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Morphology of aluminium oxide nanostructures after calcination of arc discharge Al–C soot

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Abstract

The morphology of nanostructures that were formed during the calcination of aluminium–carbon composite and synthesized by the method of electric arc spraying was studied. It was shown that based on the aluminium content in the sprayed electrode and the buffer gas pressure, nanostructures of different morphologies are formed: chains of γAl_2O_3 nanoparticles, hollow γAl_2O_3 nanoparticles, γAl_2O_3 nanotubes, and hollow nanoparticles with inner partitions.

Keywords: A. Calcination; D. Al_2O_3; Arc discharge; Powders; Gas phase reaction

1. Introduction

The interest in the synthesis of Al_2O_3 nanoparticles is due to a series of unique properties of this material, including high mechanical strength and hardness, heat resistance, insulating properties, and both chemical inertness and catalytic properties depending on the context. The above properties enable the application of nano-sized Al_2O_3 materials as the matrix of the carrier [1] and active catalyst component [2]. Synthesis of aluminium oxide nanoparticles is performed via various methods: pyrolysis in flame [3–6], plasma methods [7–9], laser ablation [10,11], explosion of wire [12], oxidation of aluminium in supercritical water [13], and wet chemistry methods [14–19].

Among the methods used for the synthesis of hollow nanoparticles of different compositions, we can distinguish between the methods based on the Kirkendall effect [20,21], templating methods [22–26], methods of thermal decomposition of salts [27,28], and CVD methods [29]. The use of agglomerated powder annealing in DC plasmatrons allows synthesis of hollow ceramic particles of micron size [30,31]. Synthesis of hollow Al_2O_3 nanoparticles was performed by Smovzh et al. [32] by the calcination of aluminium–carbon condensate (soot) obtained by electric arc spraying of the aluminium–graphite electrode. The structure of aluminium–carbon condensate is a carbon matrix with a characteristic agglomerate size of 20–30 nm and nano-sized particles of aluminium and aluminium carbide randomly distributed in this matrix. During the calcination of material, carbon from the agglomerate surface is oxidized, which leads to a reduction in their size and an increase in alumina concentration in the surface layers of particles. The process of aluminium oxidation occurs simultaneously, and at temperatures of 650–700 °C, the conversion reaction of aluminium carbide into oxide takes place. This process occurs until a dense alumina framework is formed, and the subsequent process of carbon oxidation takes place due to the diffusion of oxygen into the particle and the bringing-out of oxidation products that leads to formation of the hollow alumina shells. Formation of hollow membranes by Smovzh et al. [32] was achieved at a particular molar content of aluminium in the sprayed electrode. In this paper, experimental studies investigating the influence of mass aluminium content in the sprayed electrode and buffer gas pressure on the composition and morphology of the

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material, as obtained by the calcination of aluminium–carbon condensation at a temperature of 950 °C, are presented.

2. Experimental setup

The arc plasma reactor (Fig. 1) is vacuum chamber 1, which can be evacuated to a pressure of 10^{-2} Torr and subsequently filled with inert gas to a pressure of 3–500 Torr. There are two electrodes in the reactor, and the arc glows between these electrodes in the inert gas atmosphere. The mobile electrode 2 is a graphite pellet with a diameter of 20 mm, and the immobile electrode (anode) 3 is a rod of 7 mm in diameter and 70 mm in length. This construction allows for variation of the distance between the electrodes and maintenance of the conditions of arc glow. The electrode is moved by the bellows unit of the translation transfer 4. To introduce the current into the reactor, the high-current metal–ceramic hermetic input 5 is used. Around the electrodes, there is a copper water-cooled circuit 6. Inside, there is a removable stainless steel screen 7 for collecting the synthesized products.

The source of the DC current (80–140 A) was used for the arc discharge. Depending on the distance between the electrodes, the voltage obtained on the arc is 20–30 V. The anode is the graphite rod with an axial hole and filled with the aluminium–graphite powder in a certain ratio. Anode spraying leads to the formation of the atomic components of carbon and aluminium in the arc. Diffusion and convection of atomic components in the buffer gas lead to cooling, heterogeneous condensation and chemical reactions. Next, the products of condensation are deposited onto the cooled screen. The synthesized material consists of the nanoparticles of aluminium and aluminium carbide on the carbon matrix. The determining parameters of the synthesis are geometry and the composition of the electrodes, the buffer gas and its pressure, and the electric characteristics of the discharge.

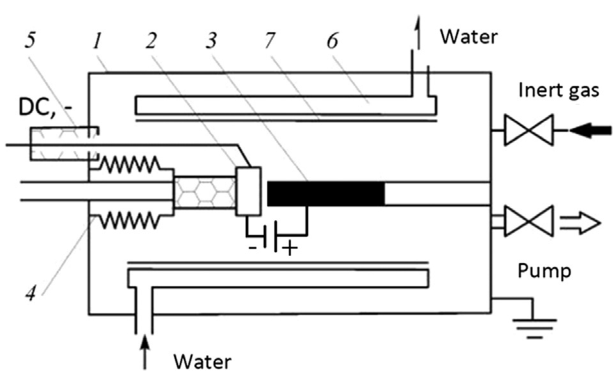

Fig. 1. Experimental setup.

All experiments were performed at a discharge current of 100 A in helium. The weight content of aluminium in the sprayed electrode was varied from 1.5% to 15%, and the pressure was varied in the range of 3–300 Torr. The synthesized material was calcinated at a temperature of 950 °C for 2 h. The phases present in the nanopowders were identified by X-ray diffraction (XRD) using a Bruker D8 Advance diffractometer operated with Cu-Kα radiation. The morphology of the powders was analyzed by transmission electron microscopy (TEM, JEOL – 2010).

3. Results

The material synthesized by electric arc spraying is the aluminium–carbon composite with a bulk density of approximately 0.05 g/cm^3. By spraying the composite anode, a portion of carbon is condensed on the cathode. This approach increases the content of aluminium in the synthesized material. The portion of carbon deposited onto the cathode depends on the buffer gas pressure. According to the measurements by the TGA method during material calcination at the temperature of 1300 K, the enrichment factor is roughly 2.5. The mass of the Al content in the soot is shown in the text below.

TEM analysis showed that this material consists of soot with a characteristic size of non-uniformities of 10–30 nm; at that scale, the particles of metal carbide or aluminium oxide on the carbon background can be distinguished only at a maximal concentration of aluminium in the composite (Fig. 2). According to the X-ray phase analysis, the chemical composition of aluminium–carbon composite changes based on a change in the portion of aluminium in the sprayed electrode. The diffraction spectrum of X-ray radiation on the material, synthesized at a maximal concentration of aluminium, used in the current experiments (38%) is analyzed in Fig. 3. According to this spectrum, the carbon material is partially

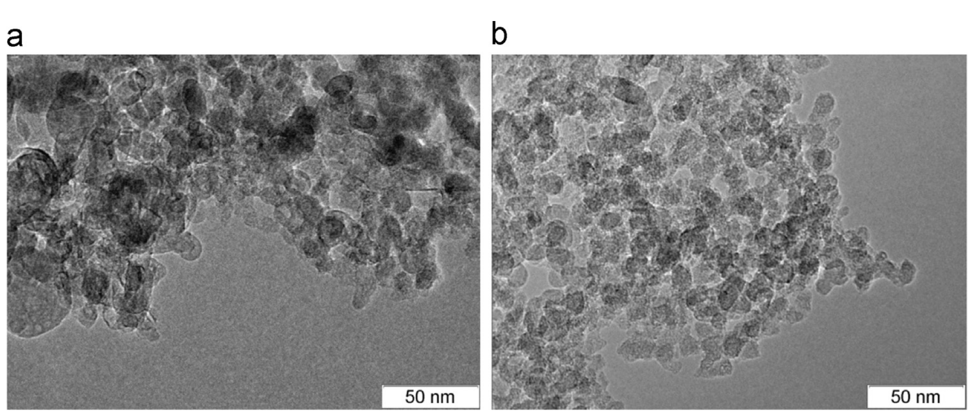

Fig. 2. TEM images of Al–C composite after electric arc dispersion. (a) Concentration of Al at 38%, and (b) concentration of Al at 21%.