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Modeling of the process of thermal polymerization of composite materials taking into account the features of the equipment

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The problem of obtaining a mathematical model of the process of thermal molding of structures from polymer composite materials in an autoclave, taking into account its technical capabilities and types of molding equipment, is considered in the article. The main task of the work was to obtain the dependence, showing the change of temperature of the internal structure of the polymerized package depending on the working environment of the autoclave and the initial conditions of the process, depending on the type of heating device and type of molding equipment. The initial realization of the problem was based on the use of Fourier equations for a triple package (auxiliary equipment, product, main molding equipment). The proposed approach used values of the initial temperature jump depending on the mass of auxiliary equipment (tooling) and the type of composite material being formed, which were obtained experimentally. The study involved real processes of manufacturing PCM products in a «Scholz» autoclave. Since the practical part of the study was carried out for a device equipped with a powerful fan unit, an assumption was made about instantaneous convection of heated air throughout the working volume of the autoclave. The second stage of the work involved solving the problem of the unsteady heat conduction problem by the method of elementary balances approximated to the classical finite element method. For this task, the method was transformed, which allowed for taking into account the heat exchange of the environment with the body. In this way, it is possible to obtain dependencies for corner points, points lying on the edges and faces of the formed package. This allows us to determine not only the amount of heat entering the package from the most developed surfaces, as is done in the analytical solution, but also from the sides of the package. In this model, the thermal conductivity coefficient and specific heat capacity are taken as linear functions of temperature. When developing the calculation model, the change in the physical parameters of the package over time during heating was taken into account, which also increased the probability of finding a more accurate solution. The main difference from other software products used to solve similar problems is the ability to solve the inverse problem, which consists in determining the required temperature of the environment for a given mode.The obtained dependencies for the total heat flows allow us to find the total heat value used for polymerization processes, the temperature of the environment in the autoclave and the corresponding value of the temperature of the middle layer of the package.

Keywords: Polymer Composite Material; Thermal process modeling; Energy Efficiency; autoclave; molding equipment; working medium.

Introduction

The processes of curing products made of polymer composite materials (PCM), or, in other words, the processes of molding, in real production conditions most often consist of giving the product an irreversible shape and properties corresponding to operational requirements on the mold. Such processes are carried out by thermal heating of the PCM package in thermal furnaces or, if it is necessary to use excess pressure in polymerization modes, in autoclaves.

The technology using an autoclave and the correct mode of its operation allows obtaining the highest quality products that have the densest structure of the formed package with minimal gas inclusions which is more difficult to provide only in conditions of vacuum forming, typical for forming in thermal furnaces. That is why the study was carried out for this type of equipment, taking into account the configuration and material of the mold, as well as the technological capabilities of a particular model of autoclave. The possibility of automatic temperature and pressure monitoring without dielectric analysis, i.e. without the possibility of binder viscosity control, was taken into account.

The main technological problem in the production of products from polymer composite materials is the adaptation of the polymerization mode of the binder, recommended by its manufacturer and obtained in laboratory conditions for a sample of small dimensions and uniform thickness, to the actual configuration of the product, the type of mold used for its shaping and the capabilities of the available equipment.

The control means available on the autoclave do not allow to control the state of the material in the entire volume of large PCM structures, which can lead to violation of the specified polymerization process in the inner layers of the package. This can lead to uneven curing, formation of dense layers preventing the escape of gaseous components of the binder and even degradation of the polymer [1, 2, 3]. As a result, there is a need to search for methods to determine the correct temperature timing. The traditional approach in most cases implied the use of trial and error method both in the design of technological equipment and in the adaptation of the polymerization cycle, which led to a significant increase in the cost of the final product.

At the present moment there are three main approaches to solving this problem. The first one is modeling of polymerization processes using mathematical apparatus; the second one is implementation by software modeling tools; the third one is using trained artificial neural networks.

The first approach is based on the development of mathematical models of polymerization, which can be further used to optimize the technological mode based on various algorithms (deterministic, stochastic or hybrid) using inherent methods [4]. Also, the approach provides an opportunity to obtain a numerical tool for heat transfer analysis and curing modeling [5].

The basis of the second approach is the numerous variants of application of the finite element method [6, 7]. Moreover, this approach is the most applicable today due to its universality. For example, in the work [8], the possibility of its integration with internal optimization algorithms using MATLAB and the multiphysical simulator COMSOL is shown. Also, in this group can be included the development of numerical models for studying temperatures in alternative methods of providing heating, such as the use of microwave curing [9, 10]. The undeniable advantage of the method is the possibility of modeling the curing process taking into account the forming matrix [11], which has a significant influence on the temperature field, and as a consequence - on the distribution of the degree of PCM curing, and also provides an opportunity to evaluate the interaction between the mold and the product [12].

When training recurrent artificial neural networks [1], numerical results are used that can also be obtained based on the three-dimensional finite volume method. This method is applicable to accelerate the repeated calls of the model during the optimization of the curing cycle in order to improve the properties and obtain a homogeneous part of a thick-walled composite product.

Due to the specifics of this work, some aspects of which are planned to be used in the continuation of the work [13] to estimate the amount of heat when assessing the activation energy level of structural transformations of PCM, the most acceptable modeling option is the method of elementary balances based on the second approach, approximated to the finite element method. The use of experimental data to determine the initial temperature jump in the autoclave medium when modeling the heating process is a classical example of the inverse problem [14].

Materials and methods

Polymerization of PCM products is inherently a non-stationary thermal process. It is caused by the change of heat content of the body and is associated with the phenomena of heating (raising the temperature of the formed package to the holding temperature and further to the polymerization temperature) or cooling (reducing the temperature of the finished product to the required level). These phenomena are realized as a result of heat exchange processes between the autoclave working medium and the filling loaded into the autoclave. When creating the mathematical model, we will consider only the directly molded package together with the main (forming) and auxiliary equipment as filling of the autoclave working volume. The rest of the filling (thermocouples and their connection systems, vacuum system hoses, etc.) due to their relatively small mass will not be taken into account.

The complexity of solving the thermal problem under the given conditions lies in the multidimensionality of the processes:

1. temperature difference of the package along x, y, i 2 axes (see Fig. 1);
2. change of the package temperature in time;
3. change of the autoclave working environment (medium) temperature in time.

Thus, it is necessary to obtain a dependence, which will demonstrate the change in temperature of the autoclave working medium in time, based on the known change in time of the temperature of the formed package (given technological mode).

Since the practical part of the study was carried out using an autoclave equipped with a powerful fan unit, the assumption of instantaneous convection of heated air through the working volume of the autoclave was accepted. thus, the temperature along the entire length of the autoclave is assumed to be the same at the considered moment of time and further we will talk about the average temperature of the working medium twe.

The theoretical model is based on the differential equation of heat conduction for solids (Fourier equation), which has the following form:

$$\frac{dt}{d\tau} = a\left(\frac{dt^2}{dx^2} + \frac{dt^2}{dy^2} + \frac{dt^2}{dz^2}\right), \tag{1}$$

where τ– the time, t – the temperature.

The problem of unsteady heat conduction will be solved by the method of elementary balances taking into account the temperature dependence of the thermal conductivity coefficient and specific heat capacity.

The study involved real processes of manufacturing PCM products in the autoclave of the “Scholz” company, the working chamber of which is a high-pressure vessel with a diameter of 3500 mm and length of 8000 mm. Air heating in the autoclave is carried out by three groups of heaters in the end of the autoclave. The limits of temperature rise rate for formed packages are 0.3 - 2 degrees per minute.

During the practical selection of the initial temperature jump value when loading molds with products into the autoclave, the type of equipment used was taken into account. Frame-type molds have a developed surface, thin walls, and good heating when washed with hot air. They can be installed directly on the autoclave floor or table (if the number of formed packages is more than one). Cast-type molds or molds milled from monolithic slabs heat up much more slowly due to the small area of hot air blowing per unit weight. To increase the washing area, they require installation on special stands. Accordingly, loading several parts into the autoclave on different molds was carried out based on the same polymerization mode, filler and binder materials, mass of parts of the same order, and a similar mold design with similar dimensions made of the same material.

Different types of products made of fiberglass were also considered: honeycomb panels with skin using film adhesive and epoxy binder, monolithic panels and panels with tubular filler made of fiberglass based on epoxy binder. The monolithic design of the mold was considered for the case of manufacturing a monolithic carbon fiber product on epoxy binder.

The realization of the mathematical model was carried out for a monolithic fiberglass structure using binder 5-211B (epoxy resin ED-20 (100 mass parts), phenol-formaldehyde resin SF-341A (70 mass parts), alcohol-acetone mixture (1:1)).

Results and discussion

The package that was considered in modeling the thermal polymerization (heating) process consists of three parts: the main forming mold, composite material and auxiliary tooling. Accordingly, the heat conduction problem solved by equation (1) was considered for each element of the package separately as a system of three equations:

$$\frac{dt_1}{d\tau} = a_1\left(\frac{d{t_1}^2}{dx^2} + \frac{d{t_1}^2}{dy^2} + \frac{d{t_1}^2}{dz^2}\right) = a_1\nabla t_1,$$

$$\frac{dt_2}{d\tau} = a_2\nabla t_2,$$

$$\frac{dt_3}{d\tau} = a_3\nabla t_3,$$

where a_1, a_2, a_3 - temperature diffusivity coefficients for auxiliary tooling, product, mold respectively.

Analytical solution of these equations requires setting the initial temperature distribution in the body and the action of the autoclave medium on the package surface. The temperature distribution is set according to the package surfaces (see Fig. 1):

$$t_1(x,y,z,\tau)_{S_2} = t_2(x,y,z,\tau)_{S_2},$$

$$t_2(x,y,z,\tau)_{S_3} = t_3(x,y,z,\tau)_{S_3},$$

$$t_1(x,y,z,0) = t_2(x,y,z,0) = t_3(x,y,z,0) = \varphi$$

Impact of the environment on surfaces:

$$\frac{dt_1}{dn_z} + h_{01}t_1(x,y,z,\tau)_{S_1} = h_{01}t_{we},$$

$$\frac{dt_1}{dn_F} + h_{01}t_1(x,y,z,\tau)_F = h_{01}t_{we},$$

$$\lambda_1\frac{dt_1}{dn_{z_{S_2}}} = \lambda_2\frac{dt_2}{dn_{z_{S_2}}},$$

$$\frac{dt_2}{dn_F} + h_{02}t_2(x,y,z,\tau)_F = h_{02}t_{we},$$

$$\lambda_2 \frac{dt_2}{dn_{z_{S_3}}} = \lambda_3 \frac{dt_3}{dn_{z_{S_3}}},$$

$$\frac{dt_3}{dn_z} + h_{03} t_3(x, y, z, \tau)_{S_4} = h_{03} t_{we},$$

$$\frac{dt_3}{dn_F} + h_{03} t_3(x, y, z, \tau)_F = h_{03} t_{we},$$

where F – lateral surface of the package; S_1, S_2, S_3 – surfaces perpendicular to the z-axis; n_F – normal to the lateral surface; n_z – normal to S_1, S_2, S_3; φ – temperature distribution at the initial moment of time.

The solution of this system of equations is carried out using Fourier series. The theoretical process is considered only in the z-axis (package thickness) direction, since the molding heating process proceeds almost equally in the x and y directions.

In this case, the general analytical solution for a flat structure is as follows:

$$t_i = b_i x + c_i + \sum_{n=1}^{\infty} A_n (cos m_n x + p_n sin m_n x) \exp(-a_i m_n^2 \tau), \quad (2)$$

where b_i, c_i – constants determined from the condition of stationarity of the mode (at $\tau = \infty$); p_n, m_n – constants determined from boundary conditions; A_n – constant determined from the initial conditions (at $\tau = 0$).

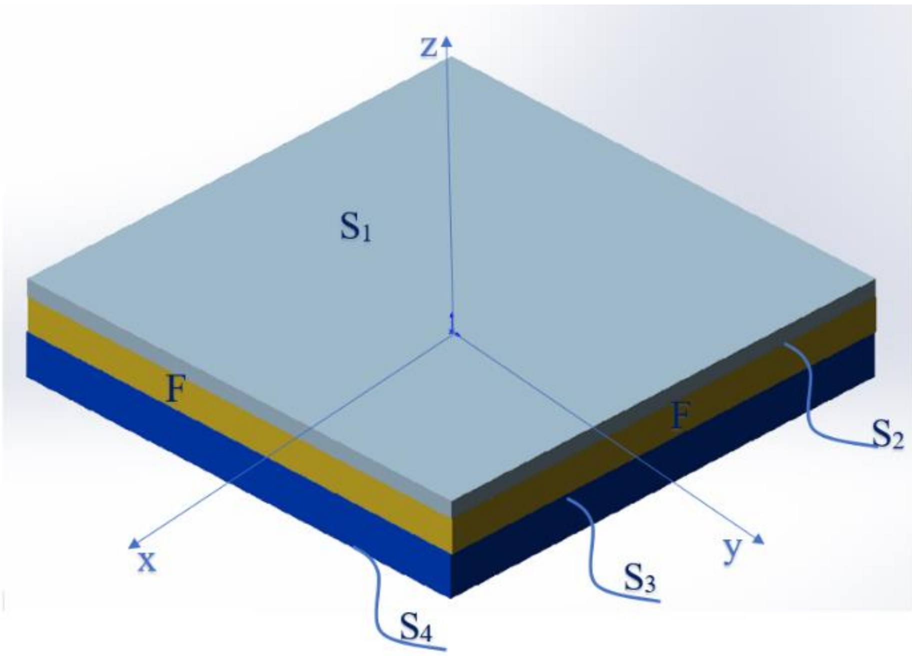

Fig. 1. Surfaces of the package layers

The obtained problem of unsteady heat conduction will be solved by the method of elementary balances, in which the body under consideration is divided into a number of elementary volumes with sides Δx, Δy, Δz. The calculation points will be the intersections of the planes of the breakdown, i.e. the corners of the elementary parallelepipeds. For this problem, we will transform the method of elementary balances, in the traditional form of which the heat exchange of the environment with the body is not taken into account. In this way, we can obtain dependencies for corner points, points lying on the edges and faces of the molded package. This will allow us to take into account not only the amount of heat entering the package from the most developed surfaces, as it is done in the analytical solution, but also from the sides of the package.

In this model, the thermal conductivity coefficient and specific heat capacity are taken as linear functions of temperature:

$$\lambda(t) = A + Bt, \quad (3)$$

$$C_p(t) = C + Dt, \quad (4)$$

where *A*, *C* – initial values of the thermal conductivity coefficient and specific heat capacity; *B*, *D* – linearity coefficients determined experimentally.

The law of temperature change of the autoclave working medium, which has the form shown in Fig. 2, was taken as a calculation model. It is necessary to choose such a value of the temperature jump t_1 and time τ_1, so that the value of the package temperature $t_i$for the inner layers of the package coincides with the given technological mode of polymerization.

The initial value of temperature $t_1$during calculations is taken from Table 1, compiled on the basis of analysis of autoclave loads.

Table 1

The value of the temperature jumps in the first approximation for PCM based on epoxy binder

Type PCM	Weight of equipment, kg	Type of construction	The value of the temperature jump,°C
fiberglass	200-400	frame	115
	500-800	frame	125
	>800	frame	135
carbon fiber	600-900	frame	100
	400-600	monolithic	140

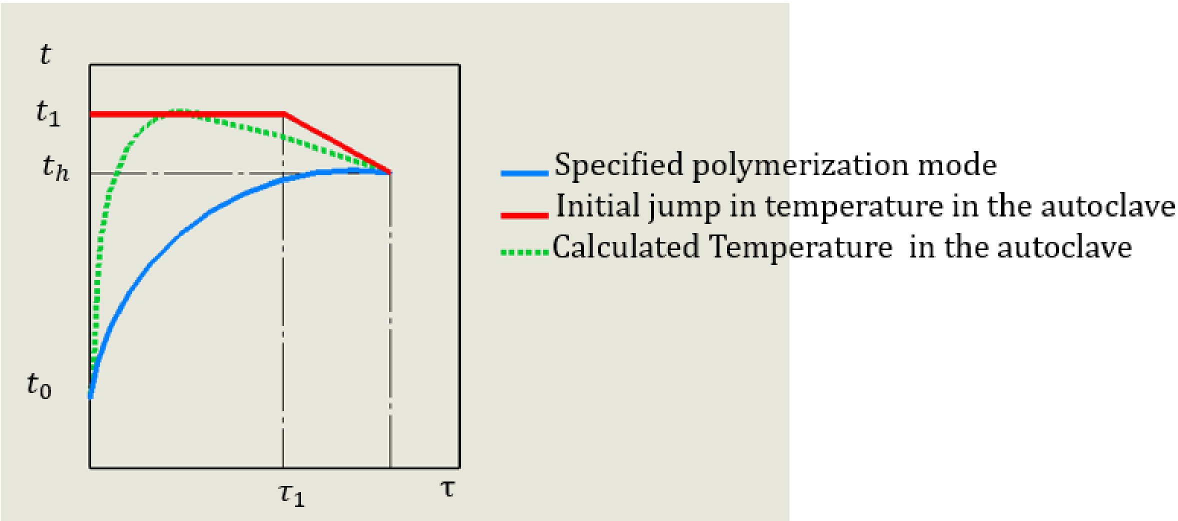

Fig. 2. Change in the temperature of the working environment of the autoclave

For a point surrounded on all sides by a homogeneous solid medium, the amount of heat entering the element through its six faces (see Fig.3) in time Δτ, based on Fourier's law and taking into account (3), is equal to:

$$\Delta Q_1 = -\left(A + B\frac{t + t_{x-\Delta x}}{2}\right)\frac{t - t_{x-\Delta x}}{\Delta x}\Delta y\Delta z\Delta\tau;$$
$$\Delta Q_2 = -\left(A + B\frac{t + t_{x+\Delta x}}{2}\right)\frac{t - t_{x+\Delta x}}{\Delta x}\Delta y\Delta z\Delta\tau;$$
$$\Delta Q_3 = -\left(A + B\frac{t+t_{y+\Delta y}}{2}\right)\frac{t-t_{y-\Delta y}}{\Delta y}\Delta x\Delta z\Delta\tau; \quad (5)$$
$$\Delta Q_4 = -\left(A + B\frac{t + t_{y+\Delta y}}{2}\right)\frac{t - t_{y+\Delta y}}{\Delta y}\Delta x\Delta z\Delta\tau;$$
$$\Delta Q_5 = -\left(A + B\frac{t + t_{z-\Delta z}}{2}\right)\frac{t - t_{z-\Delta z}}{\Delta z}\Delta x\Delta y\Delta\tau;$$
$$\Delta Q_6 = -\left(A + B\frac{t + t_{z+\Delta z}}{2}\right)\frac{t - t_{z+\Delta z}}{\Delta z}\Delta\Delta x\Delta y\Delta\tau.$$

The total amount of heat that entered the element during time Δτ is equal to the increase in its heat content:

$$\sum_i \Delta Q_i = C_p(t)\Upsilon\Delta V\Delta t = (C + Dt)\Upsilon\,\Delta x\Delta y\Delta z(t_{\tau+\Delta\tau} - t); \quad (6)$$

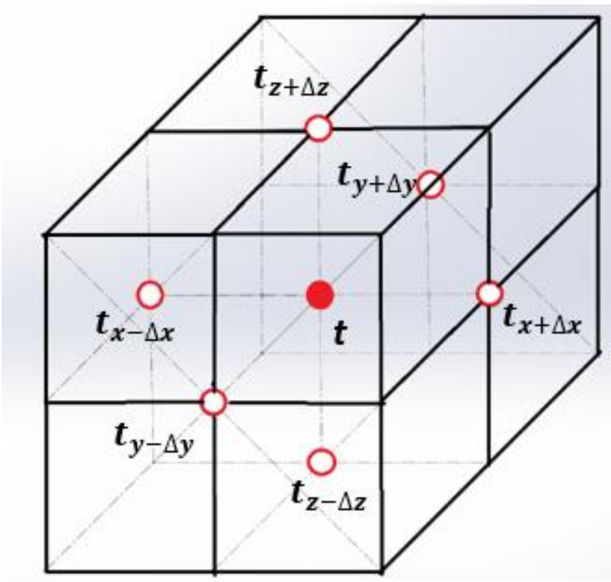

Fig. 3. Calculation points of an elementary parallelepiped

By substituting equation (5) into (6) and solving for $t_{\tau+\Delta\tau}$the following dependence was obtained:

$$t_{\tau+\Delta\tau} = R(t) + \frac{w_x(t_{x-\Delta x})t_{x-\Delta x}}{C_p(t)} + \frac{w_x(t_{x+\Delta x})t_{x+\Delta x}}{C_p(t)} + \frac{w_y(t_{y-\Delta y})t_{y-\Delta y}}{C_p(t)}$$
$$+ \frac{w_y(t_{y+\Delta y})t_{y+\Delta y}}{C_p(t)} + \frac{w_z(t_{z-\Delta z})t_{z-\Delta z}}{C_p(t)} + \frac{w_z(t_{z+\Delta z})t_{z+\Delta z}}{C_p(t)},$$

accepting

$$w_x(t) = \frac{\Delta\tau}{\Upsilon\Delta x^2}\left(A + \frac{Bt}{2}\right);$$
$$w_y(t) = \frac{\Delta\tau}{\Upsilon\Delta xy^2}\left(A + \frac{Bt}{2}\right);$$

$$w_z(t) = \frac{\Delta\tau}{\Upsilon\Delta z^2}\left(A + \frac{Bt}{2}\right);$$

$$R(t) = 1 - 2\left[\frac{w_x(t) + w_y(t) + w_z(t)}{C_p(t)}\right].$$

Implementation of the obtained model made it possible to obtain the temperature distribution in the central part of the package. On its basis it is possible to obtain the temperature value for any point of the package and to obtain the value of t_we (i.e., in fact, to obtain the theoretical value for the initial temperature jump). In the calculations, the balance equations were also similarly used, taking into account the values (5) for the vertices of the calculated elementary parallelepiped, the points lying on the edges of the formed package and the points lying on the outer planes of the formed package. If the calculated point fell on the boundary of two different layers, then the temperature value for it is calculated according to the formula for a point surrounded by a homogenous medium, taking into account in the heat flows the coefficients of the layers from the side of which they are directed.

The results of the method implementation for a fixed point in time (in the section of temperature rise to the glass transition temperature) in a given thickness of the part (fixation along the z axis - 11 calculation point corresponding to the middle layer of the package) are presented in Fig. 4, 5. For the first graph (see Fig. 4), the change in temperature along the y axis to the point of the middle of the part along its width is shown for the initial reference point x=1 (edge along the width of the part - red line) and the middle point along the width (blue line).

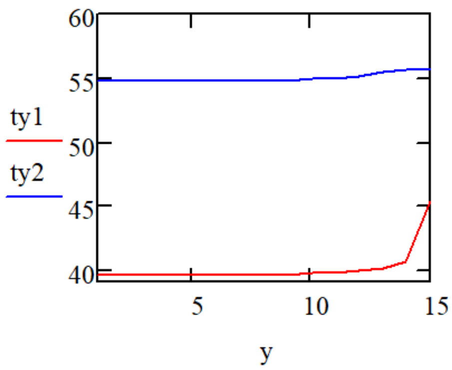

Fig. 4. The change in temperature along the y axis

The second graph (see Fig. 5) shows the results of similar calculations for evaluating the temperature change along the x-axis (along the length). The red line shows the temperature change of the middle layer of the package along the edge of the part to its middle. The blue line shows the temperature change in the middle of the width of the part to the midpoint of the length.

These parameters for outputting the results were chosen to verify the correctness of the program. These parameters for outputting the results were chosen to check the correctness of the program. As expected, the red lines of the graphs demonstrated higher rates of temperature gain along the edge surfaces of the molded part. While the inner layers along the length (the largest size of the part) showed lower rates of heat gain toward the center of the part.

The final result of this study was the determination of the time to reach the specified temperature of the polymerization of the middle layer of the part. For the product under consideration, this temperature was 128.37°C for 203.92 min, which corresponds to the technological requirements of the mode for the speed of temperature increase (1.6°C /min).

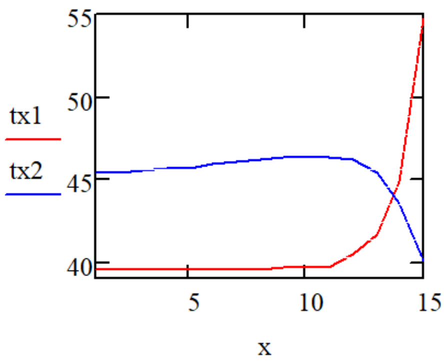

Fig. 5. The temperature change along the x-axis

Conclusion

1. In the course of the work, the formation of a model of polymerization of a product made of PCM was considered, which makes it possible to estimate the temperature in the layers of the molded product. The theoretical model was based on thermodynamic processes, which made it possible to develop the main prerequisites for the calculation model and obtain a simplified expression for estimating the temperature of the package.

2. A number of assumptions were made, such as instantaneous convection in the autoclave working environment, which made it possible to simplify the creation of the model.

3. Conducted practical research based on the analysis of autoclave loadings with products of different sizes and materials, as well as various configurations of mold, allowed us to develop recommendations for setting the initial temperature jump in the autoclave medium, necessary for the implementation of software calculation of temperature distribution in the formed package.

4. The method of elementary balances was used not in its pure form, but with transformations taking into account the effects between the medium and different layers of the package and with the possibility of determining temperatures not only in the internal elementary volumes, but also for corner points, as well as points lying on on the edges of the formed package.

5. The program developed on the basis of the considered methods is three-dimensional, i.e. it takes into account heat input into the considered element from all sides, not only from the side of developed surfaces, which is typical for most methods.

6. In developing the computational model, the change in the physical parameters of the package with time during heating was taken into account, which also increased the probability of finding a more accurate solution.

7. The main difference from other software products used to solve similar problems is the possibility of solving the inverse problem - determining the required temperature of the medium according to a given mode.

8. As a result of the implementation of the model, it was possible to track the temperature distribution in the central plane of the package.

9. In the future it is supposed to apply this model and the program based on it to determine the thermal component of the activation energy responsible for

determining the degree of PCM polymerization.

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Моделювання процесу термічної полімеризації композиційних матеріалів з урахуванням особливостей обладнання

У роботі розглянуто проблему отримання математичної моделі процесу термічного формування конструкцій з полімерних композиційних матеріалів в автоклаві з урахуванням його технічних можливостей та типів формоутворювального оснащення. Основним завданням роботи було одержання залежності, що показує зміну температури внутрішньої структури полімеризованого пакета в залежності від робочого середовища автоклава та початкових умов процесу, що залежать від типу нагрівального пристрою та виду оснастки. Початкова реалізація завдання проводилася з урахуванням використання рівнянь Фур'є для потрійного пакета (допоміжне оснащення, виріб, основне оснащення). Пропонований підхід використовував значення початкового стрибка температури в залежності від маси допоміжного обладнання (оснащення) і типу формованого композиційного матеріалу, які були отримані дослідним шляхом. У дослідженні було задіяно реальні процеси виготовлення ПКМ-виробів в автоклаві фірми «Шольц». Оскільки практична частина дослідження проводилася для пристрою з потужною вентиляторною установкою було прийнято припущення про миттєву конвекцію нагрітого повітря по робочому об'єму автоклава. Другим етапом роботи здійснювалося вирішення задачі нестаціонарної теплопровідності методом елементарних балансів, наближеним до класичного методу кінцевих елементів. Для цього завдання метод було перетворено, що дозволило здійснити облік теплообміну довкілля з тілом. Таким чином стало можливо отримати залежності для кутових точок, точок лежачих на ребрах і гранях пакету, що формується. Це дозволило врахувати як кількість тепла, що надходить у пакет із боку найрозвиненіших поверхонь (як це робиться в аналітичному рішенні), така й з боків пакета. У даній моделі коефіцієнт теплопровідності та питома теплоємність приймаються лінійними функціями температури. При розробці розрахункової моделі було враховано зміну фізичних параметрів пакета з часом при нагріванні, що також збільшило можливість отримання більш точного рішення задачі. Основною відмінністю від інших програмних продуктів, що використовуються для вирішення аналогічних задач є можливість вирішення зворотної задачі, яка полягає у визначенні за заданим режимом потрібної температури середовища. Отримані залежності для сумарних теплових потоків дозволяють знайти сумарне значення теплоти, використоване для процесів полімеризації, температуру середовища в автоклаві та відповідне значення температури серединного шару пакета. Дані параметри були розраховані для точки виходу на температуру полімеризації скловолоконної конструкції на базі епоксидного сполучного.

Ключові слова: Полімерний композитний матеріал; моделювання термічних процесів; енергоефективність; автоклав; формувальне обладнання; робоче середовище.

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