Determination of models constants for thermomechanical properties of composites based on experimental data analysis

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Annotation. This article presents an algorithm for finding the constants in the model of changing the thermoelastic properties of polymer composites. The calculation of the constants is based on the processing of the experimentally obtained data. Optimization methods are applied to minimize the discrepancy between the experimental data and the analytical dependence on the constants that need to be found. Also, a method was proposed for determining the constants in the model of density change during destruction. The paper analyzes the effect of high temperatures on the change in density and elastic modulus of composite materials. On the basis of this, an algorithm was constructed for calculating the change in elastic properties up to temperatures of thermal destruction and during thermal destruction. The error between theoretical modeling and experimental data did not exceed 5%.

1. Introduction
Polymer composite materials have found wide application in rocket-space and aviation technology, including for the creation of structures exposed to extremely high temperatures - up to 1000 °C and above. When exposed to high temperatures in polymer composites, internal physicochemical transformations occur, which are accompanied by the process of weight loss (ablation). In this case, a large number of pores filled with gaseous products of thermal decomposition of polymers are formed in the matrix, and sometimes in the fibers. Calculating the characteristics of composites under conditions of thermal decomposition is a complex problem. In a large series of works [1-6], models were proposed that describe the change in the mechanical and thermophysical properties of polymer composites at high temperatures. These models allow one to take into account the complex dependence of the properties of polymer composites on the heating history, i.e. not only from the maximum current temperature, but also from the preliminary heating-cooling process, cyclic heating, etc. One of the problems when using these models is the complexity of the problem of determining the constants of the models based on the processing of experimental data. In this work, an algorithm was developed that allows one to determine the constants of the models describing the change in the density and elastic modulus of polymer matrices during high-temperature heating in an inert medium.

Model of changes in the phase composition of polymer matrices at high temperatures.

According to the model [1-6], isotropic polymeric materials (for example, epoxy matrices, phenolic matrices, etc.) at high temperatures consist of three phases:
- solid polymer phase ($i = b$), its volume concentration - $\varphi_b$,
- solid pyrolytic phase ($i = p$), its volume concentration - $\varphi_p$,
- gas phase ($g$-phase) inside the pores, its volume concentration - $\varphi_g$.

Phase concentrations are related by the relationship

$$\varphi_b^0 + \varphi_b + \varphi_p + \varphi_g = 1,$$

where $\varphi_b^0$ is the initial concentration of fibers in the composite. The fibers will be assumed to be thermally stable in the considered temperature range. Models, in which fibers are also considered with a variable phase composition, are proposed in [6].

At the initial moment of time, the following relations are occur
\[ \varphi_f^0 + \varphi_b(0) + \varphi_g(0) = 1 \quad \varphi_p(0) = 0 \]  

where are the initial concentrations of the phases

\[ \varphi_b(0) = \varphi_b^0, \quad \varphi_g(0) = \varphi_g^0. \]  

The change in the polymer phase of the matrix is described by a multistage kinetic model

\[ \varphi_b = \sum_{i=1}^{n} \varphi_{bi}, \]  

\[ \rho_b \frac{\partial \varphi_{bi}}{\partial t} = -J_i, \]  

where \( \varphi_{bi} \) are the various components of the polymer phase of the matrix, into which the polymer phase gradually separates upon heating (for example, vapors of a solvent and hardener, monomers, etc.), \( \rho_b \) is the density of the solid polymer phase, and \( J_i \) is the mass rate of formation of each component of the polymer phase, for which it is assumed the Arrhenius's law

\[ J_i = J_{0i} \varphi_{bi} \exp \left( -\frac{E_{ai}}{R\theta} \right). \]  

Here \( E_{ai} \) is the activation energy of the process of removing each component of the polymer phase, \( R \) is the gas constant, and \( \theta \) is the temperature.

Equations (5) taking into account (6) have the following explicit solutions

\[ \varphi_{bi} = \varphi_{bi}^0 g_i(t, J_{0i}, E_{ai}), \quad G_i(t, J_{0i}, E_{ai}) = \exp(-J_{0i} \int_0^t \exp(-\frac{E_{ai}}{R\theta(\tau)}) d\tau), \]  

where \( J_{0i} = \frac{J_{0i}}{\rho_{bi}}, \quad E_{ai} = \frac{E_{ai}}{R} \) and \( \varphi_{bi}^0 \) are the initial concentrations of the constituents of the polymer phase, satisfying the relation

\[ \varphi_b^0 = \sum_{i=1}^{n} \varphi_{bi}^0. \]  

From (4) and (5) we obtain the equation for the change in the mass of the polymer phase of the matrix

\[ \rho_b \frac{\partial \varphi_b}{\partial t} = -J, \quad J = \sum_{i=1}^{n} J_i. \]  

The change in the volume concentration of the solid pyrolytic phase \( \varphi_p \) is determined by the equation

\[ \rho_p \frac{\partial \varphi_p}{\partial t} = J(1 - \Gamma), \]  

where \( \rho_p \) is the density of the solid pyrolytic phase, \( \Gamma \) is the gasification coefficient.

From (9) and (10) we find an analytical expression for the concentration \( \varphi_p \) in terms of \( \varphi_b \).
\[ \varphi_p = \left( \varphi_b^0 - \varphi_f \right) \frac{\rho_b}{\rho_p} (1 - \Gamma). \]  

(11)

The change in the concentration of the gas phase (porosity) \( \varphi_g \) is found from equation (1).

**Density change of the polymer matrix during high-temperature heating**

The change of the polymer composite density \( \rho \) is described by the following equation

\[ \rho = \rho_f \varphi_f^0 + \rho_b \varphi_b + \rho_p \varphi_p, \]  

(12)

where \( \rho_f \) is the density of the filler. Substituting (11) into (12), we obtain

\[ \rho = \rho^0 - \rho_b \left( \varphi_b^0 - \varphi_b \right) \Gamma, \]  

(13)

where \( \rho \) is the value of the initial density of the composite

\[ \rho^0 = \rho_f \varphi_f^0 + \rho_b \varphi_b^0. \]  

(14)

The system of equations (4) - (6), (13) makes it possible to calculate the change of the polymer matrix density during heating according to an arbitrary law of temperature change \( \theta(t) \). Substituting expressions (4) and (7) into (13), we obtain an explicit expression for the change in the density of the composite upon heating

\[ \frac{\rho}{\rho^0} = 1 - \frac{\rho_b}{\rho^0} \sum_{i=0}^{\infty} \varphi_b^0 \left( 1 - G_i(t,J_0,J_i) \right) \Gamma. \]  

(15)

The limiting value of the composite density, after the completion of the process of thermal destruction of the matrix, when \( \varphi_b = 0 \), has the following value

\[ \rho = \rho^\infty, \quad \rho^\infty = \rho^0 - \rho_b \varphi_b^0 \Gamma. \]  

(16)

**Description of the method for finding constants.** Let there be an experimental curve of the density change (derivatogram) \( \frac{\rho(\theta)}{\rho^0}(\theta) \) of the composite when heated at a constant rate: \( \theta = \theta_0 + \dot{\theta}_0 t \), where \( \dot{\theta}_0 = \text{const} \). We assume that the following values are known: \( \varphi_g^0, \varphi_f^0, \) and \( \rho_f \). The density of the solid polymer phase \( \rho_b \) is determined from the relation (14)

\[ \rho_b = \frac{\rho^0 - \rho_f \varphi_f^0}{1 - \varphi_f^0 - \varphi_g^0}. \]  

(17)

The gasification coefficient \( \Gamma \) can be expressed using (14) and (15)

\[ \Gamma = \frac{\rho^0 - \rho^\infty}{\rho_b \varphi_b^0}. \]  

(18)
To find the preexponential factors $J_{0i}$ and activation energies $E_{Ai}$, we use the condition for the best approximation of the experimental curve $\frac{\rho_j^{(c)}}{\rho_0^{(c)}}(\theta_j)$ with the help of (15) by the least squares method

$$\Delta (J_{0i}, E_{Ai}) \rightarrow \min,$$

(19)

where $\Delta (J_{0i}, E_{Ai})$ is the functional of the approximation error

$$\Delta (J_{0i}, E_{Ai}) = \sum_{j=1}^{K} \left( \frac{\rho_j^{(c)}}{\rho_0^{(c)}}(\theta_j) - \frac{\rho_0^{(c)}}{\rho_0^{(c)}}(t_j, J_{0i}, E_{Ai}) \right)^2,$$

(20)

and $J_{0i}, E_{Ai}$ are the control parameters of the approximation, $\theta_j$ are the temperature values, that are taken into account when minimizing the functional of the approximation error (19), $t_j = \frac{\theta_j - \theta_0}{\theta_0}$ are the times corresponding to the temperature values $\theta_j$.

To solve the minimization problem (19), (20) with respect to parameters $J_{0i}, E_{Ai}$, the coordinate descent method is used.

**Results of numerical simulation of density changes in heating.**

The proposed method for finding the constants of the density change models in heating was applied to experimental data for epoxy [7,8] and epoxy phenolic matrices [3] without filler ($\phi_0 = 0$). The initial porosity of the epoxy matrix $\phi_0 = 0.15$, epoxy phenolic - $\phi_0 = 0.06$. The heating rate was equal to $\dot{\theta} = 0.1 K$. Using the algorithm developed above for the epoxy matrix, the following values were obtained:

$$\rho_b = 1170, 21 \text{ kg} / \text{m}^3, \Gamma = 0, 25, N = 1, J_{0i} = 3, 5 \cdot 10^5 \text{ kg} / (\text{m}^3 \text{s}), \quad E_{Ai} = 7, 3 \cdot 10^3 K; \quad (21)$$

for epoxy phenolic matrix:

$$\rho_b = 1294, 12 \text{ kg} / \text{m}^3, \Gamma = 0, 66, N = 1, J_{0i} = 3, 6 \cdot 10^4 \text{ kg} / (\text{m}^3 \text{s}), \quad E_{Ai} = 8, 12 \cdot 10^3 K. \quad (22)$$
Figure 1. Curves of changes in the relative density of the epoxy matrix (a) and epoxy phenolic (b) when heated in an inert atmosphere at a heating rate $\dot{\theta} = 0.1K$. The dots denote the experimental data [7], the solid lines denote the data obtained by calculation using formula (15).

Figure 1 show the experimental density dependences $\frac{\rho}{\rho^0}(\theta)$ for epoxy and epoxy-phenolic matrices, as well as their approximation curves obtained on the basis of equation (15) with constants (21) and (22), respectively. The approximation accuracy is quite satisfactory for both types of matrices.

... Figure 2 shows thermogravimetric curves of composite based on an epoxy matrix and carbon fibers, obtained by calculation using constants (21), as well as the corresponding experimental curves at different heating rates. The approximation accuracy is quite satisfactory in the considered range of velocities.

Although, the experimental dependence of the function $\frac{\rho}{\rho^0}(\theta)$ on the heating rate manifests itself to a lesser extent than it follows from equation (15).
Figure 2. Curves of changes in the relative density of carbon/epoxy composite when heated in an inert atmosphere at different heating rates. Dots denote experimental data [8], solid lines - data obtained by calculation using formula (15).

Model of the elastic properties change for polymer matrices at high temperatures.

The constitutive relations of elasticity for polymer matrices at high temperatures relate stress \( \sigma_{ij}^{(m)} \) and small strain tensors \( \varepsilon_{ijkl}^{(m)} \)

\[
\sigma_{ij}^{(m)} = C_{ijkl}^{(m)} \varepsilon_{ijkl}^{(m)} - f_{ijkl}^{(m)} p \delta_{ij},
\]

\[
\varepsilon_{ijkl}^{(m)} = \Pi_{ijkl}^{(m)} (\sigma_{ij}^{(m)} + f_{ijkl}^{(m)} p \delta_{ij}^{(0)}) + \varepsilon_{ijkl}^{(0)}
\]

where \( C_{ijkl}^{(m)} \) is the tensor of elastic moduli, \( \Pi_{ijkl}^{(m)} \) is the tensor of elastic compliance (inverse to \( C_{ijkl}^{(m)} \)), \( \varepsilon_{ijkl}^{(0)} \) - is the tensor of thermal deformations, \( p \) - is the pore pressure of gases arising in the material at high temperatures, \( f_{ijkl}^{(m)} \) - is the coefficient of interfacial interaction between the gas and the pore walls of the material. Index (m) - means "matrix". Three tensors \( C_{ijkl}^{(m)} \), \( \Pi_{ijkl}^{(m)} \) and \( \varepsilon_{ijkl}^{(0)} \) characterize the elastic properties of matrices at high temperatures, for isotropic materials (matrices and fibers) they have the form

\[
\Pi_{ijkl}^{(m)} = \frac{1}{2E_m} \left( \delta_{ik} \delta_{jl} + \delta_{il} \delta_{jk} \right) - \frac{\nu_m}{E_m} \delta_{ij} \delta_{kl}, \quad \varepsilon_{ijkl}^{(0)} = \varepsilon_{ijkl}^{0}
\]

Three functions characterize the elastic properties of matrices at high temperatures

\[
E_m, \varepsilon_m^0, f^{(m)}, \quad \nu_m = \text{const}
\]
where $E_m$ is the modulus of elasticity, $\varepsilon_m^0$ is the thermal deformation, $f^{(m)}$ is the coefficient of interfacial interaction, $\nu_m = \text{const}$ is the Poisson's ratio, and $\delta_{ij}$ is the Kronecker symbol.

These functions (25) depend on the phase concentrations and the history of temperature changes. According to the model proposed in [6], these dependencies have the form:

$$E_m = E_b^0 \bar{a}_i$$  \hspace{2cm} (26)

$$\bar{a}_i = \left( a^{(0)} \left( S_p^2 - S_b^2 \right) + m_p \left( 1 - S_p^2 \right) + \frac{S_p - S_b}{a^{(0)} S_p^2 + m_p \left( 1 - S_p^2 \right)} \right)$$  \hspace{2cm} (27)

$$f_m = \frac{\phi_s}{E_b^0} \left( \frac{1}{a^{(0)} \left( S_p^2 - S_b^2 \right) + m_p \left( 1 - S_p^2 \right) - \frac{1}{\bar{a}_i}} \right)$$  \hspace{2cm} (28)

$$\varepsilon_m = \alpha_p \phi_s \left( \theta - \theta_0 \right) + \alpha_p \int_0^t \left[ \theta(t) - \theta(\tau) \right] \phi_p d\tau - \beta_p \phi_p$$  \hspace{2cm} (29)

where

$$S_b = \left( \frac{\phi_s}{1 - \phi_f} \right)^{1/3} ; \quad S_p = \left( 1 - \frac{\phi_p}{1 - \phi_f} \right)^{1/3},$$  \hspace{2cm} (30)

$$a^{(0)} = (1 - a_s) \exp(-a(\Delta \hat{\theta})) + a_s,$$  \hspace{2cm} (31)

$$\Delta \hat{\theta} = \Delta \theta + P \int_0^t \exp(-Q(t - \tau)) \Delta \theta(\tau) d\tau, \quad \Delta \theta = \theta / \theta_0 - 1,$$  \hspace{2cm} (32)

and $P = 1.56 \cdot 10^{-4} \text{s}^{-1}, Q = 2.5 \cdot 10^{-4} \text{s}^{-1}$ are universal constants determined experimentally [6].

This model contains the following constants

$$E_b^0, \ m_p = \frac{E_p}{E_b^0}, \ a, \ a_s, \ s,$$  \hspace{2cm} (33)

where $E_b^0$ is the modulus of elasticity of the polymer phase, $E_p$ is the modulus of elasticity of the pyrolytic phase, $a$ and $s$ are the factor and index of the degree of change in the elastic properties of the polymer phase, $a_s$ is the limiting value of the coefficient of change in the properties of the polymer phase at $\Delta \theta \to \infty$. The model also includes 3 thermal deformation constants

$$\alpha_b, \ \alpha_p, \ \beta_p.$$  \hspace{2cm} (34)

Consider the temperature range before the onset of thermal decomposition: $\theta_0 \leq \theta < \theta_t$, where $\theta_t$ is the conditional temperature of the onset of thermal decomposition, at which the relative density change
\( \Delta \rho(\theta) = 1 - \frac{\rho_0(\theta)}{\rho} \) at a certain heating rate \( \dot{\theta} \) is equal to a given value, for example, 5\% of the maximum values

\[
\frac{\Delta \rho(\Delta \theta_f)}{\Delta \rho(\Delta \theta_{\text{max}})} = 0.05 .
\]  

(35)

When the polymer matrix is heated below the temperature of thermal decomposition at \( \theta_0 \leq \theta < \theta_f \) the concentration of the solid polymer phase \( \varphi_p \) and the porosity \( \varphi_g \) can be considered unchanged, therefore, in this temperature range

\[
\varphi_p = 0, \quad \varphi_g = \varphi_g^0, \quad \varphi_b = 1 - \varphi_b^0 = \varphi_g^0 .
\]  

(36)

Then the quantities characterizing the linear dimensions of the phases \( S_p \) and \( S_b \) are also constants.

\[
S_b = S_b^0 = \left( \frac{\varphi_g^0}{1 - \varphi_f^0} \right)^{\frac{1}{3}} , \quad S_p = S_p^0 = 1.
\]  

(37)

Function \( \tilde{a}_1 \) (27), which characterizes the change in elastic properties upon heating, has the form

\[
\tilde{a}_1 = \left( \frac{S_b^0}{a^{(0)}(1 - S_b^{02})} + \frac{1 - S_b^{02}}{a^{(0)}} \right)^{-1} = a^{(0)}\Phi\left( S_b^0 \right) ,
\]  

(38)

where

\[
\Phi\left( S_b^0 \right) = \frac{1 - S_b^{02}}{1 - S_b^{02} + S_b^{03}} , \quad 0 < \Phi\left( S_b^0 \right) \leq 1 .
\]  

(39)

This function \( \Phi\left( S_b^0 \right) \) together with \( S_b^0 \) - describes the effect of the initial porosity \( \varphi_g^0 \) on the elastic modulus \( E_m \) of the polymer.

**Algorithm for calculating the constants of the model for matrix elastic properties before temperatures of thermal decomposition**

Consider the temperature range before the onset of thermal decomposition: \( \theta_0 \leq \theta < \theta_f \). Then the elastic modulus of the polymer phase \( E_{b}^0 \) is found using relations (26), (38) and the experimental values \( E_m^{(c)}(\theta_0) \) of the elastic modulus at temperature \( \theta_0 \)

\[
E_{b}^0 = \frac{E_m^{(c)}(\theta_0)}{\Phi\left( S_b^0 \right)} .
\]  

(40)

For a given heating rate \( \dot{\theta}_0 \), we calculate the function \( \Delta \dot{\theta} = R\left( \Delta \theta, \dot{\theta}_0 \right) \), where

\[
\Delta \dot{\theta} = R\left( \Delta \theta, \dot{\theta}_0 \right) \equiv \Delta \dot{\theta}\left[ 1 + \frac{P}{Q}\left( 1 - \exp\left(-\frac{\Delta \theta}{\dot{\theta}_0} \right) \right) \right]
\]  

... (41)
To find the constants $a$, $a_\infty$, $s$ in function (31), we use the procedure for minimizing the deviation of the experimental elastic modulus $E_m^{(c)}(\Delta \theta_i)$ from the theoretical $E_m^{(t)}(\Delta \theta, a, a_\infty, s)$

$$\Delta(a, a_\infty, s) = \frac{1}{N} \sum_{i=1}^{N} \left( 1 - \frac{E_m^{(t)}(\Delta \theta_i, a, a_\infty, s)}{E_m^{(c)}(\Delta \theta_i)} \right)^2 \rightarrow \text{min}, \quad (42)$$

where $E_m^{(t)}(\Delta \theta, a, a_\infty, s)$ is calculated by (26), (31) and (38)

$$E_m^{(t)}(\Delta \theta, a, a_\infty, s) = E_b^0 \Phi(S_b^0)((1-a_\infty)\exp(-a(\Delta \theta, \bar{\theta}_b)^\gamma) + a_\infty) \quad (43)$$

and $\Delta \theta_i$ are a set of experimental values of temperatures for which there are experimental values $E_m^{(c)}(\Delta \theta_i)$. For solving the problem (42) we used the coordinate descent method [2]

Algorithm for calculating the constants of the model for matrix elastic properties under temperatures of thermal decomposition

Let the experimental dependence of the elastic modulus $E_m^{(c)}(\Delta \theta)$ on temperature $\Delta \theta$ be known at $\theta > \theta_F$. Then from formula (26) we find the value of the coefficient corresponding to the temperature $\Delta \theta_{\text{max}}$ at which the process of thermal destruction of the polymer matrix is completed (i.e. when $\varphi_b = 0$ and $\varphi = \varphi_b^0 \frac{D_b}{\rho_p} (1-\Gamma)$)

$$\bar{a}_i(\Delta \theta_{\text{max}}) = \frac{E_m^{(c)}(\Delta \theta_{\text{max}})}{E_b^0}. \quad (44)$$

Then, for temperature $\Delta \theta_{\text{max}}$, we obtain an equation for calculating a constant $m_p = \frac{E_p}{E_b^0}$, which is equal to the ratio of the pyrolytic phase elastic modulus $E_p$ to the polymer phase elastic modulus $E_b^0$

$$S_b \frac{a^{(0)}(S_p^2 - S_b^2) + m_p(1-S_p^2)}{a^{(0)}S_p^2 + m_p(1-S_p^2)} + \frac{S_p - S_b}{1-S_p} + \frac{1-S_p}{m_p} = \frac{E_b^0}{E_m^{(c)}(\Delta \theta_{\text{max}})} \quad (45)$$

Equation (45) is cubic with respect to $m_p$, we find its solution on a segment $0 \leq m_p \leq 1$ using the method of dividing a segment in half [3].

Simulation results and comparison with experiments.

Let us consider the experimental data on the modulus of elasticity $E_m^{(i)}$ changing for epoxy [5] and epoxy phenolic matrices. For the epoxy matrix, using the algorithm developed above, the following values of the constants were obtained:

$a = 0.6722$, $m_p = 10^{-6}$

and for the epoxy phenolic matrix

$a = 0.74$, $m_p = 0.148$

where $\bar{a} = a\theta_b$ is the dimensionless value of the parameter.

Using the found constants, we calculated the dependence of the elastic modulus of the polymer matrix on temperature $E_m^{(c)}(\Delta \theta)$ using formula (26). The calculation results and their comparison with experiments are presented in Figure 3. For an epoxy matrix, the coincidence of the calculated and experimental graphs
$E_m(\Delta \theta)$ is quite good before the onset of thermal destruction. At $\theta > \theta_f$ (where $\theta_f \approx 590K$), when the process of thermal destruction begins, the experimental value of the elastic modulus decreases significantly more significantly than predicted.

![Graph](image1.png)

![Graph](image2.png)

**Figure 3.** Comparative experimental $E_m^{(\nu)}(\theta)$ (2) and calculated $E_m(\theta)$ (1) graphs of the dependence of the elastic modulus on temperature for an epoxy matrix (a) and epoxy-phenolic matrix (b) in an inert medium at a heating rate $\dot{\theta} = 0.1 K/c$.

**Conclusions**

The development of a model of elastic properties of polymer composites under high-temperature heating in the area of thermal destruction of the polymer matrix is proposed. The model makes it possible to take into account the multistage nature of the process of thermal destruction of the matrix. An algorithm for determining the constants of the model based on the processing of experimental data is proposed. Comparison of the calculated and experimental curves of thermogravimetric analysis at different heating rates, as well as the dependences of the change in the elastic modulus upon heating for epoxy and epoxy-phenolic matrices is carried out. It is shown that the agreement between the experimental and calculated dependences is quite good.

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