PRODUCTION OF NANOPOWDERS USING HIGH POWER ELECTRON ACCELERATOR

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Abstract

Electron-beam evaporation of various materials by an electron accelerator of ELV type in the atmosphere of various gases at atmospheric pressure can be used for the synthesis of nanopowders.

The present paper is devoted to the production of nanopowders of silica (SiO_2), magnesium oxide (MgO), alumina (Al_2O_3), copper oxide (Cu_2O), titanium dioxide (TiO_2), gadolinium oxide (Gd_2O_3), various metals (tantalum, molybdenum, nickel, aluminum, copper, silver), nitrides (AlN, TiN).

INTRODUCTION

Synthesis and characterization of highly dispersed powders of various substances is a currently important trend in modern science and technology. This importance is related, on the one hand, to the practical need for new materials that can sometimes be developed only on the basis of powdered components and, on the other hand, to the basic interest in the properties of very small particles (in particular, those with dimensions below 100 nm) within the framework of a broad and rapidly developing field of knowledge called "nanotechnology." In this context, special attention is devoted to the creation of highly effective methods for the synthesis of nanopowders. However, the efficiency of most such methods (especially those proposed for obtaining nanopowders of metals, nitrides, and carbides) is still very low.

This investigation dealt with assessing the possibility of obtaining nanopowders of a broad class of substances by means of evaporation of various materials in an electron accelerator in the atmosphere of various gases at atmospheric pressure. Previously, an electron accelerator had been used [1] for the synthesis of ultradispersed oxide powders, but the process was carried out at low power (1 kW), pressure, and efficiency. On the whole, the method used in this study can be classified into condensation technologies for obtaining ultrafine powders of refractory substances. To the best of our knowledge, the first use of a CO_2 laser for powder production was reported [2] where the process was also performed at low power and efficiency. The use of plasma generators in the condensation technology of ultrafine powders dates back to approximately the same period of time [3]. In comparison with laser and plasma technologies, the proposed method has the advantage that the energy is deposited inside the target substance rather than at its surface.

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EXPERIMENTAL

Our experiments have been performed on a commercial 100 kW accelerator of ELV type, whose special features are high electron energy (1.4 MeV) and the possibility of extracting the electron beam into the atmosphere. Electrons move in this beam at relativistic velocities and their mean free path length in air reaches 6 m. The beam power density can be as high as 5 MWt/cm^2, and this makes possible both the evaporation of refractory substances under atmospheric conditions and the high-temperature gas phase synthesis. Other advantages are high efficiency of direct conversion of electric energy into thermal energy in the heated material, high rate of heating (above 1000 K/s), and "chemical purity" of the electron beam.

The initial solid material is evaporated inside the reactor, then the vapor is transported from the zone of heating, mixed with air or some other carrier gas and cooled. Finally, the condensed and solidified nanoparticles are trapped and collected (in a powder form). Reactors of various types have been designed, in which the synthesis of nanoparticles can be performed in air, argon, nitrogen, helium and xenon. Even with incomplete (below 50%) use of the accelerator power, the nanopowders of some materials were obtained with a yield of several kilograms per hour.

RESULTS

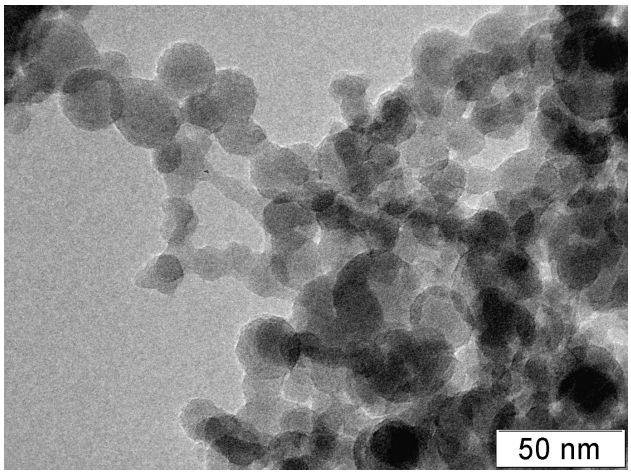
Various nanopowders were synthesized, and their physicochemical properties were studied. Part of them were compared with the properties of the materials obtained by different techniques. The samples were characterized using transmission electron microscopy (TEM), scanning electron microscopy (SEM), X-ray diffraction (XRD), specific surface measurements, chemical analysis and some other methods. The XRD measurements for all samples were performed within a long time after synthesis.

We synthesized, in particular, the following nanopowders.

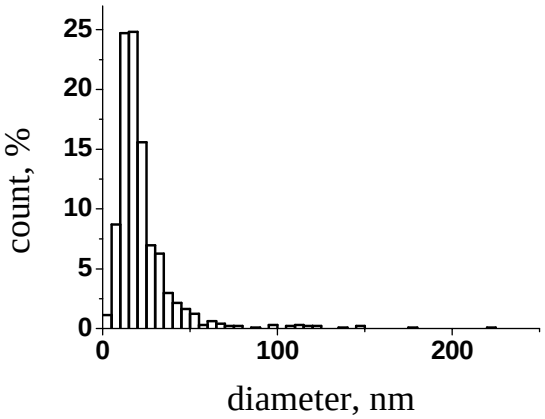
Silica Nanopowders

Silica (SiO_2) nanopowder, white with a blue tint, was obtained by evaporating high purity quartz rock (α-SiO_2; XRD data bank card number, 46-1045) or quartz sand (vaporization temperature 3000°C). The process was carried out in air at a flow rate of 900 m^3/h. The rate of

target evaporation reached 6 kg/h at the accelerator power of 50 kW.



(a)



(b)

Figure 1: (a) -TEM of silica nanopowder; (b) – particle size distribution.

Various SiO_2 powders were obtained, the product was X-ray-amorphous in all series of the samples. In addition, all samples were characterized by high chemical purity: even when the initial material had a relatively high content of impurities, their concentration in the final powder was much lower than in the target. This result indicates that electron-beam evaporation is accompanied by separation of substances and binding of impurities in the melt (probably, with the formation of low-volatile aluminosilicates). In particular, concentrations of Al, Fe, and Ti (recalculated for oxides) in the collected nanopowder were below 0.020, 0.015, and 0.002 wt %, respectively, which was several times as low as the level of these impurities in the initial material. On the whole, the content of SiO_2 in the final nanopowder was above 99.5% and almost satisfied, for example, the state standard for materials of the Aerosil type.

The results of specific surface measurements based on nitrogen adsorption showed that, depending on the regime of synthesis, this value ranged from 20 to 200 m^2/g, which implies that the average particle radius in the obtained nanopowders varied from 15 to 120 nm. For the powder with a specific surface of 116 m^2/g the particle size ranged from 6–8 to 80 nm. This is confirmed by the TEM micrograph (see Fig. 1a), which also shows that nanoparticles can form relatively large agglomerates. The particle size distribution is rather narrow (see Fig. 1b). It should be noted that SiO_2 powders did not change their specific surface and remained X-ray-amorphous upon prolonged storage in air under uncontrolled humidity conditions. The results of tests show that SiO_2 nanopowders synthesized using the proposed method can be successfully applied at least in several fields traditional for the use of Aerosil.

Magnesium Oxide Nanopowders

Analogous experiments performed with electron-beam evaporation of magnesium oxide (MgO) in air showed that nanopowders of this compound with an average particle size of 80 nm and a minimum size of about 20 nm estimated from X-ray analysis and supported by electron microscopy could also be obtained with rather high efficiency. In contrast to the case of silica, magnesium oxide particles possessed a crystalline structure (periclase; XRD, 45-496) with standard values of crystal lattice parameters. The sample contains cube-shaped nanoparticles (see Fig. 2)

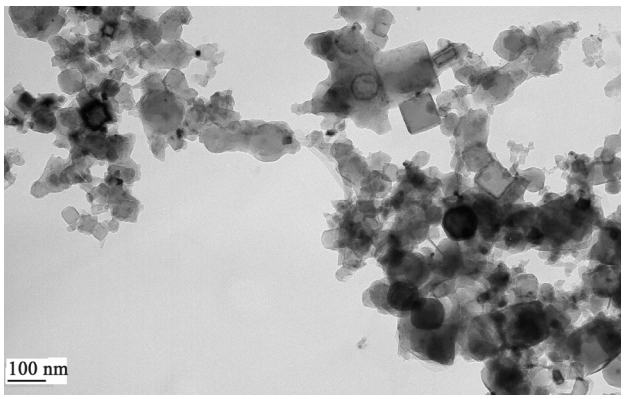


Figure 2: TEM of magnesia nanopowder.

Other Nanopowders of Oxides and Nitrides

These nanopowders were obtained in smaller amounts as compared to the yield of, for example, SiO_2. This was mostly related to a lower beam power and vapour pressure of the substance. In addition to the above two oxides, we also obtained alumina (Al_2O_3), silicon monoxide (SiO), copper oxide (Cu_2O), gadolinium oxide (Gd_2O_3) and nitrides (AlN, TiN).

In particular, at this stage gadolinium oxide (Gd_2O_3) was primarily tested to estimate the yield, which was about a few grams at 40 kW of electron beam power. Gadolinium oxide (Gd_2O_3) nanoparticles are less than 100 nm in size, and form chain-like agglomerates (see Fig. 3).

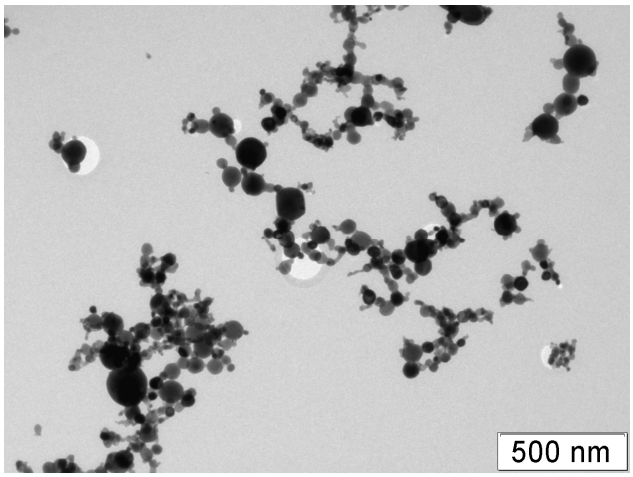


Figure 3: TEM of gadolinium oxide nanopowder.

A nanopowder of titanium nitride (TiN) with a specific surface of 28 m^2/g was obtained by evaporation of a titanium nitride target in nitrogen atmosphere. The product appeared as a black powder with blue tint and contained a pure cubic titanium nitride phase (38-1420) (see Fig. 4).

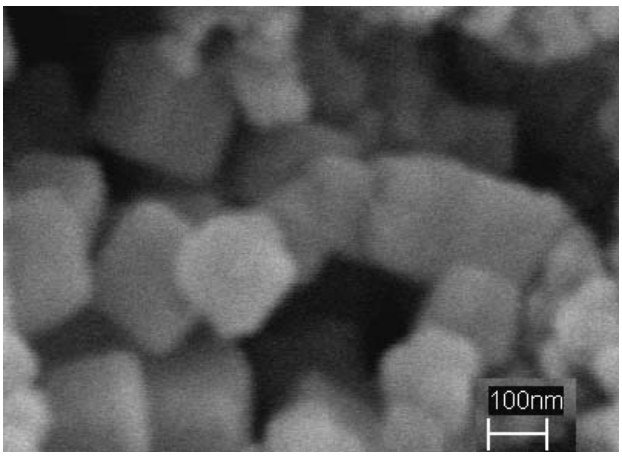


Figure 4: SEM of titanium nitride nanopowder.

Metal Nanopowders

The possibility of obtaining metal nanopowders was mostly studied during their evaporation in argon at a pressure slightly above atmospheric one. To determine limitations of the proposed technology, we tried to evaporate compact tantalum (vaporization temperature about 5500°C). The product was a black conducting powder with cubic metallic phase (XRD, 4-7883) and orthorhombic Ta_2O_5 phase (XRD, 25-922). The powder had a specific surface of 7–10 m^2/g, which corresponded to an average particle size of about 50 nm (TEM micrographs showed the presence of some coarse particles with dimensions reaching 500 nm).

Generally, the same results were obtained for molybdenum (vaporization temperature 4800°C). The powder was black with blue tint. The smallest particles were about 20 nm in size but varied in shape. The XRD data showed that the powder consisted predominantly of pure molybdenum (XRD, 42-1120) with admixtures of monoclinic MoO_3 (XRD, 47-1320), orthorhombic MoO_3 (XRD, 32-671) and MoO_2 (XRD, 32-671) phases; total impurity content was below 20%. A joint analysis of these results with allowance for TEM data suggested that particles differing in shape had different phase compositions.

After evaporation in argon and rapid extraction from the experimental setup, nickel (vaporization temperature 2900°C) was a grayish-black powder. The product volume rapidly decreased, and its specific surface changed from the initial value of 3.2 m^2/g (that corresponded to an average particle size of approximately 200 nm) to about half of this value. Nevertheless, the obtained particles were (within experimental accuracy) pure nickel (XRD, 4-850) with equilibrium crystal lattice parameters.

Silver, copper and aluminium nanopowders were also obtained. Electron-beam evaporation of aluminum target in nitrogen under certain conditions resulted in aluminum powder with a small (1 wt %) content of aluminum nitride. This aluminum powder (as well as nickel powder described above) did not exhibit self-ignition in air.

CONCLUSION

Thus, the results of our investigation showed that electron-beam evaporation of materials using high power electron accelerator in the atmosphere of various gases at atmospheric pressure can be used for the synthesis of nanopowders. These powders are characterized by high purity, exhibit unusual properties, and can be used in various technologies (electronics, catalysis, ceramics, composites, etc.).

An important feature of the proposed technology is that the main device of the technological setup (commercial electron accelerator) is capable of generating high power electron beams, which ensure the development of high temperatures necessary for the evaporation of any refractory material. The process of nanopowder synthesis is highly effective: in particular, the yield of oxides can exceed ten kilograms per hour.

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