Physicochemical Properties of Al₂O₃ Powder Produced by Explosive Synthesis

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The phase, dispersive, and morphological features of an aluminum oxide powder produced by explosive synthesis are examined experimentally. It is shown that the particle-size distribution has three distinct maxima, which are due to different combustion regimes. The relationship between the sizes and morphology of the starting powder and the product is determined. Considerable attention is given to a study of the ultrafine fraction of the product powder. The ultrafine particles are shown to have a regular spherical shape, and sinters are not revealed. Besides spherical particles, the synthesized powder contains faceted crystals. X-ray phase analysis of the ultrafine fraction of the synthesized powders shows that this fraction consists only of the metastable phases of the oxide — the δ- or oxynitride modifications. The δ-modification differs from that described in the literature.

The existing physicochemical methods for synthesis of ultrafine powders are mostly based on the evaporation of and subsequent condensation of materials. At the last stage, an additional supply of energy can be due to various chemical reactions, for example, metal oxidation [1]. The physicochemical properties of ultrafine powders are largely determined by the particle size and method of preparation. Exactly conditions of synthesis determine the characteristics of the material produced, such as the average size, degree of agglomeration, and crystal structure of the particles.

A distinctive feature of metal oxide powders produced by physicochemical methods is the polydispersity of particles. This effect was observed for powders produced by both plasmachemical synthesis [2] and electrical explosion of conductors [3]. However, these studies were focused mainly on the ultrafine fraction of the synthesized materials, which is of great scientific and practical interest. On the other hand, an extensive investigation has been performed on the morphological structure of products from metal combustion in reactive media and their relationship with oxidation regimes [4]. In this case, in contrast, the main focus was on studying coarse particles of oxides; as regards the submicron fraction, it is only reported that it is formed in gas-phase metal combustion [4].

Explosive synthesis is described in [5]. It is shown that the synthesis product contains both ultrafine and coarse (with particle sizes larger than 1 μm) fractions. Their concentrations depend on the technological parameters of synthesis and are related to the state of aggregation of the starting metal at the shock-wave front [6].

The goal of the present work is to study the phase, dispersive, and morphological features of aluminum oxide powders, including the ultrafine powder produced by explosive synthesis.

EXPERIMENTAL TECHNIQUES

Powders synthesized under conditions that are most favorable for production of the material in the ultrafine state have been studied most extensively [6]. In this case, the product of explosive synthesis is a highly porous white powder with large
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(larger than 1 μm) gray inclusions; its bulk density is ≈0.05 g/cm³.

The dispersity of the synthesized powder particles was studied by sieve and sedimentation analyses [7] and electron microscopy. The complete particle size distribution was determined as follows. An averaged powder sample was diluted in distilled water, treated by ultrasound, and poured through a standard set of sieves (GOST 3584-53). To make the extraction of the ultrafine powder fraction more complete and decrease the amount of the liquid used, the suspension was made to settle in the lower part of the vessel and the upper, more liquid, fraction was used for pouring. This operation was repeated several times. The last pouring was performed using pure distilled water. Next, the powder was dried on sieves, and the masses of particular fractions were determined. The suspension was examined by sedimentation analysis [7]. After that, we constructed the complete particle size distribution.

The morphological structure of particles of different fractions was studied by scanning and transmission microscopic techniques using a JEM-100C electron microscope with an EM-ASID-4 scanning adapter. The specific surface area of the powders was determined by the BET (Brunauer, Emmett, and Teller) method. The phase compositions of the synthesized powder and its fractions with different particle sizes were determined on a DRON-3 apparatus.

MAIN RESULTS

Figure 1 shows a typical complete particle size distribution. It is evident that the distribution has three distinct maxima for mass-averaged particle sizes $d_m = 0.25, 22,$ and $360 \, \mu m$.

To check the correctness of sedimentation measurement results for the submicron range and to refine the size distribution for ultrafine particles, we measured the particle diameters by electron micrographs and processed the results by statistical methods [7]. In all experiments, the particle size distributions are shown to be log-normal (a typical distribution is given in [5]). For the experiment considered, the parameters are as follows: number-averaged size $d_{0.5} = 70 \, nm$ and variance $\sigma = 1.9$. For log-normal distributions, the number-averaged and mass-averaged sizes are related by the formula $\ln d_m = \ln d_{0.5} + 3 \ln^2 \sigma$, which holds true in our case. This, in particular, indicates that the sedimentation of the powder in a dispersion liquid is adequately described by the equations used in sedimentation analysis. In the powder produced, the ultrafine fraction is separated from the large-size fraction (see Fig. 1). This simplifies the sizing of the powder particles by sedimentation methods.

From Fig. 2a, it is evident that the particles have a regular spherical shape, and sinters are not observed. The particles are gathered into chains and buildups, perhaps, under the action of electrostatic forces. Diluted in a dispersion medium (H₂O, C₂H₅OH, etc.), the ultrafine particles form a stable suspension. Besides spherical particles, the synthesized powder contains faceted crystals. Most often, the "shadow" of the object has 8 sides with similar sizes. Regular hexagons and irregular octagons whose opposite sides are parallel and equal are encountered rather rarely.

For fractions with particle sizes larger than 50 μm, the particle size distribution determined by sieve analysis is also log-normal. Depending on the conditions of synthesis, $\sigma = 1.2-1.4$.

The morphological structure of particles with sizes larger than 1 μm was studied by scanning electron microscopy. Typical photographs are given in Fig. 2b–d. The large fraction consists of "foam"-type formations, hollow spherical particles, and their fragments. The appearance of a spherical shell is shown in Fig. 2c. The inner diameter of the sphere is 170 μm and its wall thickness is 20 μm (see Fig. 2d). The wall has a porous structure and consists of blocks with a typical size of ≈10 μm.

Sedimentation of the synthesis product leads to separation of particles with typical sizes of 1–50 μm, which forms a considerable gray sediment. Electron microscopy show that this fraction of the powder consisted predominantly of continuous particles with a nearly spherical shape (see Fig. 2b). The number of hollow spheres and foamed agglomerates is insignificant.

In separation by sedimentation, part of the powder rose to the surface, forming a film. This film

**Fig. 1.** Typical particle size distribution ($F$ is the distribution function density).