energy costs and environmental friendliness of the process is the method of electrical discharge dispersion (EDD) [9–11]. The EDD process is the destruction of conductive material as a result of localized exposure to short-term electrical discharges between electrodes [12]. Electrical discharge method allows to obtain alumina bubble without the use of chemical reagents, which significantly affects the cost of powder and avoids contamination of the working fluid and the environment with chemicals. The average specific power consumption for alumina bubble production is about 2.1 kg/kW·h, which is lower than other methods of corundum production.

However, the properties of electrocorundum made from powders obtained by electrical discharge dispersion are not sufficiently studied, which makes their practical application difficult.

The purpose of the present work is to study the structure and properties of alumina bubble obtained by electrodispersion of AD0E grade electrical aluminum waste in distilled water.

Materials and methods of research

To obtain alumina bubble powders on the experimental unit by the method of electroerosion dispersion, precut into 5 ... 7 cm AD0E grade electrical aluminum waste, was crushed. The wire pieces were loaded into a reactor filled with dielectric fluid — distilled water.

Production of corundum was carried out according to the scheme presented in **Fig. 1** in four stages:

– Stage 1 — preparation for the process of electric discharge dispersing;

– Stage 2 — electroerosion dispersing process;

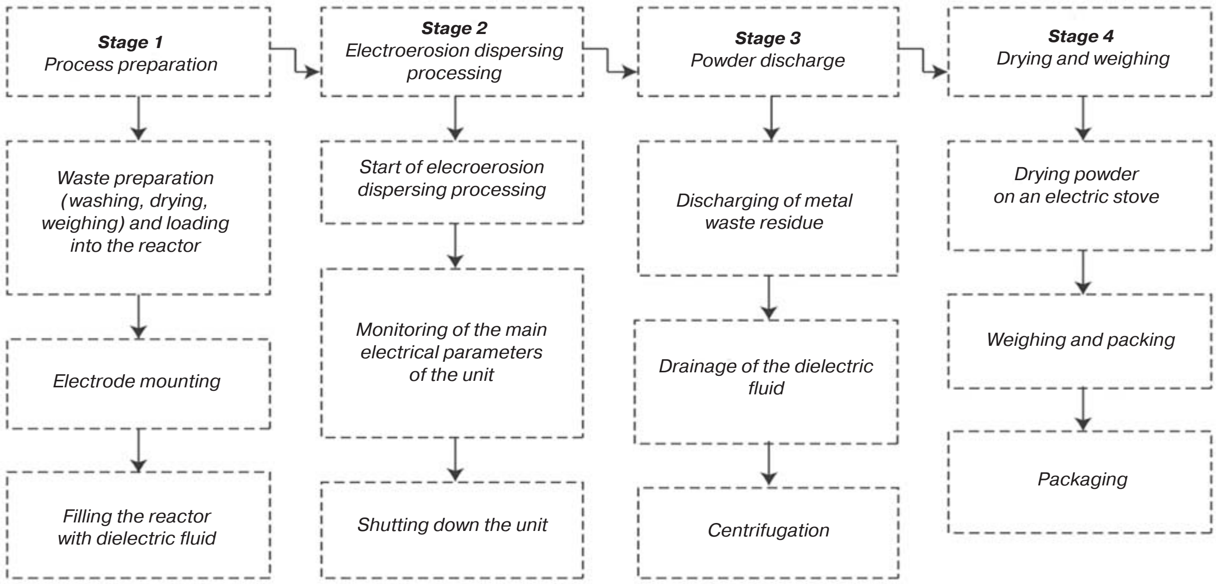

Fig. 1. Stages of the alumina bubble production process

– Stage 3 — discharging powder from the reactor;

– Stage 4 — drying, weighing of aluminum oxide powder, if necessary, certification of its properties.

In the *first stage*, aluminum waste was sorted, washed, dried, degreased and weighed. The reactor was filled with working medium — distilled water, waste was loaded into the reactor. The electrodes were mounted. The assembled electrodes were connected to the generator. The necessary process parameters were set: pulse repetition rate, voltage on the electrodes, capacitance of capacitors.

In the *second stage*, the stage of electroerosion dispersing, the installation was activated. Due to the high concentration of thermal energy, the material at the discharge point melted and vaporized, the working medium vaporized and surrounded the discharge channel with gaseous decomposition products. As a result of significant dynamic forces developing in the discharge channel and gas bubble, drops of molten material were ejected outside the discharge zone into the working medium surrounding the electrodes and solidified in it, forming drop-shaped corundum particles.

At the *third stage*, the dielectric fluid with corundum was discharged from the reactor.

The *fourth stage* involved evaporation of the solution, drying, weighing, packaging and subsequent analysis of the corundum.

When setting up the experiments it was established that the productivity of the process of alumina bubble production by electroerosion method was influenced by the electrical parameters of the plant operation, particularly the capacitance of discharge capacitors, voltage at the electrodes and pulse repetition rate. In this regard, an evaluation of their effect on alumina bubble performance was carried out.

Graphs of dependence of alumina bubble production process efficiency on voltage at constant values of capacitance of discharge capacitors and pulse repetition rate are shown in **Fig. 2**.

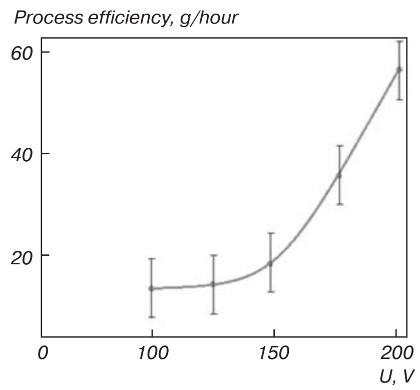

Fig. 2. Dependence of alumina bubble production efficiency on voltage (U)

A quadratic dependence of alumina bubbles production process efficiency on the voltage at the electrodes in the range up to 200 V has been experimentally established, which confirms the dependence of the pulse energy on the voltage ($E = CU^2/2$). When the voltage was increased above 200 V, increased sparking was observed, and the electrodispersion process itself was unstable. Therefore, the range of 100–200 V was chosen as the optimum voltage to obtain alumina bubbles.

The graph of dependence of alumina bubbles production process efficiency on the capacitance of discharge capacitors at constant values of voltage at the electrodes and pulse repetition rate is presented in **Fig. 3**. A directly proportional dependence of the alumina bubbles production process efficiency on the capacitance of discharge capacitors has been experimentally established.

Graphs of dependence of alumina bubbles production process efficiency on the pulse repetition rate at constant values of voltage at the electrodes and capacitance of discharge capacitors are presented in **Fig. 4**.

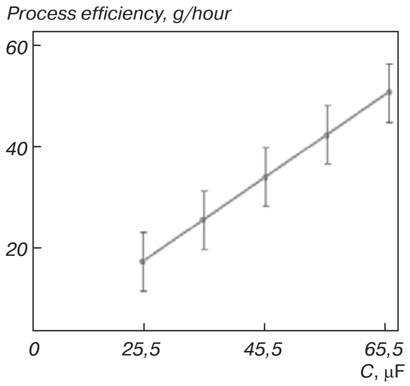

Fig. 3. Dependence of alumina bubble production efficiency on capacity (*C*)

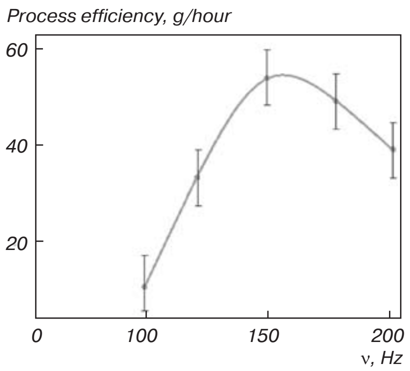

Fig. 4. Dependence of alumina bubble production efficiency on the pulse repetition rate (ν)

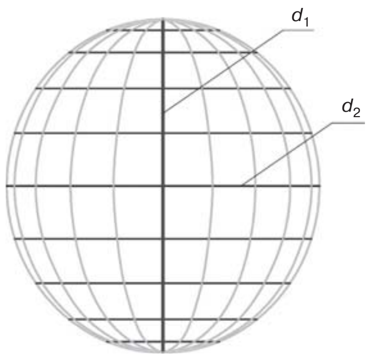

Fig. 5. Studied shape parameters of alumina bubbles particles

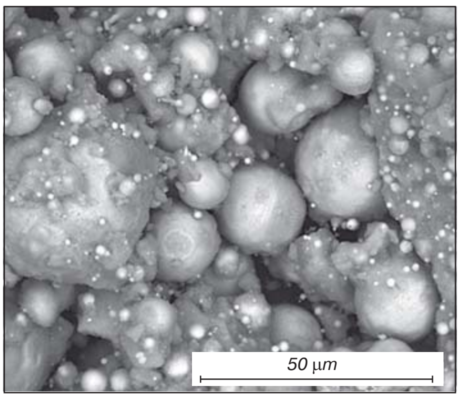

Fig. 6. Microphotograph of alumina bubbles particles

The optimal capacitance for spherocorundum production was chosen to be in the range of 25–65 μF.

As can be seen from the graph (**Fig. 5**), the increase in performance with increasing pulse repetition rate is not infinite and reaches its maximum at 150 Hz. The maximum performance is limited by the charge and discharge times of the discharge capacitors and the speed of the charge and discharge commutator thyristors.

It is noted that corundum particles obtained by electroerosion method have mainly spherical and elliptical particle shapes, i. e. the final product is alumina bubbles. Moreover, by changing the electrical parameters of the dispersing process (electrode voltage, capacitor capacitance and pulse repetition rate) it is possible to control the width and offset of the particle size interval, as well as the efficiency of the process.

For alumina bubbles, one of the main technological properties is optimal dispersibility, so the optimization of the process of its production was carried out by the average particle size by conducting a full factorial experiment. At the same time, voltage, pulse repetition rate and capacitance of discharge capacitors were taken as the main factors. The optimization criterion was the average size of alumina bubbles particles. According to the results of the optimization it was found that to obtain alumina bubbles particles with an average size of 40 ... 50 microns it is necessary to use the following electrical parameters of the installation: pulse repetition rate 50 Hz; voltage at the electrodes 90 V; capacitance of capacitors 65 μF.

The tasks set in this work, aimed at studying the structure and properties of alumina bubbles obtained by electrodispersing AD0E grade electrical aluminum waste in distilled water, were solved using modern equipment and complementary methods of physical materials science: the shape and surface morphology of fused alumina particles were investigated on an "QUANTA 600 FEG" electron-ion scanning microscope with field emission of electrons (Netherlands); the elongation coefficient and particle size distribution of powder particles were investigated on a "Analysette 22 NanoTec" laser particle size analyzer (Germany); *X*-ray spectral microanalysis of powder particles was performed on the "EDAX" energy dispersive *X*-ray analyzer (Netherlands), integrated into the "QUANTA 200 3D" scanning electron microscope (Netherlands); phase analysis of powder particles was performed on the "Rigaku Ultima IV" *X*-ray diffractometer (Japan).

Results and discussion

Quite important characteristics of alumina bubbles particles, on which the area of their practical application depends, are dimensional characteristics such as elongation coefficient and fractional composition.

The results of the study of the elongation coefficient of electrocorundum particles are presented by studies on a laser analyzer (**Fig. 5**) and images of particles obtained on an electron-ion scanning microscope (**Fig. 6**).

The average value of the elongation coefficient (K_e^{av}) of particles was calculated by the ratio of their maximum (d_1) and minimum (d_2) diameters according to the formula (1):

$$K_e^{av} = d_1/d_2 \quad (1)$$

The results of the study of the shape and morphology of electrocorundum particles produced in water are presented by the images of particles obtained on the "QUANTA 600 FEG" electron-ion scanning (scanning) microscope with field emission of electrons (Netherlands) (**Fig. 6**).

Analysis of shape parameters of alumina bubbles particles with an average size of 25...100 microns by images from a scanning microscope, in conjunction with studies of the elongation coefficient equal to 1.0 to 1.25, suggests that these particles have mainly spherical and elliptical shape, i.e. the final product is alumina bubbles. In the process of electrodispersing, alumina bubbles particles, which are ejected from the electric discharge channel in molten form into the reactor filled with dielectric fluid, crystallize very quickly. The process of rapid crystallization of the molten material in the liquid working medium contributes to the sphere and ellipse shape of the particles.

The formation of corundum particles of spherical shape in the process of electrical discharge metallurgy is promoted by the conditions of crystallization of molten metal droplets in the liquid working medium.

For electrocorundum particles, besides the elongation coefficient, the most important characteristic is the fractional composition, which allows to determine the percentage of particles with a certain size range (**Fig. 7**).

These histograms give a clear picture of the degree of polydispersity of the analyzed electrocorundum particles and the content of each fraction in it, since the diameter intervals in the fractions are taken the same.

It was found experimentally that the electrocorundum particles have sizes ranging from 0.15 to 100 μm, the volume average diameter of the particles is 42.6 μm.

The presence of two extremes of the most probable sizes of alumina bubbles particles was noted: small fraction (0.25 ... 25.0 μm) is formed due to condensation of vapor phase and large fraction (25.0 ... 100 μm) is formed due to condensation of liquid phase. The ratio of volumes formed during dispersion of vapor and liquid phases is determined by the thermophysical properties of the dispersed metal waste. The dependence showing that the average particle size of alumina bubbles increases with increasing momentum energy has been established.

Determination of the elemental composition of microobjects of electrocorundum particles was carried out according to the characteristic *X*-ray radiation excited in them with the help of the energy dispersive *X*-ray analyzer of "EDAX" company, built into the scanning electron microscope "QUANTA 200 3D".

As the results of the analysis of alumina bubbles elemental composition spectrograms (**Fig. 8**) showed, when dispersing AD0E grade electrical aluminum waste in distilled water, the surface of particles is clad with oxygen.

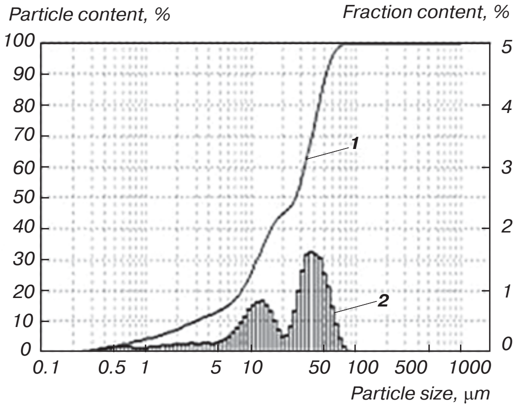

Fig. 7. Integral curve (*1*) and histogram (*2*) of alumina bubbles particle size distribution

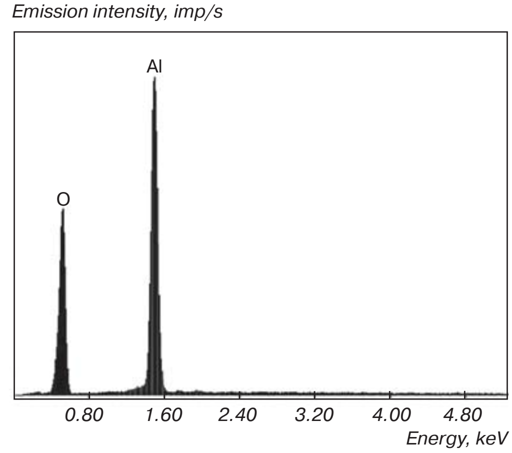

Fig. 8. Spectrogram of alumina bubbles particles elemental composition

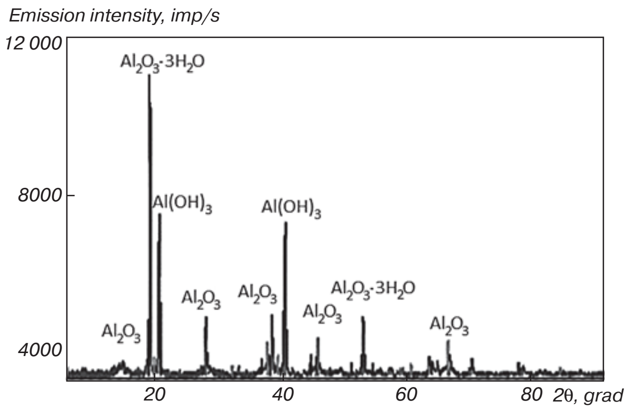

Fig. 9. Diffractogram of alumina bubbles particles phase composition

When analyzing the phase composition of electrocorundum (**Fig. 9**) on a "Ultima IV" Rigaku high-resolution diffractometer, powder particles were poured into a 20 mm diameter cuvette with a depth of 0.5 mm and pressed. Then the cuvette was installed in the holder and analyzed under the following parameters: range: imaging angle 2θ — 10–95 deg; step — 0.020 deg, speed — 1 deg/min, operating voltage — 40 kV, current intensity — 40 mA. The diffractogram profile was processed using the PDXL program; smoothing was performed using the Savitzky-Golay method; background calculation was performed using the Sonneveld-Visser method; peak search was performed using the peak top method.

Analysis of the phase composition of alumina bubbles particles obtained by dispersing AD0E grade electrical aluminum waste in distilled water showed the presence of Al_2O_3 and $Al(OH)_3$ phases in it.

Conclusion

On the basis of the conducted experimental research aimed at studying the structure and properties of alumina bubbles obtained by electrodispersing AD0E grade electrotechnical aluminum waste in distilled water, the high efficiency of electrodispersing technology application is shown, which provides at low power consumption obtaining alumina bubbles powders suitable for processing soft elements, which include rubber, plastic, leather, aluminum, copper products and others. Economic efficiency from the use of the obtained electrocorundum is due to the use of waste and low-energy technology for their production.

It is noted that alumina bubbles, has the following characteristics: shape — spherical, elliptical and agglomerates; volume average particle diameter is 42.6 μm; the surface of the particles is clad with oxygen; phase composition consists of Al_2O_3 and $Al(OH)_3$.

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