

INTUMESCENT FIREPROOF COATINGS BASED ON ZEOLITE-LIKE CEMENT MATRICES

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Abstract

Concrete and reinforced concrete building structures (for example, such as tunnels) lose carrying ability in case of high-temperature fire action. The aim of the research is to study the prevention of reinforced concrete structures (for example, such as tunnels) under fire action in case of using the proposed coating based on the alkaline aluminosilicate binder, which would not consist of organic components dangerous to health. The ratios between constituent oxides in the binder which ensure the ability to bloat the coating under fire action were determined. The performance properties of developed fire protective coating were defined after artificial aging (cycles of alternate drying and cooling) and fire action: bloating factor - 2.0...5.1, adhesion strength - 6.6...8.0 MPa, compressive strength - 2.3...4.5 MPa, cohesive strength of 1.2...1.5 MPa, thermal conductivity coefficient - 0.042...0.066 W/m·°C, total porosity - 92...97 %. The temperature at which the coating starts to bloat = 200...250 °C has been developed. The results of the test held in the open air suggested drawing a conclusion that with a coating thickness of 6 mm protection of the reinforced concrete from fragile fracture and from plastic deformations in the metal of the reinforcement they provided under fire exposure for a period of 3 hours.

Keywords

Adhesion, Aluminosilicate pellets, Cohesion, Intumescent coating, Tunnel, Zeolite-like cement matrices

1 Introduction

Concrete and reinforced concrete building structures themselves have a certain fire resistance, which depends on the concrete grade and its reinforcement, the size of the structure, the type of aggregate, the loads, the support pattern, and the moisture content of the concrete under the operating conditions of the building. Reinforced concrete structures in the loaded state are subjected to high temperatures while fire action, which changes the properties of concrete and reinforcement. Loss of carrying ability of reinforced concrete elements can be caused by different factors: deformations arising due to asymmetrical change of physical-mechanical properties of concrete as a result of uneven heating in the cross-section of bearing elements; reduction of calculated section height due to heating of concrete to high temperatures; slippage of reinforcement on the support when the contact layer of concrete and reinforcement is heated to critical temperature, etc. Large plastic deformations occur in the reinforcement at high heating temperatures, which ceases to accept the forces of external loads. Concrete with a moisture content of approx. 3.5% has the highest fire resistance. However, moistened concretes with a density above 1200 kg/m³ are subject to brittle fracture even when exposed to short-

term fire action. Moisture that expands at the temperature rises and the phase transition of water to vapor creates pressure on the pore walls, resulting in considerable stress in the concrete. The brittle deterioration of concrete in a fire can quickly cause concrete or reinforced concrete structures to collapse. In this case, the fire resistance limit of structures can be significantly lower than required due to the reduction in the size of the cross-section of the structure, the reduction in thickness or complete elimination of the protective layer of the working reinforcement, and the formation of a through-hole. In general, the fire resistance limit of reinforced concrete structures can be up to 2.5 hours, fire protection for them is applied in case of higher (more than 2.5 hours) requirements for fire resistance limits. This is typical for objects such as high-rise buildings or road tunnels.

2 State-of-the-art and literature analysis

The design of safety systems and fire protection of structures in EU countries is carried out separately for each tunnel [1-3] taking into account different fire scenarios (standard temperature regime, hydrocarbon fire regime, according to German standards (RABT/ZTV with cooling stage), according to Dutch standards (RWS) [4, 5]. These

measures are primarily aimed at preventing limited states of the concrete itself and its reinforcement when exposed to fire.

The most common methods of additional protection of reinforced concrete structures from long-term exposure to elevated temperatures now are cladding with fire-resistant panels and the application of flame-retardant coatings and plasters.

The use of passive means of protection in the form of fire- and heat-resistant panels and coatings on cement bases is reflected in [6, 7], on geopolymer bases - in works [8, 9]. A common disadvantage of the proposed solutions is the increase of additional distributed load on the concrete tunnel structures due to the increased mass and thickness of the material, as well as the low fire resistance limit, not more than 1.5 hours in a standard fire [10-12].

It is advisable to use polymeric coatings of the intumescent type as active agents [13-16]. The authors pay more attention to the modification of polymers aimed at the thermal stabilization of the foam coke layer. The main disadvantage of such coatings is low adhesion to the protected surface and the release of toxic gases at the time of bloating.

Silicate coatings are known [17, 18], which are effective in terms of adhesion to the concrete base, but are characterized by short shelf life and the ability to bloat. The analysis of the publications on intumescent aluminosilicate coatings [19-23] makes it possible to determine their general orientation, which is manifested in the study of the processes of structure formation, revealing the mechanism of bloating, formation of porous structures, etc. However, from their side, there is no data on the development of the compositions of coatings, their protective effectiveness and the ability to prevent the heating of the concrete surface to brittle failure, and the reinforcement to the limit state.

A breakthrough in the development of intumescent coatings based on zeolite-like cement matrices is noted in the classic works of scientists of the school named after V.D. Glukhovskiy of the Kyiv National University of Construction and Architecture. The authors established the main regularities of synthesis of zeolite-like phases capable of two-stage dehydration with the evolution of free and chemically bound water [24-26], they revealed a mechanism of creating porous structures not only in the binder but also in aggregates [27-29], determined the durability [30, 31], conducted fire tests [32], and confirmed the effectiveness of their development in this area, the fire-resistant materials were produced by JSC Geofip (Ukraine).

The authors in [33] provided data on the protection of concrete structures of tunnels from hydrocarbon fire with fire retardant coatings based on alkali aluminosilicate binders. The issue of protection of concrete structures in a standard fire is not investigated.

Therefore, the purpose of the work is to investigate the basic properties of the filled flame retardant coatings based on zeolite-like cement matrices after artificial aging and fire exposure under standard fire conditions for their

use for fire protection for 3 hours of concrete tunnel structures.

To fulfill the goal, the following tasks we are supposed to them solved:

- determination of the bloating ability of flame retardant coatings after artificial aging;
- determination of coating protective properties concerning concrete under the conditions of standard fire development for 3 hours;
- determination of the basic physical-mechanical and thermal-physical properties of the coatings before and after fire action.

3 Materials and methods of research

3.1 Materials

To reach the goals, the dispersion based on zeolite-like cement matrices of the composition $\text{Na}_2\text{O} - \text{K}_2\text{O} - \text{Al}_2\text{O}_3 - \text{SiO}_2 - \text{H}_2\text{O}$ was used as the binder, the ratios of its structural oxides being as follows: $(0.8\text{Na}_2\text{O}+0.2\text{K}_2\text{O})/\text{Al}_2\text{O}_3 = 1.0\text{...}1.3$, $\text{SiO}_2/\text{Al}_2\text{O}_3 = 4\text{...}5$ and $\text{H}_2\text{O}/\text{Al}_2\text{O}_3 = 13\text{...}24$. The calculation of the ratios between oxides in the alkaline aluminosilicate binder for the synthesis of zeolites was carried out taking into account the recommendations given in [34, 35].

Also, the aluminosilicate pellets-based zeolite-like cement matrices of the $(0.8\text{Na}_2\text{O}+0.2\text{K}_2\text{O})\cdot\text{Al}_2\text{O}_3\cdot(4-5)\text{SiO}_2\cdot(13-24)\text{H}_2\text{O}$ composition and with a particle size of 0.63...4.0 mm were added as intumescent and fillers. These pellets were prepared by granulation of the alkaline aluminosilicate binder in the CaCl_2 -solution ($\rho = 1350 \text{ kg/m}^3$); the appearance of these pellets is shown in Fig. 1.

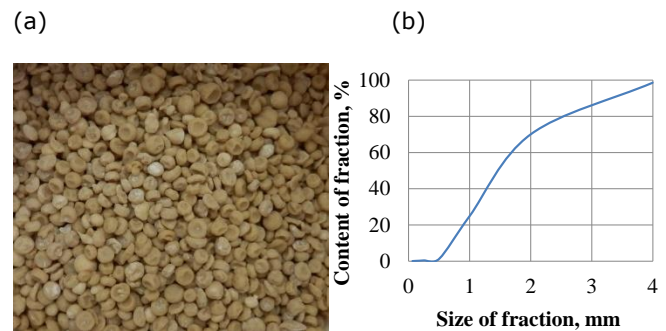


Figure 1 Appearance of the pellets (a) and their distribution by a fraction (b)

Metakaolin was used as a solid phase component of the binder. The composition of metakaolin, % by mass of mixture: $\text{CaO} - 0.52$, $\text{SiO}_2 - 53.67$, $\text{Al}_2\text{O}_3 - 43.61$, $\text{Fe}_2\text{O}_3 - 0.77$, $\text{MgO} - \text{traces}$, $\text{TiO}_2 - 0.74$, $\text{K}_2\text{O} - 0.75$, $\text{Na}_2\text{O} - 0.25$, other - 0.14, $\text{LOI} < 0.5$. The specific surface area of the ground metakaolin was 300...350 m^2/kg (by Blaine).

Sodium silicate solution with a silicate modulus $M_s = 3.05$ and $\rho = 1420 \pm 10 \text{ kg/m}^3$ was used as an alkaline component.

Modification of the zeolite-like cement matrices has been done by adding NaOH , KOH , and a rotten stone (by mass, %: $\text{CaO} - 3.6$, $\text{SiO}_2 - 88.4$, $\text{Al}_2\text{O}_3 - 2.3$, $\text{Fe}_2\text{O}_3 - 1.1$, $\text{MgO} - 0.9$, $\text{TiO}_2 - 0.2$, $\text{K}_2\text{O} - 0.9$, other - 0.6, $\text{LOI} < 2.0$). The

specific surface area of the ground rotten stone is 250...280 m²/kg (by Blaine).

Limestone (by mass, %: CaO – 44.15, SiO₂ – 7.6, Al₂O₃ – 2.92, Fe₂O₃ – 2.64, MgO – 2.89, TiO₂ – 0.22, K₂O – 1.18, LOI < 39.71) was used as an intumescent and a filler [36, 37]. Its specific surface area after grinding is 70...80 m²/kg (by Blaine).

3.2 Testing Methods

The coating, obtained after mixing the based on zeolite-like cement matrices with fillers in appropriate proportions, was applied by hand using a trowel to the vertical surface of concrete cube samples (150 mm) with a thickness of 6 mm. After the coating had cured, 2 TT-K-24-SLE type K thermocouples were installed, temperature range 20...1100 °C, accuracy ± 0.05 °C (Czech Republic). These three thermocouples (T_s , T_c , and T_a) (Fig. 2), connected to a KIMO HD 200 HT multifunction device (France), were inserted into specially drilled holes in the concrete samples to a depth of 75 mm and at a distance of 25 mm of reinforcement. A DT 8867H infrared pyrometer (Germany) was used to measure the temperature (T_s) on the surface of uncoated and coated concrete specimens as well as in the depth of the concrete specimen (temperature field distribution). A gasoline burner with a flame temperature at the coating surface of not more than 1100 °C was used in the fire test (Fig. 2). The distance of the burner flame to the coating surface was 200 mm.

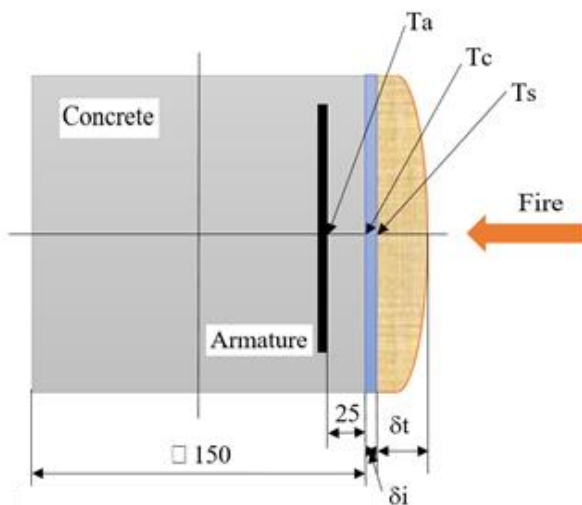


Figure 2 Location of thermocouples in standard fire tests: T_s – the temperature at the surface of the coating; T_c – the temperature at the surface of the concrete under the coating; T_a – temperature of the concrete at the depth of laying the reinforcement

The surface heating temperature T_s , which corresponds to the standard fire temperature, was determined by the formula [38] and provided by changing the distance of the flame of the gasoline burner within 160...200 mm:

$$T = 20 + 345 \log_{10}(8t + 1) \quad (1)$$

where T is the flame temperature, °C; t is the duration of thermal exposure during the fire test, min.

To determine the preservation of performance properties of flame retardant coatings on coatings based on zeolite-

like cement matrices, an accelerated aging method was applied, given in [39]. The tests were carried out on 6 samples, three main samples and three control samples were randomly selected. Three control samples were used to determine the flame retardant properties. The three main samples were consecutively kept for 8 h in a desiccator at 60±5 °C, 16 h in a desiccator with relative humidity of 100 % at normal temperature, 8 h in a desiccator at 60±5 °C, 16 h in normal conditions. These operations constitute one cycle, which corresponds to one year of operation under normal conditions. The tests included 10 cycles according to the specified scheme. At the end of the specified period, the samples were kept under normal conditions for at least 48 h.

Bulking capacity after aging and ageing the samples under normal conditions was determined by the formula:

$$P = \frac{(m_1 - m_2) \cdot 100}{m_1} \quad (2)$$

where m_1 is mass of the sample before tests, g; m_2 is mass of the sample after testing, g.

The coating is considered to have passed the aging resistance test if its integrity is preserved (no cracks, fractures, peeling, etc.) and the fire protection properties are reduced by no more than 20 % of the values determined for the control samples. In parallel, we tested 3 samples with coatings after exposure for 10 years in natural conditions.

Bloating coefficient (bloating factor), was determined by the formula:

$$F_s = \frac{\delta t}{\delta i} \quad (3)$$

where δt is the thickness of the blown coating, mm; δi is the thickness of the original coating, mm.

The thermal conductivity coefficient of the coating after the fire action was determined in steady-state mode by the thermal probe method on the device ITP-MG4 "250" (Ukraine).

The porosity of the foamed coatings was determined by the BET method using low-temperature nitrogen gas adsorption on a NOVA 2200e apparatus (Germany).

The adhesion strength of the fire-retardant coating before and after the fire action with the concrete surface was determined by tearing off the steel plates with the help of the adhesion meter ONIKS-1 AP (Ukraine).

Compressive strength of the coating after fire action was determined on samples-cubes with dimensions of 10x10x10 mm on a hydraulic press according to the standard procedure [40, 41].

4 Results and discussion

At the first stage of the research, the bloating ability of initial and filled flame retardant coatings based on zeolite-like cement matrices after artificial aging by indicators of

weight loss and changes in the bloating coefficient was determined. After the completion of tests a visual inspection of the surface of the coatings and, it was noted that in the unfilled (original) coating observed network of cracks, which is characteristic of the passage of processes of recrystallization of zeolite phases of heulandite-clinoptilolite type into the analcime phase, as a more thermodynamically stable [24, 26, 28, 34]. The coating mass loss at the end of the aging term was 7.8 %, which is 2.5 times less than the critical values indicated in the test requirements (Fig. 3).

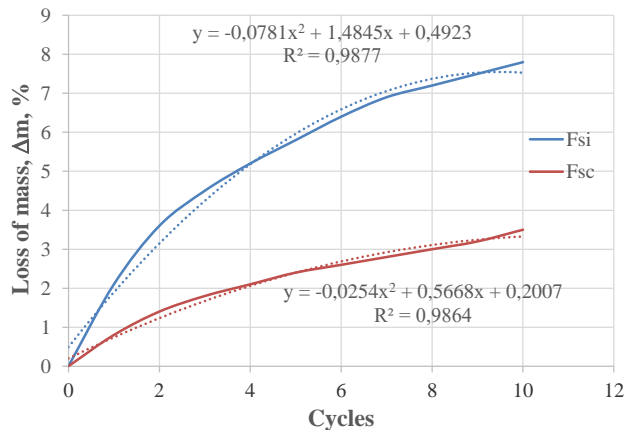


Figure 3 Weight loss of flame retardant coatings after 10 cycles of artificial aging

This means that the unfilled coating is capable of bloating. Visual inspection of the filled coating showed no cracks in the mesh, which noted a slight white patina on the surface, represented by sodium hydrogen carbonates [34]. The mass loss at the end of aging was 3.5% and equaled the equilibrium moisture content of the concrete substrate. The weight loss of the filled coating was 5.7 times less than the critical values outlined in the test requirements. This means that the filled coating has a sufficient reserve for bloating. The changes in the coating masses they quite correctly described by second-order polynomials.

Fire tests of the unfilled coating showed at the end of 10 cycles of artificial aging a 2.9-fold decrease in the swelling coefficient compared to the original (Fig. 4).

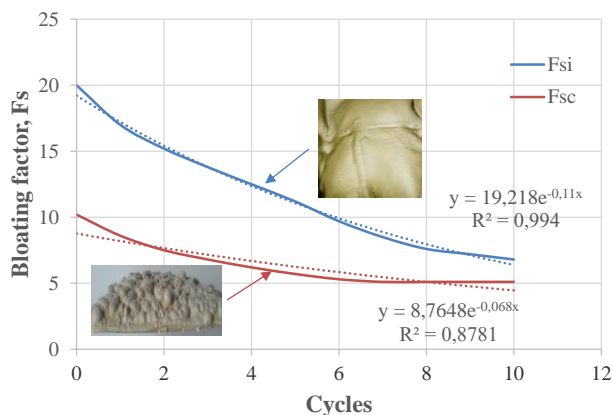


Figure 4 Change in the bloating coefficient of flame retardant coatings after 10 cycles of artificial aging

Externally, after temperature exposure, the aluminosilicate coating is a hollow bubble with an insignificantly thick

layer of porous aluminosilicate framework. This coating structure is not able to provide effective fire protection to concrete surfaces against fire. In the filled coating, at the end of 10 cycles of artificial aging, there was also a 2-fold decrease in the swelling coefficient compared to the initial one. However, the swelling of the coating occurs evenly over the entire area, and the formed highly porous aluminosilicate structure quite slows down the heat fluxes in the upper layers of the coating, leaving a reserve for the lower layers (the difference in the colors of the whiteness of the coating layers). This coating retains its fire-retardant effectiveness within 10 years or more. The data we also confirmed by the results of fire tests of samples that have been in storage under natural conditions. This ability they provided by the preservation of chemically bound water in capsules of aluminosilicate filler, the surface layer of which is protected by a film of water-soluble low-base calcium hydrosilicates [36]. Changes in the values of the coefficients of swelling of coatings they quite correctly described by exponential functions.

In the second stage of research, the fire protection efficiency of the filled coating based on zeolite-like cement matrices was determined under conditions as close as possible to the spread of a standard fire. The heating curves of a sample of unprotected concrete are shown (Fig. 5).

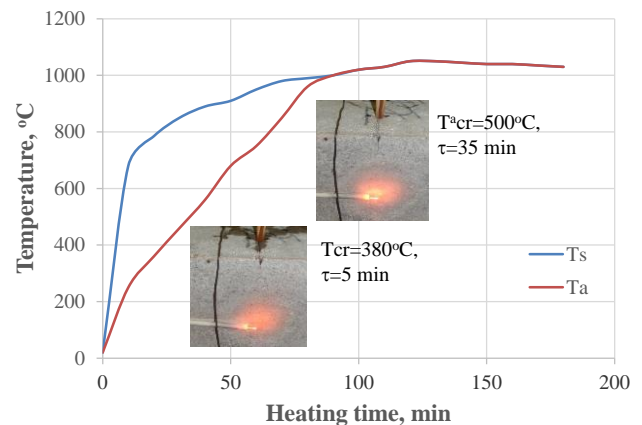


Figure 5 Temperature distribution over the depth of an uncoated concrete specimen depending on the duration of exposure to fire: T_s - surface temperature; T_a - the temperature at a distance of 25 mm from the exposure surface (at the depth of rebar embedding)

It can be seen from the figure that already at 5 min of test the surface of the concrete heated up to the limiting condition (critical temperature) – 380 °C, the concrete went into the mode of brittle failure (appearance of cracks), caused by a sharp release from the surface layers of water vapor. At the depth of occurrence of the reinforcement (25 mm from the surface of the concrete) heating up to the limiting condition (critical temperature) of the metal – 500 °C reached already on 35 min of the test. Traces of water vapor seeping from the deep layers of the concrete are also noted. The heating curves between 0...80 min of the fire test are different, which is due to the passage of dehydration of cement matrix hardening products occurring in the temperature range from 20 to 1000 °C. Further, in the interval from 80 to 180 min of the test, temperature equalization occurs both at the surface and at the depth of the reinforcement. The structure of the cement stone compacts, despite the presence of cracks.

The heating curves of a protected concrete specimen with a filled coating based on zeolite-like cement matrices with a thickness of 6 mm are shown (Fig. 6).

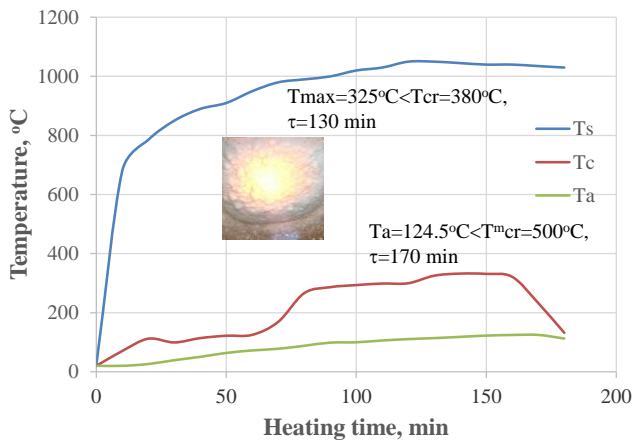


Figure 6 Temperature distribution along the depth of the concrete specimen with coating depending on the duration of exposure to fire: T_s - surface temperature; T_c - temperature under the coating; T_a - the temperature at a distance of 25 mm from the exposure surface (at the depth of rebar embedding)

The distribution of temperature fields during the test (Fig. 7) confirms the above laws, namely, that the temperature at the surface of the concrete is about 1000 °C, and at the depth of reinforcement laying – 500 °C, which is a critical value for the metal. It is noted that at 152 °C the coating begins to swell (Fig. 8 a). We are primarily interested in the temperature values under the coating and the reinforcement's depth.

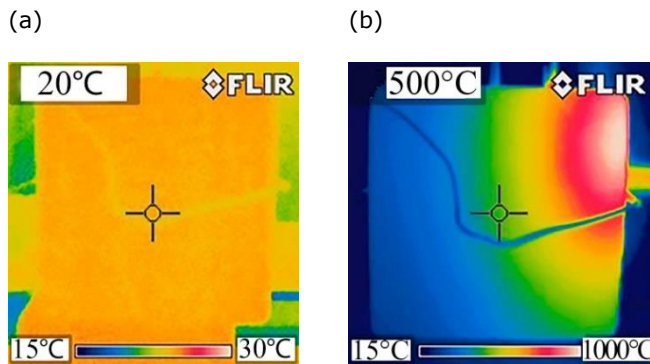


Figure 7 Changes of temperature fields in an uncoated concrete specimen as a function of fire exposure time: a - beginning of tests; b - when the critical surface temperature is reached ($T_{cr} = 380$ °C)

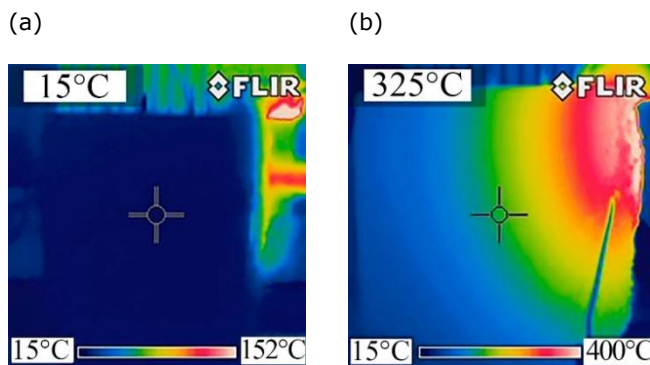


Figure 8 Changes of temperature fields in a coated concrete specimen depending on the time of fire exposure: a - beginning of tests; b - when reaching the critical surface temperature ($T_{cr} = 380$ °C)

During the fire test from 0 to 180 min, the temperature at the surface of the concrete did not exceed the heating limit of 380°C. The maximum value of temperature on the concrete substrate we recorded at 325°C at 135 min of the test, which is 1.2 times less than the limit (critical) value. At the depth of rebar placement (at a distance of 25 mm from the surface) during the fire test from 0 to 180 min, no values of the limiting temperature for the metal were recorded. The maximum temperature of rebar heating was 124.5 °C at 170 min of the test, which is 4 times less than the limiting (critical) value. The data are also confirmed by the distribution of temperature fields (Fig. 8 b), which record the values of the stated temperatures in the corresponding minutes of the fire test. The bloating coefficient is in the range of 2.0...5.1.

In the third stage of the research, we studied the physical-mechanical and thermal-physical properties of the protective coating after the completion of the fire test. It was shown that the porous aluminosilicate framework material has a compressive strength in the range of 2.3...4.5 MPa, cohesive strength of 1.2...1.5 MPa, and an adhesive strength (before fire exposure) in the range of 6.6 - 8.0 MPa. The coefficient of thermal conductivity of the coating is in the range of 0.042...0.066 W/(m·°C) at total porosity of 92...97 %.

5 Conclusions

The results of the study showed a possibility to protect concrete and reinforced concrete tunnel structures under the standard fire regimes by using the intumescent coating based on zeolite-like cement matrixes. The developed coating does not lose the ability to bloat and is characterized by a bloating coefficient from 2.0 to 5.1 with a weight loss of no more than 3.5 % after artificial aging for 10 cycles of alternate drying and cooling. The coating prevents the heating of surface structures of concrete and reinforcement of concrete to the limit state, i.e. 380 and 500 °C. The protective coating in a thickness of 6 mm provides fire resistance of concrete structures for 180 minutes with a service life of about 10 years. The average temperature of heating of concrete at the depth of the reinforcement (25 mm) is 124.5 °C, which is 4 times lower if compared with the limit temperature of heating of the metal armature. This effect is attributed to the formation of a porous aluminosilicate framework of low density with a coefficient of thermal conductivity in the range of 0.042...0.066 W/(m·°C) with total porosity of 92...97 %. The foamed aluminosilicate material is characterized by a compressive strength of 2.3...4.5 MPa as well as cohesive strength of 1.2...1.5 MPa. The adhesion to the concrete substrate of the cured intumescent fireproofing material prior to fire exposure is in the range of 6.6...8 MPa. The bloating coefficient of the coating without artificial aging is in the range of 15...20.

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