Plasma way for oxide nanoparticles obtaining

A A Safronov, V E Kuznetsov, Yu D Dudnik, V N Shiryaev and O B Vasilieva
Institute for Electrophysics and Electric Power, Russian Academy of Sciences, 191186, St. Petersburg, Russia
E-mail: julia_dudnik-s@mail.ru

Abstract. The paper deals with various designs of alternating current plasma torches that can be used in the technological cycle for production of nanoscale materials. The results of experimental studies on the production of ultrafine powders of metal oxides (iron and aluminum) by plasma methods with the help of a laboratory installation based on an alternating current plasma torch are presented.

1. Introduction
Plasma production of ultradispersed and nanosized materials is one of the promising areas of application of knowledge about the physical processes occurring during the interaction of an electric arc and the high-enthalpy gas flow with various materials.

Plasma technology is a promising one for solution of such problems. The high values of the enthalpy of arc plasma allow virtually evaporation of any material, provided that the reactant is correctly introduced into the plasma and has a sufficient residence time in the hot zone. High temperature gradients available for plasma-thermal reactors can result in homogeneous nucleation, which allows creation of nanoscale materials. It is known about production of nanoscale metal powders of transition metals in a plasma reactor [1]. The studies [2, 3] also describe the processes of obtaining nanomaterials using a direct current plasma arc or high-frequency plasma arc.

It can be concluded from data analysis that for plasma-thermal production of ultrafine materials, direct current plasma torches with power up to 100 kW or high-frequency plasma torches with power up to 50 kW are used. At the same time, the particle size varies in a wide range from 2 nm to 2 μm, and there is no exact data on throughput. In this connection the use of an alternating current plasma torch with power of up to 100 kW may be more economically feasible because this class of plasma torches have high efficiency and provide a long life time of continuous operation. This opens up the possibility of producing significant quantities of ultrafine materials in a continuous cycle while ensuring a high chemical purity of the process. In addition, the high enthalpy of the arc plasma allows evaporation of almost any material, providing the possibility to produce nanophase structures, even refractory materials.

2. Experimental installation
Among the existing designs of alternating current plasma torches, developed in Institute for Electrophysics and Electric Power Russian Academy of Sciences, the single-phase plasma torches are the most suitable for the creation of a laboratory installation. These plasma torches are capable of working not only on inert and oxygen-free (reduction) gases, but also on oxidizing media in the power range from 5 to 50 kW and the plasma-forming gas flow rate from 0.5 to 30 g/s. At the same time, the
life time of the electrodes is more than 200 hours, and the efficiency is 90%. General view of the experimental installation and its block diagram are shown in figure 1 and 2 respectively.

Figure 1. Experimental installation: 1 – single-phase plasma torch; 2 – input device; 3 – nozzle; 4 – reaction chamber; 5 – separator; 6 – output nozzle; 7 – nozzle for hardening; 8 – container for receiving product.

The installation consists of a single-phase plasma torch connected to the input device with the nozzle that is connected to the reaction chamber, which goes in the separator, and then closes on a hardening unit with the container for receiving product with the output nozzle and the nozzle for hardening. Also, a characteristic feature of the used solution is the application of an aqueous solution of the precursor of metal salts.

Figure 2. The block diagram: 1 – power source; 2 – supply of plasma-forming gases; 3 – valve; 4 – control and flow measurement unit; 5 – reactor; 6 – plasma torch; 7 – quencher; 8 – feeder; 9 – cooling system; 10 – water pump; 11 – water flow rate measurement system; 12 – filter; 13 – output system; 14 – temperature measurement system; 15 – parameter recording system.

3. Results and discussion
To date, exploratory experiments have been carried out, the purpose of which was to demonstrate the possibility of production of ultradispersed oxide powders by the indicated method on the example of iron (III) oxide. When using iron (III) nitrate solution as a precursor, the following operating
parameters of an alternating current plasma torch were established, contributing to the formation of Fe₂O₃ nanopowder: power 6 kW, operating voltage 1000 V, total plasma gas flow rate 1 g/s, precursor flow rate from 0.5 to 1.5 g/s. A fine brown powder was produced as a result of plasma-chemical synthesis under the specified conditions. This powder, according to the results of X-ray phase analysis, is almost a single-phase α-Fe₂O₃ [4, 5].

The average crystallite size was estimated by the Scherrer formula, based on the broadening of X-ray diffraction lines, which was 57 ± 6 nm. Also, experiments were carried out on plasma production of aluminum oxide particles with the same parameters of the plasma torch power and plasma gas flow rate. Figure 3 shows micrographs of samples at various degrees of magnification. As one can see, they consist of particles of irregular shape of various sizes and contain spherical, highly porous and rod particles.

![Micrographs of α-Fe₂O₃ nanopowder taken by a scanning electron microscope in various scale: (a) – 2 μm; (b) – 2 μm; (c) – 50 μm; (d) – 200 μm.](image)

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

4. Conclusions
As a result of this work, the possibility of α-Fe₂O₃ nanopowder production under conditions of plasma-chemical synthesis using a continuous reactor based on a high-voltage alternating current
plasma torch was shown. The obtained iron oxide nanocrystals have an average size of $57 \pm 6$ nm and are weakly agglomerated with each other, which makes it possible to consider this synthesis method as promising one for the synthesis of oxide nanopowders from aqueous solutions of their salts.

The possibility of plasma production of ultrafine particles of aluminum oxide has also been demonstrated. It is determined that the nitrates are completely decomposed under the action of thermal electric arc plasma. The size and shape of the particles depend on the temperature of the plasma jet and the steady supply of an aqueous solution of precursors. Decreasing of the nitrates concentration reduces the probability of the interaction of particles forming in a hot stream.

References

[1] Girardin D and Manrer M 1990 *Mat. Res. Bull* **25** 119–25
[2] Uda M 1989 *Nisshin Steel Tech. Rep.* **61** 573–604
[3] Kikukawa N, Kobayashi M, Sugasawa M and Sakamoto H 1992 *J. High Temp. Soc. Jpn* **18** 235–47
[4] Kuchina Yu A, Subbotin D I, Kumkova I I, Dudnik Yu D, Kuznetsov V E, Popkov V I, Popov V E, Cherepkova I A, Pavlova E A, Shiryaev V N and Obraztsov N V 2018 *Journal of Physics: Conference Series* **1135** 012095
[5] Subbotin D I, Surov A V, Kuznetsov V E, Pavlova E A, Azartsova VV, Kuchina J A and Dudnik J D 2018 *Journal of Physics: Conference Series* **1115** 032093