Plasma synthesis of Al2O3 from related nitrate

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Abstract. The article deals with plasma synthesis of aluminium oxide particles using a high-voltage alternating current plasma torch. Air plasma is generated between two annular copper electrodes. Behind the plasma torch there is a reaction volume, into which an aqueous solution of aluminium nitrate is fed. Under the influence of high temperature, nitrate decomposes to form an oxide. The composition and properties of the obtained particles were determined using an electron scanning microscope.

1. Introduction

Nanosized aluminium oxide is widely used in the production of resistant coatings [1], many functional catalysts [2], refractory materials, etc. The most valuable properties are nanoscale particles obtained by coprecipitation [3], hydrothermal synthesis [4], self-propagating high-temperature synthesis [5], sol-gel technology [6] and other methods.

All these methods are characterized by the periodicity of the process and low unit productivity, which limits the use of nano-sized aluminium oxide. There are a number of plasma methods for obtaining it: precipitation of thin aluminium oxide films at low pressures (up to 20 torr) at 13.56 MHz by RF generator [7]; treatment of powdered oxides by an electric arc plasma of inert gases [8]. Plasma torch DC (800 V, 35 A) was used as the plasma source, the powder flow rate was 600-900 g/h, the flow rate of the plasma-forming argon was 75 l/min. In this case, nanoscale particles were formed, which melted and formed large grains of aluminium oxide.

Spheroidization of aluminium oxide was carried out at a higher power plant [9]. In this case, a plasma torch with a power of 40 kW worked on a mixture of argon and nitrogen (transport gas for the powder is argon). In the plasma flow, spherical particles with a diameter of about 15 mkm are formed, and with increasing plasma torch power (plasma jet temperature), the fraction of unstable Y-Al2O3 increases.

From the foregoing, it can be concluded that the efficiency of electric arc plasma is high in the synthesis of oxide powder materials. Electric arc plasma torches also showed their effectiveness in the processing of organic compounds [10] and oil-free ignition of pulverized coal boilers [11].
2. Experimental part

Studies on the production of powdered aluminium oxide were carried out in a laboratory facility consisting of a single-phase high-voltage AC plasma torch and a reaction chamber. The plasma torch is two coaxial annular copper electrodes with insulators between them. The plasma torch is powered by an AC power source (50 Hz) with an idle voltage of 6 kV \[12\]. This voltage allows to re-ignite the electric arc without missing current. To stabilize the electric arc in the gap between the electrodes, the tangential supply of plasma-forming gas (air) is organized. One side of the plasma torch is muffled, and through the second side the air plasma flow (1 g/s) enters to the reaction volume. In the reaction volume, the thermal plasma of air is mixed with the flow of an aqueous solution of aluminium nitrate (0.01 g/s). A solution of 0.1 mol/liter aluminium nitrate is prepared from Al(NO\(_3\))\(_3\)\(\cdot\)9H\(_2\)O of high purity. When the aqueous solution interacts with the plasma, water evaporates and the nitrate decomposes to aluminum oxide by the reaction equation (1).

\[4\text{Al(NO}_3\text{)}_3 = 2\text{Al}_2\text{O}_3 + 12\text{NO}_2 + 3\text{O}_2\] (1)

By changing the flow rate of the plasma-forming gas, it is planned to change the temperature of the plasma jet, which will lead to a change in the phase composition of the reaction products. The particles withdrawn from the reaction volume were collected on a cooled surface of stainless steel. The samples were analyzed using the electron microscope with the elemental analysis prefix.

3. Results and discussion

During the continuous operation of the plasma torch at a power of 6 kW, three samples of oxide powders with a condensate were obtained. The condensate was separated by filtration, and the samples were dried at a temperature of 50 °C. Table 1 shows that the composition of dried oxide particles includes products of erosion of the case and electrodes of the plasma torch. Especially a lot of iron in them, eroding from the wall of a plasma torch made of stainless steel. The content of copper in the samples is much smaller, which indicates the stable operation of the electrode assemblies of the plasma torch.

| Sample | O   | Al  | Ti  | Cr  | Mn  | Fe  | Ni  | Cu  | Zn   |
|--------|-----|-----|-----|-----|-----|-----|-----|-----|------|
| 1      | 37.40 | 23.77 | 0.25 | 5.60 | 0.48 | 20.45 | 2.50 | 2.77 | 6.77  |
| 2      | 35.35 | 18.09 | 0.17 | 5.68 | 0.51 | 22.16 | 3.73 | 3.23 | 11.08 |
| 3      | 40.44 | 33.13 | 0.00 | 2.82 | 0.18 | 14.80 | 1.23 | 2.47 | 4.93  |

Figure 1 shows the photomicrographs of the samples at different degrees of magnification. As can be seen, they consist of irregularly shaped particles of different sizes and contain spherical, highly porous and rodlike particles.

The main reason for obtaining particles of different shapes is a significant temperature change in the reaction chamber due to the uneven flow of an aqueous solution of aluminum nitrate.

4. Conclusions

It has been established that decomposition of nitrates using thermal electric arc plasma occurs completely. In this case, the size and shape of the particles depends on the temperature of the plasma jet and the steady supply of an aqueous solution of precursors. Lowering the concentration of nitrates reduces the probability of interaction of particles forming in the hot stream.

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Figure 1. Scanning electron micrographs of a scanning electron microscope.

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