Phase Transitions and Dimensional and Morphological Changes in an “Aluminum Hydroxide–Alumina” System under a Shock-Wave Action

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A method is proposed to study the sequence of phase transitions in powdered materials under a shock-wave action. It is shown that the “aluminum hydroxide–alumina” system has the following sequence of phase transformations under a shock-wave action: bayerite → boehmite → γ-Al2O3 → α-Al2O3. It is found that there are no transitional high-temperature modifications of alumina. A method is developed for obtaining a submicron alumina powder, which allows obtaining materials with a controlled phase composition, including a thermodynamically stable α-modification of Al2O3. The specific features of the morphological structure and the phase and structural characteristics of powders after a shock-wave action are considered.

Key words: shock-wave action, phase transitions, “aluminum hydroxide–alumina” system, nanocrystallite size.

INTRODUCTION

Phase transitions in the “aluminum hydroxide–alumina” system under a thermal action and normal pressure have been studied in much detail [1–3]. The phase diagram of the Al2O3–H2O system [1] allows one to analyze the phase and chemical transformations in aluminum hydroxides and low-temperature (water-containing) aluminum oxides. The temperatures of phase transitions depending on powder-production conditions were determined for high-temperature modifications of Al2O3 in [1, 2]. The dimensional and morphological changes in the material being heated were considered in [3].

The influence of shock-wave treatment on the γ-modification of Al2O3 was studied in [4, 5]. The experiments were performed by the traditional ampoule scheme of substance conservation. It was shown that the degree of transition of the γ-phase to a thermodynamically stable α-modification depends on the amplitude and duration of the shock-wave action. A powerful charge of pressed TNT is necessary for complete stabilization of the α-Al2O3 phase. The changes in size and morphology of individual particles were not considered because the work was aimed at obtaining a compact material.

There are some papers [6, 7] on synthesis of various modifications of alumina formed via combustion or detonation-induced decomposition of an RDX–gibbsite mixture in thick-walled metallic containers (Bichel bombs). The possibility of stabilization of α-Al2O3 and new oxide phases, e.g., γ-Al2O3, was demonstrated. The use of massive metallic shells around the charge leads to formation of complex oxides [7]. The shock-wave action on gibbsite by the ampoule scheme was considered in [8]. If an HMX charge was used, α-Al2O3 was not formed; the material was stabilized in a new aluminum-oxide modification called γ1-Al2O3 by the authors. Nevertheless, x-ray data made it possible to identify this phase as aluminum oxynitride.
Other possible sequences of phase transitions of aluminum hydroxides and oxides under pulsed actions have not been considered. The significance of these studies is caused both by practical importance of this material (alumina) and by the possibility of obtaining new knowledge about the substance behavior under pulsed high-energy actions. In addition, it is of interest to obtain powder materials with new properties under extreme (explosive) conditions of synthesis.

The objective of the present work was to study the sequence of phase transitions in aluminum hydroxides (bayerite and boehmite) under a shock-wave action and the physicochemical properties of synthesized powders.

**RAW MATERIALS AND EXPERIMENTAL LAYOUT**

The raw material — aluminum hydroxide — is a white powder with a bulk density $\rho = 0.75$ g/cm$^3$. The material to be examined was placed into the outer cylinder.

The powder or its mixture with water in ratio of 3 : 2 was subjected to a shock-wave action. The density of the suspension was $\rho = 1.4$ g/cm$^3$.

The experimental assembly was placed to the center of a leakproof explosive chamber made of stainless steel and closed by a cover; the chamber volume was 24 dm$^3$. The explosion was initiated by an electric-spark method from the upper end of the high explosive (HE). After the explosion, the excess pressure was released through tap 5, and the explosive chamber was ventilated. The synthesized powder was collected through tap 7 in the bottom part of the explosive chamber.

The raw and explosion-processed powders were examined by methods of electron microscopy (JEM-100S electron microscope with an EM-ASID-4 scanning attachment), powder x-ray diffraction analysis (XDA) and structural (DRON-3 automated system), and differential-thermal analysis (derivatograph of the MOM company).

**BASIC RESULTS AND DISCUSSION**

The results of electron-microscopic studies revealed that the raw powder consists of coarse (up to 20 µm) and submicron particles (Fig. 2a). The coarse particles have a layered structure. The mass content of the coarse fraction in the powder is $\approx 50\%$. The shape of submicron particles is predominantly irregular; faceting is observed for some of them.

Based on the XDA data, the raw powder is a mixture of two phases: aluminum trihydroxide (bayerite) and aluminum monohydroxide (boehmite) (Fig. 3a). The width of diffraction maximums for boehmite (Bm) is greater than for bayerite (Br). The reason is that boehmite formation during bayerite annealing proceeds with removal of crystallization water from the lattice, which is accompanied by material fragmentation [3]. According to [1–3], the following sequence of phase transitions is typical of these hydroxides:

$$\text{Br} \rightarrow \text{Bm} \rightarrow \gamma\text{-Al}_2\text{O}_3 \rightarrow \delta\text{-Al}_2\text{O}_3$$

$$\rightarrow \theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3.$$  \hspace{1cm} (1)

The transition temperatures and the presence of intermediate phases strongly depend on the method of obtaining the raw material and its properties, as well as on heating conditions [2]. For instance, boehmite can be absent in sequence (1) but only if bayerite is contained for a long time in vacuum at a temperature $T = 200$ °C. In this case, stabilization of $\gamma\text{-Al}_2\text{O}_3$ proceeds via formation of the amorphous modification of the oxide.