Виявлення механізму роботи покриття в будівельних конструкціях дозволить оцінити умови експлуатації виробів, визначити ефективність застосування методів і засобів вогнезахисту. Розроблена математична модель та розраховано процес просування фронту фазових перетворень при спучуванні вогнезахисного покриття. Наведено результати експериментальних досліджень фронту фазових перетворень при спучуванні вогнезахисного покриття та встановлено вплив наповнювачів на нього

Ключові слова: спучуючі покриття, температура печі, втрата маси, оброблення поверхні, фронт фазових перетворень

Определение механизма работы покрытия в строительных конструкциях позволит оценить условия эксплуатации изделий, определить эффективность применения методов и средств огнезащиты. Разработана математическая модель и рассчитан процесс продвижения фронта фазовых превращений при вспучивании огнезащитного покрытия. Приведены результаты экспериментальных исследований фронта фазовых превращений при вспучивании огнезащитного покрытия и установлено влияние наполнителей на него

Ключевые слова: вспучивающиеся покрытия, температура печи, потеря массы, обработки поверхности, эффективность покрытия

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SIMULATION OF THE PHASE TRANSFORMATION FRONT ADVANCEMENT DURING THE SWELLING OF FIRE RETARDANT COATINGS

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1. Introduction

Currently, the most common building material is wood, which belongs to the group of average-flammability combustible materials. To reduce these shortcomings, fireproofing provides the wood with the ability to withstand flame exposure and propagation.

One way of wood flameproofing is application of a fireproof coating, which for a while prevents the access of heat to wood.

Swelling of the coating is accompanied by softening of components while endothermic decomposition of fire retardants and blowing agents, leading to the formation of a dense foam coke layer, which causes the fireproof properties of the coating. In this case, the outer coating surface moves towards the influence of flame temperature, which provides the necessary insulation of the material, and the other coating layer, which has not undergone changes and is separated by a line of phase transition, is in reserve.

The main reactions that lead to the formation of a protective foam coke layer occur in the high-temperature region and are complicated to establish the role of the components in the swelling process and advancement of the phase transformation front of foam coke. So, there is the need to study the formation conditions of a barrier for heat conduction and reveal a mechanism of phase transition from the coating film to the coke layer.

2. Literature review and problem statement

In recent years, fire protection research works, aimed at synthesis of coatings using organic paints, refractory oxides and silicates have been known. These materials form temperature and heat-resistant ceramic phases during heating [1, 2]. The most common are crystal glass and enamel coatings [3], but they can't provide reliable protection of structures at long-term temperatures as they are unable to form a necessary coke layer, heated and destroyed.

Modern fire protection methods include the use of intumescent coatings, which are complex systems of organic and inorganic components [4] and have a high intumescent ability. The effectiveness of flame retardant coatings based on organic substances has been shown in [5]. Flame retardants based on polyphosphoric acids and blowing agents may significantly influence the porous layer of foam coke. The above works present the component compositions, efficiency, and thermal characteristics of the coating, but not the mechanism of coke formation, phase and thermal transitions of the coating in foam coke.

Description of the behavior of intumescent coatings, one of the objectives of which is integration of experimental data with available theoretical models, has been considered in [6]. It allows at least a general estimation of simplifications made. Therefore, a thermophysical model, whose solution is given by polynomials that are not associated with a physical content has been examined.

The mathematical model of heating of fire-proof coatings, based on the laws of conservation of matter and energy has been considered. The models immediately assume a particular type of functional relationships with a set of uncertain factors and the problem is reduced to determining the numerical values of these factors, due to the high inaccuracy [7].

On the basis of the kinetics considered, the coating model is represented, which is a mixture of starting materials and products of their transformations, including gas [8]. An increase in the volume of gas when heated causes swelling, but the impact of temperature on the coating churning zone movement has not been determined. The coefficient of thermal conductivity of fire-resistant coatings in fire has been estimated on the basis of numerical simulation, where the phase transitions in the coating have not been taken into account [9]. The effect of inorganic fillers on fire-retardant water-based coatings has shown to be effective. However, the coating swelling mechanism has not been specified and the coating phase transitions in coke have not been found [10]. The authors have presented the analytical model to calculate the thermal conductivity of the porous structure of foam coke of the fireproof coating, which allows for the shapes of pores. However, the model does not account for what phase transitions of the coating occur. Therefore, simulation of the phase transformation front during the swelling of the fireproof coating, under the impact of the components is the unresolved element of ensuring fire resistance of building structures. This led to the need for research in this area.

3. The purpose and objectives of the research

The aim is to study the process of phase formation of foam coke during the swelling of fire-proof coatings.

To achieve this aim, the following objectives were accomplished:

– to conduct simulation of the process of advancement of the phase transformation front of the intumescent coating under thermal exposure;

– to determine timing features of full swelling of fire-proof coatings under thermal exposure.

4. Materials and methods of the research of the phase transformation front during the swelling of fireproof coatings

4. 1. Experimental materials

The study was conducted using a system of ammonium polyphosphate (PFA), melamine pentaerythritol (PER) and a binder-based on PVA dispersion. Titanium dioxide, talc, aluminum and magnesium hydroxide and mixtures thereof were added to the above mixture.

Experimental samples of coatings were based on the system containing 18÷20 % PFA, 12÷14 % melamine, 10÷12 % PER, 16 % PVA dispersion and water. The resulting mass was stirred with injecting the fillers in an amount of up to 10 % and applied on a 0.5÷0.01 mm steel plate (Fig. 1).

The fillers do not significantly after the appearance and structure of the coating, slightly increasing its viscosity.

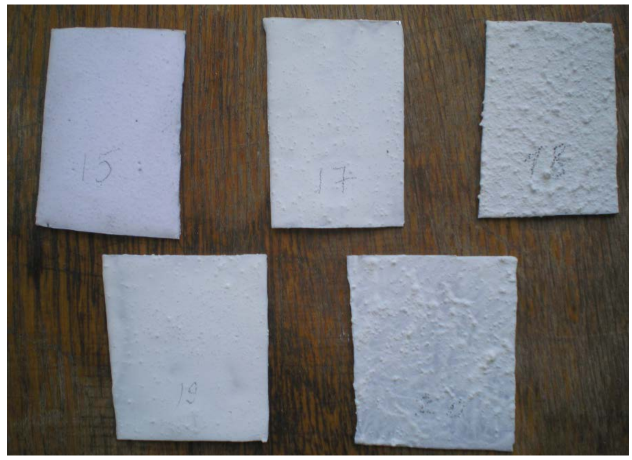


Fig. 1. Samples of the coating on a steel plate

4. 2. Method of determining the properties of samples

Simulation of the advancement process of the phase transformation front of the intumescent coating under thermal exposure was performed with the use of the main provisions of mathematical physics.

Experimental determination of the swelling process was performed according to the method given in [11]. The method consists in the fact that a sample with the flame retardant coating is placed in the sample holder and inserted into a test chamber. The sample was fixed so that the end of the control thermocouple touched the back surface of the sample. The heating of the test chamber was turned on, the temperature on the reverse surface and in front of the sample was measured with a thermocouple and the coating was expanded by a ruler. According to the measured values, the heat-insulating properties and changes of the coating were determined.

The criterion for determining the phase transformation front of the intumescent coating under thermal exposure is the foam coke formation of the coating thickness. Herewith, the phase transformation front is observed in the form of a thin layer between the foam coke layer and source material.

5. Simulation of the phase transformation front advancement during the swelling of fire-proof coatings

The model for obtaining the dependencies for calculating the rate of advancement of the phase transformation front of the intumescent coating under thermal exposure is proposed. The focus was on three areas (Fig. 2):

1 – environment, $x<0$;

2 – intumescent foam coke layer area, $0<x\leq Z(\tau)$ (Z – the coordinate of the front of phase transformation of the coating film in the swollen foam coke layer, m);

3 – material sample with solid material (coating film), $Z\leq x\leq h$ (h – half the thickness of the sample, m).

The differential equation that describes the process:

$$\frac{\partial^2 T_i}{\partial x^2}-\frac{1}{\phi_i^2}\frac{\partial T_i}{\partial \tau}=0, \tag{1}$$

where T_i is the temperature in the “i” region, °C; $\phi_i=\sqrt{a_i}$; a_i is the thermal diffusivity in the “i” region, m²/s; τ is the sample residence time in the high-temperature environment, s.

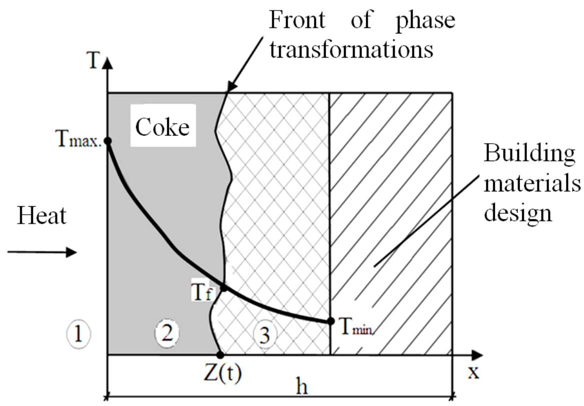


Fig. 2. The scheme of the fire-proof coating swelling process: 1 — environment; 2 — foam coke layer; 3 — starting material

At the initial time (τ=0), the temperature in the environment is maximum $\hat{T}_{max}$, and in the sample material – minimum $\breve{T}_{min}$, thus, the initial and boundary conditions can be written as follows:

$$T_1(x,0)=\breve{T}_{max};$$

$$T_3(x,0)=\hat{T}_{min};$$

$$Z(0)=0. \quad (2)$$

At the border of the sample and the environment, the boundary condition of the fourth kind, which means maintaining the heat flow at the transition from the environment into the inner region of the sample (the law of conservation of heat flow) is given [12]:

$$\lambda_1 \left.\frac{\partial T_1}{\partial x}\right|_{x=0} = \lambda_2 \left.\frac{\partial T_2}{\partial x}\right|_{x=0}, \quad (3)$$

where λ_1, λ_2 are the coefficients of thermal conductivity of the environment (1) and the foamed coke layer (2), W/(m·°C).

Given the continuity of the heat flow fields, the temperatures on the sample surface and the front of the phase transformation of the intumescent coating into the foam coke layer coincide:

$$T_1(0,\tau)=T_2(0,\tau); \quad (4)$$

$$T_2[Z(\tau),\tau]=T_3[Z(\tau),\tau]=T_f. \quad (5)$$

Due to the phase transformation (solid into in foam coke), the temperature is absorbed at a rate q, W/(m^3·s), which is proportional to the speed of advancement of the phase transformation front of the coating [13], namely:

$$q=\vartheta\frac{dZ}{d\tau}, \quad (6)$$

where ϑ is the proportionality factor showing the heat flux absorption rate per unit depth of advancement of the phase transformation front of the coating, W/m^4.

The absorption rate of the heat flow q, coming from region 1 to region 2 is absorbed through the formation of the expanded layer of foam coke [6]. Therefore, on the border of phase transformation, the condition should be satisfied:

$$\lambda_2\frac{dT_2}{dx}=q+\lambda_3\frac{dT_3}{dx}$$

or

$$\lambda_2 \left.\frac{dT_2}{dx}\right|_{x=Z(\tau)} - \lambda_3 \left.\frac{dT_3}{dx}\right|_{x=Z(\tau)} = \vartheta\frac{dZ}{d\tau}. \quad (7)$$

Thus, the problem is reduced to the solution of the differential equation (1) with the boundary conditions (2)–(5), (7).

To solve the differential equation (1), we introduce dimensionless variables:

$$\xi=\frac{x}{h};$$

$$\bar{\tau}=\frac{\phi_3^2}{h^2}\tau;$$

$$T_i=\frac{T_i-\breve{T}}{\hat{T}-\breve{T}}, \quad (8)$$

and then the problem is formulated as follows:

– to solve the differential equation:

$$\frac{\partial^2\bar{T}_i}{\partial\xi^2}=\frac{1}{\bar{\phi}_i^2}\frac{\partial\bar{T}_i}{\partial\bar{\tau}}, \quad (9)$$

with the boundary conditions:

$$\bar{T}_1(\xi,0)=0;$$

$$\bar{T}_3(\xi,0)=1; \quad (10)$$

$$\bar{\lambda}_1 \left.\frac{\partial\bar{T}_1}{\partial\xi}\right|_{\xi=0} = \bar{\lambda}_2 \left.\frac{\partial\bar{T}_2}{\partial\xi}\right|_{\xi=0}; \quad (11)$$

$$\bar{T}_1(0,\tau)=\bar{T}_2(0,\tau); \quad (12)$$

$$\bar{T}_2(\bar{Z},\bar{\tau})=\bar{T}_3(\bar{Z},\bar{\tau})=\bar{T}_f; \quad (13)$$

$$\bar{\lambda}_2 \left.\frac{\partial\bar{T}_2}{\partial\xi}\right|_{\xi=\bar{Z}} - \bar{\lambda}_3 \left.\frac{\partial\bar{T}_3}{\partial\xi}\right|_{\xi=\bar{Z}} = \bar{\vartheta}\frac{\partial\bar{Z}}{\partial\bar{\tau}}, \quad (14)$$

where

$$\bar{\phi}_i=\frac{\phi_i}{\phi_3},(\bar{\phi}_3=1);\ \bar{\lambda}_i=\frac{\lambda_i}{\lambda_3},(\bar{\lambda}_3=1);$$

$$\bar{Z}=\frac{Z}{h};\ \bar{T}_f=\frac{T_f-\breve{T}}{\hat{T}-\breve{T}};\ \bar{\vartheta}=\frac{\phi_3^2}{\beta_3}\frac{\vartheta}{\hat{T}-\breve{T}}.$$

To determine the limits of phase transformations and the speed of advancement of the phase transformation front, the solution of equation (9) takes the following form taking into account [14]:

$$\bar{T}_i=c_i+b_i\Phi\left(\frac{\xi}{2\bar{\phi}_i\sqrt{\bar{\tau}}}\right), \quad (15)$$

where the error integral is

$$\Phi(\sigma)=\frac{2}{\sqrt{\pi}}\int_0^{\sigma}e^{-\sigma^2}d\sigma. \quad (16)$$

The coefficients c_i, b_i are determined from the boundary conditions (10)–(14). After substituting (15) to (10), we obtain the equation:

$$\overline{T}_1(\xi,0)=c_1+b_1\Phi(\infty)=c_1+b_1=0; \tag{17}$$

$$\overline{T}_3(\xi,0)=c_3+b_3\Phi(\infty)=c_3+b_3=1. \tag{18}$$

Since

$$\frac{\partial\overline{T}_i}{\partial\xi}=\pm\frac{b_i}{\overline{\phi}_i\sqrt{\pi\overline{\tau}}}e^{-\frac{\xi^2}{4\overline{\phi}_i^2\overline{\tau}}}, \tag{19}$$

where the sign of the right side coincides with the sign ξ, then from (11) it follows that:

$$b_1=-\frac{\overline{\phi}_1\overline{\lambda}_2}{\overline{\phi}_2\overline{\lambda}_1}b_2. \tag{20}$$

Given (15) and F(0)=0, the condition (12) is converted to the form:

$$c_1=c_2. \tag{21}$$

After substituting (15) to (13), we get the equation for the swelling front boundary:

$$c_2+b_2\Phi\left(\frac{\overline{Z}}{2\overline{\phi}_2\sqrt{\overline{\tau}}}\right)=c_3+b_3\Phi\left(\frac{\overline{Z}}{2\sqrt{\overline{\tau}}}\right)=\overline{T}_f. \tag{22}$$

Since the right side of equation (22) does not change, the arguments of the function F are also constant. This is possible only if the coordinate of the coating swelling front is proportional to the square root of time, provided that:

$$\overline{Z}=\overline{Z}(\overline{\tau})=2\beta\sqrt{\overline{\tau}}, \tag{23}$$

where for convenience the proportionality factor is taken as being equal to the doubled, yet unknown constant β. As a result, (22) can be written in the form of two equations:

$$c_2+b_2\Phi\left(\frac{\beta}{\overline{\phi}_2}\right)=\overline{T}_f;$$

$$c_3+b_3\Phi(\beta)=\overline{T}_f. \tag{24}$$

After substituting (24), (23) in the heat flow continuity condition given $\overline{Z}=\xi$, the equation (14) is converted to the following form:

$$\frac{\overline{\lambda}_2}{\overline{\phi}_2}e^{-\frac{\beta^2}{\overline{\phi}_2^2}}b_2-e^{-\beta^2}b_3=\sqrt{\pi}\,\overline{\vartheta}\beta. \tag{25}$$

From the system of equations (12), (17), (18), (20), (24), (25), with respect to the unknowns c_i, b_i, β, we find the solutions:

$$c_1=c_2=\frac{\eta\overline{T}_f}{\eta+\Phi\left(\frac{\beta}{\overline{\phi}_2}\right)};$$

$$c_3=\frac{\overline{T}_f-\Phi(\beta)}{\Phi^*(\beta)}; \tag{26}$$

$$b_1=-\frac{\eta\overline{T}_f}{\eta+\Phi\left(\frac{\beta}{\overline{\phi}_2}\right)};$$

$$b_2=-\frac{\overline{T}_f}{\eta+\Phi\left(\frac{\beta}{\overline{\phi}_2}\right)};$$

$$b_3=\frac{1-\overline{T}_f}{\Phi^*(\beta)}; \tag{27}$$

$$\frac{\overline{\lambda}_2}{\overline{\phi}_2}\frac{\overline{T}_f}{\eta+\Phi\left(\frac{\beta}{\overline{\phi}_2}\right)}e^{-\frac{\beta^2}{\overline{\phi}_2^2}}-\frac{1-\overline{T}_f}{\Phi^*(\beta)}e^{-\beta^2}=\sqrt{\pi}\,\overline{\vartheta}\beta, \tag{28}$$

where

$$\eta=\frac{\overline{\phi}_1\cdot\overline{\lambda}_2}{\overline{\phi}_2\cdot\overline{\lambda}_1};\quad \Phi^*(\sigma)=1-\Phi(\sigma).$$

It follows that all the constants c_i, b_i depend on the parameter β, which in turn can be determined on the basis of the solution (28) only by approximate calculations [15].

Thus, the calculated dependences, allowing to get changes in the swelling dynamics of fire-retardant coatings are obtained. However, they provide an opportunity to directly calculate the coating swelling time and the phase transformation front movement depending on the effects of temperature.

6. The results of determining the phase transformation front during thermal coating swelling

Fig. 3 shows the calculation of β depending on the dimensionless complex $\overline{\vartheta}$, characterizing the heat-insulating properties of intumescent coatings on the border of the phase transformation front in a situation where half of the heat is absorbed by the coating:

$$\overline{T}_f=\eta=0{,}5. \tag{29}$$

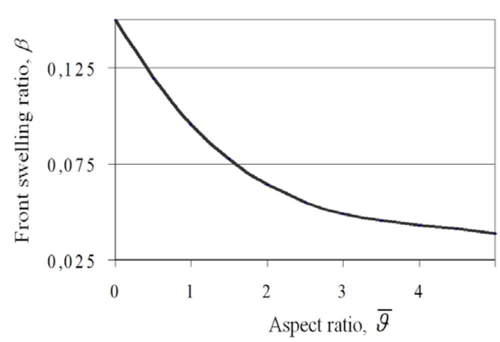


Fig. 3. Dependence of the coating swelling front coefficient on the heat flux absorption proportionality coefficient

The results correspond to the physical content of the problem solved, namely the rate of advancement of the phase transformation front decreases given the constant swelling rate of fire-proof coatings.

We solve graphically the equation (28) and obtain the value β=0.062 (Fig. 4).

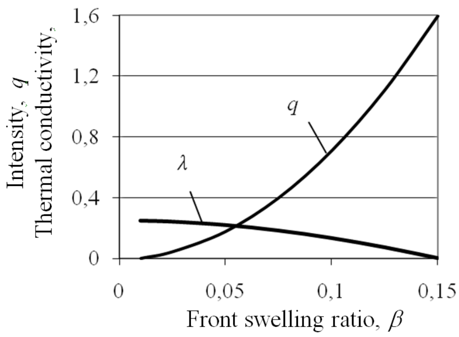


Fig. 4. Graphical solution of equation (28): the dependence of the fire-proof coating swelling rate and thermal conductivity on the swelling coefficient.

So, the dependencies, which allow getting a picture of the swelling dynamics and provide an opportunity to directly determine the phase transformation front, are given.

7. Experimental studies of the phase transformation front during the swelling of fire-proof coatings and their results

To find the time of full swelling of fire-proof coatings (Z=h), we get the value of:

$$\tau = \frac{h^2}{4\beta^2 \cdot a}. \tag{30}$$

Given h=1 mm and a=2.9·10^{-5} m^2/s, the time of full swelling of fire-proof coatings is 2.2 s.

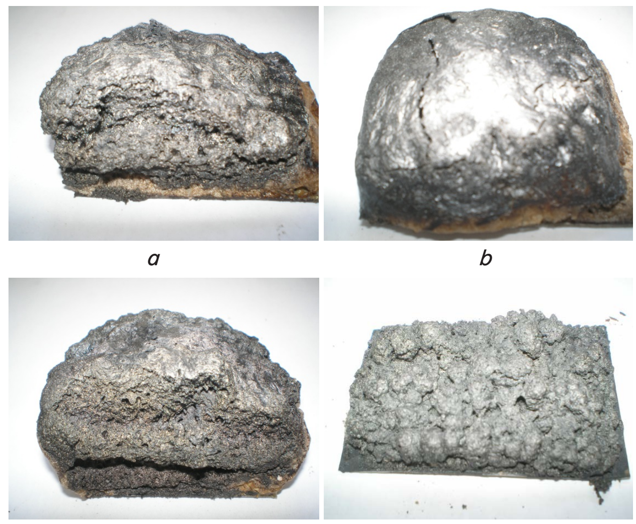
Experimental studies of the phase transformation front during the expansion of a flame retardant coating are shown in Fig. 5, 6.

Fig. 5. The process of coating swelling

Exposure of the coating samples to a heat flow at temperatures of about 190÷200 °C in front of the sample caused intensive swelling and a slight temperature increase on the back sample surface, which lasted upon reaching a temperature of about 360÷390 °C.

The height of the expanded layer of foam coke increased to 22÷38 mm and the phase transformation front line appeared on the surface under the foam coke layer.

As a result of the tests, it is found that under thermal exposure the coating foams and moves towards the high temperature to form coke. The foaming front boundary line in the form of a thin layer, which is slightly shifted towards the temperature, divides the coating into two parts. On the one side, there is a swollen coke layer (dark), the outer part of which moves at a certain speed, on the other side – the layer of the source material, where the temperature is not sufficient to start the foaming process and the speed of transformations is zero (white), Fig. 6, *a, b*.



a *b*

c *d*

Fig. 6. Test results of the intumescent coating:
a — organic and inorganic + (10 %) TiO_2;
b — organic and inorganic + (10 %) $Al(OH)_3$;
c — organic and inorganic + (10 %) $Mg(OH)_2$;
d — organic and inorganic + (5 %) TiO_2 + (10 %) $Al(OH)_3$

However, in the organic-inorganic coating with the addition of TiO_2 and $Al(OH)_3$, a relatively low swelling rate and the absence of the starting material, as well as the phase transition boundary were observed (Fig. 6, *d*). A large amount of $Al(OH)_3$ led to the suppression of the coating swelling process.

8. Discussion of the results of research of the advancement process of the phase transformation front

When swelling of fire-proof coatings under the thermal effect of high-temperature flame, as indicated by the research results (Fig. 3, 5, 6), there is a natural process of the coating swelling under the influence of temperature and the formation of the phase transition line, due to the mechanism of the foam coke layer formation, which slow down the heat transfer processes. It should be noted that the presence of more than 10 % of additives, such as aluminum hydroxide, reduces the swelling process and leads to the lack of the phase transition line. Obviously, such a mechanism of the effect of additives is a factor regulating the degree of coke destruction and thermal insulation efficiency. But at the same time, this mechanism adversely affects the intumescent ability of the coating. This agrees with the data of the known works [6, 9], the authors of which have also attributed the changes in the swelling process to the addition of mineral fillers. In contrast to the results of the studies [6, 9], the data on the influence of fillers on the process of phase transition of the coating into the foam coke layer and changes in the insulation properties suggest the following:

– the main process regulator is not only the formation of the foam coke layer, but also heat resistance of the fire retardant coating;

– correct addition of mineral fillers has a significant influence on the phase transition of the fireproof coating into the vermiculite foam coke layer.

The results of determining the phase transition of the fireproof coating into the vermiculite coke layer (Fig. 6) indicate a mixed impact of fillers on the intumescent ability of the coating. Such uncertainty can't be resolved within the present study because this would need additional experiments to obtain more reliable data. In particular, this requires sufficient data for carrying out the effective swelling process and identifying the time of the beginning of heat resistance decline. Such identification will allow exploring the conversion of the coating, which moves in the direction of high temperature to form coke, and identifying the variables that significantly affect the beginning of the process transformation.

This work is a continuation of the research presented in [11], which fully shows the formation mechanism, movement and high-temperature heat insulation of foam coke.

9. Conclusions

As a result of the studies, the process of phase formation of foam coke during the swelling of fire-proof coatings was revealed, in particular:

– the simulation of the advancement process of the phase transformation front during the swelling of the fireproof coating was performed, the swelling front ratio was determined and settlement dependences allowing to get changes in the swelling dynamics of fire-proof coatings under the influence of temperature were obtained. According to the dependencies, the foam coke formation time under thermal decomposition of the coating, which makes up 2.2 s, subject to the double heat absorption, was calculated;

– the phase transformation front moves in the direction of high temperature to form coke, the foaming front boundary line in the form of a thin layer divides the coating into two parts. On the one side, there is a swollen coke layer, the outer part of which moves at a certain speed to the source of heat. On the other side – a layer of starting material, where the temperature is insufficient to start the foaming process and the speed of transformations is zero. Further research may be aimed at studying the process of establishing the relationship between the components and properties of coatings and their effect on the heat resistance of building structures.

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