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Regimes of Connecting Materials with the Help of Synthesis in the Solid Phase

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Abstract

Mathematical models of connecting materials using a mixture of exothermal composition with inert filler in the regime of thermal explosion and in the regime of combustion are proposed. The first problem is formulated as thermal inflammation in a vessel with thick walls, the second one as the propagation of the front of exothermal reaction in a slit between two thick plates. In general, the addition of an inert filler to the connecting mixture causes an increase in inflammation time, a decrease in temperature and rate of reaction, which coincides with the known experimental data. Within a definite region of the model parameters, numerical investigation of the inflammation problem revealed different regimes of inflammation: thermal explosion and a slow transformation under weakly changing temperature. When investigating the problem of reaction propagation in a slit, also different regimes of transformation with variations of thermal properties of the substances were discovered. The region of parameters in which the self-sustaining regime of transformation occurs was found. For real technologies, low-temperature synthesis regimes are of interest, because in this case thermal strain decreases.

INTRODUCTION

Connecting materials by means of the solid-phase synthesis has been attracting attention of researchers and technologists for years. An example may be thermit welding of metal materials (rails, tubes, *etc.*), when the materials to be connected are preliminarily heated to some temperature $T_s >> T_0$, where T_0 is the reagent temperature. The conditions of inflammation of the connecting composition can be estimated on the basis of thermal theory of inflammation [1]. Assuming that the materials to be connected have rather large size, we will come across the problem of ignition of a layer by a body with a large amount of heat stored [2]. Such a problem may be considered in the conjugated formulation taking into account temperature redistribution in the system due to heat conductivity [3]. Non-metal compounds can be connected in the regime of thermal explosion or in the combustion regime. Such a problem arises, for example, for the construction of electrolyzers in aluminium industry. The formulation of the problem will depend on the experimental conditions. In any case, high temperature in the reaction zone is often undesirable because it is accompanied by high thermal strain causing fracturing of the materials to be connected. To avoid this, one should use the connecting compositions containing the reagents that interact producing not very high exo-effects. In doing this, it is necessary to determine the necessary conditions for the solid-phase reactions to proceed, and their dependence on the properties of the materials to be connected. One of the possible methods of reducing the transformation temperature is to use an inert filler in the connecting composition; on the one hand, this filler takes a part of total heat thus decreasing heat evolution during reaction; on the other hand, it helps maintaining the transformation due to the accumulated heat. Possible melting of the inert filler also can help achieving the required result. If the reagent and the materials to be connected are heated by the starting moment of time to the same temperature

($T_0 >> T_e$, where T_e is temperature of environment), we will have the problem of thermal inflammation in a thick-walled vessel. The effect of heat loss into the environment can be different depending on sample geometry.

The problem of determining favourable conditions for connecting the materials with the help of an exothermal composition in the combustion regime can be formulated as a problem of flame propagation (the propagation of a front of the solid-phase transformation) in a slit between two inert materials. In this case, the materials to be connected play a dual role: on the one hand, they remove heat from the zone of chemical reactions, on the other hand, they prevent deformation of the reagent layer in the cross direction and promote heating of the reagent before the reaction zone due to thermal conductivity.

The goal of the present work was to investigate possible transformation regimes during connecting the inert materials.

A PROBLEM OF THERMAL EXPLOSION IN A THICK-WALLED VESSEL

Let us consider the problem of thermal explosion in a vessel with thick walls formulated as follows. Let a mixture of the compound capable of undergoing exothermal transformation and an inert material be present between two inert plates with thickness h (Fig. 1).

Let us denote the thickness of the reagent-containing layer as $2r$, and the fraction of the inert filler as z. The point of origin is in the centre of the narrow layer. The problem is symmetrical, so the symmetry condition

$$\partial T/\partial x = 0 \tag{1}$$

is fulfilled in the centre at $x = 0$.

The thermal conductivity equation for a mixture of the reagent with an inert filler taking into account heat evolution during the chemical reaction is:

$$|x| \le r : \left[c_1\rho_1(1-z) + c\rho z\right]\frac{\partial T}{\partial t} = \left[\lambda_1(1-z) + \lambda z\right]\frac{\partial^2 T}{\partial x^2} + (1-z)Qk_0\varphi_1\varphi_2 \tag{2}$$

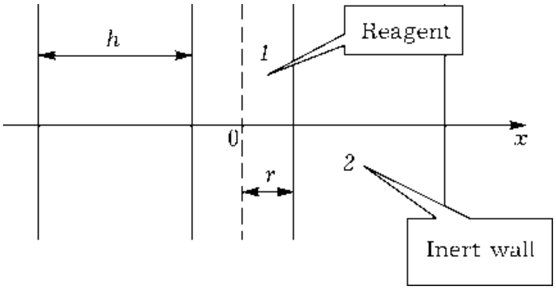

Fig. 1. Illustration to the formulation of the problem of thermal inflammation in a thick-walled vessel: *1* – reagent, *2* – materials to be connected. For designations, see text.

The thermal conductivity equation for inert plates does not contain the sources and sinks of heat:

$$r + h \ge x \ge r : \quad c_2\rho_2\frac{\partial T_2}{\partial t} = \lambda_2\frac{\partial^2 T}{\partial x^2} \tag{3}$$

We assume that this is the first-order reaction and its rate depends on temperature according to the Arrhenius law. Then, for the mass fraction of the reaction product a (or the transformation degree), we obtain:

$$\frac{\partial a}{\partial t} = k_0\varphi_1(a)\varphi_2(T) = k_0(1-a)\exp\left(-\frac{E}{RT}\right) \tag{4}$$

The conditions of the ideal thermal contact will be considered true at the boundary between the substances; this requirement is represented mathematically in the following form:

$$x = r : \quad \left[\lambda_1(1-z) + \lambda z\right]\frac{\partial T_1}{\partial x} = \lambda_2\frac{\partial T_2}{\partial x}, \; T_1 = T_2 \tag{5}$$

At the external boundary of the inert material, heat exchange with the environment occurs according to Newton's law:

$$x = r + h : \quad -\lambda_2\frac{\partial T_2}{\partial x} = \alpha(T_2 - T_e) \tag{6}$$

At the initial moment of time, temperature of the substances T_0 and environmental temperature T_e are predetermined:

$$T_1 = T_2 = T_0 \tag{7}$$

at that, $T_0 >> T_e$.

It is necessary to determine critical conditions separating inflammation and extinction, or different regimes of transformation depending in the thickness and properties of the inert material.

The following designations are accepted in (1)–(7): c_i, ρ_i, λ_i are heat conductivity, density and coefficient of thermal conductivity, respectively (the values without indices refer to the inert filler), T_i is their temperature, x is the spatial coordinate, t is time, Q is heat effect, E, k_0 are activation energy and pre-exponential factor for the overall reaction, respectively; α is heat-transfer factor.

With dimensionless variables,

$$\theta = \frac{T - T_0}{RT_0^2}E\,,\quad \xi = \frac{x}{r}\,,\quad \tau = \frac{t}{t_a}$$

where $t_a = \dfrac{c_1\rho_1 RT_0^2}{EQk_0}\exp\left(\dfrac{E}{RT_0}\right)$ is the period of adiabatic induction at temperature T_0, the problem (1)–(7) takes the form:

$$\xi \le 1:\quad \frac{\partial\theta_1}{\partial\tau} = \delta^{-1}\frac{\left[(1-z)+B_\lambda z\right]}{\left[(1-z)+B_c z\right]}\frac{\partial^2\theta_1}{\partial\xi^2} + \frac{1}{\left[(1-z)+B_c z\right]}\exp\left(\frac{\theta_1}{1+\beta\theta_1}\right) \tag{8}$$

$$\frac{\partial a}{\partial\tau} = \gamma\exp\left(\frac{\theta_1}{1+\beta\theta_1}\right)(1-a) \tag{9}$$

$$1 \le \xi \le h:\quad K_c\frac{\partial\theta_2}{\partial\tau} = K_\lambda\frac{\partial^2\theta_2}{\partial\xi^2} \tag{10}$$

$$\xi = 0:\quad \frac{\partial\theta_1}{\partial\xi} = 0 \tag{11}$$

$$\xi = 1:\quad \frac{\partial\theta_1}{\partial\xi} = \frac{K_\lambda}{\left[(1-z)+B_\lambda z\right]}\frac{\partial\theta_2}{\partial\xi}\,,\quad \theta_1 = \theta_2 \tag{12}$$

$$\xi = H+1:\quad -K_\lambda\frac{\partial\theta_2}{\partial\xi} = \mathrm{Nu}\left(\theta_2 + \theta_e\right) \tag{13}$$

$$\tau = 0:\quad \theta_1 = 0,\ \theta_2 = 0,\ y = 0 \tag{14}$$

where $H = h/r$, $\sigma = r^2/(t_a\kappa_1)$, $\mathrm{Nu} = \alpha r/\lambda_1$, $\beta = RT_0/E$, $K_c = c_2\rho_2/(c_1\rho_1)$, $K_\lambda = \lambda_2/\lambda_1$, $B_\lambda = \lambda/\lambda_1$, $B_c = c\rho/(c_1\rho_1)$, $\theta_0 = (T_0 - T_e)E/(RT_0^2)$, $\theta_0 = (T_0 - T_e)E/(RT_0^2)$, $\gamma = c_1\rho_1 RT_0^2/(EQ)$, $\kappa_1 = \lambda_1/(c_1\rho_1)$.

The problem (8)–(14) was investigated in detail in [4, 5]. Here we should mention the following.

Unlike the classical problem of thermal explosion [6–8], under the conditions of conjugated heat exchange the role of burnup increases even for large θ_0, which is illustrated in Fig. 2, *a*, *b*.

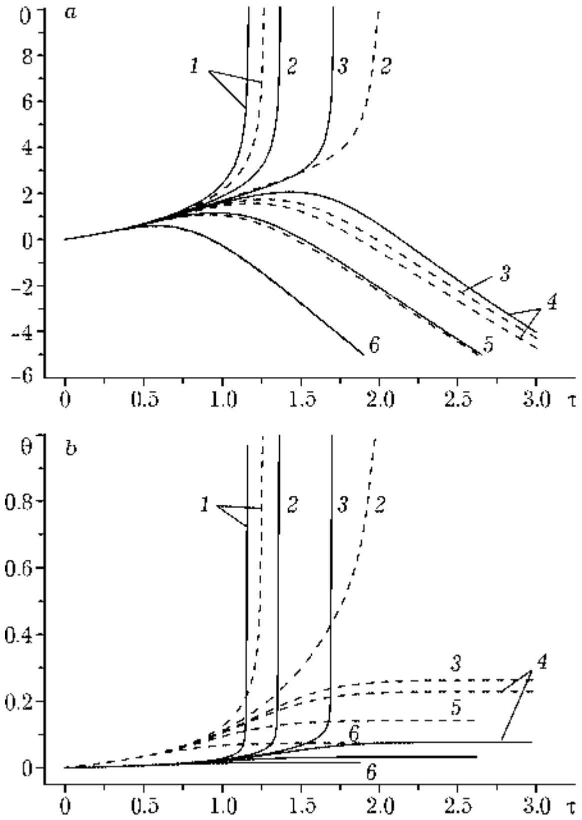

Fig. 2. Dependence of temperature (*a*) and transformation degree (*b*) on time ($K_c = 5$, $K_\lambda = 10$, Nu = 100, $H = 2$, $\theta_0 = 20$): continuous curves − $\gamma = 0.01$ ($\delta^* = 4.6$), dashed curves − $\gamma = 0.05$ ($\delta^* = 4.88$); δ values: 5 (*1*), 4.9 (*2*), 4.88 (*3*), 4.6 (*4*), 4.5 (*5*), 4 (*6*).

Similarly to the classical heat explosion problem, for small δ and $\theta_0 = 20$ temperature in the centre of the reagent mass drops either at once or after a small increase (continuous curves *4–6* and dashed ones *5*, *6* in Fig. 2); for δ larger than some critical value δ^* temperature rises sharply, that is, ignition occurs (continuous curves *1, 2,* dashed ones *1–3* in Fig. 2). Such a character of the process development is typical for the normal inflammation regime. For inflammation, $a(0,\tau_B)$ approached unity even in the case of small γ values. If $\gamma = 0.05$, substantial a values are observed also in the case of temperature drop, that is, in the case of extinction. With an increase in γ, the δ^* value increases; this is connected with a decrease in heat generation due to burnup (elimination) of the substance. An increase in the role of burnup is explained by the specific character of the problem − the additional heat storage in the vessel walls, which promotes transformation.

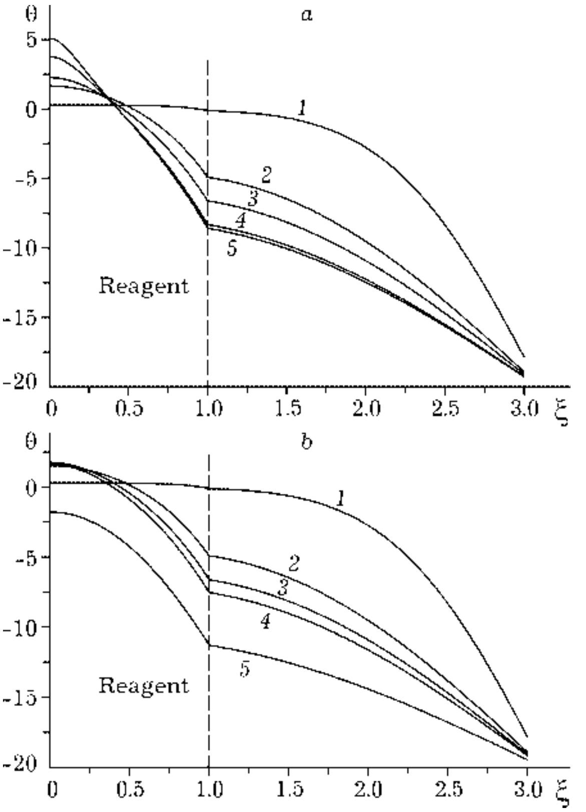

Fig. 3. Spatial distribution of temperature for $\delta = 4.6$ ($\theta_0 = 20$, $K_c = 5$, $K_\lambda = 10$, $\mathrm{Nu} = 100$, $H = 2$): *a* – $\gamma = 0.01$ (inflammation), τ values: 0.25 (*1*), 1 (*2*), 1.28 (*3*), 1.62 (*4*), 1.68 (*5*); *b* – $\gamma = 0.05$ (extinction), τ values: 0.25 (*1*), 1.01 (*2*), 1.45 (*3*), 1.62 (*4*), 2.4 (*5*).

The higher are the walls of the vessel, the larger amount of heat is accumulated in them, the less essential is heat emission from the ends of the materials to be connected, the lower is δ^* critical value. The effect of γ is shown in Fig. 3. While for $\gamma = 0.01$ and $\delta = 4.6$ inflammation occurs (see Fig. 3, *a*), extinction is observed for $\gamma = 0.05$ and the same δ (see Fig. 3, *b*). Figure 3, *a* corresponds to continuous curves in Fig. 2 and dashed curves in Fig. 3, *b*.

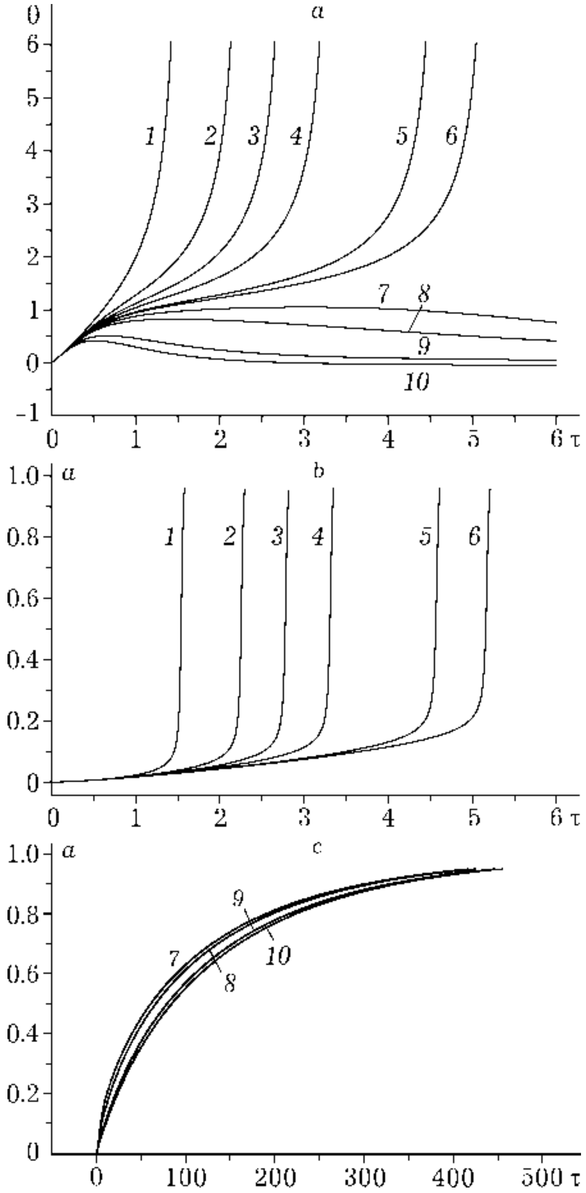

Fig. 4. Dependence of temperature (*a*) and transformation degree (*b*, *c*) on time for the rapid (*b*) and slow (*c*) regimes: continuous curves – $\theta_0 = 5$ ($\delta^* = 2.4$), dashed curves – $\theta_0 = 20$ ($\delta^* = 3.07$); δ values: 2 (*1*), 1.5 (*2*), 1.4 (*3*), 1.35 (*4*), 1.3 (*5*), 1.29 (*6*), 1.25 (*7*), 1.2 (*8*), 1 (*9*), 0.9 (*10*).

It was discovered during a detailed numerical examination of the problem that the addition of an inert filler into the connecting composition brings substantial changes into the qualitative character of the process. Within some region of the parameters of the problem, varying δ we may distinguish different regimes of transformation: thermal explosion and a slow transformation. In the second regime, transformation occurs at almost constant reagent temperature. time of complete transformation differs by two orders of magnitude for different regimes. In this case, the critical δ value takes another sense when the inert material is added into the connecting mixture.

Dependencies of temperature and transformation degree on time for different regimes in the centre of the reagent ($\xi = 0$) with the set of parameters $K_c = 5$, $K_\lambda = 10$, $H = 2$, $\theta_0 = 5$, $B_c = B_\lambda = 0.5$ taking into account the inert filler $z = 0.4$ are shown in Fig. 4, *a*–*c*. In this case, the critical value $\delta^* = 1.28$ separates two different regimes: thermal explosion and slow transformation. For inflammation (see Fig. 4, *a*, *b*, curves *1–6*), the complete transformation of the substance occurs within a short time interval following the induction period. For the second regime (curves *7–10*), gradual accumulation of the reaction product occurs.

CONNECTING TWO MATERIALS IN THE COMBUSTION REGIME

Here we are considering the following formulation of problem of connecting two materials with the help of solid-phase synthesis in the combustion regime. Let a layer of a mixture of the reacting substance and an inert filler, with thickness h, be placed between two inert plates with thickness H (Fig. 5). The mixture is brought to firing from the left end where temperature increases at the moment of time $t = 0$ from T_0 to $T_s >> T_0$. It is necessary to determine possible regimes of transformation in this system.

The problem is symmetric, so it is sufficient to restrict consideration the upper half-plane. The mathematical formulation includes an equation for heat conductivity in the reagent:

$$c_{\text{eff}}\rho_{\text{eff}}\frac{\partial T}{\partial t} = \frac{\partial}{\partial x}\left(\lambda_{\text{eff}}\frac{\partial T}{\partial x}\right) + \frac{\partial}{\partial y}\left(\lambda_{\text{eff}}\frac{\partial T}{\partial y}\right) + (1-a)Qk_0\varphi_1(a)\varphi_2(T) \qquad (15)$$

where $c_{\text{eff}}\rho_{\text{eff}} = c_1\rho_1(1-z) + c\rho z$, $\lambda_{\text{eff}} = \lambda_1(1-z) + \lambda z$, and equation for thermal conductivity in inert materials:

$$c_2\rho_2\frac{\partial T}{\partial t} = \frac{\partial}{\partial x}\left(\lambda_2\frac{\partial T}{\partial x}\right) + \frac{\partial}{\partial y}\left(\lambda_2\frac{\partial T}{\partial y}\right) \qquad (16)$$

In the general case, thermophysical characteristics can depend on temperature. We assume that the reaction is of the first order and its rate depends on temperature according to Arrhenius law similarly to (4).

The conditions at the external boundaries are:

$$x = 0: \begin{cases} T_i = T_s,\ t \le t_1, \\ \dfrac{\partial T_i}{\partial x} = 0,\ t > t_1 \end{cases} \qquad (17)$$

$$x \to l: \ \frac{\partial T}{\partial x} = 0, \quad i = 1,\ 2 \qquad (18)$$

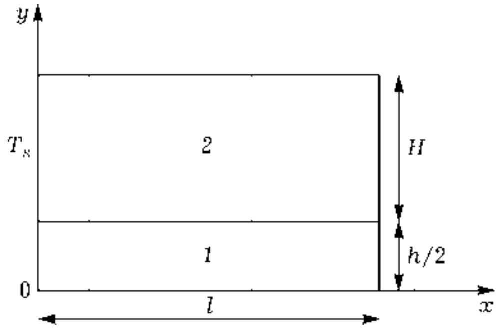

Fig. 5. Illustration to the formulation of the problem of reaction propagation in a slit between two inert plates: *1* – reagent, *2* – materials to be connected. For designations, see text.

The symmetry condition is fulfilled at the $y = 0$ axis:

$$y = 0: \ \frac{\partial T_1}{\partial y} = 0 \qquad (19)$$

while heat exchange with the environment occurs according to Newton's law at the external boundary of the materials to be connected:

$$y \to h/2 + H: \ -\lambda_2\frac{\partial T_2}{\partial y} = \alpha\left(T_e - T_2\right) \qquad (20)$$

where t_1 is time of action of the pulse source.

The conditions of ideal thermal contact are considered to be true at the boundary between the substances; this is similar to (5):

$$y = h/2: \left[\lambda_1(1-z) + \lambda z\right]\frac{\partial T_1}{\partial y} = \lambda_2\frac{\partial T_2}{\partial y},\ T_1 = T_2 \qquad (21)$$

It is necessary to carry out the analysis of possible transformation regimes in this system.

Using dimensionless units,

$\theta = (T - T_s)E/(RT_s^2)$, $\xi = x/x^*$, $\eta = y/x^*$, $\tau = t/t_a$,

where $t_a = \dfrac{\gamma}{k}\exp\left(\dfrac{E}{RT_s}\right)$, $x^* = \sqrt{\kappa_1 t_a}$, $\kappa_1 = \dfrac{\lambda_1}{c_1\rho_1}$,

the problem (15)–(21) may be written down as

$$0 \le \eta \le \sqrt{\delta}: \ \frac{\partial\theta_1}{\partial\tau} = F\left(\frac{\partial^2\theta_1}{\partial\xi^2} + \frac{\partial^2\theta_1}{\partial\eta^2}\right) + G\varphi_1(a)\varphi_2(\theta)$$

$$\frac{\partial a}{\partial\tau} = \gamma(1-a)\exp\left(\frac{\theta_1}{1+\beta\theta_1}\right)$$

$$\eta \ge \sqrt{\delta}: K_c\frac{\partial\theta_2}{\partial\tau} = K_\lambda\left(\frac{\partial^2\theta_2}{\partial\xi^2} + \frac{\partial^2\theta_2}{\partial\eta^2}\right)$$

$$\xi = 0: \quad \theta_i = 0, \quad \tau \le \tau_1 \text{ и } \frac{\partial \theta_i}{\partial \xi} = 0, \quad \tau > \tau_1$$

$$\xi = L: \frac{\partial \theta_i}{\partial \xi} = 0, \; \eta = 0: \frac{\partial \theta_1}{\partial \eta} = 0$$

$$\eta = \sqrt{\delta}: \left(\frac{\partial \theta_1}{\partial \eta}\right)_{\sqrt{\delta}-0} = K_\lambda \left(\frac{\partial \theta_2}{\partial \eta}\right)_{\sqrt{\delta}+0}, (\theta_1)_{\sqrt{\delta}-0} = (\theta_2)_{\sqrt{\delta}+0}$$

$$\eta = \Delta + \sqrt{\delta}: K_\lambda \frac{\partial \theta_2}{\partial \eta} = \mathrm{Nu}(\theta_2 + \theta_e)$$

$$\tau = 0: \theta_1 = -\theta_s, \theta_2 = -\theta_s$$

where $L = l/x^*$, $\Delta = H/x^*$, $\delta = h/x^*$, $\mathrm{Nu} = \alpha\sqrt{\delta}/\lambda_1$, $\tau_1 = t_1/t_a$, $\beta = RT/E$, $\gamma = c_1\rho_1 RT_s^2/(QE)$, $\theta_0 = (T_s - T_0)E/(RT_s^2)$.

Numerical investigation of the problem reveals different steady and non-steady transformation regimes [9] (Fig. 6).

1. If heat conductivity and heat capacity of the materials to be connected are much lower than heat conductivity and heat capacity of the reagent ($K_c << 1$, $K_\lambda << 1$), after a short time interval the quasi-steady transformation regime gets established in the system (see Fig. 6, *a*). It is discovered that in this case the maximal heat emission occurs in the reagent in the zone of its contact with the materials to be connected. Due to the low heat conductivity of the materials to be connected, far away from $\eta = \sqrt{\delta}$ the inert substance is almost non-heated; however, the amount of heat is sufficient to maintain the steady transformation regime. We may estimate the rate of the steady regime using the results of numerical calculation.

2. In the case of large heat capacity ($K_c > 1$) and low heat conductivity ($K_\lambda << 1$) of the

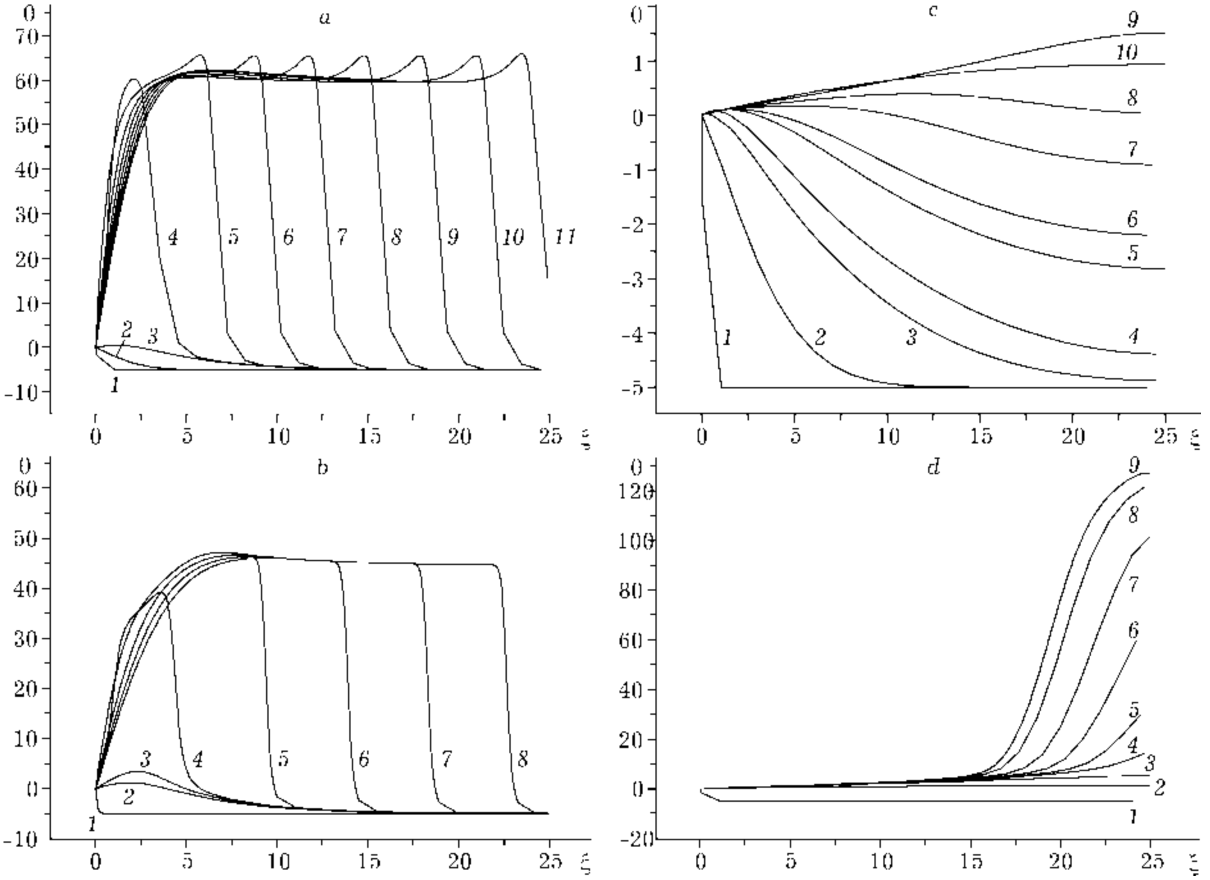

Fig. 6. Spatial distribution of temperature at different moments of time for different transformation regimes: *a* − τ values: 0.01 (*1*), 2 (*2*), 20.5 (*3*), 21.5 (*4*), 21.75 (*5*), 22 (*6*), 22.25 (*7*), 22.5 (*8*), 23 (*9*), 23.1 (*10*), 23.2 (*11*); $z = 0$, $\theta_0 = 5$, $K_\lambda = 0.1$, $K_c = 0.1$; *b* − τ values: 0.01 (*1*), 31 (*2*), 33 (*3*), 34 (*4*), 35 (*5*), 37 (*6*), 38 (*7*), 39 (*8*); $K_c = 5$, $K_\lambda = 0.1$, $\theta_0 = 5$, $z = 0$, $\tau_c = 38.57$; *c* − τ values: 0.01 (*1*), 5 (*2*), 25 (*3*), 45 (*4*), 100 (*5*), 125 (*6*), 175 (*7*), 200 (*8*), 225 (*9*), 299 (*10*); $K_c = 5$, $K_\lambda = 10$, $\theta_0 = 5$, $z = 0$, $\gamma = 0.01$; *d* − τ values: 0.01 (*1*), 7.5 (*2*), 8.15 (*3*), 8.2 (*4*), 8.21 (*5*), 8.22 (*6*), 8.23 (*7*), 8.24 (*8*), 8.245 (*9*); $K_c = 0.1$, $K_\lambda = 10$, $\theta_0 = 5$, $z = 0$, $\gamma = 0.01$.

materials to be connected, the steady stage of the process development also exists, but the reaction front propagates with lower rate than that for the previous case (see Fig. 6, *b*); the reaction zone is wider. Due to an increase in the amount of heat accumulated and low heat conductivity, the materials to be connected have no sufficient time to get heated; the front of chemical reaction leaves behind heating of the materials to be connected.

3. With an increase in heat conductivity of the materials to be connected, steady regimes of transformation may be absent. For example, if $K_\lambda = 10$, $K_c = 5$, slow transformation occurs within the entire volume of the material at low temperature; the heat evolved in the reaction zone gets transferred to the inert walls (see Fig. 6, *c*) due to their high heat conductivity. The materials to be connected are heated more rapidly, thus promoting heating of the reagent.

4. For a sample of finite dimensions, the regime of transformation in the form of thermal explosion after a long delay was discovered. The regimes of this kind are observed for very low heat capacity ($K_c << 1$) but high heat conductivity ($K_\lambda >> 1$) of the materials to be connected (see Fig. 6, *d*).

The dependence of the time of complete transformation τ_c on the problem parameters characterizing thermophysical properties is presented in Fig. 7. Time τ_c was determined using the condition $a(L,0) \geq 0.95$, which is correct for normal combustion regimes.

Parametric analysis showed that with the inert filler added ($z \neq 0$) time τ_c increases, combustion rate and temperature of combustion products decrease, which corresponds to the notions arising from the analysis of experimental data.

The discovered steady transformation regimes in such a system are similar to those known from theoretical consideration of the combustion of layer systems [10, 11]. For the actual technology of connecting materials, of interest are low-temperature regimes 2 and 3; judging from the results of theoretical investigation, these regimes can be realized is the connecting composition possesses the necessary thermophysical properties.

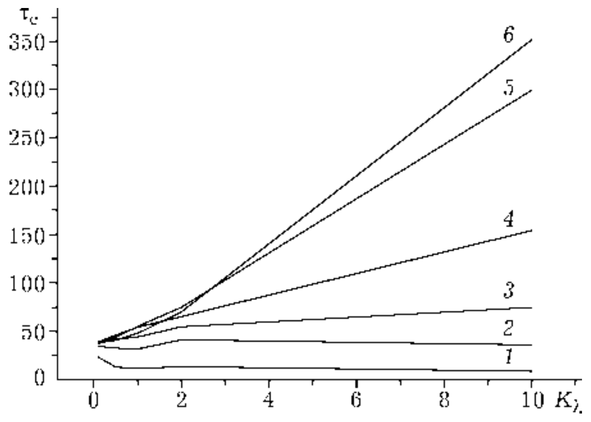

Fig. 7. Dependence of the total time of transformation on the parameters of the problem. $\theta_0 = 5$, $z = 0$, K_c values: 0.1 (*1*), 0.5 (*2*), 1 (*3*), 2 (*4*), 5 (*5*), 10 (*6*).

The numerical investigation of the problem with the external pulsed source showed [12] that self-sustaining transformation regimes are observed in such a system. The rate of front propagation depends on the relations between thermophysical properties of different materials and on the parameters that characterize the reaction. It is discovered that self-sustaining regimes are realized in a wider region of parameters θ_0, γ, β, than that deduced from the classical notions; the difference is connected with the effect of the materials to be connected.

CONCLUSIONS

Thus, different transformation regimes observed when connecting the materials with the help of solid-phase synthesis are described in the work. It is possible to control the synthesis temperature by choosing the connecting composition, fraction and type of inert filler in it. An additional possibility to decrease temperature arises with the use of inert filler with low melting point. It is evident that the materials connecting procedure involves the formation of a transition diffusion zone and adsorption compounds, the formation of a porous structure of the connecting joint, which has not been taken into account in the proposed models but should be of interest for further investigation.

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