

## Regulation of the Dispersed Composition of Aluminum Oxide Nanopowders Produced by Electrical Explosion

Young-Soon Kwon\*, Olga B. Nazarenko\*\* and Alexander P. Ilyin\*\*

\*Research Center for Machine Parts and Materials Processing, School of Materials and Metallurgical Engineering, University of Ulsan, San-29, Mugeo-2Dong, Nam-Ku, Ulsan 680-749, Korea

\*\*High Voltage Research Institute at Tomsk Polytechnic University, 2a, Lenin Ave., Tomsk 634050, Russia

(Received 16 May 2003 ; Accepted form 9 June 2003)

**Abstract** The feasibility of obtaining highly dispersed aluminum oxide powders by the electrical explosion of aluminum conductors in an inert gas atmosphere and the subsequent oxidation of aluminum particles by water prior to their contact with air is demonstrated. For a specific surface area of the initial aluminum powder of 6.5 m<sup>2</sup>/g, the corresponding specific surface area of the resultant aluminum oxide nanopowder was as large as 300 m<sup>2</sup>/g.

**Keywords :** Aluminum oxide powder, Electric wire explosion, Oxide nano powder

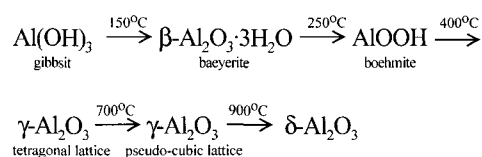
### 1. Introduction

Aluminum oxide nanopowders are widely used to produce inorganic adsorbents, catalysts, ceramics, etc. One of the methods of producing aluminum oxide nanopowders (NP) is the electrical explosion of conductors (EEC) in oxygen-containing atmospheres including air, water, and oxygen and argon mixture<sup>1,2)</sup>.

A study of the properties of powders produced by the EEC in gases demonstrates that their phase contents and degrees of dispersion are determined by the amount of energy deposited into the conductor and by the partial oxygen pressure in the gaseous mixture<sup>1)</sup>. The degree of dispersion of nanopowders increases (the average surface particle diameter  $a_s$  decreases down to 0.05  $\mu$ m) as the energy  $e$  deposited into the conductor increases to  $e/e_s \sim 1.5$ , where  $e_s$  is the sublimation energy of the conductor material. With further increase in  $e/e_s$ ,  $a_s$  becomes virtually independent of the amount of the deposited energy (Fig. 1). With increasing gas pressure, the particle size grows fast (Fig. 2). EEC in gaseous media can be used to produce nanopowders with not so high degree of dispersion (with specific surface areas as large as 40–60 m<sup>2</sup>/g); moreover, the energy consumption in the process of their production also increases.

Coarsely dispersed aluminum powders form aluminum hydroxide in the process of relatively slow interaction with water<sup>3)</sup>. The scheme of aluminum hydroxide

transformations on heating is described by the following sequence of reactions<sup>4,5)</sup>:



The interaction of fine powders with water is accompanied by particle self-heating. It was established that when aluminum NP, produced in a gaseous argon atmosphere and passivated under conditions of slow air intake, are oxidized in water at temperatures 60–100°C. The conditions of forming  $\beta\text{-Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ , AlOOH, and amorphous aluminum oxide are realized on self-heating<sup>6)</sup>. Cooling and passivation of aluminum powders after their production decrease their activity.

To exclude a number of intermediate operations from the aluminum oxide production process, it was suggested to set off an electrical explosion of aluminum conductors immediately in water<sup>7)</sup>. Unlike gases, liquids are more dense media, which causes the formation of larger particles compared to explosions in gases. In addition, the electrical explosion of aluminum conductors in water complicates the technological setup in connection with increased hydrodynamic loads on its parts. Severe problems arise with high-voltage insulation of the setup.

The present paper studies the feasibility of increasing the degree of dispersion of aluminum oxide pow-

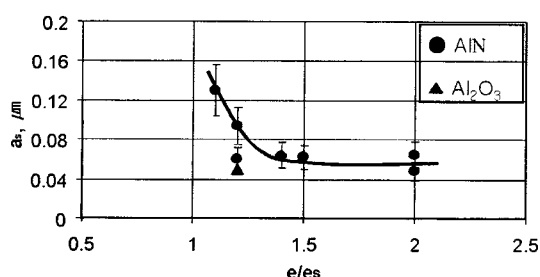


Fig. 1. Dependence of the average particle diameter on the energy content of explosive conductors.

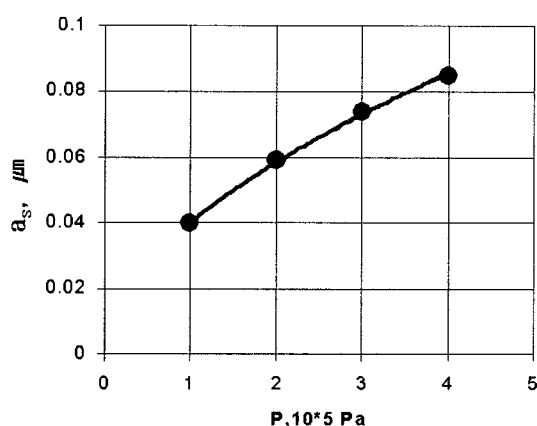


Fig. 2. Dependence of the average diameter of  $Al_2O_3$  particles on the pressure of the gas medium.

ders produced by the explosion of aluminum conductors above the water surface in an inert gas atmosphere with subsequent interaction of hot unpassivated aluminum NP with water.

## 2. Materials and Experimental Procedure

Investigations were performed using an electrical setup comprising a high-voltage power source, a capacitive energy storage device, a reaction chamber, a gas-supply system, and a controllable spark discharger. Before the experiment, the reaction chamber was evacuated and then filled with argon. An aluminum conductor of 0.25 mm in diameter 100 mm long was placed 30 mm above the water surface. An electrical explosion was set off with the deposited energy  $0.9 e_s$ . The phase contents of the resultant powders were analyzed using a DRON-3.0 x-ray diffractometer. The particle shapes and sizes were observed with a JSM-840 scanning electron microscope. The specific surface area was measured by the method of low-temperature

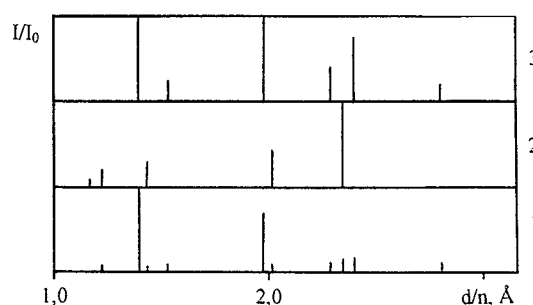


Fig. 3. X-ray diffraction patterns of the electrical explosion products of aluminum conductors in argon above the water surface (curve 1).  $Al^0$  (curve 2) and  $\gamma-Al_2O_3$  (curve 3) patterns are given together (from the ASTM data).

argon adsorption (the BET method).

## 3. Results and Discussion

According to the data of x-ray diffraction analysis, the end product formed by the electrical explosion of the aluminum conductor above the water surface showed low-temperature modification of  $\gamma-Al_2O_3$  with a contaminant of nonreacted aluminum of about 15% (Fig. 3). The particle shapes were close to spherical ones, and the particle sizes were between 3–5  $\mu m$  and 0.1  $\mu m$  and smaller. The measured specific surface area of the aluminum oxide powder samples was  $S_{sp} = 34.8 m^2/g$ . The average particle diameter calculated for the given value of  $S_{sp}$  was 47 nm. After heating of the sample at 800°C, the specific surface area increased to 146.3  $m^2/g$ , which corresponded to an average particle diameter of 11 nm.

A comparison of powders obtained by the EEC above the water surface in the protective gas atmosphere and in water with approximately identical amounts of energy deposited into the conductors demonstrates that the EEC above the water surface leads to a decrease in the residual aluminum content and better retaining of spherical particle shapes. At the same time, the presence of the low-temperature modification  $\gamma-Al_2O_3$  indicates that the temperatures of interaction of aluminum dispersed by the electrical explosion with water are almost the same in both cases ( $\sim 400^\circ C$ )<sup>7</sup>. Moreover, the maximum of the particle size distribution is shifted toward ultradispersed particles (Fig. 4). Aluminum particles formed during the electrical explosion above the water surface fall into water, having no time to coagulate. In fact, indi-

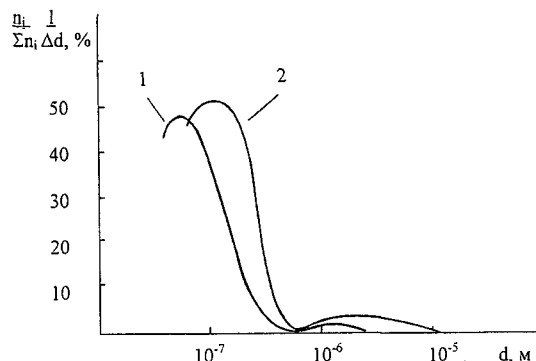


Fig. 4. Average particle size distribution: 1) the EEC above the water surface and 2) the EEC in the bulk of water.

vidual powder particles interact with water producing highly dispersed aluminum oxide and gaseous hydrogen. In this case, the concentration of agglomerated particles decreases significantly and the concentration of ultradispersed particles with sizes in the range 0.1–0.01  $\mu\text{m}$  increases.

In the process of production of the aluminum oxide powder, hydrogen by-product gradually substitutes argon. This obviates the need for addition of argon into the reaction chamber; only the hydrogen pressure should be periodically decreased in it.

#### 4. Conclusions

1. The electrical explosion of aluminum conductors in the protective gas medium above the water surface with the subsequent oxidation of the resultant aluminum particles leads to the formation of highly dispersed aluminum oxide powder with the specific surface area as large as 300  $\text{m}^2/\text{g}$ .

2. The process of producing aluminum oxide pow-

der can be combined with that of pure hydrogen.

#### Acknowledgements

This work was supported by the Korea Research Foundation Grant(KRF-2002-042-D00074) and was performed by using the facilities of ReMM(Research Center for Machine Parts and Materials Processing) at University of Ulsan.

#### References

1. M. I. Lerner: Regulation of the process of forming highly dispersed particles by the electrical explosion of conductors, Candidates Dissertation in Technical Sciences, Tomsk (1988).
2. O. B. Nazarenko: Specific features in the formation of products during the electrical explosion of conductors in condensed media, Candidates Dissertation in Technical Sciences, Tomsk (1996).
3. V. N. Ananin, V. V. Belyaev and V. E. Romanenkov: Hydrothermal oxidation of aluminum powders with different degrees of dispersion. *Izv. Akad. Nauk Bel.SSR, Ser. Khim.*, (1988) 17.
4. N. A. Toropov, V. P. Barzakovskii and I. A. Bondar: Phase Diagrams of Silicate Systems, A Handbook, Issue 2, Metal-Oxygen Compounds of Silicate Systems, Nauka, Leningrad, (1969).
5. R. Rinan and I. Chetyanu: Inorganic Chemistry. Chemistry of Metals. Vol. 1. Mir, Moscow.
6. A. P. Lyashko, G. G. Savelev and A. P. Ilin: Specific features of the interaction of submicron aluminum powders with liquid water, *Kinet. Katal.*, **31** (1990) 967.
7. A. P. Ilin, O. B. Nazarenko, V. Y. Ushakov: Production of high-temperature modification  $\gamma\text{-Al}_2\text{O}_3$  by the electrical explosion of conductors in water, *Zh. Tekh. Fiz.*, **66** (1996) 131.