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THE TEMPERATURE DEPENDENCE OF THE THERMAL CONSTANTS OF COMPOSITE POLYMER MATERIALS

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UDC 536.21

We developed a method for determination of the thermal-conductivity temperature dependence of organic and fiberglass plastics at temperatures up to 1000°C from thermocouple measurements by solving the inverse heat-conduction problem.

In the region of temperatures exceeding the minimum temperature of thermal decomposition of composite polymer materials, the macrostructure and the chemical composition of the material change and the thermal effects of the physicochemical transformations appear. These factors depend crucially on the rate and conditions of heating and as a result, the traditional methods of measuring the thermal constants [1] are largely inapplicable since these are based on the solution of the heat equation without taking into account the features mentioned above. The determination of the thermal constants in this temperature region is made possible using the temperature measurements in heating conditions close to those occurring in real situations by the method of the inverse heat-conduction problem (IHCP).

The mathematical model describing the heat propagation in the composite polymer materials at high temperatures should describe all relevant features of the process, and at the same time be sufficiently simple from the point of view of practical applications. These requirements are satisfied by the heat equation written in the form

$$F\frac{\partial t}{\partial \tau}=\frac{\partial}{\partial x}\left(\lambda\frac{\partial t}{\partial x}\right)+G\frac{\partial t}{\partial x};$$
$$F=(1-f)c_2\rho_2+\rho_0(1-K_{\text{mc}})Q\frac{\partial \chi}{\partial t};\quad G=c_1\int\limits_{x_{\text{bd}}}^{x}\frac{\partial \chi}{\partial \tau}dx. \tag{1}$$

The majority of the physical parameters appearing in the heat equation (1) (χ, K_{mc}, f, Q, ρ_0, ρ_2, c_1) can be determined by existing methods. To determine the specific heat c_2 and the thermal conductivity λ it is necessary to use IHCP.

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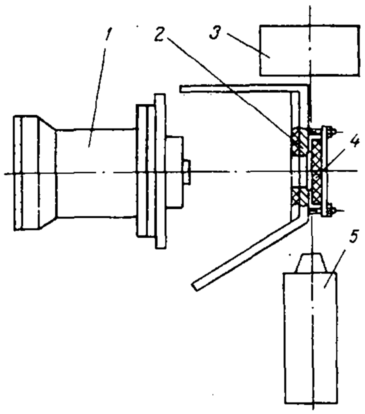

Fig. 1

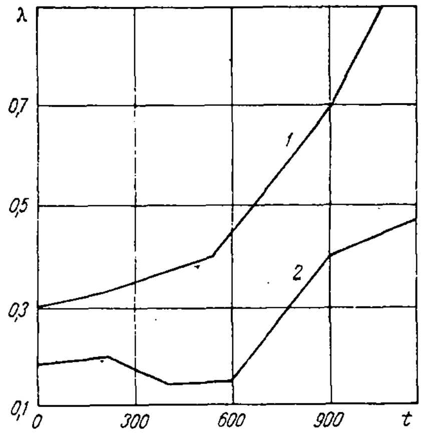

Fig. 2

Fig. 1. Layout of the experimental apparatus: 1) gas generator, 2) screen; 3) roentgen photorecording device; 4) sample; 5) roentgen IRA-2D installation.

Fig. 2. Thermal conductivity $\lambda(t)$ of glass-fiber plastic (1) and organic plastic (2) vs temperature t.

It follows from the analysis of the thermal mass transport in the composite polymer materials that the thermal conductivity and the specific heat must be considered as functions of temperature and the degree of decomposition: $\lambda(t, \chi)$ and $c_2(t, \chi)$.

In many situations of practical importance the rate of heating of the composite polymer materials is sufficiently fast and the degree of thermal decomposition depends only weakly on the rate of heating [2]. In these cases one may with sufficient accuracy assume that λ and c_2 depend only on temperature. This simplifies IHCP considerably.

The simplest solution of IHCP and the most useful in experimental application is the thermal scheme: an infinite plate from the material in question, with conditions of the first kind on the "hot" and "cold" surfaces. The boundary conditions of the heat equation of this scheme have the form

$$t(x,\ 0) = t_0(x);\ t(0,\ \tau) = u_0(\tau);\ t(\delta,\ \tau) = u_I(\tau). \tag{2}$$

The IHCP for determining the thermal constants is therefore formulated in this situation as follows: We obtain the time dependence of the temperature at the surface of the plate $u_0(\tau)$, and the temperatures at several points through the thickness $u_i(\tau)$. We need to determine those functions $\lambda(t)$ and $c_2(t)$ for which the temperature behavior in the interior of the plate, calculated from Eqs. (1) and (2), reproduces in the best way the experimentally measured values.

We shall use the direct method for the solution of this problem. The possible solution instability which can take place as a consequence of the mentioned incorrect formulation of IHCP problem is removed thanks to natural regulating properties of the algorithm and to the appropriate choice of the parameters [3]. The direct-solution methods of the IHCP are based on the scheme which gives, subject to the imposed constraints, the best approximation of temperatures which are calculated from a direct-heating problem and measured experimentally [4]. This approach makes it possible to obtain a stable solution, even taking into account the unavoidable errors in the input data and in the intermediate calculations. The choice of the best solution is obtained by the method [5] using the expression

$$\Phi = \sqrt{\sum_{i=1}^{I}\left[\frac{1}{\tau^*}\int (u_i(\tau) - t(x_i,\ \tau))^2\, d\tau\right]} = \Delta. \tag{3}$$

Approximating the functions $\lambda(t)$ and $c_2(t)$ by linear or cubic splines reduces the problem to finding the minimum of a function of K independent variables.

To find the minimum of the functional (3) we used a method of random search which uses the information about the minimum function obtained during the search and has a faster convergence and simple algorithm.

A preliminary investigation of the solution algorithm of the IHCP was carried out by analyzing the solutions of model problems. In this analysis we established the following result. For the same boundary conditions and the same number of variables the convergence of the process is worse and the length of the calculation longer in the case of simultaneous determination of $\lambda(t)$ and $c_2(t)$ than when determining only $\lambda(t)$.

For a simple function $\lambda(t)$ (quadratic parabola) the convergence of the process is the same when approximating the function by cubic or linear splines. In the case of a more complicated function (third-order polynomial) the approximation by cubic splines gives a somewhat better result. The length of the calculation, however, is longer when using the cubic splines. In addition we note that the convergence is improved when the boundary moments of the spline are included in the optimized parameters. By this the length of the calculation is increased still further.

For the methods above the optimum number of approximation intervals lies between 2 and 5, the smaller number referring to the approximation by cubic splines.

The experimental temperature fields in composite polymer materials necessary for the calculation of the thermal constants by the IHCP were worked out taking into account the formulation of the problem and the preliminary investigation of the solution algorithm.

1. The experiment has to reproduce with sufficient accuracy the thermal scheme of the "infinite plate with the conditions of the first kind on the hot and cold surfaces."

2. The heating conditions of the specimen at arbitrary heating regimes have to be appropriate for the thermal flow towards the surface of the specimen (convective flow is the best), for the rate of heating and for the pressure of the surrounding medium.

3. The thermal flow inside the specimen has to be uniform and perpendicular to the heated surface.

4. The filtration of the volatile thermal-decomposition products has to be carried out in the direction perpendicular to the heated surface and the outflow of the volatile products must not be impeded.

5. The measuring system must make it possible to measure the temperature as a function of time in the interior points of the specimen with accuracy not worse than the required accuracy for the determination of the temperature dependence of the thermal constants.

6. The separations between the temperature sensors must be measured with sufficient accuracy during the whole heating process.

The experimental investigations of the temperature fields in flat composite polymer material samples were conducted by heating them from one side by the stream of high-temperature burning products in the experimental set-up shown in Fig. 1. The experimental set-up is notable for the following reason: By using sufficiently thick samples, whose outer surface remains cold during the experiment and whose heated surface is fixed by means of a perforated plate from a heat-resistant metal, it is possible to minimize the movement of the thermocouples during the heating process. The x-ray survey of the specimen performed by means of the IRA-2D apparatus increases the accuracy and reliability of the temperature-field measurements in the specimen. We used an electrically heated stainless-steel plate as the source of heating.

For the specimen we used a typical composite polymer material widely used in large-scale constructions: fiberglass from modified epoxy resin reinforced by a fiberglass fabric and an organic plastic from an epoxy resin reinforced by a high-strength thread. The physical parameters appearing in equation (1) were determined in the following way. The mass concentration of the solid residue in the thermal decomposition products and the degree of completion of the process were determined experimentally by means of a thermogravimetric analysis at heating rates close to those used in the experimental determination of the temperature fields. The initial density of the material and the instantaneous density of the solid residue were found experimentally until the solid residue was reduced to fragments. The instantaneous porosity was found from the degree of thermal decomposition, density of the solid residues and from the relative deformation which were found experimentally. The specific heat of the volatile thermal-decomposition products and the specific heat of the solid residue were calculated from the state and specific heats of the components. The calorific effect of the thermal decomposition was calculated from the formation temperatures of the initial resin and of the thermal-decomposition products.

The thermal conductivity as a function of temperature, calculated by the methods described above, is shown in Fig. 2. The comparison of these thermal conductivities with those determined for the same materials by monotonic heating at temperatures below the thermal decomposition region showed a reasonable agreement.

NOTATION

τ, time; x, thickness coordinate; δ, sample thickness; x_{bd}, isothermal coordinate of the beginning of decomposition, $t(x, \tau)$, calculated temperature field in the specimen; $u(x, \tau)$, experimentally determined temperature field in the specimen; $t_0(x)$, initial temperature profile in the specimen; χ degree of thermal decomposition; ρ_0, initial density of the material; ρ_2, instantaneous density of the solid residue; $c_1(t)$, specific heat of the volatile thermal-decomposition products; $c_2(t)$ and $\lambda(t)$, specific heat and thermal conductivity of the material; K_{mc}, mass concentration of the solid residue in the thermal decomposition products; Q, calorific effect of thermal decomposition at the temperature of decomposition; Δ, root-mean-square error of the temperature measurements; f, porosity of the coke; τ^*, duration of the thermal treatment. Indices: $k = 1, 2, \ldots, K$ is number of the approximation interval, and $i = 0, 1, 2, \ldots, I$ is the number of the point through the thickness.

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A MODIFIED METHOD OF DETERMINING THE THERMAL CONSTANTS OF NONMETALLIC MATERIALS

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We describe a method and the experimental set-up for a comprehensive determination of thermal constants. The method is applied to reinforced composite materials.

In the current technology there is some interest in composite materials reinforced by metallic inclusions in the form of a foil or wire. In some cases, a contact zone is formed between the composite material and the inclusion which is different from the reinforcing material.

To find the thermal conductivity of such a system it is necessary to know the thermal constants of the initial materials and of the contact zone. The properties of the initial materials are often known fairly reliably; to determine the properties of the contact zone by the usual methods, however, is usually difficult because the thickness of the zone is small and its separation is practically impossible.

Physical Basis of the Method

In the present combined method of determination of the thermal constants of nonmetallic materials we used as a source of heat and as the temperature sensor a small strip of metallic foil or a small-diameter wire from aluminum, copper, or silver. For the above-mentioned composite materials this element can be the metal inclusion.

The method is based on the combination of the method of regular thermal regime of the third kind [1] and of the cylindrical-probe method [2]. This makes it possible to determine in one experiment the coefficients of thermal activity, thermal diffusivity, and thermal conductivity. The experiment is internally consistent since from any two of the coefficients it is possible to find the third.

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