LASER SYNTHESIS OF ULTRADISPERSE POWDERS OF ALUMINUM OXIDE

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We have studied laser synthesis and the structure and properties of ultradisperse aluminum oxide powders. We show that the laser radiation intensity affects the phase composition. We find that the following conditions are optimal for production of these powders: power 500 W, pressure 0.1 atm (1-10 kPa), helium as the synthesis atmosphere.

In the field of physical materials science of oxide ceramic systems, major directions have been devised for development of new materials with a unique set of physicochemical characteristics. The most promising method is based on the use of nanocrystalline powders. Consequently, the problem of manufacture of materials with special mechanical properties requires development of methods for obtaining nanocrystalline powders.

Nanocrystalline powders of oxide ceramic may be obtained by various methods. One method is vaporization of an oxide ceramic target by concentrated energy fluxes. Vaporization occurs upon heating of the target with an electron beam or a laser beam in air or in an inert gas medium [1-3]. A second method is plasmochemical synthesis, based on combustion of a metal in an oxygen medium with formation of oxide ceramic particles [4, 5]. There are also other methods for obtaining ultradisperse powders, such as (sol--gel) technology [3], laser-initiated chemical vapor-phase deposition [6].

In this paper, we consider a method rather widely used in industry: laser synthesis of nanocrystalline and ultradisperse quasicrystalline powders of aluminum oxide [5, 7]. Our major goal was to establish the effect of the basic parameters of the process (the power of the laser radiation, the pressure and composition of the gas in the reactor) on the phase and fractional composition of the ultradisperse aluminum oxide powder.

Vaporization of the material occurred according to the following scheme. The CO2 laser emission from an LTU-0501 was transported to the target by a system of rotating mirrors and focused on the end of a cylindrical specimen placed in the reactor. The sample was mounted using a conical copper holder. The spot of the focused laser beam had an area of 3·10^{-4} cm^{2}. The power of the laser radiation was registered by the RSI 105-5 power meter. The powders were investigated on the DRON-3M x-ray phase analysis apparatus with cobalt radiation, and also on the JEM-10SX and UEMB-100K electron microscopes. The specific surface was determined by low-temperature adsorption of nitrogen.

Upon action of the laser beam on the target, an erosion jet is formed whose length is determined by the radiation power. An increase in the length of the erosion jet, which is a consequence of a change in the temperature gradient of the medium and hence the cooling rate, affects the formation of the structure of the incipient particles.

In Fig. 1, we present the x-ray diffraction patterns of ultradisperse aluminum oxide powder, taken under identical conditions; in Fig. 2, we present the x-ray diffraction pattern of the γ phase of aluminum oxide. Up to a scattering angle of 20-40°, we observe a halo which can be explained by nanocrystalline or x-ray amorphous structure of the powder particles.
Fig. 1. X-ray diffraction patterns of Al₂O₃ powder obtained for radiation power 300 W (1), 500 W (2), and 750 W (3).

Fig. 2. X-ray diffraction pattern of the γ phase of ultradisperse aluminum oxide powder.

Fig. 3. Particles of ultradisperse aluminum oxide powders obtained at a pressure of 10 kPa (a) and 101 kPa (b).

Data on the interplanar distances in the γ phase of aluminum oxide are presented in the American system ASTM [8]. Two crystalline γ modifications of aluminum oxide exist, which in this paper we call the high-temperature γ and the low-temperature γ phases, and they are distinguished by the crystal lattice parameters.

We should note the characteristic features of the x-ray diffraction pattern of the powder obtained for a radiation power of 300 W. All the lines are identified with γ, δ, and χ phases; the most intense lines belong to the high-temperature γ modification of Al₂O₃, with which we identify the reflections 2-4, 6, 9, 10, 15; the low-temperature γ phase is represented by reflections 7, 9, and 15, and the maximum of line 15 is located at the center between the lines from the high-temperature γ phase and the low-temperature γ phase; the γ-phase has a cubic lattice, yielding intense lines even for a concentration of the given phase on the order of 3%; an intense χ phase is present, represented by reflections 4, 8, 14, 15; we can distinguish traces of δ phase (lines 1, 4, 6, 15); lines 9 and 15 are the superposition of reflections: 9 — from the high-temperature γ phase and the low-temperature γ phase, 15 — from the high-temperature phase, the low-temperature γ phase, the χ phase, and the δ phase; since each reflection has its own characteristic Wulff—Bragg angle, superposition of the reflections leads to formation of a synthetic line with a certain contour. The disappearance of individual phases will be accompanied by a shift of the line, a change in its halfwidth and the shape of the contour.

The x-ray diffraction pattern taken from the powder which was obtained for a laser beam power of 750 W has the following features: the χ phase is characterized by weak lines, splitting of the lines at 20-54° and 80° is not observed; the line in the region of the 80° scattering angle was shifted by 0.5° toward smaller angles, i.e., the ratio of the intensities of the reflections from the different phases forming the given line changed; only traces of high-temperature γ phase are present; the low-temperature γ phase becomes intense.