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Optimization of Effectiveness Evaluation Method for Intumescent Fire Retardant Coating

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Abstract. An optimized method for assessing the fire protection efficiency of intumescent coatings has been proposed, which can be applied during the development and research of new formulations of fire retardant compositions. To achieve this goal, a critical analysis of existing methods for evaluating the fire protection efficiency of intumescent fire retardant coatings has been conducted, both those approved by regulatory documents and those used by researchers for the fire protection agents effectiveness rapid assessments. Based on the analysis of the studied methods advantages and disadvantages, an optimized method for evaluating the intumescent fire-resistant coatings efficiency has been proposed to reduce the time for preparing and processing experimental results. The proposed optimized method involves the use of an electrical furnace with an insulated test chamber for heat accumulation as a source of thermal radiation, which allows obtaining temperatures on the reverse side of the metal plate exceeding 950 °C. As a criterion for fire protection efficiency, it is proposed to use the comparison of the time to reach the critical temperature (500 °C) on the outer side of metal plates protected by fire retardant coatings. The efficiency of fire protection of the metal plate has been investigated using the proposed method for three samples of intumescent fire protection agents: a coating based on epoxy oligomer, ammonium polyphosphate, aluminum hydroxide, and intercalated graphite, a coating on a styrene-acrylic basis of industrial production, and a well-known coating based on epoxy oligomer filled with monoammonium phosphate and intercalated graphite. The results of the experiment allowed a comparative assessment of the studied coatings fire protection efficiency. The use of the optimized method significantly simplifies the experiment and reduces the time spent on sample preparation and processing of its results.

Introduction

Metal structures are widely used in industry and construction. However, under fire conditions, unprotected metal structures lose their bearing capacity within 0.1 to 0.4 hours [1–4], since the critical temperature for structures made of various steels averages 470-550 °C, and for aluminum alloys – 165–225 °C. Thus, the fire protection efficiency of coatings for fire protection of metal structures will be determined by the time interval from the onset of temperature impact to the moment the structure reaches critical temperature [5–7]. The most promising way to achieve the regulatory fire resistance limit of building structures, which does not burden the structures and can be applied to complex configurations, is fire protection using intumescent fire retardant coatings (IFRC) [8–11]. These types of agents swell under flame action, forming a fireproof thermal insulation layer on the surface of the building structure. Intumescent fire retardant coatings are complex in terms of the interaction of their main components: film-forming agent, carbonizing agent, catalyst (acid source), carbon source, and blowing agent. This causes certain difficulties during research for developing new formulations of IFRC, particularly in studying the multiplicity of their swelling under flame temperature effects, the structure and strength of the formed coked layer, as well as fire protection efficiency. The complexity of developing new IFRC formulations lies in the necessity of preparing a large number of samples for processing the experimental plan and directly in the complexity of testing methods. This is especially true for research for determining the effectiveness of fire protection for building structures, particularly metal ones [12–14].

Thus, the imperfection of existing methods for assessing the efficiency of intumescent fire retardant coatings is a pressing issue.

Main Part

In the European Union, the classification of fire protection efficiency of intumescent coatings for bearing steel structures is based on EN 13381-8:2013, IDT "Test methods for determining the contribution to the fire resistance of structural members. Applied reactive protection to steel members", which corresponds to the state standard of Ukraine DSTU EN 13381-8:2022. This regulatory act governs the dependence of the fire protection material minimum thickness on the cross-section coefficient of the steel profile and the prescribed fire resistance limit for the bearing steel structure. The essence of the method is to heat a set of samples in a standard temperature regime with subsequent assessment of the test data using mathematical analysis methods to obtain the characteristics of the fire protection capacity of the coating. The method allows to evaluate the fire protection efficiency of fire retardant coatings under standard fire temperature conditions. During the tests, metal samples are placed in a fire furnace, and using thermocouples located at specified points, the temperature data of the samples are taken for varying durations of fire exposure without load. Based on the test results, dependencies are established between the temperature affecting the sample under study, the thickness of the fire protection material layer, and the limiting temperature of the structural material to which the fire retardant coating is applied.

The determination of the fire protection efficiency of intumescent coatings during the development and research of new formulations of fire protection materials according to standard EN 13381-8 is not always practical, as tests must be conducted on full-sized samples, which requires appropriate equipment, but can be used in the final phase of the experiment to confirm the efficiency of the developed fire protection coatings [15].

At the same time, a number of experimental studies [16, 17] are dedicated to determining the processes of thermochemical transformations of fire protection coatings and its fire protection efficiency using non-standard methodologies. Such methodologies also allow for the assessment of the fire protection materials effectiveness by determining the fire protection coefficient as a function of the coating thickness and the heating rate in the furnace. However, the use of a furnace and samples with complex configuration complicates the preparation for conducting the experiment and increases the time for processing its results.

The authors [18–20] present a methodology that can take into account different fire protection properties of intumescent coatings during tests at various temperature regimes. The model is based on experimental research conducted in a cone calorimeter. It has been shown that the method allows for the comparison of fire protection efficiency by determining the temperature of the plate on the opposite side to the heating device at different values of heat radiation from the heating element. The results of processing the experimental data allow to determine the thermal conductivity of the expanded coke layer of the fire retardant coating after its stabilization. One of the advantages of this method is the ability of studying the behavior of intumescent fire retardant coatings under different heating conditions that differ from the standard fire temperature regime. However, the issue of assessing the efficiency of the fire retardant coating by determining the time until the limiting state is reached due to the loss of thermal insulation capacity remains unresolved.

The regulatory document DSTU EN 13381-8 provides for determining the fire protection capability of expandable fire protection materials (which increase in volume during thermal exposure). The testing is conducted by determining the volumetric and/or linear expansion coefficient of the fire protection material. The assessment of the fire retardant material effectiveness can be determined by comparing the volumetric and/or linear expansion coefficients of the studied intumescent materials. Testing is carried out in an electric furnace at a fixed temperature of 340±5 °C, which is sufficient to initiate the physical-chemical transformations between the components of the intumescent materials but does not take into account the processes in the reactive environment at temperature, what are critical for steel building structures (470–550 °C) [21, 22].

In the paper [23], a comparison of temperatures after 30 min of flame impact from the burner on the reverse side of metal plates without fire protection agents and metal plates that were previously protected by the studied fire retardant agents was used as a criterion for the fire protective effectiveness of intumescent coatings. A gas burner with a maximum flame temperature of 1150 °C was used as a heat source. As the studies have shown, the tests are also conducted at a temperature regime that differs from the standard fire regime – the maximum temperature on the reverse side of even the unprotected metal plate does not exceed values of 470 °C.

The analysis indicates the impracticality of using existing methods when developing and researching new formulations of intumescent fire retardant coatings. This issue could be resolved by optimizing existing testing methods that would take into account the time it takes for critical temperatures to be reached by metal structures protected by the corresponding intumescent coatings.

The purpose of the work is to optimize the method for evaluating the fire protective effectiveness of intumescent coatings for use during the development and research of new formulations of fire protection compositions and to assess the possibility of using simplified methods for evaluating the fire protective effectiveness of intumescent fire retardant coatings in the process of its development. In this case, the proposed effectiveness criterion is the comparison of the time to reach the critical temperature (500 °C) on the outer side of metal plates protected by fire protective coatings.

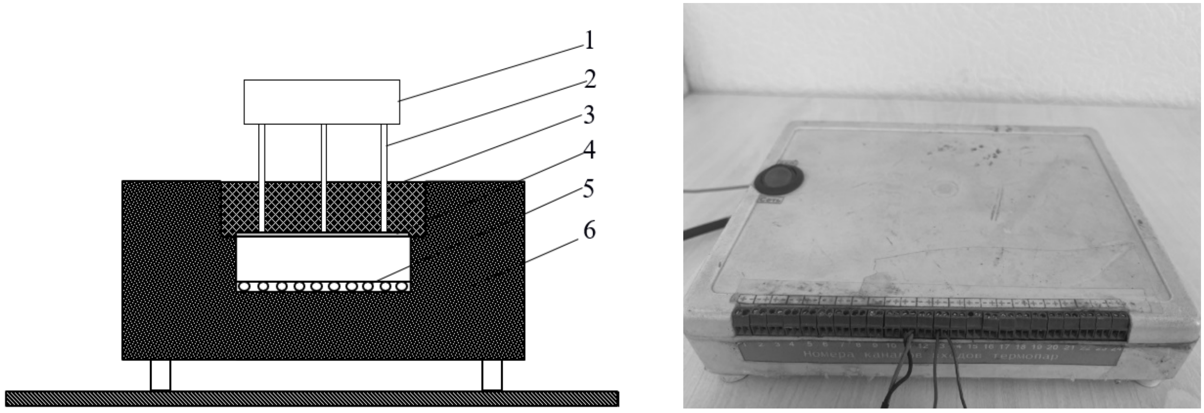
Materials and Methods of Research

The method described in [23] was chosen for optimization. Unlike the provided testing method, the effectiveness criterion chosen is not a comparison of temperatures on the reverse side of the metal plate, but a comparison of the time it takes to reach the critical temperature (500 °C) on the outer side of metal plates protected by fire retardant coatings. An electric furnace with an insulated test chamber, unlike [23], which promotes heat accumulation (Fig. 1, a), is proposed as the source of thermal radiation. The temperature regime of the furnace is slower than the standard temperature regime but allows achieving a temperature on the reverse side of the metal plate of over 950 °C, which is quite sufficient for a comparative evaluation of the fire protective effectiveness of intumescent coatings for metal structures. Research under such conditions is justified, as generally, with a decrease in the intensity of heating, the effectiveness of fire protection of intumescent coatings decreases, which is explained by the slowing down of the physical and chemical transformations between the components of intumescent coatings.

It is proposed to conduct tests on 3 samples of metal plates measuring 120×120×3 mm for each experiment.

Temperature measurements are proposed to be carried out using type L thermocouples and a twenty-four-channel temperature recording device (Fig. 1, b). One thermocouple is used to control the temperature inside the heating chamber. To control the temperature on the reverse side of the plate from the heating chamber, instead of one thermocouple, five thermocouples are proposed to be used, positioned at five points: the first in the geometric center of the sample, and the other four equidistant from the center point diagonally, at a distance equal to 0.25 times the length of that diagonal.

Testing procedure. The pre-prepared plate measuring 120×120×3 mm with the applied fire retardant coating (1 mm thick) is placed in the testing furnace and the thermocouples are fixed as shown in Fig. 1, a. Simultaneously with the activation of the electric heating elements, the temperature recording device is turned on. The test continues until the temperature on the outer side of the metal plate reaches 500 °C in at least 3 points.



a) b)

Fig. 1. Equipment used for the comparative evaluation of the fire protective effectiveness of intumescent coatings: a – scheme of the testing furnace: 1 – temperature recording device, 2 – thermocouples, 3 – sample holder block, 4 – test sample (plate coated with fire retardant agent), 5 – heating element, 6 – thermal insulation; b – temperature recording device.

To construct the dependence of the temperature on the external side of the plate over time, it is suggested to take the average temperature measured at five points averaged over the results of three tests. The assessment of the effectiveness of fire retardant coatings using the optimized method can be carried out by comparing the test results of the studied coating with the test results of other fire retardant coatings, provided that the tests are conducted under the same conditions.

Discussion

Obtaining intumescent coatings is usually achieved by using three components: a source of inorganic acid, a source of carbon, and a gas-producing agent.

The model system chosen for testing the optimized method consisted of samples of fire retardant coatings based on polymer film formers with different compositions of fire-retardant additives, obtained as a result of previous developments based on:

– epoxy oligomer filled with ammonium polyphosphate as a source of inorganic acid, aluminum hydroxide as a gas-producing agent, and intercalated graphite as an additional source of carbon (ER-1) [24, 25],

– epoxy oligomer filled with ammonium monophosphate as a source of inorganic acid and intercalated graphite (ER-2) [24, 25],

– styrene-acrylic dispersion filled with ammonium polyphosphate and pentaerythritol as a carbon source (SAD) [26, 27].

The dependence of temperature change inside the experimental plant (1), on the external side of the metal plate (2), and on the external side of the metal plate with experimental fire retardant coatings is presented in Fig. 2.

As seen from the presented research results (Fig. 2), the heating time of the external side of the unprotected metal plate is about 12 minutes. The use of a styrene-acrylic based coating allows this time to be delayed to 22.6 minutes. When using a coating based on an epoxy oligomer filled with ammonium monophosphate and intercalated graphite, the time to reach the critical temperature is 29.1 minutes. The use of a coating based on an epoxy oligomer filled with ammonium polyphosphate, aluminum hydroxide, and intercalated graphite for the fire protection of metal structures increases the heating time of the metal plate to 36.4 minutes, which is 1.3–1.6 times higher compared to other test samples.

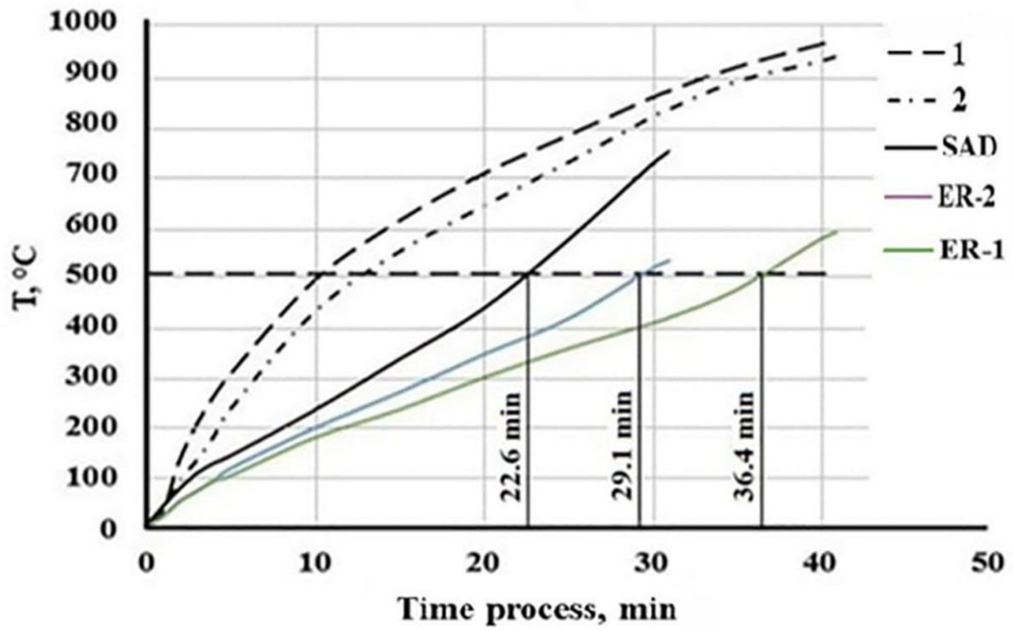


Fig. 2. Dependence of temperature change inside the experimental plant (1), on the external side of the metal plate (2) and also on the external side of the metal plate protected with fire protection coatings: SAD, ER-2, ER-1.

The specifics of transformations in coatings of this type indicate that during heating in modes slower than standard fire temperatures, there is a tendency for reduced fire protection effectiveness, which is due to the shift of temperature limits for synchronizing physicochemical processes in the condensed phase of the system «film former – source of inorganic acid – source of carbon – gas-producer» (ER-1) towards lower temperatures. The nature and consistency of the chemical reactions and physical processes at this stage are important, as they must occur in a certain sequence and at defined temperatures; only in this case can a strong thermal insulating carbon layer be formed. Therefore, testing under a temperature regime slower than the standard temperature is justified, as it more closely corresponds to the conditions typical of a real fire.

The use of an optimized method for evaluating the fire protection effectiveness of intumescent fire retardant coatings allows for a significant reduction in sample preparation time when planning and conducting the experiment. Using the heating time to critical temperature (500 °C) on the external side of the metal plate protected with fire protection coatings as the effectiveness criterion, significantly simplifies the experiment and reduces the time spent on processing its results. The use of an electric furnace with a temperature regime different from the standard fire regime allows, firstly, to assess the effectiveness of fire protection in conditions of slow fire development, and secondly, significantly simplifies the conditions of the experiment, which is extremely important during research aimed at the development and study of new formulations of intumescent fire retardant coatings.

Conclusion

The method for assessing the fire protection effectiveness of intumescent fire retardant coatings has been optimized. A critical analysis of existing methods for evaluating the fire protection effectiveness of intumescent fire retardant coatings has been carried out. It has been established that during research aimed at developing new fire retardant formulations, it is advisable to use simplified testing methods. The criterion for effectiveness in this case can be the time to reach the critical temperature (500 °C) on the outer side of the metal plate protected by the fire retardant coating. The use of the optimized method for assessing the fire protection effectiveness of intumescent fire retardant coatings, based on comparing the results of tests on metal samples protected by fire retardant compositions, has been proposed. The use of the optimized method allows for a reduction in the time required for sample preparation when planning and conducting the experiment, as well as investigating the effectiveness of intumescent fire retardant coatings under temperature regimes different from the standard fire regime.

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