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MODELING AND IDENTIFICATION OF HEAT- AND MASS-TRANSFER PROCESSES IN INTUMESCENT HEAT-INSULATING MATERIALS

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The development and use of intumescent heat-insulating materials is important for ensuring fire safety and refractoriness of various structures exposed to powerful sources of heat energy [1–5]. Under heat loading such materials can increase in thickness by a factor of greater than several tens [3–5], forming a heat-insulating layer with a low thermal conductivity and thereby protecting the wall of the structure against damage. The number of such materials and the cost of thermal tests for their certification and prediction of heat-insulating properties constantly increase [5]. In this connection, it seems reasonable to use an approach that includes a study of the physical and chemical processes occurring in heated layers of intumescent coatings and their identification and mathematical description. This makes it possible to construct physically plausible mathematical models that can give well-founded predictions in the development of new, more effective coatings.

Attempts to realize this approach have been undertaken by Buckmaster et al. [1–4]. Thus, for example, some heat- and mass-transfer mechanisms in a fire-insulation coating based on chlorosulfonated polyethylene (CSPE) and thermally expansible graphite (TEG) have been studied [3, 4]. Intumescence and thermal destruction of the binder were found to proceed in the same temperature interval. This feature and also the high porosity of the heat-insulating layer significantly complicate identification of the observed physicochemical processes; therefore, it is necessary to split them into simpler processes, study them sequentially, and identify each of them separately.

This experimental procedure is described in detail in [6], where a diagram of preliminary and basic experiments is given. In the first stage, the thermophysical characteristics of intumescent material were determined [8] over a wide temperature range by solution of the inverse problem of heat conduction for each individual stabilization temperature using the method of stabilized states [7]. The macrokinetic constants for the first step of thermal destruction of the polymer binder were found using the results of dynamic experiments [4]. This process is exothermic and can be described by a first-order solid-state reaction with a temperature dependence in the form of Arrhenius' law. The adiabatic regime of the first step of this reaction was found to be similar to the ignition process of condensed substances [9, 10].

In this paper, a most complete model of intumescent material is proposed and the thermal destruction of the polymer binder and intumescence of the filler are identified over the studied temperature range using experimental data on the temperature fields and the dynamics of growth of the heat-insulation layer under heat loading.

1. Experimental Procedure and Physical Concepts of the Processes. In accordance with the procedure of [6], we split spatially the processes in the physicochemical experiment and consider the diagram of the experiment shown in Fig. 1. The studied specimen 1 in the form of a tablet of an intumescent material based on CSPE and TEG with initial thickness $h_i = 2Oz_1 \leqslant 6\cdot 10^{-3}$ m is placed in a reaction unit. The unit is a hollow cylinder 2 and allows one to study the intumescence process only along the longitudinal z coordinate. In this case the condition of symmetry about the Or axis is satisfied. The material (quartz, stainless steel) and dimensions of the cylinder were varied to determine the influence of the walls on the magnitude and velocity

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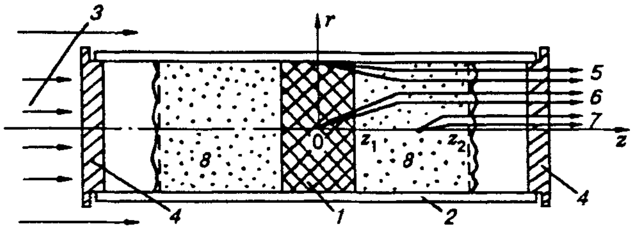

Fig. 1

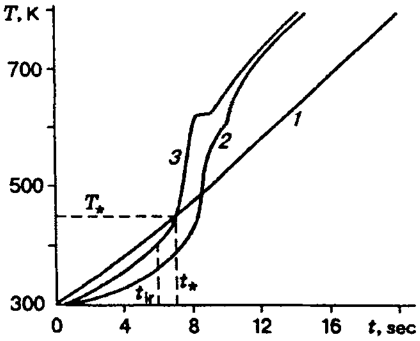

Fig. 2

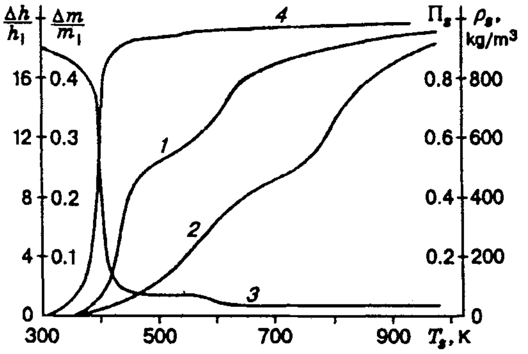

Fig. 3

of intumescence. It turned out that these factors influence only slightly the final result and can be considered insignificant. The reaction unit is heated in the longitudinal flow 3 of the heated gas (nitrogen) at fixed temperature. The ends of the unit are stopped by plugs 4 of annealed asbestos board to prevent gas inflow into the unit. The quantities measured in the experiment are as follows: the temperature T_c at the center of the specimen, the temperature T_{Oz} on the axis of the hollow cylinder, and the temperature T_w on the boundary between the specimen and the cylinder wall. The measurements were performed by microthermocouples 5–7 of KhA type with a junction size of $2 \cdot 10^{-4}$ m. The signals from the microthermocouples were recorded on an N-115 light-beam oscillograph [4, 10].

Figure 2 shows a typical oscillogram of the temperatures T_w, T_{Oz}, and T_c, (curves 1–3) as functions of time t for a specimen of SGK-1 intumescent material [3, 4]. It is seen that the unit is heated linearly (i.e., $q_w = \text{const}$) at a speed of ~25 K/sec. Beginning with $t > t_{ir}$ (t_{ir} is the onset of thermal destruction of the polymer binder), the temperature $T_c(t)$ increases abruptly and equals $T_w(t)$ for $t = t_*$. Such a behavior of the curve of $T_c(t)$ shows that the thermal destruction proceeds with volume heat release and is qualitatively similar to the solid state reaction in ignition of condensed substances [9, 10]. The adiabatic regimes of such reactions have been much studied [9, 10] and were used [4] to find zeroth approximations for kinetic parameters. The method of stabilized states was used to determine the intumescence ratio and the mass ratio as functions of temperature [7]. The essence of this method is that a specimen is annealed in a heated-gas flow (nitrogen) at fixed stabilization temperatures T_{si} ($i = 1, 2, \ldots, n$), which are increased sequentially $T_{s1} < T_{s2} < T_{s3} < \ldots < T_{sn}$, and then is cooled (frozen) rapidly in a flow of cold nitrogen. It is assumed that in rapid cooling the physicochemical processes in the material are fixed and correspond to the equilibrium state at the given temperature T_{si}. The process of heating of the specimen to a new, higher stabilization temperature is recorded by oscillograph as a function of time and is used subsequently to determine thermophysical characteristics by solution of the coefficient inverse problem of heat conduction [8].

Figure 3 shows the intumescence ratio $\Delta h/h_i$ and the mass ratio $\Delta m/m_i$ (curves 1 and 2) as functions of the temperature T_s. Here $\Delta h = h - h_i$ and $\Delta m = m_i - m$ are the changes in the dimensions and mass of the specimen from the initial to the final times. The quantity Δh corresponds to the elongation of the specimen due to intumescence between initial state 1 and final state 8 (see Fig. 1).

Let us consider the second stage of physicochemical conversions, which takes place at $T_s > 550$ K. At these temperatures the intumescence ratio is $\gtrsim 12$ (curve 1), and the porosity reaches $\gtrsim 0.9$ (curve 4). It seems likely that heterogeneous oxidation or chlorination with possible carbon-black formation proceed in a highly developed porous structure. Since the mechanism of this overall reaction is unknown, we use a formal kinetic approach and write this reaction as [10]

$$W_2 = -k_{02}[(m/m_\mathrm{i}) - (m_2/m_\mathrm{i})]\exp(-E_2/RT), \tag{1.1}$$

where $k_{02} = \overline{k_{02}}(S/V)(P_{e2}M_{e2}C_{e2}/RT_*)^\nu$ is an effective preexponential factor.

A distinctive feature of reaction (1.1) is the presence of a developed porous structure in the intumescent layer 8 (see Fig. 1), in which an element with specific surface S/V can be chosen as a characteristic size. Figure 3 gives graphs of the density ρ_s and porosity Π_s of the heat-insulating layer (curves 3 and 4) as functions of the stabilization temperature T_s. It is seen that at $T_s > 600$ K, the porosity reaches a limiting value. The size of a characteristic element, for example, the radius of the fibrous structure of the intumescent layer, can be taken into account in Eq. (1.1) in calculation of the heterogeneous reaction rate W_2. We assume that reaction (1.1) proceeds from $t \geqslant t_*$, which corresponds to the point of intersection of the curves $T_c(t)$ and $T_w(t)$ (see Fig. 2). The characteristic temperature at $t = t_*$ is denoted by T_*.

2. Mathematical Model and Formulation of Inverse Problems. Let us formulate the inverse problem of transient heat-and-mass transfer for a cylindrical body (see Fig. 1). The thermal-flux density vector is assumed to be dependent on the longitudinal z coordinate and nondependent on the radial angle φ. The temperature $T_w(z,t)$ is given on the outer heated surface of the body. Free convective heat exchange is specified by a boundary condition of the third kind with the heat-transfer coefficient α_z at the right boundary. Symmetry conditions are specified on the left boundary $z = 0$ and on the axis $r = 0$. The initial temperature distribution in the specimen and the mass of the specimen are known. The problem is to determine the thermophysical characteristics of the body and macrokinetic constants of the chemical reactions occurring in the solid body upon heating, using the initial and boundary conditions and also additional experimental information on the temperature $T_c(t)$ at the center of the specimen.

The heat-and-mass transfer in the intumescent material is described by a system of nonlinear equations of energy and chemical kinetics:

$$\rho c\frac{\partial T}{\partial t} = \frac{1}{r}\frac{\partial}{\partial r}\Big(r\lambda\frac{\partial T}{\partial r}\Big) + \frac{\partial}{\partial z}\Big(\lambda\frac{\partial T}{\partial z}\Big) - \rho c U\frac{\partial T}{\partial z} + \frac{\rho_\mathrm{i}}{f(T)}(Q_1W_1 + Q_2W_2), \qquad f(T) = 1 + \frac{\Delta h}{h_\mathrm{i}}; \tag{2.1}$$

$$\frac{\partial(m/m_\mathrm{i})}{\partial t} + U\frac{\partial(m/m_\mathrm{i})}{\partial z} = W_1 + W_2 \quad [0 \leqslant t \leqslant t_\mathrm{f}, \quad 0 \leqslant r \leqslant r_w, \quad 0 \leqslant z \leqslant z_2(r,t)] \tag{2.2}$$

with the initial and boundary conditions

$$T(r,z,0) = T_\mathrm{i}, (m/m_\mathrm{i})(r,z,0) = 1; \tag{2.3}$$

$$T(r_w,z,t) = T_w(z,t); \tag{2.4}$$

$$\frac{\partial T(0,z,t)}{\partial r} = \frac{\partial T(r,0,t)}{\partial z} = 0; \tag{2.5}$$

$$\lambda(T(r,z_2,t))\frac{\partial T(r,z_2,t)}{\partial z} = \alpha_z(T_{ez}(r,z_2,t)) - T(r,z_2,t)). \tag{2.6}$$

Here the rate of the first stage of thermal destruction has the form

$$W_1 = -k_{01}\Big(\frac{m}{m_\mathrm{i}} - \frac{m_1}{m_\mathrm{i}}\Big)\exp\Big(-\frac{E_1}{RT}\Big), \tag{2.7}$$

and the heterogeneous reaction rate W_2 (second stage) is determined from (1.1). The intumescence rate U is calculated taking into account the experimental dependence $\Delta h/h_\mathrm{i} = f(T_s) - 1$ (Fig. 3, curve 1).

Since reaction (1.1) proceeds beginning from $t = t_*$ at $T_c(t) \geqslant T_*$ (Fig. 2, curve 3), we have $W_1 = 0$ and $W_2 \neq 0$ for $m/m_\mathrm{i} \leqslant m_1/m_\mathrm{i}$, and $W_1 = W_2 = 0$ for $m/m_\mathrm{i} \leqslant m_2/m_\mathrm{i}$. The values $m_1 > m_2$ are taken

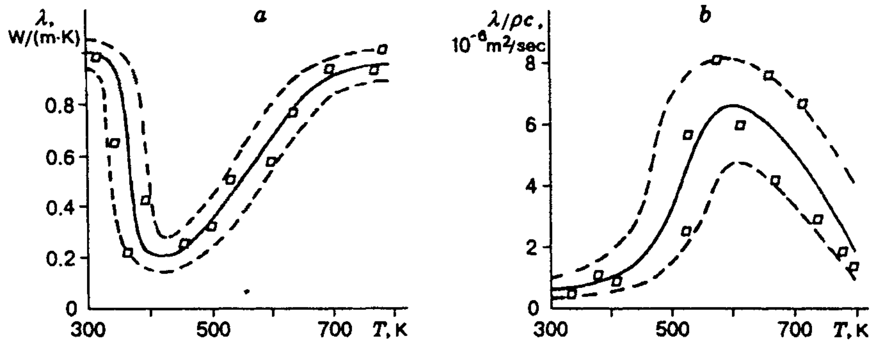

Fig. 4

from the curve of mass ratio $\Delta m/m_i(T_s)$ (Fig. 3, curve 2). In expressions (1.1) and (2.1)–(2.7), the notation is as follows: c is the specific heat; λ is the thermal conductivity; E_1, k_{01}, and Q_1 are the activation energy, preexponent, and heat effect of the first stage of thermal destruction; E_2, k_{02}, Q_2, and ν are the activation energy, preexponent, heat effect, and order (for the oxidizer) of the heterogeneous reaction; P, M, and C are the pressure, molecular mass, and concentration of the oxidizer in the porous structure; $z_2(r,t)$ is the free boundary of the intumescent layer; R is the gas constant; subscripts i, f, $*$, and e refer to the initial, final, characteristic, and gaseous states, respectively; subscript s refers to stabilization at a given temperature, $e2$ to the porous structure in the intumescent layer, and w to the heated surface of the reaction unit.

In the mathematical model (2.1)–(2.7) taking into account (1.1), it is required to determine the thermophysical characteristics λ and ρc and the macrokinetic constants of the first and second stages of the thermal-destruction reaction E_i, k_{0i}, and Q_i ($i = 1, 2$), which identify the heat-and-mass transfer in the intumescent heat-insulation material.

3. Solution Algorithm for the Inverse Problems. The thermophysical characteristics of SGK-1 intumescent material [8] were refined by solution of the two-dimensional, coefficient, inverse problem of heat conduction using the principle of splitting complex processes into simple components [6], the method of stabilized states [7] with the experimental diagram in Fig. 1, and the experimental data of Fig. 2. As a zeroth approximations, we used the values of thermal conductivity λ and volume specific heat $c\rho$ from [8], where they were obtained by solution of the corresponding inverse one-dimensional, coefficient, problem [8, 11]. The coefficients λ and ρc are shown in Fig. 4a and 4b as functions of the stabilization temperature T_s, which are nonlinear and represent the entire spectrum of processes observed in the intumescent material during heating. The solid curves are approximation functions, the boxes are values of the thermophysical characteristics obtained by the method of stabilized states [7, 8], and the dashed curves are confidence intervals of the determined values.

The macrokinetic constants of both stages of thermal destruction E_i, k_{0i}, and Q_i ($i = 1, 2$) were determined by minimizing the functional

$$\Phi(E_i, k_{0i}, Q_i) = \int_0^t (T_c^{\text{exp}}(0,0,t) - T_c(0,0,t))^2 dt, \tag{3.1}$$

which is the root-mean-square difference between the experimental (T_c^{exp}) and calculated (T_c) temperatures at the center of the specimen; the latter were found by solution of two-dimensional boundary-value problem (2.1)–(2.7) with (1.1).

System (2.1)–(2.7) is difficult to integrate because of significant variations in the length of the specimen in the Oz direction. Therefore, we introduce Lagrangian variables [12] in the form

$$t = \tau, \qquad z = \int_0^\xi f\,d\tau. \tag{3.2}$$

The relations

$$\frac{\partial}{\partial t} = \frac{\partial}{\partial \tau} - f^{-1} \int\limits_0^{\xi} \frac{\partial f}{\partial \tau} d\xi \frac{\partial}{\partial \xi}, \quad \frac{\partial}{\partial z} = f^{-1} \frac{\partial}{\partial \xi} \tag{3.3}$$

are valid for the derivatives, and the rate of intumescence is given by the formula

$$U = \int\limits_0^{\xi} \left(\frac{\partial \xi}{\partial \tau}\right) d\xi. \tag{3.4}$$

Two-dimensional boundary-value problem (2.1)–(2.7) in the new variables ξ and τ from (3.2) with allowance for (3.3) and (3.4) takes the form

$$\rho c \frac{\partial T}{\partial \tau} = \frac{1}{r} \frac{\partial}{\partial r}\left(r \lambda \frac{\partial T}{\partial r}\right) + \frac{1}{f} \frac{\partial}{\partial \xi}\left(\frac{\lambda}{f} \frac{\partial T}{\partial \xi}\right) + \frac{\rho_{\text{i}}}{f}(Q_1 W_1 + Q_2 W_2); \tag{3.5}$$

$$\frac{\partial (m/m_{\text{i}})}{\partial \tau} = W_1 + W_2, \quad 0 \leqslant \tau \leqslant \tau_{\text{f}}, \quad 0 \leqslant r \leqslant r_w, \quad 0 \leqslant \xi \leqslant \xi_2 \quad \left(\xi_2 = \frac{h_{\text{i}}}{2}\right); \tag{3.6}$$

$$T(r, \xi, 0) = T_{\text{i}}, \quad (m/m_{\text{i}})(r, \xi, 0) = 1; \tag{3.7}$$

$$T(r_w, \xi, \tau) = T_w(\xi, \tau); \tag{3.8}$$

$$\left(\frac{\lambda}{f}\right) T(r, \xi_2, \tau) \frac{\partial T(r, \xi_2, \tau)}{\partial \xi} = \alpha_z (T_{ez}(r, \xi_2, \tau) - T(r, \xi_2, \tau)). \tag{3.9}$$

Problem (3.5)–(3.9) can be solved by any of the well-known numerical methods, for example, by the splitting method [13]. The one-dimensional equations resulting from splitting in each time half-step are solved effectively by the iteration-interpolation method of [11] with iterations for coefficients.

The functional (3.1) is minimized using the conjugate gradient method [14]. The iterative process of searching for the macrokinetic constants is terminated when the condition

$$\Phi(E_i, k_{0i}, Q_i) \leqslant \delta^2 \tag{3.10}$$

is satisfied. Here $\delta^2 = \delta_T^2 + \delta_a^2$; δ_T^2 and δ_a^2 are integral errors in the determination of experimental and calculated temperature values, respectively. As a rule, $\delta_a \ll \delta_T$ [11]. The quantity δ_T^2 is estimated using the formula $\delta_T^2 = \int\limits_0^t \sigma^2(t) dt$, where $\sigma^2(t)$ is the variance of the function $T_c^{\text{exp}}(t)$. The number of iterations consistent with condition (3.10) serves as a parameter of natural regularization in the chosen method.

4. Results of Numerical Calculations. A FORTRAN program for an IBM PC AT 386 was developed to solve the problem. The thermophysical characteristics presented in Fig. 4, and the intumescence ratio function $\Delta h/h_{\text{i}}(T_s)$ (Fig. 3, curve 1) were used in the numerical calculations. The values of macrokinetic constants from [4] were taken as zeroth approximations for the first stage of the thermal destruction reaction. They were obtained under the assumption of the adiabatic progress of the reaction at the characteristic points (t_*, T_*), which are determined experimentally for different heating rates. The time step Δt was 0.1 sec, and the number of points in the Or and Oz directions was $n_r = n_z = 11$ in the solution of the direct heat-and-mass transfer problem (3.5)–(3.9). The characteristic temperature was $T_* = 450$ K. The calculation time for one variant of the problem did not exceed 5 min. Subsequent doubling of the number of points n_r and n_z increased insignificantly (~0.5%) the accuracy of the solution of the problem, although the calculation time increased by a factor of greater than two.

Numerical investigations were performed to estimate the solutions obtained within the frameworks of one-dimensional (for $z = 0$) and two-dimensional formulations of the problem. We used the values of macrokinetic constants obtained in [15] by solution of the one-dimensional inverse problem. Curves 1–3 in Fig. 5 show, respectively, the experimental temperatures at the center and on the surface of the specimen, and the gas temperatures in the reaction unit, and curves 4 and 5 represent the calculated temperatures on the specimen axis at the center $z = 0$ (upper curves) and on its free boundary $z = z_2(0, t)$ (lower curves) for

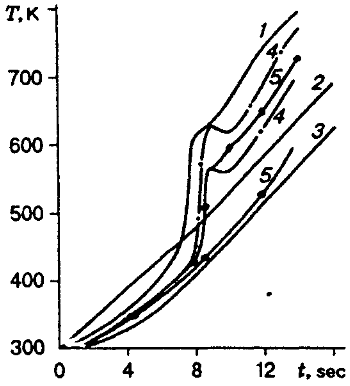

Fig. 5

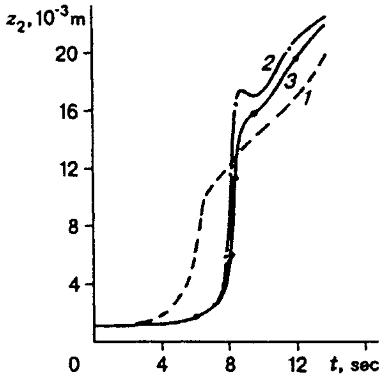

Fig. 6

$\alpha_z = 0$ and 50 W/(m^2· K). Analysis of the curves in Fig. 5 shows that the temperature values at the center of the specimen obtained by solution of the one-dimensional problem are higher than the values found by solution of the two-dimensional problem. This is due to neglect of heat flow along the z axis. The temperature difference across the thickness of the specimen increases with increasing α_z.

Figure 6 gives the calculated coordinate of the free boundary $z_2(r,t)$ at $r = r_w$ (curve 1) and at $r = 0$ for $\alpha_z = 0$ and 50 W/(m^2·K) (curves 2 and 3). Nonuniform intumescence of the specimen along the radius is observed, which corresponds to the temperature variation along the radius. At initial times to $t \leqslant 8.4$ sec, the intumescence on the specimen axis lags behind but then leads the intumescence on the heated surface of the specimen.

This analysis of the curves in Figs. 5 and 6 shows that it is necessary to use the two-dimensional formulation and also take into account heat exchange on the free boundary with a sufficiently accurate heat-transfer coefficient α_z in order to determine the macrokinetic constants of the studied reactions by solution of the inverse problems. In this connection, we determine the free-convective heat-transfer coefficient α_z in the gas space between the free boundary $z_2(r,t)$ and the plug (see Fig. 1) by the formula [16] $\alpha_z = (\lambda_e/d)\mathrm{Ra}^{1/8}$, where Ra is the Rayleigh number; d is the characteristic size of the space; and λ_e is the heat conductivity of the gas in the space.

If we take the radius of the hollow cylinder as the characteristic size d (see Fig. 1), we obtain $\alpha_z \sim$ 16–25 W/(m^2· K) in the temperature range T_{ez} = 300–800 K and for Rayleigh numbers Ra = 8–80 at $P_e = 0.1$ MPa. The mean value $\alpha_z = 20$ W/(m^2· K) was used in numerical calculations by the model (3.4)–(3.9). Curves 4 in Fig. 7 show the calculated temperature functions on the specimen axis at the center $z = 0$ (upper curve) and at the free boundary $z = z_2(0,t)$ (lower curve) as compared with experimental curves 1–3 (notation the same as in Fig. 5) for the given values of the heat-transfer coefficient $\alpha_z = 20$ W/(m^2· K) and the macrokinetic constants from [15]. A noticeable difference between the experimental (1) and calculated (4) temperature values at the center of the specimen was observed. Therefore, we used a two-dimensional mathematical formulation with $\alpha_z = 20$ W/(m^2· K) to determine the thermokinetic constants.

The errors of the thermokinetic constants due to the error in the solution algorithm for the inverse problem and inaccurate temperature measurements at the center of the specimen $T_c^{\mathrm{exp}}(0,0,t)$ were found by solution of a model problem close to the real one. The initial data on the experimental temperatures at the center of the specimen used in the two-dimensional inverse problem were obtained by solution of the two-dimensional direct problem with the data given in the paper. The calculated temperature values $T_c(0,0,t)$ were determined with an accuracy of 0.5%. Cubic splines [11] were used to decrease the errors caused by interpolation of temperature values at the grid points.

The numerical calculations show that the maximum relative error of the thermokinetic constants at unperturbed experimental temperature values at the center of the specimen does not exceed 2%. The obtained error is close to the error of determination of the effective thermokinetic constants of heterogeneous chemical

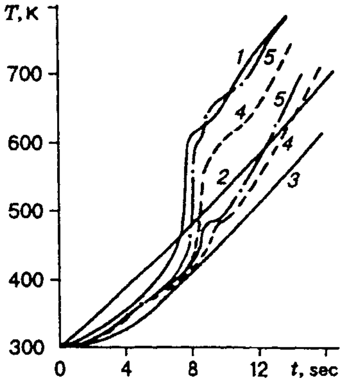

Fig. 7

reactions [11]. Actually, the experimental temperature values at the center of the specimen were measured by thermocouples with an error not greater than 4% [4, 10]. Specifying the temperature from the range of errors with limits of $(1 \pm 0.04)T_c(0,0,t)$ [$T_c(0,0,t)$ is the unperturbed temperature], we obtained the relative errors of the thermokinetic constants. Taking into account these errors, the solution of the two-dimensional inverse problem with real temperatures at the center of the specimen (curve 1 in Fig. 7) at $\alpha_z = 20$ W/(m$^2\cdot$ K) yields corrected values of the macrokinetic constants for the studied reactions: $E_1 = (74 \pm 10)$ kJ/mole, $Q_1 = (1.2 \pm 0.2) \cdot 10^6$ J/kg, $k_{01} = 1.3 \cdot 10^{8\pm1}$ sec^{-1}, $E_2 = (43.5 \pm 5)$ kJ/mole, $Q_2 = (3.5 \pm 0.5) \cdot 10^7$ J/kg, $k_{02} = 5 \cdot 10^{2\pm0.5}$ sec^{-1}.

Curves 5 in Fig. 7 show the calculated temperature functions on the axis $r = 0$ at the center of the specimen $z = 0$ (upper curve) and at its mobile boundary $z = z_2(0,t)$ (lower boundary) obtained by solution of the direct problem using the macrokinetic constants found. The root-mean-square difference between the experimental and calculated temperature values at the center of the specimen does not exceed 30 K, which is ~5–6% for a confidence level of 0.95.

Thus, a set of thermophysical and macrokinetic characteristics that identify with sufficient accuracy the experimentally observed physicochemical processes was determined on the basis of a two-dimensional mathematical model of heat- and mass transfer in intumescent materials using the principle of splitting into physical processes and effective solving algorithms for the direct and inverse problems.

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