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PLASMA-CHEMICAL PROCESS OF OBTAINING NANOSILICON FOR LITHIUM-ION BATTERIES

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ABSTRACT

The process of complete plasma evaporation of the initial solid material for the synthesis of Si nanoparticles as applied to lithium-ion batteries and energy storage devices was studied in this work. The use of numerical modeling methods made it possible to determine the flow parameters of a two-phase high-temperature flow — temperature fields, velocities and concentrations. To study the processes of evaporation and subsequent synthesis of nanopowders, a plasma reactor with an electric arc plasmatron with a linear circuit and using an argon-hydrogen mixture as a plasma-forming gas was developed. The influence of the external magnetic field on control of the plasma jet parameters was studied in a series of experiments using an electric arc plasmatron in plasma laboratory installations of 30 and 150 kW power. The influence of the magnetic field on the configuration, geometric dimensions and structure of the initial section of the plasma jet was determined. The initial dispersed material — silicon powder was fed to the section of the plasmatron nozzle in a radial pattern. Experimental confirmation of the phenomenon of elongation of the high-temperature initial section of the plasma jet in an axial magnetic field was obtained. It was experimentally established that the creation of a peripheral gas curtain significantly improves the characteristics of heat and mass transfer in the reactor. The influence of two-phase flow, heat exchange and mass flow of nanoparticles, including on the surface of a plasma reactor with a limited jet flow, in the processes of obtaining silicon nanopowders was studied. The obtained regularities can be used to develop and put into operation a pilot plant for high-performance production of nanosilicon powders.

KEYWORDS: plasma-chemical synthesis; arc reactor; plasma jet; nanosilicon; lithium-ion battery; numerical modeling

INTRODUCTION

Rapid growth of sales of electrocars outlines the problem of increase of batteries manufacture. The most powerful source of autonomous electric supply is the batteries based on lithium-ion technologies. Theoretical boundaries of a battery efficiency are always limited by key components, i.e. anode, cathode, electrolyte and separator. Modern anode-electrodes of lithium-ion batteries (LIB) based on graphite materials have capacity around 372 mA·h/g. Theoretically replacement of the standard carbon anodes by silicon-based materials increases anode capacity almost by order to 3579 mA·h/g [1]. It is expected that in the nearest future the silicon anodes will lead to the most significant lithium-ion breakthrough, since graphite is a weak link in the battery, which takes more space than any other component. Appearance of silicon (Si) anodes of ultra high capacity, which can completely replace graphite, increases energy density of ion-lithium elements and can dramatically reduce cost of lithium-ion batteries, in particular in energy sector [2]. It is proved on practice that application of silicon instead of graphite as a negative electrode in the lithium batteries allows rising battery capacity as minimum in three times. The battery of the same size and weight could be able to provide several times more capacity or vice versa at the same capacity reduction of the battery size ensure several times. The main disadvantage of silicon is its swelling in saturation with lithium at recharging with concurrent increase of mechanical stresses in the volume of electrode layer, which promote loss of electric contact of active material with current supply and increase of corrosion. This results in low stability of cycling of electrodes. In order to increase the specific indices of power and capacity of LIB the main efforts of the researchers are directed on development of silicon nanomaterials [3]. In general silicon is one of the most perspective elements for the next generation of electrode materials in lithium-ion batteries due to its high theoretical specific power.

Up to the moment scientific community dramatically succeeded in development of silicon-containing anodes, which can provide significant improvement of energy density. The crucial moment is regulation of the volumetric expansion of silicon, i.e. necessity in nanoparticles application.

The most promising proposals in literature are related with application of nanostructuring in connection with the structures which can adjust to the change of volume during lithiumization such as yolk-shell or porous structures [4]. The final electrode materi-

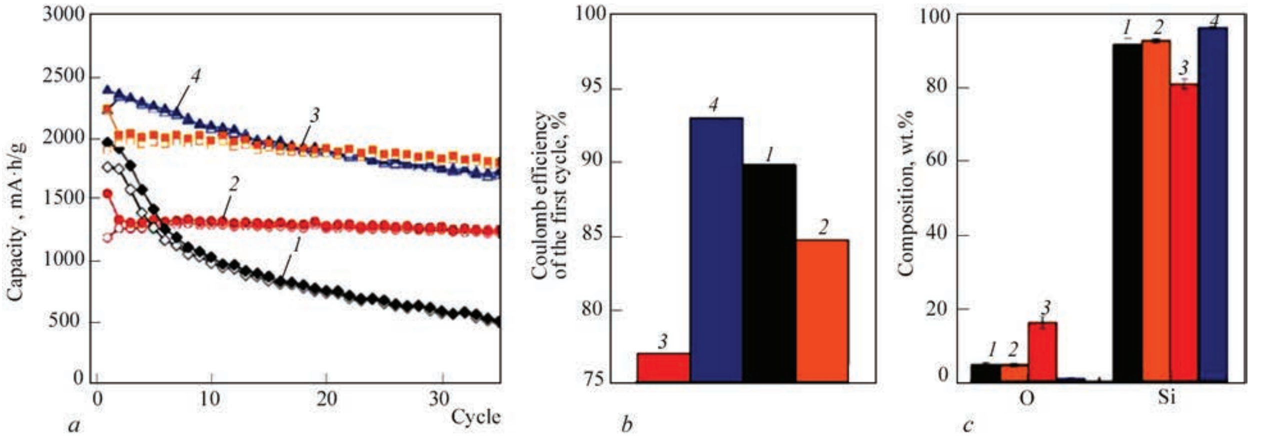

Figure 1. Characteristics of anode material for different commercially available companies (Tekna (*1*), Nano Amor (*2*), Sigma (*3*), GNM (*4*)) manufacturers of silicon nanoparticles [5]: *a* — comparison of stability during cycling between commercially available silicon nanoparticles; *b* — Coulomb efficiency of the first cycle for four considered powders; *c* — element analysis of tested commercially available powders

al consists of agglomerated silicon nanoparticles of 5 nm size, encapsulated inside hollow carbon structures of microsize. At that high specific capacity of the electrode is provided, namely 1570 mA·h/g, with preservation of 65 % capacity after 250 cycles of deep discharge.

The work [1] states that even at solution of the problem of optimum nanomaterials for LIB their commercialization is still not satisfactory because of two reasons. The first one is a complex and expensive methods of manufacture of nanomaterials, in particular of complex morphology. The second one is non-commercial standards, which are used for testing of novel nanostructures. Any proposed solutions finally have to stand commercialization test. For this it is necessary to take into account the issue of scaling at early stage of technology development. Cost and quality of silicon powders are the main problems, which require attention at further investigations. At that it is proposed to make more efforts in development of a system of nanoparticles manufacture, which can provide a set distribution by size as well as compatibility with processing scales, i.e. tones per year. Figure 1 shows characteristics of anode material for different currently commercially available companies–manufacturers of silicon nanoparticles.

For indices on Figure 1, *a* testing was carried out in the semi-centers with different output powders under similar conditions.

Nanopowders of elements and their inorganic compounds can be synthesized by different methods in gas-phase, liquid-phase and solid-phase processes that include physical and chemical deposition from gas phase (so called aerosol methods), solution deposition, mechanical chipping and others. Production of nanoparticles in thermal plasma of electric discharges (arc, high-frequency (HF) and ultra high frequency) is one of the leading directions of researches and developments on creation of basics of the new plasma technologies. Plasma-jet processes [6] are acceptable in all aspects for large-scale production of nanosilicon powders of different shape.

The silicon nanoparticles with carbon coating are considered as a perspective anode material for lithium-ion batteries of the next generation (Figure 2, *a*, *b*) [3]. At the same time development of economic and eco-friendly method of their high-efficient synthesis is still complicated that prevents practical realizing. Such investigations are important for deep understanding of the processes of plasma synthesis (Figure 2, *c*) and development of batteries with perfect characteristics [7].

It is believed that the technology of gas-phase plasma-chemical synthesis is of great potential for production of silicon nanoparticles. In comparison with other methods of synthesis the gas-phase plasma-chemical process has unique advantages. This is a one-stage production with possibility of high efficiency of synthesis with application of output material in any desirable shape (solid, liquid or gas phase). Besides, application of the plasma parameters provides regulation of material modification, product morphology and surface chemistry. Thus, these methods, when developed, can be considered a step ahead for promotion of large-scale production.

The problem of LIB development is determined in general [2]. As for technological peculiarities of plasma the work [8] provides a 3D modeling using a calculation method depending on time of plasma jet. It is shown that superposition of a uniform magnetic field due to Lorentz force and Joule heating promotes laminarization of flow, extension of plasma jet and temperature profile becomes more filled. This results in more effective heating of powder

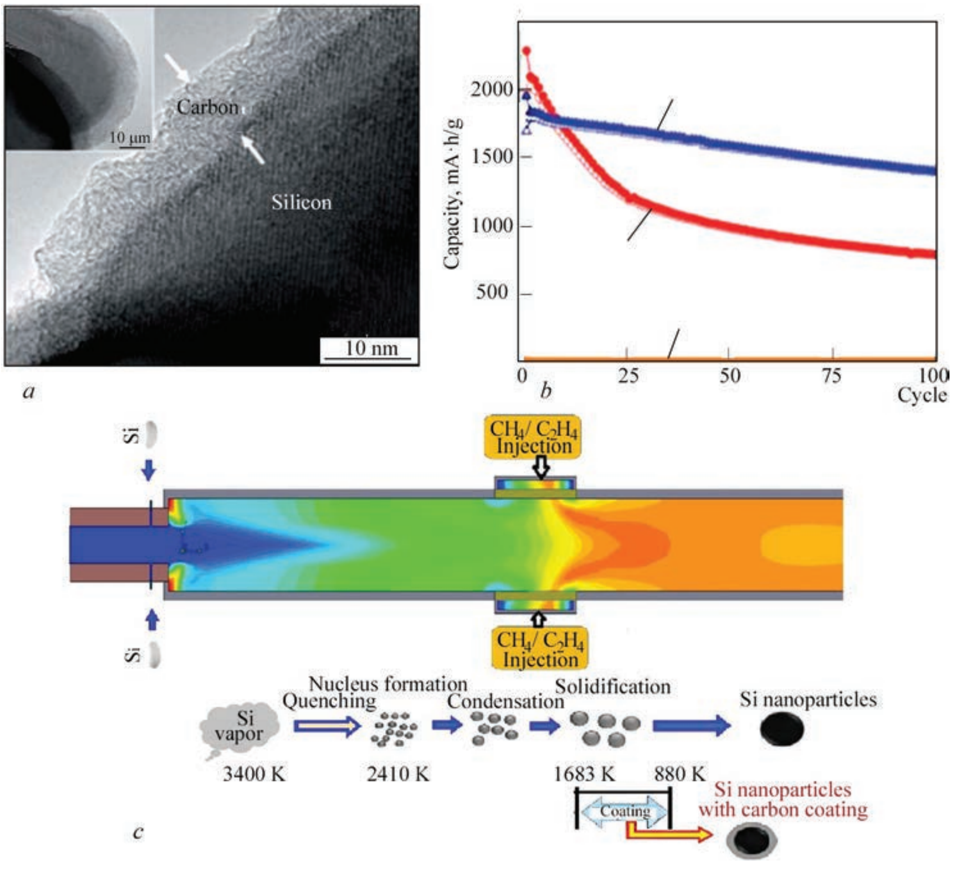

Figure 2. Properties and synthesis of nanosilicon particles with carbon coating produced in thermal plasma per one pass: *a* — TEM image of silicon nanoparticle in carbon coating; *b* — comparison of cyclic loss of LIB capacity with different silicon nanoparticles on anode (*1* — silicon nanoparticles; *2* — silicon nanoparticles with amorphous carbon; *3* — silicon nanoparticles with graphitized coating); *c* — process of obtaining of plasma synthesis of nanoparticles in carbon coating

particles and suppression of turbulent diffusion of silicon vapors and nanoparticles by vortexes that in turn affects their shape. In scope of the first task it is necessary to perform experimental check of efficiency of theoretically described phenomenon with application of hydrogen plasmatron of 30 [9] and 150 kW [10] power.

It is necessary to consider that without special measures deposition of the nanoparticles on a wall in a reactor will take place in the plasma process of production of nanopowders at gas-dispersed flow passing. Generation of a layer of sintered material results in overlaying of reactor section and complete violation of technological mode of the process [11].

The aim of the research is derived from the fact that there are problems to be solved on a way of creation of a large-scale production of nanosilicon for lithium-ion batteries.

AIM AND TASKS OF THE RESEARCH

The aim of this research lies in overcoming the issue of high productive manufacture of cheap silicon nanostructures for LIB including process scales. The next tasks are to be fulfilled in order to reach the set aim, namely increase the efficiency of evaporation/dissociation of a precursor material of silicon nanopowders in the area of high-temperature initial section of a plasma jet; provide the conditions for continuous removal of synthesized nanoparticles from a reactor working zone.

RESEARCH METHODS

In order to realize the tasks set there was developed a plasma reactor using electric arc plasmatron of linear scheme with application of argon-hydrogen mixture as a plasma-forming gas.

Using a system of electromagnet control the possibility is provided for change of magnetic induction by set law. The researches on effect of the magnetic field on the processes of formation and evaporation of a gas-powder flow in a plasma jet were carried out by means of determination of configuration, geometry sizes and structure of the initial jet section. The dispersed material is the silicon powder being fed to plasmatron nozzle section on a radial scheme.

A series of experiments using electric arc plasmatron on laboratory plasma units of 30 and 150 kW power (Figure 3) was carried out for analysis of the effect of external magnetic field on regulation of plasma jet parameters.

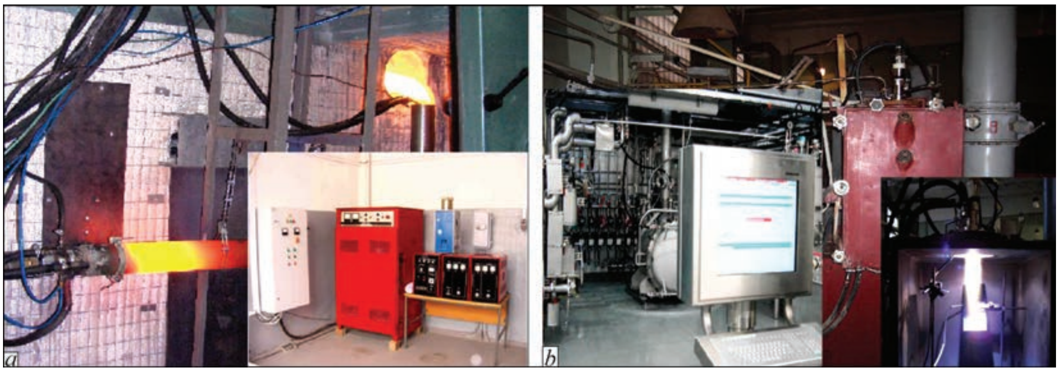

Figure 3. General view of the laboratory plasma units for nanosilicon synthesis of power, kW: *a* — 30; *b* — 150

Numerical modeling was used as a tool in designing the process and reactor for the nanopowders synthesis. It provided the information on temperature fields, velocities and concentrations.

RESEARCH RESULTS AND THEIR DISCUSSION

A series of the experiments using the electric arc linear scheme plasmatron [9] was carried out for detection of the effect of external magnetic field on regulation of the plasma jet parameters. The plasmatron is oriented on application of argon-hydrogen mixture as a plasma-forming gas. The electromagnet is fixed in relation to the plasmatron nozzle system in such a way that a part of arc column, its area with connection spot to the electrode, initial area of plasma-forming jet and nozzle part of arc channel were located in a zone of effect of magnetic field.

A value of magnetic induction is determined by the current value in a coil and can be changed by set law using the electromagnet regulation system. The result of directed orientation of the column part and arc end area is rebuilding of the temperature profile and velocities of plasma jet, which is formed in the nozzle part of arc channel. It is well represented on Figure 4, where appearance of the plasma jet with and without magnetic field at the same operation mode is shown.

Regulation of the parameters of plasma jet is particularly relevant on the stage of evaporation/dissociation of the precursor (in form of powder or gas) in the process of nanoparticles synthesis.

It is known that in general case the channels of transfer of gas and solid phases in two-phase flows of such type do not match. This results in coming of part of the material being processed in the area of relatively low temperatures and velocities of working medium. Instead the consequence of difference in the conditions of heating and acceleration of the particles is a decrease of a coefficient of raw material use. Correction of mutual position of phases of the two-phase flow will allow to some extent improving this index of the process efficiency. The investigations of effect of the magnetic field on the process of formation and evaporation of gas-powder flow in the plasma jet was carried out by means of determination of configuration, geometry and structure of the initial jet section. Dispersed material (silicon powder) was fed on the nozzle end section of plasmatron by radial scheme.

Electric power of the plasmatron is 30 and useful one is 22 kW. Consumption of the components of plasma-forming gas: $G_{Ar} = 3.3$; $G_{H_2} = 0.7$ m^3/h. Based on obtained estimations the specific efficiency of evaporation of 5–20 μm size particles of silicon powder in argon-hydrogen electric-arc plasma jet makes up to

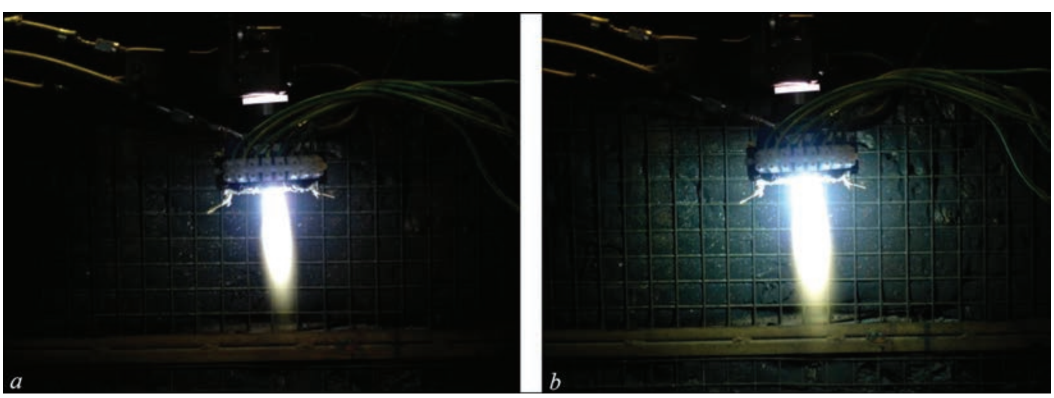

Figure 4. Appearance of plasma jet*: *a* — without external magnetic field; *b* — with external magnetic field

*Photo is provided by Dr. of Tech. Sci. Pashchenko V.M.

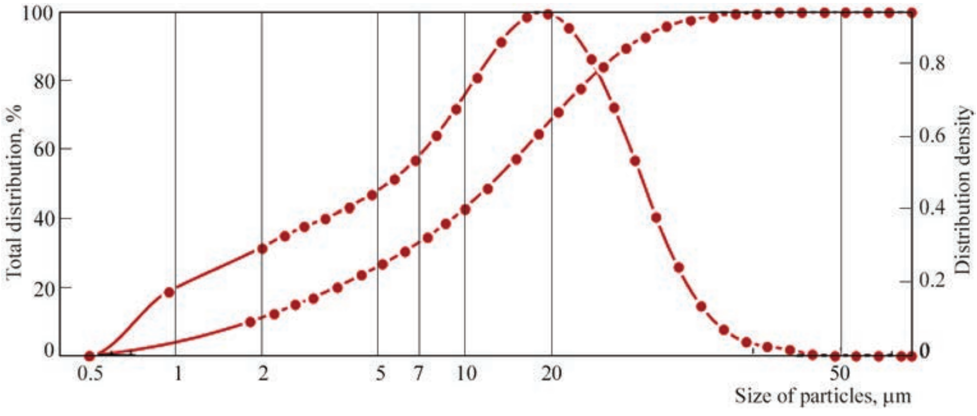

Figure 5. Size of the particles of raw silicon powder

10 kW per 1 kg. Silicon powder with consumption of G_{Si} = 2.0 kg/h (Figure 5) was used in the experiments. Consumption of a transporting gas argon was optimized for 2 kg/h efficiency for the purpose of blowing of the powder on the axis of plasma jet and it was kept constant. The final aim of the experiments was to provide stable evaporation of all powder without presence of tracks of melted particles shining at the output of initial section of the plasma jet (Figure 6).

Also the work was directed on the examination of two-phase flow, heat exchange and mass flow of nanoparticles, including on a wall of plasma reactor with limited jet flow in the processes of silicon nanopowders production. These dependencies are important for optimizing the technological parameters and design of the processes of plasma synthesis of nanopowders.

The reactor is a stationary working flow-through device at atmospheric pressure (Figure 7). It includes a zone of discharge of plasmatron of indirect action, assembly for raw material supply into a high-temperature flow, reactive volume, quenching device, imbedded heat-exchanger and filter for release of condensed products from gas-dispersed flow.

Current production methods of nanopowders of metallic silicon are expensive and make 30 000 US dollars per 1 kg. The most powerful unit (to 200 kW) Teknano-200 Plasma Nanopowder Synthesis of Canadian Company Tekna based on induction plasma technology with working gases (Ar, O_2, N_2, H_2, He and etc.) provides efficiency up to several kg/h of nanomaterials depending on their properties [12].

Following from this, the present work is an attempt to demonstrate the possibility of high-productive manufacture of nanosilicon for LIB from cheap and available raw material, namely silicon powder of metallurgical property with grain-size composition (Figure 5). The electric-arc reactor is made on traditional scheme (Figure 7). Issues of movement of dispersed phase, its heating, melting, evaporation and further condensing into nanoparticles in a plasma jet are sufficiently studied and have many years of history [6, 9, 11]. On practice realization of this condition lies in providing the sufficient time for staying of polydispersed powder flow in the high-temperature jet till complete evaporation. The reactor realizes the limited jet flow with abrupt channel expansion. A channel zone, located behind abrupt expansion (section of flow detachment and to section of flow attachment), is a zone of flow recirculation formed by vortexes. The vortexes are also formed in the plasmatron channel below the flow from powder input, which is transported by gas, into the plasma jet. These vortexes and the next turbulent dissipation down the flow are the most critical source of heat- and mass exchange of dusty plasma with the reactor walls. This as a result leads to deposition of the particles on the reactor walls and forced stop of the process. Removal of these phenomena will provide stable continuous work of the reactor.

Numerical modeling was used as a tool in design of the process and reactor for nanopowders synthesis. It provided the information on temperature fields, velocities and concentrations. Figure 8 shows typi-

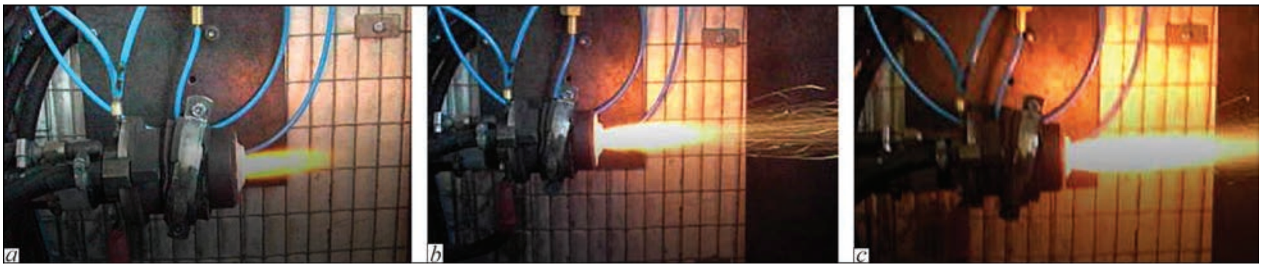

Figure 6. Appearance of plasma jet: *a* — at absence of powder feed without magnetic field; *b* — powder feed without magnetic field; *c* — powder feed with magnetic field

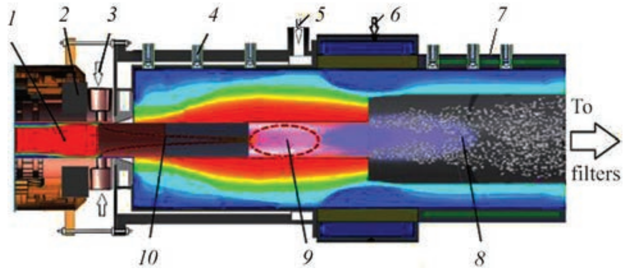

Figure 7. Electric arc reactor for plasma chemical synthesis of nanopowders: *1* — zone of attachment of arc anode spot; *2* — magnet; *3* — powder + transporting gas; *4* — heat flow and dust deposition probe; *5* — concurrent blow in, gas shield; *6* — granulating gas; *7* — water cooling; *8* — zone of nanoparticles growth; *9* — zone of powder particles evaporation; *10* — zone of heating and melting of powder particles

cal results of modeling of the plasma reactor in zone of powder injection into a flame jet and behind the abrupt channel expansion of outlet of plasmatron anode nozzle and reactor inlet.

Based on modeling results the protection was put for the wall in a zone of recirculation flow behind the abrupt channel expansion of reactor and behind quenching device from the effect of high-enthalpy two-phase flow using wall mounted gas shields. There is a twist of a peripheral flow in the reactor (Figure 7) or blowing in through porous wall. Down the flow efficiency of the shield is reduced, however, it should protect the surface of the reactor wall until the quenching device.

Based on preliminary estimations the specific efficiency for target product makes around 10 kW·h/kg, at which complete evaporation of the silicon powder in argon-hydrogen plasma jet is achieved. Using the plasmatron with electric power 150 kW (Figure 3, *b*)

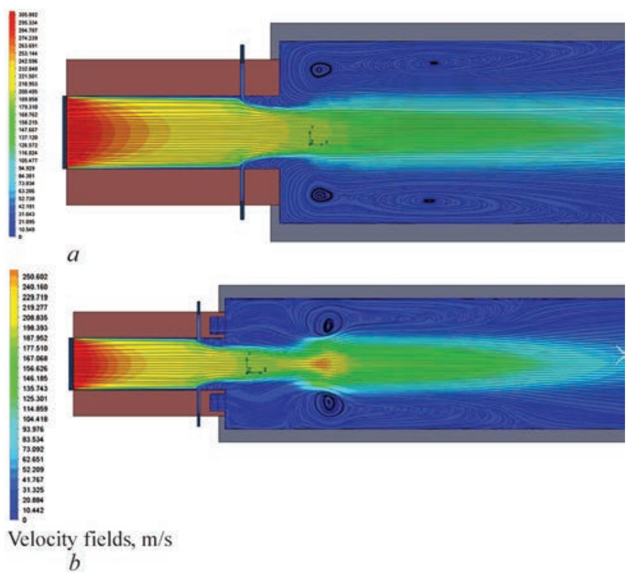

Figure 8. Vortex flows in nozzle channel of plasmatron anode behind the place of powder injection and abrupt expansion at reactor inlet: *a* — without shield; *b* — with gas shield through porous blow in

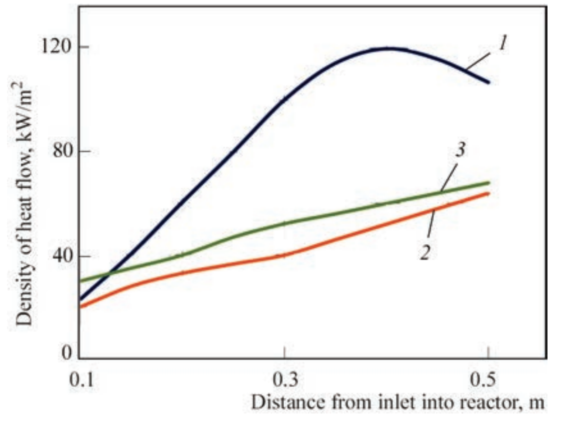

Figure 9. Distribution of density of heat flows on reactor wall under different working conditions: *1* — without gas shield; *2* — with gas shield; *3* — with gas shield and powder feed

and useful 100 kW the silicon powder was fed into the reactor through two injectors with 5 kg/h consumption (Figure 7). Diameter of the outlet nozzle of plasmatron anode made 20 mm. Plasma-forming gas is argon (75 %) + hydrogen (25 %) with 25 m^3/h consumption. Temperature of jet axis is 15000 K. Shielding gas is argon (75 %) + hydrogen (25 %) with 10 m^3/h consumption. Quenching gas is argon (75 %) + hydrogen (25 %) with 100 m^3/h consumption.

Inner reactor diameter is 200 mm, length of quenching device is 500 mm. Wall of the reactor is water-cooled with partial recuperative heat removal in shielding gas.

Formation of nanoparticles in the plasma reactors with limited jet flow takes place as a result of condensation from gas phase and usually accompanied by deposition of obtained nanoparticles on the reactor surfaces that limit high-temperature gas-dispersed flow. The issues of local heat and mass transfer in the plasma reactor are of high importance for performance of directed plasma synthesis of nanopowders with set properties. Due to this there was carried out

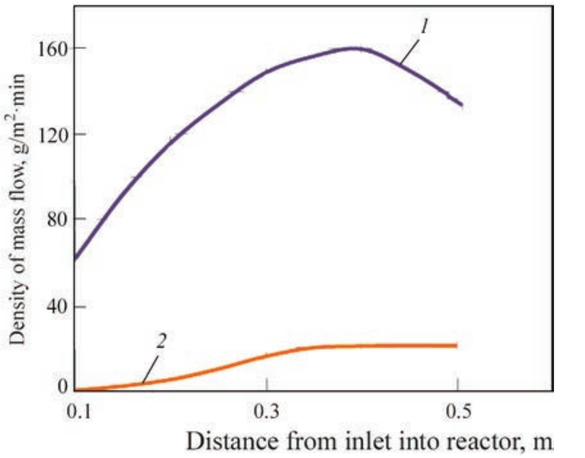

Figure 10. Distribution of density of mass flows on reactor wall in synthesis of silicon nanopowders: *1* — without gas shield; *2* — with gas shield

an experimental investigation of distribution of density of heat (Figure 9) and mass (Figure 10) flows of nanoparticles on the plasma reactor surface. Measurement of a value of heat flow on the reactor wall was carried out by means of registration of the values of loss of cooling water and variation of its temperature in the probes of heat flow installed on the reactor wall. The powder was collected separately from each probe and weighed after experiment for evaluation of distribution of density of mass flows on the reactor wall.

The first stage of the process is evaporation of the raw material at high temperature of high-enthalpy thermal arc plasma. Output particles of silicon move along the flow up to a tail of plasma jet, heat and evaporate. Temperature of plasma flow is quickly reduced transferring energy to the raw material. On the second stage a saturated vapor in the process of quenching undergoes homogeneous nucleation and heterogeneous condensation as a series of processes of nanoparticles production. Thermal arc plasma is a corresponding tool for silicon processing with its unique properties such as high conductivity in comparison with metallic materials, high latent heat of evaporation and high temperature of vapor formation. Besides, synthesis of silicon nanomaterials is affected by unique characteristics of electric arc plasma jet, namely sufficient heat emission from thermal plasma to silicon in the initial section and fast temperature drop beyond it that is favorable for the second stage of the process.

Phenomenon of extension of a high-temperature initial area of the plasma jet in longitudinal magnetic field was experimentally proved (see Figure 4). At that there is an improvement of heating of polydisperse phase with concurrent increase of efficiency and possibility of application of larger powder particles. Complete evaporation of such silicon raw material requires sufficiently long time of staying in a high-temperature zone. The external magnetic field suppresses turbulent vortexes and formation of coarser nanopowders is expected due to suppression of turbulent diffusion of silicon vapors and nanoparticles by the vortexes on the plasma periphery.

Distribution of density of heat flow along the reactor length is nonuniform. Without gas shield it has a maximum in the area of attachment of near-boundary layer of the jet to reactor wall. The value of heat flow density is determined by radiant and convective heat exchange and depends on plasma power. Distribution of heat flow density with gas shield and dusty flow is greatly varied.

Distribution density of mass flows on the reactor wall at silicon nanopowders synthesis without gas shield also has extreme nature with a maximum in the zone of attachment of near-boundary layer to the wall. Relative parts of mass flows on the reactor wall remained unchanged even at increase of duration of synthesis in the scope of up to 60 min under given consumption of the output raw material. Increase of thickness of deposited nanoparticles layer reduces heat flow on the reactor wall due to increase of layer thermal resistance. Moreover, increase of experiment duration to 60 min provides rise of the average size of nanoparticles, in particular in a zone of maximum heat flow.

A gas shield in a form of peripheral vortex flow was used for stabilizing the high-temperature zone of flow in the reactor (see Figure 7) and decrease of intensity of dissipation due to reduction of the turbulent pulsations of velocity (laminarization of flow) and, respectively, increase of time of reagents staying in this zone. Presence of the vortex flow created by swirler results in significant change of distribution of heat and mass flows to the reactor wall (Figures 9, 10) and decrease of their value. The results of the experiments show that creation of the peripheral vortex flow significantly change the characteristics of heat- and mass exchange in the reactor. It should be expected that optimizing can result in elimination of deposition of powder nanoparticles on the reactor wall and provide the conditions of continuous work.

CONCLUSIONS

1. An important result of the carried investigations is the experimental proof of the possibility of significant increase of efficiency of plasma-chemical reactor using electric-arc plasma. This was reached thanks to two conditions, namely possibility of maximum application of energy of plasma jet due to its laminarization in the magnetic field and multijet introduction of raw material; provision of nonequilibrium process when velocity of evaporation of the particles of source silicon in the plasma jet exceeds vapor diffusion with temperature equalizing. This provides efficiency of the process more than equilibrium.

The second important result of the investigation is positive application of the gas shield for provision of continuous process of synthesis of silicon nanoparticles.

2. The result of performed modeling and experimental check on the plasma units of 30 and 150 kW power showed that application of additional effects on the plasma jet by magnetic field at the outlet of nozzle-anode of the plasmatron and gas shield at the inlet of reactor provide complete evaporation of source silicon powder with specific energy consumption 10kW·h/kg.

3. Carried investigations showed that the vortex flow of gas shield results in significant change of distribution of heat and mass flows to the reactor wall

and 2 and 7 times decrease of their value in this performance, respectively. It is expected that optimizing can result in elimination of deposition of nanosilicon powder on the reactor wall and provide the conditions for continuous work.

Further development of the process based on this investigation should be performed in a direction of synthesis of nanosilicon particles with carbon coating in thermal plasma per one pass.

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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