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CONTROL OF THE SYNTHESIS OF COMPOSITE CORE–SHELL-TYPE PARTICLES IN A CONTINUOUS PLASMACHEMICAL REACTOR

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Characteristics of composite titanium dioxide and silicon dioxide nanoparticles with a structure of the core–shell type, synthesized by the chloride method in a continuous plasmachemical reactor with supply of reagents by different schemes, were investigated. For the composite nanoparticles obtained by these schemes, data on the size of the core of the particles, the thickness of their shell, and the ratio between the number of the particles covered with a shell and the number of the particles with no shell in a unit volume of the reactor have been obtained.

Keywords: *titanium dioxide, silicon dioxide, composite particle, plasmachemical reactor, multicomponent medium, homogeneous and heterogeneous reactions, coagulation.*

Introduction. Nanocomposite powders of oxide ceramics, including those with nanocomposite particles having a structure of the core–shell type, e.g., $TiO_2 + SiO_2$ particles, are used widely in modern technologies [1, 2]. Among the methods of obtaining nano-dimensional particles, the gas-phase method of their synthesis stands out because it possesses a number of advantages over the other analogous methods. In particular, the gas-phase processes of synthesis of nanocomposite powders are more environmentally safe than the liquid-phase ones (as a rule, solvents include traces of minerals), provide a higher rate of obtaining a powder compared to that of the liquid-phase processes (which is of importance for the mass production of powders), and are simple in their control. In [3, 4], the synthesis of composite particles in the flame of a $CH_4 + O_2$ mixture was simulated, and particles with a TiO_2 core of size 40 nm and a SiO_2 shell of thickness 2–4 nm have been obtained. In [5, 6], such particles were synthesized in two stages on the basis of the TiO_2 powder obtained in advance. In [7, 8], the one-stage synthesis of composite $TiO_2 + SiO_2$ nanoparticles by the chloride method, in which titanium tetrachloride and silicon tetrachloride are oxidized separately but at one and the same time, was simulated. The calculated size of these nanoparticles was 40 nm, and the thickness of their shell was not larger than 2 nm. In [9], the synthesis of composite $TiO_2 + SiO_2$ nanoparticles in a plasmachemical reactor with simultaneous supply of reagents to it was simulated, and composite nanoparticles of size 6–7 mm with a shell of thickness not larger than 2 nm have been obtained in the calculations. The indicated works were performed on the basis of the experimental and theoretical investigations of the convection of the titanium tetrachloride and silicon tetrachloride vapors in the process of synthesis of titanium dioxide and silicon dioxide nanoparticles [10–12].

In the present work, some variants of control of the synthesis of composite nanoparticles in a continuous plasmachemical reactor were investigated for the purpose of obtaining nanoparticles with a core and a shell of definite sizes.

Formulation of the Problem. Two variants of control of the sizes of the core and the shell of the composite nanoparticles synthesized in a continuous plasmachemical reactor are possible. In the first variant, the ratio between the flow rates of the reagents supplied to the reactor is varied or the regime parameters of the reactor are changed with no changes in its design, and the composition of the final product is controlled. In the second variant, the regime of flow of reagents in the reactor is controlled with the use of side gas jets. In the present work, the second variant of control of the sizes of nanoparticles synthesized in a continuous plasmachemical reactor is considered, and the control parameters are the positions of the side gas jets in the reactor as well as the flow rates and compositions of the reagents in it because the presence or absence of side gas jets in the flow of reagents correspond to their separate or combined oxidation.

Figure 1 shows three schemes of the working zone of a continuous plasmachemical reactor. In scheme 1 (Fig. 1a), a nitrogen having temperature T_1 inflows with rate Q_1 through the channel, attached to the reactor, to the reactor working

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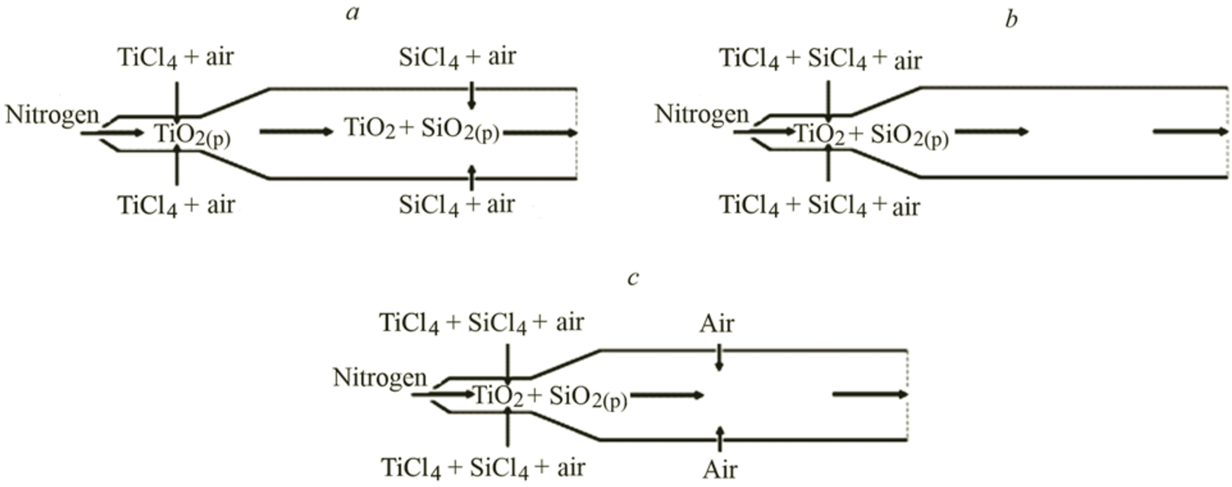

Fig. 1. Schemes of the working zone of a continuous plasmachemical reactor: a) separate supply of reagents; b) combined supply of reagents; c) combined supply of reagents in the presence of a side air jet.

zone. A mixture of titanium tetrachloride and air, having temperature T_2, inflows with rate Q_2 to the reactor through the first side slot. In the zone of mixing of the main gas flow with the side gas jet there takes place a reaction proceeding with the formation, first, of the gas-phase TiO_2 and, then, TiO_2 particles. As these particles move along the reactor, they coagulate and grow due to the reaction proceeding on their surface. A mixture of silicon tetrachloride and air, having temperature T_3, inflows with rate Q_3 to the reactor through the second side slot. In the zone of mixing of the main gas flow with the side gas jet there takes place a homogeneous reaction proceeding with the formation of the gas-phase SiO_2 which condenses on the surface of the TiO_2 particles and the surface of the composite $TiO_2 + SiO_2$ particles formed. In scheme 2 (Fig. 1b), a nitrogen having temperature T_1 inflows with rate Q_1 through the channel to the working zone of the reactor. A mixture of titanium tetrachloride, silicon tetrachloride, and air, having temperature T_2, inflows with rate Q_2 to the channel through the slot in its side surface, and the fraction of silicon tetrachloride is several times smaller than the fraction of titanium tetrachloride in this mixture. In the zone of mixing of the main gas flow with the side gas jet and downstream there take place a homogeneous reaction proceeding with the formation of the gas-phase TiO_2, the nucleation of TiO_2 particles, the formation of their monomers, the coagulation of TiO_2 particles, the growth of them due to the reaction proceeding on their surface, and a homogeneous reaction proceeding with the formation of the gas-phase SiO_2 which condenses on the surface of the TiO_2 particles and the surface of the composite $TiO_2 + SiO_2$ particles formed. Scheme 3 (Fig. 2c) differs from scheme 2 in that an air having a temperature T_3 inflows with rate Q_3 to the reactor through the slot in its side surface.

The calculations were performed using two models of synthesis of composite particles in a continuous plasmachemical reactor. In the first model, the complete coverage of the core of TiO_2 particles with a silicon dioxide layer is provided. In this model, the surface area of the TiO_2 particles covered with a SiO_2 layer of thickness not smaller than the diameter of a SiO_2 monomer is calculated, and the fraction of the TiO_2 particles covered completely with this layer in a unit volume of the reactor is determined [8]. In the second model, the fraction of the TiO_2 particles covered partially with a SiO_2 layer in a unit volume of the reactor is determined [9]. It is assumed that the composite particles covered completely with a SiO_2 layer do not coagulate, and the particles covered partially with a SiO_2 layer can coagulate. The indicated models of synthesis of composite nanoparticles with a TiO_2 core and a SiO_2 shell are based on the fact determined experimentally that the time of agglomeration of TiO_2 nanoparticles is smaller by two orders of magnitude than the time of agglomeration of TiO_2 nanoparticles [13].

A flow of a heat-conducting viscous mixture of the gases O_2, N_2, $TiCl_4$, $SiCl_4$, TiO_2, SiO_2, and Cl_2 in a continuous plasmachemical reactor is considered. In this flow, TiO_2, SiO_2, and Cl_2 appears as a result of the generalized chemical reactions

$$\mathrm{TiCl_4 + O_2 \xrightarrow{k_{tr}} TiO_2 + 2Cl_2}\ , \quad \mathrm{SiCl_4 + O_2 \xrightarrow{k_s} SiO_2 + 2Cl_2}\ .$$

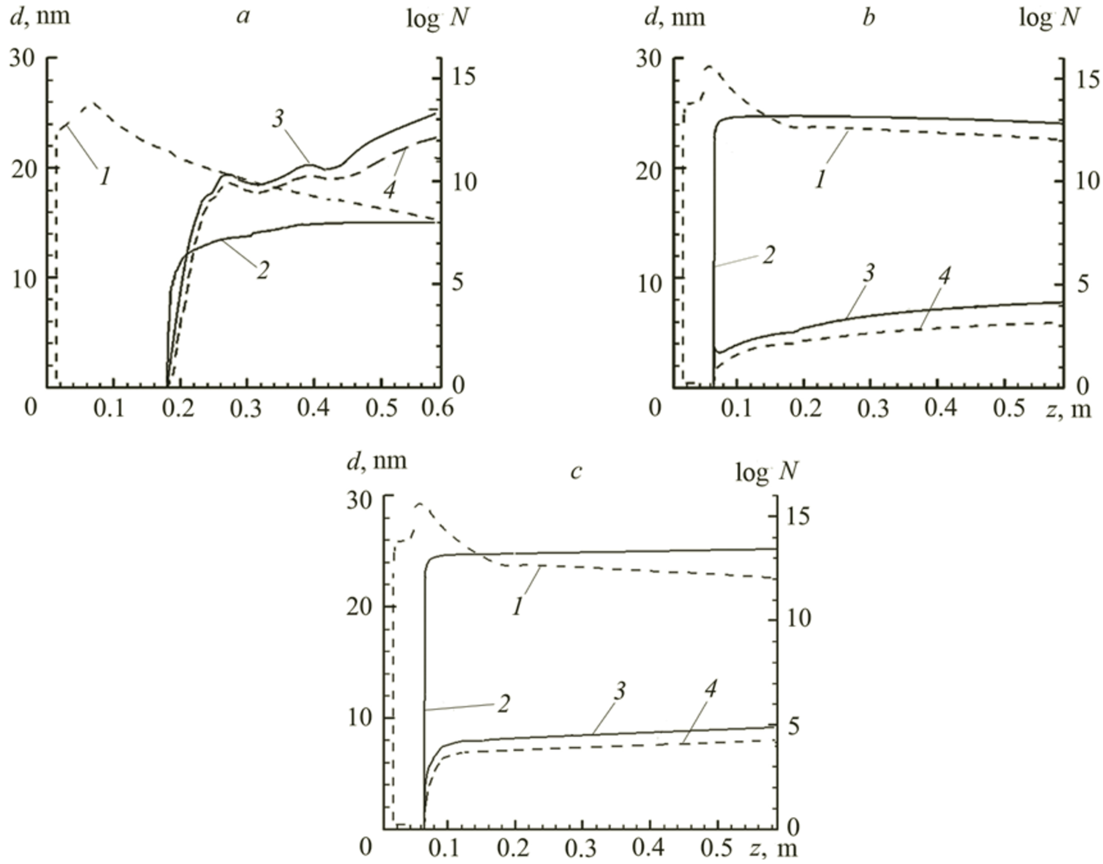

Fig. 2. Logarithm of the density of distribution of the TiO_2 particles (1) and the composite particles (2) with respect to their number and the distributions of the composite particles with respect to their diameter (3) and the diameter of their core (4) along the reactor in the model of synthesis of composite particles covered completely with a shell: a) separate supply of reagents; b) combined supply of reagents; c) combined supply of reagents in the presence of a side jet.

The indicated flow is defined by the system of quasi-gasdynamic equations with account of the external forces acting on the flow and the heat sources in it [14]

$$\frac{\partial \rho}{\partial t} + \operatorname{div} \mathbf{j} = 0 , \quad \frac{\partial (\rho \mathbf{u})}{\partial t} + \operatorname{div} (\mathbf{j} \otimes \mathbf{u}) + \nabla p = \rho \mathbf{F} + \operatorname{div} \Pi ,$$
$$\frac{\partial E}{\partial t} + \operatorname{div} (\mathbf{j} H) + \operatorname{div} \mathbf{q} = \mathbf{j F} + \operatorname{div} (\Pi \mathbf{u}) + Q , \tag{1}$$

where Π is the normalized viscous stress tensor, and E, $\mathbf{F}$, H, and $\mathbf{u}$ are the normalized energy of a unit volume of the mixture, mass force density vector, total specific enthalpy of the mixture, and its velocity vector. The density vector of the mass flow of the mixture is determined by the relation

$$\mathbf{j} = \rho \mathbf{u} - \tau [\operatorname{div} (\rho \mathbf{u} \otimes \mathbf{u}) + \boldsymbol{\nabla} p - \rho \mathbf{F}] , \quad \tau = \frac{M}{\operatorname{Re} \operatorname{Sc}} \frac{T}{p} .$$

To the system of equations presented are added the continuity equation for the mixture components

$$\frac{\partial \rho_i}{\partial t} + \operatorname{div} \mathbf{j}^i = \sum_j J^{(ji)} , \tag{2}$$

and the continuity equation for the volume concentration of the solid phase in the mixture

$$\frac{\partial c_{\mathrm{p}}}{\partial t} + \operatorname{div}\,(c_{\mathrm{p}}\mathbf{u}) = \sum_{j} J^{(j\mathrm{p})} \,, \tag{3}$$

where $J^{(ji)}$ and $J^{(j\mathrm{p})}$ are the normalized rates of transformation of the jth component of the mixture into its ith component and into the solid phase in a unit volume of the mixture, and $\mathbf{j}^{i}$ is the normalized density vector of the mass flow of the ith mixture component. The right sides of Eqs. (2) and (3) involve the kinetic relations defining the changes in the concentrations of titanium tetrachloride and titanium dioxide in the gas and solid phases in the mixture and the changes in the concentrations of silicon tetrachloride and silicon dioxide in the gas and solid phases due to the homogeneous and heterogeneous reactions and the phase transitions in it:

$$\begin{gathered}\frac{dC^{1}}{dt} = -k_{\mathrm{tr}}^{\mathrm{g}}C^{1} = -(k_{\mathrm{tr}}^{\mathrm{h}} + k_{\mathrm{tr}}^{\mathrm{sur}}A)C^{1} \,, \quad \frac{dC^{2}}{dt} = k_{\mathrm{tr}}^{\mathrm{h}}C^{1} - k_{\mathrm{tr}}^{\mathrm{ph}}C^{2} \,, \quad \frac{dC^{3}}{dt} = k_{\mathrm{tr}}^{\mathrm{sur}}C^{1}A + k_{\mathrm{tr}}^{\mathrm{ph}}C^{2} \,, \\ \frac{dC^{4}}{dt} = -k_{\mathrm{s}}^{\mathrm{g}}C^{4} = -(k_{\mathrm{s}}^{\mathrm{h}} + k_{\mathrm{s}}^{\mathrm{sur}}A)\,C^{4} \,, \quad \frac{dC^{5}}{dt} = k_{\mathrm{s}}^{\mathrm{h}}C^{4} - k_{\mathrm{s}}^{\mathrm{ph}}C^{5} \,, \quad \frac{dC^{6}}{dt} = k_{\mathrm{s}}^{\mathrm{sur}}C^{4}A + k_{\mathrm{s}}^{\mathrm{ph}}C^{5} \,.\end{gathered} \tag{4}$$

The system of equations is closed by the additional relations

$$p = \rho R_{\mathrm{m}}T\,\frac{m_{\mathrm{gas}}}{1 - c_{\mathrm{p}}} \,, \quad \alpha_{i} = \rho_{i}/\rho \,, \quad R_{\mathrm{m}} = R_{\mathrm{gas}}\left(\sum_{i} \alpha_{i}m_{i}\right).$$

The addition of the equation for the number of particles in a unit volume of the mixture in the reactor

$$\frac{dN}{dt} = k_{\mathrm{tr}}^{\mathrm{h}}C^{1}N_{\mathrm{av}} - \frac{\beta N^{2}}{2} \,, \tag{5}$$

to relations (1)–(4) makes it possible to calculate the sizes of the particles in each computational cell at each instant of time by their initial diameter d_0, mass, number, and volume concentration. The parameter of coagulation of the particles in the mixture is calculated by the formula [5]

$$\beta = 8\pi d_{\mathrm{B}}d_{\mathrm{p}}\left[\frac{d_{\mathrm{p}}}{d_{\mathrm{p}} + g\sqrt{2}} + \frac{4d_{B}\sqrt{2}}{u_{\mathrm{p}}d_{\mathrm{p}}}\right]^{-1} , \quad g = \left(\frac{1}{3d_{\mathrm{p}}l}\right)[(d_{\mathrm{p}} + l)^{3} - (d_{\mathrm{p}} + l)^{3/2}] - d_{\mathrm{p}} \,,$$

$$l = \frac{8d_{\mathrm{B}}}{\pi u_{\mathrm{p}}} \,, \quad d_{\mathrm{B}} = \frac{3\sqrt{mkT/2\pi}}{2\rho d_{\mathrm{p}}^{2}(1 + \alpha\pi/8)} \,.$$

The conditions of adhesion and absence of heat flows at the walls of the reactor are set. It is assumed that the derivative of the pressure in the reactor with respect to the normal to its wall is equal to zero (the necessity of introduction of this additional condition is explained by the specificity of the quasi-gasdynamic system of equations used). The temperature and flow rate of the gas jets, supplied to the reactor, are predetermined. The pressure, density, and velocity of the gas in the jets at the inlet boundaries are calculated using the Riemann invariants for the Euler equations. Since the problem being considered is axisymmetric, Eqs. (1)–(3) written in cylindrical coordinates are numerically integrated and are brought to the dimensionless form. As the main dimensional parameters of the problem, the radius of the reactor channel, the velocity of sound in the air having a temperature of 300 K, and the air density are used.

The system of equations is solved by the difference scheme explicit with respect to the time. The time derivatives are approximated by the forward differences with the first order of accuracy, and the space derivatives are approximated by the centered differences of the second order of accuracy.

Results of Calculations. The calculations were performed for the regimes of work of a continuous plasmachemical reactor with a separate supply or a combined supply of reagents to it with comparable flow rates. A reactor with the following geometric characteristics was considered: $L_{\mathrm{r}} = 600$ mm, $d_{\mathrm{r}} = 32$ mm, $L_{\mathrm{ch}} = 38$ mm, $d_{\mathrm{ch}} = 7$ mm, $L_{\mathrm{tr}} = 33$ mm, and $\alpha^{\mathrm{o}} = 15^{\mathrm{o}}$. The coordinates of the middle lines of the side slots, through which gases are injected to the reactor, are

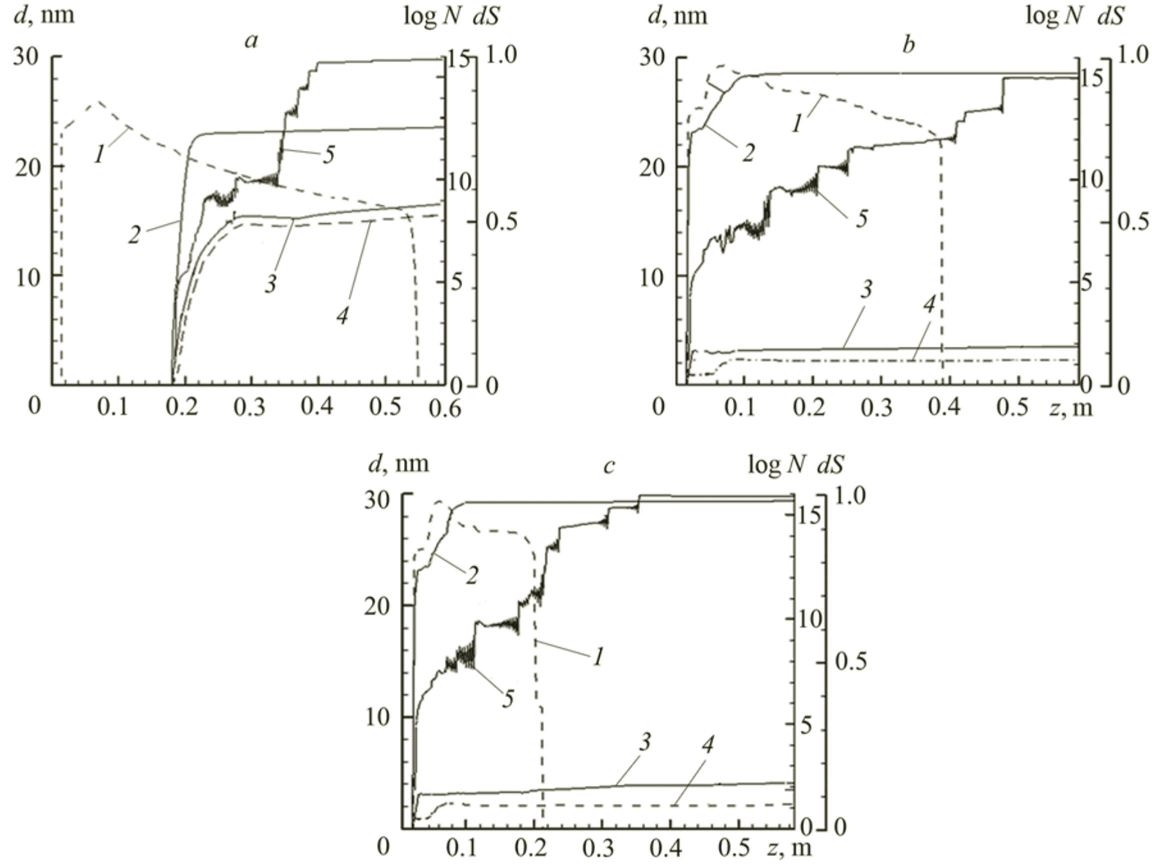

Fig. 3. Logarithm of the density of distribution of the TiO_2 particles (1) and the composite particles (2) with respect to their number and the distributions of the composite particles with respect to their diameter (3), the diameter of their core (4), and the fraction of their surface area covered with SiO_2 (5) along the reactor in the model of synthesis of composite particles covered partially with a shell: a) separate supply of reagents; b) combined supply of reagents; c) combined supply of reagents in the presence of a side jet.

z_1 = 28 mm and z_2 = 192 mm. The regime parameters of the reactor are as follows in the cases of its operation by scheme 1: T_1 = 4500 K, Q_1 = 1 g/s, T_2 = 300 K, Q_2 = 2.5 g/s, T_3 = 300 K, Q_3 = 1 g/s, the flow rate of $TiCl_4$ in the first side jet is 0.0125 g/s, and the flow rate of $SiCl_4$ in the second side jet is 0.0064 g/s, by scheme 2: T_1 = 4500 K, Q_1 = 1 g/s, T_2 = 300 K, Q_2 = 4.0 g/s, T_3 = 300 K, Q_3 = 0 g/s, the flow rate of $TiCl_4$ in the first side jet is 0.016 g/s, and the flow rate of $SiCl_4$ in the second side jet is 0.004 g/s, and by scheme 3: T_1 = 4500 K, Q_1 = 1 g/s, T_2 = 300 K, Q_2 = 4.0 g/s, T_3 = 300 K, Q_3 = 20 g/s, the flow rate of $TiCl_4$ in the first side jet is 0.016 g/s, and the flow rate of $SiCl_4$ in the second side jet is 0.004 g/s.

The values of the constants in Eqs. (4) and (5) were prescribed in accordance with [6, 7]:

$$k_{\rm tr}^{\rm g} = 8.26 \cdot 10^4 \exp\left(\frac{-10{,}681}{T}\right), \quad k_{\rm tr}^{\rm sur} = 4.9 \cdot 10^3 \exp\left(\frac{-8993}{T}\right), \quad k_{\rm tr}^{\rm ph} = 1.2 \cdot 10^{10} \exp\left(\frac{-10{,}681}{T}\right),$$
$$k_{\rm s}^{\rm g} = 8.0 \cdot 10^{14} \exp\left(\frac{-400{,}000}{T}\right), \quad k_{\rm s}^{\rm sur} = 4.0 \cdot 10^{13} \exp\left(\frac{-40{,}828}{T}\right), \quad k_{\rm s}^{\rm ph} = k_{\rm tr}^{\rm ph} . \tag{6}$$

Figure 2 shows the distributions of the particles in the reactor with respect to their weight average diameter $d_i = \frac{1}{N_i} \sum\limits_j d_{ij} N_{ij}$, where $N_i = \sum\limits_j N_{ij}$, and the logarithm of the density of distribution of the particles along the reactor with respect to their number, obtained by the model of synthesis of composite particles covered completely with a shell. It is seen from Fig. 2a that in the reactor there is a region in which, due to the coagulation of TiO_2 particles and the increase in their sizes, composite particles having a large core and a relatively thin shell are formed. In scheme 2, the indicated region is much smaller compared to that in scheme 1, and, particles with a core of small size compared to the thickness of their shell are synthesized (Fig. 2b). In this case, due to the combined supply of the reagents to the reactor, the degree of mixing of the TiO_2 and SiO_2 particles with the gas phase is larger than that in the zone of mixing of the second side jet with the main gas flow in scheme 1. Therefore, the number of composite particles at the outlet of the reactor in scheme 2 is larger than that in scheme 1. Because of the presence of the second side gas jet in scheme 3 (Fig. 2c), the gas flow upstream of the injection zone is decelerated, which increases the time of residence of particles in the indicated region. Consequently, due to the coagulation of TiO_2 particles, the composite particles with a core of increased size are formed. In scheme 3, the thickness of the shell of the composite particles is smaller than that in scheme 2 with no the second side gas jet.

Figure 3 presents results of the analogous calculations performed by the model of synthesis of composite particles covered partially with a shell. It is seen from Figs. 2 and 3 that, in the model in which the core of TiO_2 particles is completely covered with a SiO_2 film, the number of composite particles at the outlet of the reactor is smaller by an order of magnitude compared to that in the model in which TiO_2 particles are partially covered with a SiO_2 film. The results of calculations by these two models differ also qualitatively. The data presented in Fig. 3 show that, at the outlet of the reactor there is no titanium dioxide particles. Because of the coagulation of TiO_2 particles and the formation of a SiO_2 shell on them, the gas flow in the reactor becomes free of pure titanium dioxide particles (curve 1 in Fig. 3). In scheme 3, realized by the model in which TiO_2 particles are covered partially with a SiO_2 film, in the zone of deceleration of the main gas flow upstream of the second side jet, the process of formation of composite $TiO_2 + SiO_2$ particles (even if the fraction of the surface of TiO_2 particles covered with a SiO_2 film is small) prevails over the coagulation of TiO_2 particles. Therefore, the size of the core of the composite particles synthesized by scheme 3 is smaller than that in scheme 2 (Fig. 3b and c), and the thickness of the shell of the composite particles synthesized by scheme 3 is larger than that in scheme 2. This situation is opposite to the situation characteristic of the model in which TiO_2 particles are completely covered with a SiO_2 film (Fig. 2b and c). The nonmonotony of curve 5 in Fig. 3, defining the distribution of the weight-average fraction of the particles covered partially with a shell in a unit volume of the reactor along its length, and the presence of steps in it are explained by the fact that, in the model of synthesis of composite particles covered partially with a shell, the minimum thickness of this shell is limited and is equal to the diameter of a SiO_2 monomer as well as by the possible decrease in the fraction of the particles with a small surface area covered with a shell in the process of change of the new TiO_2 particles to the category of composite particles.

Conclusions. The influence of the regime parameters of a continuous plasmachemical reactor on the total size of the composite particles synthesized in it and the thickness of their shell was investigated. The results of calculations of the parameters of the composite particles synthesized in this reactor by the models and the algorithm presented with respect to the working regimes of the reactor show that these models and the algorithm proposed provide an adequate qualitative definition of the synthesis of composite particles.

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NOTATION

A, relative surface area of particles, cm^2/cm^3; C^1, C^2, and C^3, concentrations of titanium tetrachloride and dioxide in the gas phase and of titanium dioxide in the solid phase, $mole/cm^3$; C^4, C^5, and C^6, concentrations of silicon tetrachloride and dioxide in the gas phase and of silicon dioxide in the solid phase, $mole/cm^3$; c_p, volume concentration of the solid phase; d_B, diffusion coefficient of a Brownian particle, m^2/s; d_{ch} and d_r, diameters of the channel and the reactor, mm; d_p, diameter of a particle, nm; *k*, Boltzmann constant, J/K; k_{tr}^{g}, k_{s}^{g} and k_{tr}^{h}, k_{s}^{h}, rates of the generalized and homogeneous reactions, 1/s; k_{tr}^{sur}, k_{s}^{sur} and k_{tr}^{ph}, k_{s}^{ph}, rates of the surface reactions and the phase transitions, cm^3/s; L_{ch}, L_r, and L_{tr}, lengths of the channel, the reactor, and the transitional region, mm; M, Mach number; *m*, mass of a carrying gas molecule; m_{gas}, mass fraction of a gas; m_i, molecular weight of the *i*th mixture component; m_{ij}, mass of the particles in the *j*th computational cell at the *i*th cross section of the reactor; *N*, number of particles in a unit volume of the reactor; N_A, Avogadro number; N_i, number of particles at the *i*th cross section of the reactor; N_{ij}, number of particles in the *j*th computational cell at the *i*th cross section of the reactor; *p* and *Q*, normalized pressure and heat; Q_1, rate of the nitrogen flow at the outlet of a plasma generator, g/s; Q_2 and Q_3, rates of the first and second side jet flows, g/s; Re, Reynolds number; R_{gas}, specific gas constant, J/(kg·K); R_m, specific gas constant of a mixture, J/(kg·K); Sc, Schmidt number; *t*, time, s; *T*, normalized temperature; T_1, temperature of the nitrogen jet produced by the plasma generator, K; T_2 and T_3, temperatures of the side jets, K; u_p, velocity of movement of particles, m/s; z, axis of the cylindrical coordinate system; z_1 and z_2, coordinates of the middle lines of the slots for injection of side jets, mm; α, accommodation coefficient; α^o, angle of inclination of the transitional region, deg; α_i, mass fraction of the ith mixture component; β, coagulation parameter, cm^3/s; ρ and ρ_i, normalized density of a mixture and of its ith component; τ, normalized relaxation parameter. Subscripts and superscripts: ch, channel; g, generalized; h, homogeneous; m, mixture; p, particle; ph, phase; s, particle surface; sur, surface reaction; r, reactor; tr, transition.

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