

<https://doi.org/10.36719/2707-1146/47/44-48>

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Plasma Chemical Synthesis Research

Abstract

When using uranium – 238, thorium – 232 and plutonium – 239 isotopes, there is no need for expensive isotopic enrichment, and the use cycle of such nuclear fuel can be extended to 10-15 years. At the same time, the predicted reserves of thorium in the earth's crust are 3-5 times greater than uranium, and the use of ceramic nuclear fuel from oxide compositions based on thorium will make it possible to create ultra-small and small power plants for use in remote and hard-to-reach regions, in mines and quarries. However, ceramic nuclear fuel still has a significant drawback – low thermal conductivity. One of the promising areas for the further development of nuclear energy is the use of dispersion nuclear fuel, in which inclusions of fissile metals (uranium, thorium, plutonium) in the form of granulated oxide compositions (microspheres) are placed in a matrix with a high thermal conductivity coefficient.

Keywords: *plasma, chemical, synthesis, methods, nitrides*

Introduction

One of the most common chemical methods for obtaining highly dispersed powders of nitrides, carbides, borides and oxides is plasma-chemical synthesis. The main conditions for obtaining highly dispersed powders by this method are the reaction occurring far from equilibrium and a high rate of formation of new phase nuclei at a low rate of their growth. In real conditions of plasma-chemical synthesis, it is advisable to obtain nanoparticles by increasing the cooling rate of the plasma flow in which condensation from the gas phase occurs; due to this, the size of the resulting particles is reduced, and the growth of particles by merging during collisions is suppressed. In plasma-chemical synthesis, low-temperature (4000-8000K) nitrogen, ammonia, hydrocarbon, argon plasma of arc, glow, high- or ultra-high-frequency discharges is used; elements, their halides and other compounds are used as feedstock (Parkhomen, 1991, p. 166-170).

The characteristics of the resulting powders depend on the raw materials used, the synthesis technology and the type of reactor. Particles of plasma-chemical powders are single crystals and have sizes from 10 to 100-200 nm and more. Plasma-chemical synthesis ensures high rates of formation and condensation of the compound and is characterized by fairly high productivity (Dorzet, 1978, p. 103-110).

The main disadvantages of plasma-chemical synthesis are a wide distribution of particle sizes and, as a result, the presence of fairly large (up to 1-5 μm) particles, i.e. low selectivity of the process, as well as a high content of impurities in the powder. To date, the plasma-chemical method has been used to obtain highly dispersed powders of titanium, zirconium, hafnium, vanadium, niobium, tantalum, boron, aluminum and silicon nitrides, titanium, niobium, tantalum, tungsten, boron and silicon carbides, magnesium, yttrium and aluminum oxides (Dulnev, 1974, p. 264).

The plasma-chemical method is most widely used for the synthesis of nitrides of transition metals of groups IV and V. The plasma temperature, reaching 10,000 K, determines the presence of ions, electrons, radicals and neutral particles in an excited state. The presence of such particles leads to high rates of interaction and rapid reactions. High temperature ensures the transition of almost all initial substances to a gaseous state with their subsequent interaction and condensation of products. Plasma-chemical synthesis includes several stages. Powders obtained by plasma-chemical synthesis have a regular shape and particle size from 10 to 100 nm and more (Misnar, 1968, p. 464).

Plasma-chemical powders of metal, boron and silicon carbides are usually obtained by the interaction of chlorides of the corresponding elements with hydrogen and methane or other hydrocarbons in argon high-frequency or arc plasma; nitrides are obtained by the interaction of chlorides with ammonia or a mixture of nitrogen and hydrogen in low-temperature microwave plasma. Using plasma-chemical synthesis, it is also possible to obtain multicomponent submicrocrystalline powders, which are mixtures of carbide and nitride, nitride and boride, nitrides of different elements, etc. Synthesis of oxides in electric arc discharge plasma is carried out by evaporation of metal with subsequent oxidation of vapors or oxidation of metal particles in oxygen-containing plasma (Polmear, 2008, p. 464).

Plasma-chemical synthesis of aluminum oxide nanoparticles with an average size of 10-30 nm is described. It follows from the results of this work that the formation of aluminum oxide nanopowders with a minimum particle size is achieved by the interaction of metal vapors with atmospheric oxygen under conditions of intense air injection, due to which a rapid decrease in temperature occurs. Plasma-chemical synthesis with the oxidation of aluminum particles in a flow of oxygen-containing plasma leads to the formation of larger oxide particles compared to the oxidation of pre-obtained metal vapor. Gas-phase synthesis using laser heating of the reacting gas mixture is quite closely related to plasma-chemical synthesis (Calliot, 1963).

The reliability and cost-effectiveness of the synthesis of nanopowders in laser-stimulated gas-phase reactions make this method quite competitive compared to other methods. Laser heating ensures controlled homogeneous nucleation and eliminates the possibility of contamination. Such a material should consist of hollow spherical molecules, the shell of which is built of graphite sheets, and 5-membered cycles should be included in the network of hexagonal rings of C₆₀ for stability. However, no one noticed that a similar design had already been proposed in 1951 by the famous American architect (Samsonov, 1978, p. 472).

Fuller, who patented a structural basis for the construction of spherical structures - the so-called geodesic dome. Such a dome design was used, for example, in the construction of the US pavilion at the Expo 67 World Exhibition in Montreal. Fullerenes are obtained by electric arc spraying of graphite in an He atmosphere; the gas pressure is 1.33 · 10⁴ Pa. As a result of the combustion of the arc, soot is formed, which condenses on the cold surface (Alekseev, 2013, p. 240).

After evaporation of the solution, a black condensate is formed, which consists of about 10-15 % of a mixture of fullerenes. To obtain fullerenes, instead of an electric arc, electron beam evaporation and laser heating are also used. The central place among fullerenes belongs to the C₆₀ molecule, which has the highest symmetry and, as a consequence, the greatest stability. In shape, the fullerene molecule resembles a football cover and has the structure of a regular truncated icosahedron (Fig. 1).

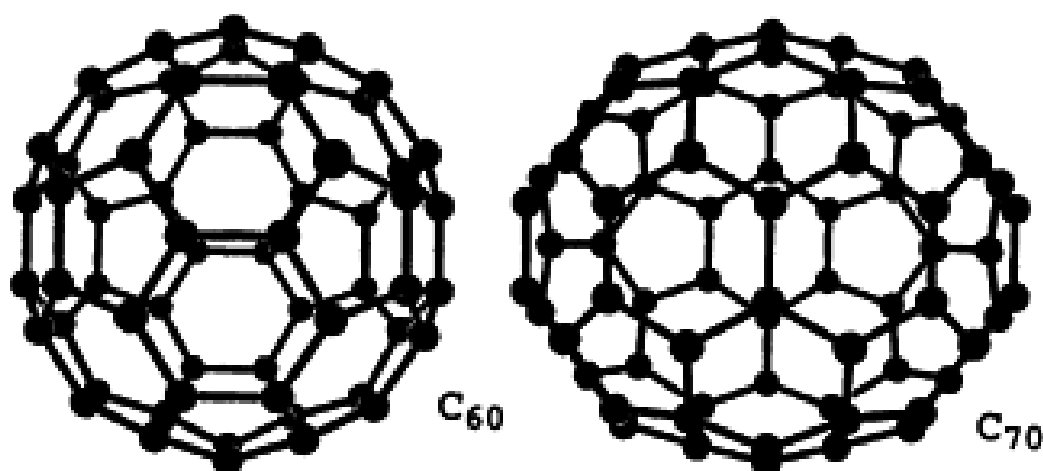


Figure 1. The structure of the most important fullerenes C60 and C70

The C60 molecule is built like a football and has a diameter of about 0.7 nm. All fullerenes contain hexagonal six-membered and pentagonal five-membered rings of carbon atoms. In the C60 fullerene molecule, the carbon atoms form a closed hollow spherical surface consisting of 5- and 6-membered rings, with each atom having a coordination number of three and located at the vertices of two hexagons and one pentagon. The diameter of the fullerene molecule is 0.72-0.75 nm. When C60 crystallizes from a solution or gas phase, molecular crystals with an FCC lattice are formed; the lattice parameter is 1.417 nm. Fullerene in the solid state is called fullerite. Fullerene (270, which has the shape of a closed spheroid, also has high stability. Fullerenes can be considered as a spherical form of graphite, since the mechanisms of interatomic bonding in fullerene and bulk graphite are very similar to a large extent (Hollek, 1988, p. 319).

In early 2001, a group of scientists discovered a new fullerene-like form in which, compared to ordinary fullerene, a fifth of the carbon atoms are replaced by nitrogen atoms (Fig. 2). If in fullerene crystals the molecules are united by weak van der Waals forces, then the presence of nitrogen atoms leads to the appearance of strong covalent bonds. For this reason, the fullerene-like crystalline material has unique very unusual properties of fullerenes.

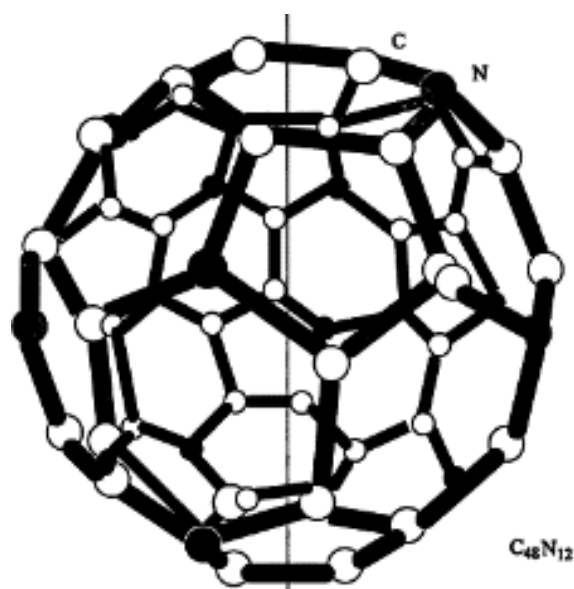


Figure 2. Fullerene structure, the solid line shows the position of the C60 symmetry axis

Thus, crystalline fullerenes are semiconductors and have photoconductivity, and crystals doped with alkali metal atoms.

In general, plasma-chemical synthesis with different methods of creating plasma is one of the most promising methods for obtaining various nanostructured materials.

The undoubted advantages of using plasma for plasma-chemical synthesis of oxide compositions for dispersion nuclear fuel from dispersed mixed aqueous nitrate solutions compared to the sol-gel process and technology based on separate production and mechanical mixing of metal oxides include: single-stage; high speed; the ability to actively influence the size and morphology of particles; compactness of process equipment. Figure 3 shows a diagram of plasma processing of dispersed solutions (Matzke, 1999).

The initial solution is dispersed and fed into the plasma jet. High dispersion of the droplets facilitates their faster and deeper processing. The formation of the target product can be achieved in two ways. The first is that the solution droplets evaporate completely, homogeneous chemical reactions occur in the gas phase, and when the reaction gases are cooled, nanosized particles of the target product are formed. The second way is the formation of particles due to the emergence of crystallization centers and their subsequent growth directly in a liquid drop of the initial solution, which can lead to the formation of larger particles. Mixed aqueous solutions or suspensions are used as feedstock.

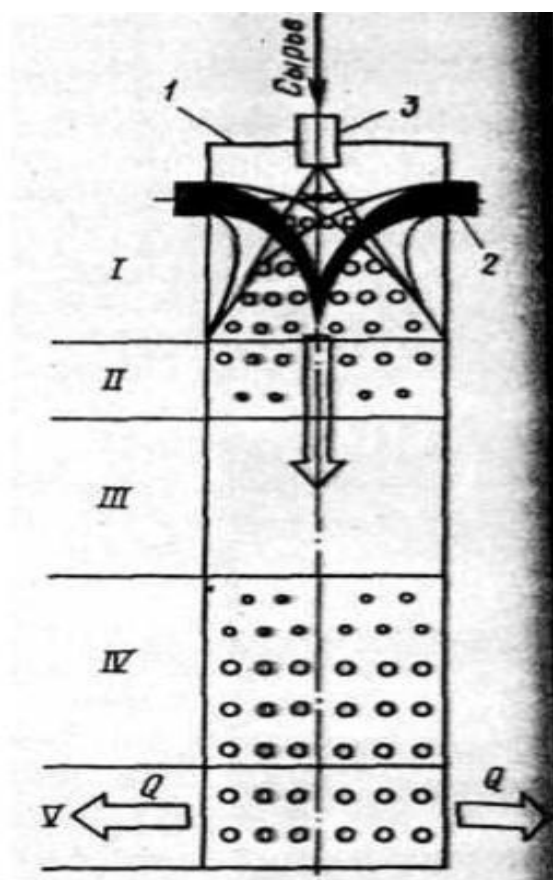


Figure 3. Scheme of plasma processing of dispersed solutions

Thermolysis of aqueous solutions of salts or suspensions is a flexible and universal method for obtaining nanosized powders of simple and complex metal oxides. Its main advantages are a large number of channels for influencing the physicochemical properties of the product, the possibility of synthesizing complex oxide compounds, as well as the high chemical activity of the resulting target products. Nitrates, sulfates, acetates, carbonates, and hydroxides of metals are used as initial compounds for the preparation of aqueous solutions and suspensions. Complex oxides are obtained

from aqueous solutions and suspensions that include compounds of several metals (CSNCT, 2001, p. 24).

However, plasma treatment of only mixed aqueous solutions and suspensions requires significant energy consumption (up to 4 MW h/t) and does not allow obtaining oxide compositions of the required stoichiometric composition in one stage without additional hydrogen reduction. Direct plasma-chemical synthesis in an air-plasma flow of complex oxide compositions (including oxides of fissile materials and a matrix of refractory metal oxides with a high thermal conductivity coefficient and low resonance absorption of neutrons) from dispersed combustible aqueous-organic nitrate solutions containing an organic component (alcohols, ketones, etc.) and having a net calorific value of at least 8.4 MJ/kg is proposed (SPR, 2001, p. 43).

Conclusion

Plasma processing of such solutions leads to a significant reduction in specific energy consumption for their processing (up to 0.1 MW h/t), will significantly increase the productivity of plasma installations, and will also provide conditions in the plasma installation reactor for direct synthesis in air plasma of nanosized complex oxide compositions with a homogeneous phase distribution, high thermal conductivity, and the required stoichiometric composition without additional hydrogen reduction.

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Received: 11.05.2024

Accepted: 18.07.2024