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PARTICLE GROWTH IN LASER RADIATION-INDUCED CHEMICAL DEPOSITION OF A SUBSTANCE FROM THE GAS PHASE

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Consideration is given to problems of the kinetics of laser radiation-induced growth of aerosol particles in gas deposition of molecules with allowance for the chemical reaction.

Unique properties of resonance laser radiation that enable us, at the molecular level, to selectively affect the character of the interaction of both molecules in the gas phase and gas molecules with a surface make it possible to control transfer phenomena in different media and to initiate transfer of mass and energy in systems that are in equilibrium before exposure to the radiation [1-6]. In recent years, laser radiation has been successfully used to produce nanosized aerosol particles by its action on a gas mixture in which chemical reactions are realized [7, 8]. Problems of production of such particles are timely for a number of fields of modern technology (manufacture of catalysts with a large interior surface for the pores, production of new materials, etc.). Construction of a sufficiently accurate mathematical model for a real process of laser chemical deposition faces considerable obstacles. As has been noted in [9], at the moment, there is no realistic model that completely describes the very complicated process of laser chemical deposition. This is due to lack of information on the mechanisms of all the processes in the system (in particular, processes associated with chemical transformations). In a number of cases, we do not know the values of the constants that characterize the course of the processes or the exact conditions under which the experiment is carried out (geometry and size of the reaction zone etc.). Furthermore, the statement of the problem itself, in the general case, calls for self-consistent consideration of the action of radiation on a medium whose parameters change as a result of this action (the problem of self-action of radiation [10]). In the case of resonance radiation, where the interaction of molecules with each other and with the surface is dissimilar for molecules excited by the radiation and unexcited molecules, the problem of calculating the kinetics of molecular transitions from one state to another is included here. Therefore, in analyzing laser-deposition processes, attention usually focusses on isolated aspects of the problem [9] (chemistry, mass transfer, issues of the stability of a steady-state regime, coagulation of aerosol particles, etc.). However, to better understand the phenomena in the system, their relationship with each other, and the possibility of controlling the process of laser deposition, we need to have some model (naturally, with allowance for the aforesaid, a most simplified model) that includes the basic features of the process occurring. Therefore in what follows we restrict ourselves to such a model of the process under study that makes it possible, however, to qualitatively assess the role of various phenomena. Joint manifestation of thermal and resonance effects in growth of particles from the gas phase in a resonance-radiation field is discussed.

We consider the following model problem. Let a mixture of a carrier gas with an initial gas (in what follows all the parameters that pertain to this gas will be denoted by the subscript A) enter a quasiclosed volume. A-type molecules, by the action of laser radiation, can be decomposed into a number of components, giving rise, in particular, to molecules (of the B-type) capable of depositing with formation of aerosol particles. Examples of this

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kind of reaction for the case of production of aerosol particles in thermal decomposition of the initial gas are given in [11]:

$$
\begin{aligned}
&\mathrm{Si\,(OC_2H_5)_4} \rightarrow \underline{\mathrm{SiO_2}} + 4\mathrm{C_2H_4} + 2\mathrm{H_2O}\,,\\
&\mathrm{Ti\,(OC_3H_7)_4} \rightarrow \underline{\mathrm{TiO_2}} + 4\mathrm{C_3H_6} + 2\mathrm{H_2O}\,,\\
&2\mathrm{Al\,(OC_4H_9)_3} \rightarrow \underline{\mathrm{Al_2O_3}} + 6\mathrm{C_4H_8} + 3\mathrm{H_2O}\,.
\end{aligned}
\tag{1}
$$

The underlined components are the initial product for production of aerosol particles (in the scheme presented above, these are B-type molecules).

In addition to ordinary physical condensation, molecular deposition on the surface of particles can also be realized through a heterogeneous chemical reaction directly on the particle surface. First, let us consider the first scheme, where radiation initiates a chemical reaction in the gas phase, thus producing the particles for which the process of ordinary physical condensation is realized.

We note that when a gas is exposed to resonance radiation, molecules of the starting material and reaction-product molecules can be in both the ground state and the excited state. Next, allowing for the scheme of possible reactions (1), we assume that molecules capable of subsequent deposition appear as a result of a chemical reaction of the first order. For the model of an ideal-mixing flow reactor, in view of [12], we can write the following system of equations to determine the densities of the initial molecules (n_A) and the reaction-product molecules (n_B) in the reactor:

$$\frac{dn_\mathrm{A}}{dt} = \frac{n_\mathrm{A0} - n_\mathrm{A}}{\tau} - k_\mathrm{r}\,(1 - \gamma_\mathrm{A})\,n_\mathrm{A} - k'_\mathrm{r}\gamma_\mathrm{A}n_\mathrm{A}\,, \tag{2}$$

$$\frac{dn_\mathrm{B}}{dt} = -\frac{n_\mathrm{B}}{\tau} + k_\mathrm{r}\,(1 - \gamma_\mathrm{A})\,n_\mathrm{A} + k'_\mathrm{r}\gamma_\mathrm{A}n_\mathrm{A} - J_\mathrm{B} - J_{\mathrm{B}S}\,. \tag{3}$$

Here n_A0 is the density of the molecules of the starting material outside the reactor; τ is the time of stay of the gas in the reactor ($\tau = L/v_L$, where L is the reactor length; v_L is the linear velocity of flow [12]); k'_r and k_r are, respectively, the reaction-rate constants for A-type molecules excited by the radiation and unexcited molecules (in what follows, parameters of excited molecules will be primed). Usually, $k_\mathrm{r} < k'_\mathrm{r}$, and in the case of reaction initiation by means of resonance effects we can assume that only $k'_\mathrm{r} \neq 0$. The quantities k_r and k'_r have the form

$$k_\mathrm{r} = k_\mathrm{r0}\exp\left\{-E/kT\right\},\quad k'_\mathrm{r} = k_\mathrm{r0}\exp\left\{-(E - \Delta E)/kT\right\},$$

where E is the activation energy for the chemical reaction without the effect of radiation; ΔE is the change in the activation energy for the chemical reaction in the radiation field; k_r0 is the pre-exponential factor, which is assumed to be a constant quantity. The parameter γ_i is the fraction of excited molecules: $\gamma_i = n'_i/(n_i + n'_i)$, where n_i and n'_i are, respectively, the densities of unexcited and excited molecules of component i (consideration is given to a two-level model for molecular transitions from the ground state to the excited state and vice versa). The quantities n_i and n'_i in (2) and (3) are found from the system of kinetic equations for molecular transitions from one state to the other. Assuming the radiation to be acting uniformly throughout the entire volume, to find n_i, n'_i (and, accordingly, γ_i) we have [13]

$$\frac{dn'_i}{dt} = \frac{I\sigma_i}{h\nu}\,(n_i - n'_i) - \frac{n'_i}{\tau_{\nu i}}\,,\quad \frac{dn_i}{dt} = \frac{I\sigma_i}{h\nu}\,(n'_i - n_i) + \frac{n'_i}{\tau_{\nu i}}\,, \tag{4}$$

where I and ν are, respectively, the radiation intensity and frequency; σ_i is the cross section of photon absorption by a molecule of component i; h is the Planck constant; $\tau_{\nu i}$ is the relaxation time for molecules of the i-th component (it is assumed that $\tau_{\nu i} << \tau$).

In the steady state, for γ_B, the expression

$$\gamma_B = \frac{(\sigma_B I/h\nu)\,\tau_{\nu B}}{1 + (2\sigma_B I/h\nu)\,\tau_{\nu B}} \tag{5}$$

follows from (4).

The quantity J_B in (3) describes the flow of deposited molecules (in our case, B-type molecules) into the condensed phase (aerosol particles) in unit volume of the reactor and J_{BS} describes the flow of B-type molecules onto the walls of the reactor, normalized to its volume. We note that both molecules of material B and particles already formed can be deposited on the reactor walls (in the case of "cold walls," where the temperature in the gas phase decreases toward the walls, the thermophoresis efffect can be of substantial importance in particle deposition). Naturally, loss of material B on the reactor walls lowers the yield of produced particles from the reactor. Therefore, in practice, special precautions are taken to decrease the deposition of material B on the walls. The term J_B in (3) can be associated with both homogeneous and heterogeneous condensation. Here, we need, however, to note the following. Since the reactor is usually not insulated hermetically from the atmosphere and there are many condensation centers in it, we can assume that the probability of deposition on them will be much higher than the probability of "pure" homogeneous condensation. For homogeneous condensation, the term J_B is determined by the probability of deposition of B-type molecules on condensation centers and their re-evaporation from the latter.

In the case of the uniform-temperature approximation (where the gas-phase and particle temperatures are assumed to be the same), the temperature of the aerodispersion medium is determined by the balance equation

$$\rho c \frac{dT}{dt} = k_\nu I - \beta\,(T - T_0) + Q'_{e.r}\,k'_r \gamma_A n_A + Q_{e.r}\,k_r\,(1 - \gamma_A)\,n_A + J_Q\,, \tag{6}$$

where ρ and c are, respectively, the averaged values of the mass density and heat capacity of the aerodisperse system, considered as a quasihomogeneous medium; $Q_{e.r}$ is the energy release of the reaction; T_0 is the ambient temperature (if the temperature at the inlet to the reactor is different from the ambient temperature, a new averaged value of T_0 should be introduced, similarly to [12]); the coefficient β describes both heat transfer and removal of heat by the departing flow [12]; k_ν is the effective coefficient of absorption of radiation by the aerodispersion medium; J_Q is the energy flux into the growing aerosol particles, calculated per unit volume of the aerodispersion medium and associated with the energy released in molecular condensation.

In a quasistationary approximation where the last three terms in the right-hand side of (16) are ignored, for the temperature of the system we have $T = T_0 + Ik_\nu/\beta$. It follows from this that as I increases and β decreases, the temperature of the system will increase, and accordingly the rate constant of the reaction that gives rise to the B molecules and the rate of aerosol-particle evaporation will increase.

The effective absorption coefficient k_ν depends in the general case on the densities n_A and n_B and on the density and size distribution of the aerosol particles in the system, which in turn is determined by I and, accordingly, the temperature of the system.

As follows from (2)-(6), the process considered is described by a rather complex system of nonlinear equations that are typical of laser photochemistry [14]. In it, different fluctuating regimes can be realized and the system can be studied for stability similarly to [14]. In what follows, however, the parameters will be considered to be such that the steady-state solution of the considered system of equations is stable (a necessary condition for the stability of such systems is the possibility of removing the heat released in them [12]). As has been noted above, because of the complexity of the problem of laser chemical deposition of a substance, one usually restricts oneself to a certain aspect of the problem for a more detailed investigation. First we consider the effect of the pumping rate on the density of molecules of component B in the system. In a quasistationary approximation, from (2) and (3) we obtain for n_B the expression

$$n_B = \frac{n_{A0}}{1 + \left\{\tau\,[k_r\,(1 - \gamma_A) + k'_r \gamma_A]\right\}^{-1}} - \tau\,(J_B + J_{BS})\,, \tag{7}$$

which, in view of the relation $\tau = L/v_L$, yields that as $v_L \to 0$ the sum $J_B + J_{BS}$ must also tend to zero (lest n_3 become negative). As $v_L \to \infty$, n_B (by which J_B and J_{BS} are determined) also tends to 0. Thus, n_B and, accordingly, the number and size of the aerosol particles formed can be characterized by the maximum as v_L increases. This is confirmed by results of experiments on the action of the radiation from a frequency-tuned CO_2 laser on the mixture of $Si(OC_2H_5)_4$ (tetraethylorthosilicate; in what follows, TEOS) vapor with nitrogen, used as a carrier gas, that were conducted at the Institute of Chemical Processes of the Academy of Sciences of the Czech Republic. When radiation similar in frequency to the TEOS absorption line was introduced into the reactor, aerosol particles appeared in it. To measure the number and the size distribution of the particles formed, use was made of a DMPS (differential mobility particle sizer) aerosol analyzer. In the number of formed particles and their total volume (normalized per cm^3) as functions of the pumping rate, a maximum was observed for certain values of the parameters.

We consider the growth of an aerosol particle in a volume filled with an inert gas. Allowing for the condition of strong dilution of the reactant gas by the inert gas, in calculating the molecular flow to the growing particle, we will restrict ourselves just to a diffusion mechanism of transfer, disregarding the convective component of mass transfer. The solution to the diffusion equation for the density distribution of the molecules of the condensing substance (B) that are deposited on a spherical particle, in a quasistationary approximation, has the form [15]

$$n_B = b - a/r , \tag{8}$$

where r is the coordinate counted off from the center of the particle; a and b are integration constants determined from the boundary conditions.

For $r \to \infty$ we assume $n_B = n_\infty = \text{const}$, from which $b = n_\infty$. In formulating the second boundary condition we note the following. As noted in [15], if the diffusion equation is assumed to hold true right up to the particle surface and the free-molecular and diffusion fluxes are matched on the surface itself, a functional dependence on the Knudsen number ($Kn = \lambda/R$, where λ is the mean free path of the molecules; R is the particle radius) is obtained that yields the limiting transitions for the free-molecular and continuous regimes. However the coefficient of Kn turns out to be inaccurate. In the model of a "boundary sphere," matching is performed at a distance from the sphere surface that is proportional to the mean free path λ. Here a result that is very similar to the accurate result for small Kn numbers, and an incorrect functional dependence for $Kn >> 1$ are obtained [15]. Without dwelling on the numerous theoretical approaches available in the literature by means of which expressions are obtained that describe, to a certain degree of accuracy, evaporation-condensation processes for arbitrary Kn numbers, in what follows we will state a boundary condition directly on the particle surface to obtain rather simple expressions that yield correct limiting transitions for the free-molecular and continuous regimes.

With allowance for the above, for $r = R$, we can write the following condition (we assume that the deposited molecules can, in the general case, be in the excited state):

$$D_B \frac{dn_B}{dr} = [\gamma_B \alpha'_B + (1 - \gamma_B)\alpha_B] \frac{n_\infty v_B}{4} - A \exp\left\{-\frac{Q - \Delta Q}{kT}\right\} . \tag{9}$$

In writing (9), for simplicity we assume that the excited and unexcited molecules are described by one diffusion coefficient D_B. Otherwise, we should consider the excited and unexcited molecules as individual components and, for each, calculate the fluxes of deposited molecules (the influence of a difference in the diffusion coefficients for the excited and unexcited molecules was discussed in [16]). It is also assumed that the molecules incident on the particle surface are characterized by the Maxwell velocity distribution function. In (9), Q is the evaporation energy per molecule; ΔQ is a quantity that characterizes the change in the evaporation energy in the resonance-radiation field; v_B is the average velocity of the molecules of component B. The pre-exponential factor A is determined by the employed model for evaporation kinetics [17, 18].

The evaporation energy, in the general case, depends on the particle size. As a first approximation this dependence is expressed in the form (for simplicity, in which follows we assume $\Delta Q = 0$)

$$Q = Q_0 - \frac{2\sigma V_m}{R}, \tag{10}$$

where Q_0 is the energy of evaporation from a flat surface; V_m is the volume of a molecule; σ is the surface tension, which, in principle, for rather small particles, depends on the particle radius, just like the sticking coefficient α [18].

With allowance for (7)-(10) and the specified assumptions, for the density of the resultant flux of molecules deposited on an aerosol particle we obtain the expression

$$j_B = \frac{[\alpha_B - \gamma_B(\alpha_B - \alpha'_B)]\frac{n_\infty v_B}{4} - A\exp\left\{-\frac{Q_0}{kT}\left(1 - \frac{2\sigma V_m}{RQ_0}\right)\right\}}{1 + \left[\alpha_B - \gamma_B(\alpha_B - \alpha'_B)\frac{v_B R}{4D_B}\right]}. \tag{11}$$

For $\alpha_B = \alpha'_B = 1$, by replacing the kinetic expression for evaporating molecules by $n^e_B v_B/4$, where n^e_B is the density of molecules of saturated vapor of component B, and expressing D_B in terms of the velocity v_B and the mean free path λ_B (which yields $v_B R/4D_B = 3/4\mathrm{Kn}_B$) we obtain an expression that is given in [15] and coinsides with Sherman's interpolation formula.

The rate of particle growth is determined as $dR/dt = j_B/n_s$, where n_s is the particle density in the condensed phase.

Let us consider the rate of aerosol-particle growth in the system as a function of its temperature (and, accordingly, the radiation intensity). In the case where the term $\tau(J_B + J_{BS})$ in (7) can be disregarded (i.e., when the deposition of the molecules of material B practically does not affect their density in the reactor), for the rate of particle growth the following expression follows from (7) and (11):

$$\frac{dR}{dt} = \frac{1}{n_s}\left\{\frac{n_{A0}d}{1 + \left\{\tau\,[k_r(1 - \gamma_A) + k'_r\gamma_A]\right\}^{-1}} - g\exp\left(-\frac{Q}{kT}\right)\right\}, \tag{12}$$

where

$$d = [\alpha_B - \gamma_B(\alpha_B - \alpha'_B)]\frac{v_B}{4}\left\{1 + \left[\alpha_B - \gamma_B(\alpha_B - \alpha'_B)\frac{v_B R}{4D_B}\right]\right\}^{-1};$$

$$g = A\left\{1 + \left[\alpha_B - \gamma_B(\alpha_B - \alpha'_B)\frac{v_B R}{4D_B}\right]\right\}^{-1}.$$

From (12) it is evident that the process of deposition (this is expressed in the positive value of the quantity in the braces of (12)) can be initiated both by excitation of A-type and B-type molecules by radiation (in the case where $k'_r > k_r$ and where $\alpha'_B > \alpha_B$) and by heating the system by the incident radiation (an increase in the reaction-rate constant). The importance of the joint manifestation of thermal and resonance processes should be noted. Thus, for example, excitation of the initial molecules by resonance radiation ($\gamma_A \neq 0$) leads to a stronger effect of a temperature increase on the reaction rate (for $k'_r > k_r$). We note that in the above-mentioned experiments on initiating the process of deposition under the action of the radiation of a frequency-tuned CO_2 laser on a mixture of TEOS vapor with nitrogen, formation of particles occurred only near the absorption line of TEOS.

From (12) it is also evident that, for certain values of the parameters, dR/dt can be characterized by its maximum, depending on T (and, accordingly, I). Thus, the radiation that initiates the process of deposition (which is realized through molecular excitation and heating of the system), as the intensity increases further, can begin to play the role of a factor that leads to the opposite effect: an increase in the probability of the transition of molecules to the gaseous state (evaporation of aerosol particles).

Above, consideration has been given to the simple single-step kinetics of gas deposition of a substance on the aerosol-particle surface with normal condensation of B-type molecules, which appeared in the system as a result of chemical decomposition of the initial-gas molecules (A-type molecules) in the gas phase. If there is no reaction in the gas phase and only a heterogeneous chemical reaction of the initial molecules (A) on the particle surface is realized (decomposition according to scheme (1) with deposition of the underlined terms only), to determine n_A, we have the equation

$$\frac{dn_A}{dt} = \frac{n_{A0} - n_A}{\tau} - J_A - J_{AS},$$

where J_A and J_{AS} are analogous to J_B and J_{BS} but for the A-type particles.

In the case where the effect of the flow of molecules into the particles on their density in the reactor can be disregarded (an assumption that is similar to the assumption for B-type molecules made above), in a quasistationary approximation, for the rate of particle growth we obtain expression (12) in which $k_r = k'_r = 0$, the subscript B should be replaced by the subscript A, and α_A and α'_A mean coefficients that characterize the probabilities of the chemical reaction in collision with the wall of unexcited and excited A-type molecules. For $\gamma_A = 0$ and the absence of a flux of molecules that evaporate from the surface of the particle, for its growth rate (allowing for the fact that $n_A v_A / 4 = P_A / (2\pi m_A kT)^{1/2}$, where P_A is the gas pressure of component A), we obtain the expression given in [19]. The statement of [19] that the rate of particle growth can be increased only by increasing the pressure of the reacting gas and cannot increase as a consequence of an increase in the surface temperature remains, however, unclear. It is known, however, that the probability of the first-order reaction is proportional to the rate constant of the heterogeneous reaction [20], which depends exponentially on the temperature; the coefficient of molecular chemisorption on the surface is characterized by the same relationship with the temperature [21]. This enables us to assume that it is precisely an increase in the particle temperature that can serve as a factor that leads to initiation of the growth of particles in a heterogeneous chemical reaction on their surface, similarly to the above-considered case of a chemical reaction in the gas phase that leads to the appearance of condensing molecules. We note that, in heterogeneous chemical deposition, foreign particles (condensation centers) serve as a distinctive kind of "aerodispersed catalyst" similarly to a regular porous catalyst. Here, laser radiation, as in the above-considered case, can "prepare" molecules for deposition, exciting them in the gas phase and heating the system (including the condensation centers). For a more detailed investigation of the process of deposition with heterogeneous chemical transformations on the surface itself, we should allow for the presence of the molecules in the adsorption layer. The probability of the heterogeneous chemical reaction becomes dependent on the composition of the gas mixture, including the buffer component, not involved in the chemical reaction but capable of affecting the component concentration in the adsorption layer. In this case, the conclusion of the rate of particle growth in a free-molecular flow regime being independent of the buffer-gas pressure, which is made in [19] starting from an expression similar to (12), will be contrary to fact. We note that for the normal evaporation-condensation process, adsorption of impurity molecules on the surface of a phase transition can have a substantial effect on its rate. Thus, in [3], some problems of a possible effect of resonance radiation on the "blocking" role of foreign molecules in phase-transition processes were discussed.

Naturally, as has been pointed out above, the schemes considered give only a basic account of the investigated phenomena. The chemical reactions themselves can be more complicated than (1). This, in particular, is demonstrated by the results of the above-mentioned experiments on the action of CO_2-laser radiation on TEOS vapor. From them it follows that in a number of cases addition of oxygen to a mixture of TEOS vapor with nitrogen increases the number of aerosol particles formed. This indicates that the reaction of $Si(OC_2H_5)_4$ decomposition follows a more complicated scheme than (1). In particular, it can be assumed that, aside from SiO_2, Si is also formed, whose reaction with O_2 leads to an increase in the number of condensing SiO_2 molecules. Of interest is the fact that in the action of laser radiation on the above oxygenous mixture aerosol particles are formed at a radiation frequency detuned from the resonant frequency of TEOS, at which no particle formation in a mixture without oxygen was observed. This suggests that when oxygen is added to the starting mixture of TEOS vapor with nitrogen a substance that is resonant to the radiation frequency used forms (we note that oxygen itself is not resonant with respect to the radiation frequency used). Warming up of the system by absorption of radiation occurs that initiates the reaction of TEOS decomposition, which in turn leads to formation of aerosol particles. It should also be noted that in the first of the above-considered schemes, B-type molecules can be deposited on the surface chemically (i.e., the chemical reaction can proceed simultaneously both in the gas phase, producing deposited molecules, and on the surface of aerosol particles). Representing, however, the basic picture of possible mechanisms of the action of laser radiation on an aerodispersed system, we can, in principle, supplement it with newly emerging facts that characterize the process.

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