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Fire Resistance Tests of Various Fire Protective Coatings

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Tests were carried out on more than 14 different samples of fire protective coatings in order to investigate a relation between the thickness of the intumescent fire protection coating and the time of exposure to heat. A number of coatings of different chemical composition enabled to determine the fire resistance behaviour patterns. During test the one-side and volumetric methods were employed in observance of the standard temperature-time curves. For one-side method, the coating was applied on one side and all edges of the specimen, whereas for volumetric test the specimens were completely covered with fire protective coating. It is shown that a layer of coating protects the specimen's surface from heat exposure for a certain period of time until full oxidation of the coating occurs. The efficiency of fire protective coatings also depends on thickness of the charred layer of the side exposed to heat.
Keywords: fire protective coatings, intumescent coatings, fire resistance, steel, thermoinsulative properties.

1. INTRODUCTION

Nowadays thermoinsulative materials find broad application in construction business: they are used both for insulating the buildings and increasing the fire-resistance of steel structures. Application of film-making materials is one of the ways to increase the fire-resistance of steel structures. Fire resistance tests are carried out in order to determine, the numeric values of the fire resistance of building structures with appropriate coating. Fire resistance is understood as ability of building structures to resist high temperature for a certain time under simulated fire environment. Where structural steel members are required to have enhanced fire resistance, they can be protected by applying insulating materials [1].

The most important factor in determining the efficiency of fire protective coating of structure is its resistance to the effect of standard fire temperature for a certain period of time. Fire resistance of building construction product is marked R and measured in minutes for example R15, R240 [2]. When exposed to intense fire even incombustible materials such as steel can be weakened, so that building constructions may be damaged and finally destroyed [3].

One of the most popular of such measures is fire protective paint (i. e., coating applied on steel surfaces) which forms a thin, hard and smooth film which under exposure to high temperature (400 °C) becomes intumescent expands to form a thermoinsulative layer of char and char foam [4, 5]. After expansion and simultaneous charring of the coating, a small-grain layer forms which, due to its low thermal conductivity, significantly delays the heating of metal structures.

A lot of organic solvent and water based paints have been developed with different components combinations [7]. Fire protective coatings intended for the protection of steel structures vary in thickness from 250 μm to several millimetres. A thickness of coating depends upon the required fire resistance class.

The use of fire protection coating on steel structures may prolong the fire resistance time from 15 to 120 minutes. The tests have demonstrated that fire resistance of the structure is directly dependent on the thickness of its coating. This theory was also confirmed by the tests made by applying intumescent coatings compositions in the powder form on to steel I-sections [8]. Different polymers binders influence to the formation of foam was examined and tested on aliuminium plates in international patent WO 2004/061020 A1, results show that chemical reactions that take place during the formation of coating also are involved [9]. It was observed that the addition of different particles to these coatings changes the char formation process, the height, the mass, the structure and also the fracture behaviour of the protecting shield, and therefore the fire protection of the expanded char [10].

Determination of fire resistance of building structures in the European Union is regulated by a number of standards. The main criteria are provided in EN 1363-1:2004. This standard contains a description of the test equipment and main conditions of testing with permitted tolerances [11]. However, the differences in test results are conditioned by different test equipment and skills [12].

The main instruments used in the test: a furnace (3 m × 4 m × 6 m) capable to ensure the necessary test conditions, equipment for applying load (if applicable), thermocouples used for measuring the mean temperature of the specimen and furnace interior. Chromel/Alumel type thermocouples were used to measure specimen surface temperature and plate type thermocouples were used to measure temperature in the furnace [13].

The increase of temperature in the furnace should remain within the limits of permitted tolerances and conform to the below dependency [11, 14].

$$T - T_0 = 345 \lg(8t + 1), \tag{1}$$

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where t is the time in minutes, T is the furnace temperature (°C) within time t, T_0 is the initial furnace temperature (°C).

The main criterion in determining the fire resistance of building structures is the time within which the member of structure achieves critical temperature and is no longer suitable for its function. In Lithuania, the critical temperature for steel structures is 500 °C, whereas, for instance, in Great Britain the critical temperature for steel columns is 550 °C and for steel beams 620 °C [15, 16]. In Lithuania, like in a number of other European Union member states, the fire resistance of steel structures is evaluated according to standard ENV 13381-4:2003 [17]. The standard contains a description of the test methods and procedure of calculations. Properties of the fire protective coating may be evaluated after performing calculations with the obtained test results. Calculations may be performed using one of the four methods described in the standard. It should be noted that the results of calculations performed in different methods are not identical. Open profile members of structures are used in the test, since open profile enables to more consistently evaluate the thermoinsulative properties of coatings [17]. Studies made on steel plates with different content of fire retardant additives (pentaerythrihol, 5,5-dihydroxymethyl-1,3-dioxane, monopentaerythritol) in the fire protective paints, show that at least 25 % of weight should be in the fire protective paints, but these test were performed with the uncontrolled heat flame Bunsen burner [18].

Main components of the tested fire protective coatings are [19]:

– synthetic binders (based on vinyl acetate, vinyl chloride and other homopolymer and copolymer resins, chloroparaffin, trichlorethylphosphate, chlorinated resins and various halogen-containing copolymer substances);

– fire retardant additives (ammonium, orthophosphates, boron acid, ammonium polyphosphates, pentaerythrite);

– functional additives (starch, dextrin, sacharosis, dicyandiamide, melanin, titanium oxide, zinc oxide, silica oxide).

A bigger part of scientific works deal with the investigation based on different chemicals compositions of intumescent paints to get better fire resistance results [4 – 10, 18 – 20]. Object of the test are intumescent thin-film forming fire protective coatings. Film forming substances for the test were provided by representatives of manufacturers or purchased at the points of sale: Steelguard FM 550 and Steelguard FM 585 (PPG Protective Coatings, Netherlands), Teknosafe 100 and Teknosafe 2002-00 (Teknos Oy, Finland), Interchar 404 and Interchar 963 (International Farg AB, Sweden), Char21 and Char22 (Iris Vernici S.r.l., Italy), Pyro-tech SPX (E. Wood Limited, United Kingdom), Sika Unitherm ASR (Sika Korrosionsschutz GmbH, Germany), Protherm Steel (Italvis protection s.r.l., Italy), Nonfire S104 (Tikkurila Coatings B.V., Netherlands), Pyroplast-ST 200 and Pyroplast-HW (Rutgers Organics GmbH, Germany).

Aim of the current study is:

– to evaluate the changes in temperature of fire protective coatings using one-side and volumetric methods of heating;

– to determine the relationship between the thickness of foam layer and the time of achieving a certain temperature using one-side and volumetric methods of heating.

2. SPECIMENS AND TEST METHODS

Commonly, thermoinsulative properties of fire protective coatings are evaluated by performing tests during which fire protected structures are exposed to high temperatures. In the event of fire in a premise, the structures are exposed to flame from one side or several sides and only after the spread of fire the structures are exposed to fire from all sides. Low consumption of time and costs of test are essential when developing or improving fire retardant coatings; for this purpose small size furnaces are suitable.

2.1. Description of specimens

Specimen plates (200 mm × 250 mm) were cut out using guillotine from 1 mm thickness steel sheet (steel grade St3) in order to obtain the largest solidity ratio of the cross section (3600 m^{-1}). Calibrated Vernier calliper and ruler were used to measure the side lengths and thickness. The said thickness was selected subject to obtaining the minimum thermal capacity which would ensure the maximum precision of temperature recordings. Moreover, the selected ratio of cross section minimises the effect of thermal capacity of the steel plate. The ratio of cross section was calculated according to the instructions provided in Clause 4.2.5.1 LST L ENV 1993-1-2:2000 [21] and is as follows:

$$A_p/V = 0.9 \div 0.0025 = 3600\ \text{m}^{-1}, \qquad (2)$$

where A_p is the cross section perimeter of the member of structure, m; V is the cross section area of the member of structure, m^2.

Alkyd anticorrosive primer GF-021 (red colour) was used to enhance the binding of the steel plate surface with fire retardant paint. The primer is used on wooden and metal surfaces before applying paint. Commonly, the output for one layer is (60 – 100) g/m^2. Drying time of one layer is 24 hours at 20 °C ±2 °C and 65 % relative humidity. The plates were cleaned and degreased with dissolvent “Skiediklis 646”. After the traces of dissolvent evaporated, the plates were sprayed with anticorrosive primer “GF-021” (output 150 g/m^2, dry layer thickness 40 μm – 50 μm). Film-forming material “GF-021” forms a film based on glyphtalic resin. The primed plates were inserted into drying chamber with preset temperature 25 °C ±2 °C. After 24 hours the plates were taken out of the drying chamber, visually inspected for coating damages and left for 180 hours in the conditioning room at 20 °C ±2 °C temperature and 65 % ±5 % relative humidity. After conditioning, the plates were taken out and measured in 24 points to record the mean thickness of the primer coating. The permitted variation range is from 40 μm up to 50 μm. Coating thickness measurements were made using magnetic induction device “Mega Check“ operating on non-destructive measurement technology.

The device is used to measure thickness of impermeable and dry film applied on magnetic metal base.

Measuring device accurate is ±4 mm, when the total thickness is up to 100 mm, ±1 % when the coating thickness from 100 mm to 1000 mm and ±3 % where the total thickness is more than 1000 mm. Prior to measurement the device was calibrated according to the manufacturer's instructions using a proper calibration standard.

For one-side test, the plates were coated on one side and edges, and for volumetric test the plates were coated on both sides and edges. Fourteen fire protective intumescent coatings with different compositions and different solvent base were used for testing on the plates.

Each type of coating was applied on 3 steel plates. The coated plates were put into the drying chamber for 24 hours and then kept in the conditioning room for approximately 7 days. After conditioning the paint coating has completely hardened. A thickness of coating was measured and varied from 980 μm to 1050 μm. Fig. 1 shows the steps of preparing the plates.

a b c

Fig. 1. Steps of preparing the steel plates for test: a – clean steel plate; b – primed plate; c – plate after application of fire protective coating

The tests were performed on steel plates of the same thickness coated with from 40 μm up to 50 μm layer of anticorrosive primer and from 980 μm up to 1050 μm layer of fire protective coating.

Testing was stopped and time recorded after reaching 500 °C on the unexposed side. After taking out the plate from the furnace, the intumescent coating thickness was measured at twelve points.

2.2 Test equipment

One-side heating method was selected in order to determine the thermoinsulative properties of fire protective paint under exposure to temperature [12], i. e., the various structures were tested by this method, when one side is exposed to heat [22, 23]. This method of testing building materials was first used in the nineties and was intended to predict the fire resistance of various structures by several scientists [12, 24, 25]. One-side heating method more realistically simulates the environment of fire than Bunsen burner flame [18]. This method was also used to evaluate the thermal conductivity properties of building materials [12]. The test is based on one-side heating of the specimen in observance of the temperature regime and recording the time of reaching critical temperature (500 °C) on the unexposed side of the plate, since steel structures lose their strength properties after reaching 500 °C [26]. A basic scheme of the equipment is shown in Fig. 2.

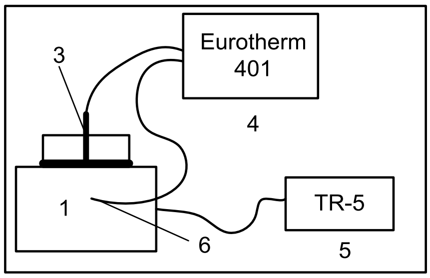


Fig. 2. Basic scheme of the equipment for one-side heating: 1 – one–side heating furnace; 2 – specimen with holder; 3 – thermocouple for measuring specimen temperature on the unexposed side; 4 – Eurotherm 401 device for recording thermocouple readings; 5 – TR-5 furnace temperature regulator; 6 – thermocouple for measuring furnace temperature

When performing tests based on volumetric principle, the furnace doors were installed in the furnace to replace the specimen holder. Throughout the test, the standard temperature was maintained in the furnace according to the dependency (1). The temperature of the furnace interior was measured using K type thermocouples (Ø 0.8 mm) subject to the permitted tolerances. The permitted tolerances of thermocouple readings: 1.5 °C in the range from 40 °C up to 375 °C and 0.4 % in the range from 375 °C up to 1300 °C. The temperature was recorded using "Eurotherm 401" device which can measure and record readings in 1 s intervals.

Our investigation has shown that the structure of thermocouple used for measuring has a considerable impact on the temperatures of the standard fire curve. To get more precise results of the specimen temperature we measured it with wire thermocouples.

The volumetric tests have shown that correct selection of the primer layer thickness is of great importance (Fig. 3 and Fig. 4).

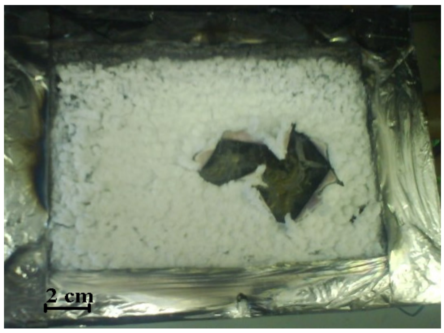


Fig. 3. Specimen after test with damaged fire protective coating top view

If thickness of the primer layer is about 150 μm and thickness of the solvent-borne coating Steelguard FM 550 (PPG Protective Coatings, Netherlands) is 160 μm, the localised decomposition of the coating occurred rapidly after reaching the critical temperature in 15 minutes.

A thickness of the coating has effect on the time of fire resistance: the thicker is the coating, the later is the critical temperature reached.

Fig. 4. Specimen after test with damaged fire protective coating side view

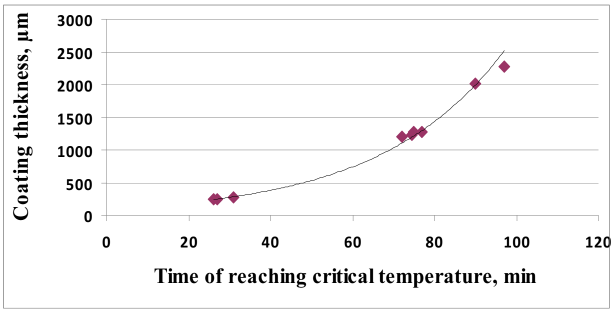


Fig. 5. Relationship between the thickness of coating and the fire resistance of a steel plate

Further tests were performed in order to optimise the thickness of fire protective coatings in the case of one-side heating when the plate is coated on one side and all edges. As the test results presented in Fig. 5 show, the dependency is of parabola type, where the thickness of the primer and coating varies from 300 µm up to 2400 µm. Thus, irrespective of the fire resistance of the structure itself, R90 is the maximum fire resistance value that can be reached.

3. EXPERIMENTAL RESULTS AND DISCUSSION

The fire resistance tests results which best reflect the differences between volumetric and one-side heating tests are presented in the tables below. Steel plates were covered with solvent-borne coating CHAR22 (Iris Vernici S.r.l., Italy), water-borne coatings Steelguard FM 585 (PPG Protective Coatings, Netherlands) and TEKNOSAFE 2002-00 (Teknos Oy, Finland). From Table 1 it can be observed that differences between the results of tests using different methods become more evident after reaching 200 °C. Specimens with the same thicknesses of coating temperature rise in the volumetric heating test shows, that up to 200 °C is near the same [9]. The tests performed with coating CHAR 22 (Table 2) also show the same behaviour, except for the time of reaching the critical temperature, which is different for the two coatings tested.

Also it was stated with intumescent coating compositions in the powder form, actual film thicknesses varies from 500 µm up to 680 µm, time of reaching a mean temperature of 550 °C was from 23 minutes up to 30 minutes [8].

When comparing the results of tests performed using one-side heating method and volumetric heating method, the initial temperature obtained during one-side heating is lower, but subsequently it starts rapidly increasing until protective foam layer is completely formed, however, final results show a considerable difference of ≈12 min.

Table 1. Temperature changes on the surface of steel plates with Steelguard FM 585 coating, using different heating regimes

	One-side heating			Volumetric heating		
	Coating applied on one side and edges			Coating applied on both sides and edges		
Specimen No.	I	II	III	I	II	III
Initial *T*, °C	19	22	20	18	21	20
Time of reaching *T* = 100 °C (min)	3.75	3.5	3.45	1.5	2	1.75
Time of reaching *T* = 200 °C (min)	8.5	8.25	8.25	3.5	4	3.75
Time of reaching *T* = 250 °C (min)	15	14.5	14.75	5	6,5	6
Time of reaching *T* = 300 °C (min)	25	24.75	23	13	14	13
Time of reaching *T* = 500 °C (min)	59	60	59	48	46	47.5

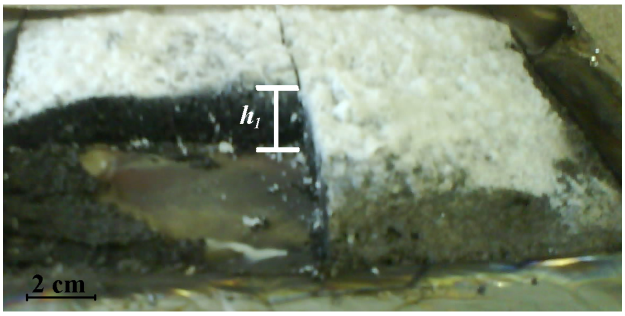
Table 2. Temperature changes on the surface of steel plates with CHAR 22 coating, using different heating regimes

	One-side heating			Volumetric heating		
	Coating applied on one side and edges			Coating applied on both sides and edges		
Specimen No.	I	II	III	I	II	III
Initial *T*, °C	19	22	20	18	21	20
Time of reaching *T* = 100 °C (min)	3.75	3.5	3.5	2	1.5	1.75
Time of reaching *T* = 200 °C (min)	9	8.75	8	5,5	3	3.25
Time of reaching *T* = 250 °C (min)	11.75	11.5	11	6.75	4	4.5
Time of reaching *T* = 300 °C (min)	16.5	16.25	15.75	8	6	5.75
Time of reaching *T* = 500 °C (min)	35	34.5	35.5	24	21.25	20.5

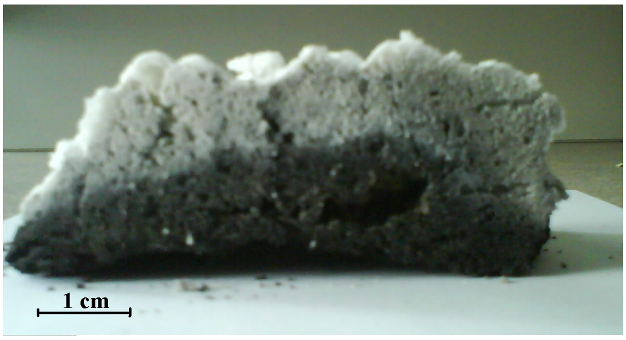
It can be stated that during one-side heating test before 250 °C is reached, the behaviour of temperature change is the same irrespective of the pattern of the coating application. The situation is different in the range from 300 °C up to 500 °C, because fire protective coating foam has been totally formed and the layers of foam protect steel surface from the heat. The three stages (stability stage 20 °C – 230 °C, formation stage of char layer 230 °C – 450 °C, stage of char loss 455 °C – 760 °C) of intumescent paint working mechanism were described by Zhenyu et al in [27]. When using one-side heating on the coating applied on the inner side, the results of the temperature change are close to those of the volumetric heating, whereas, when using one-side heating on the coating applied on all sides and edges, it is evident that the fire resistance time starts increasing (Table 3).

Table 3. Temperature changes on the surface of steel plates with TEKNOSAFE 2002-00 coating, using different heating regimes and methods of coating application

	One-side heating						Volumetric heating		
	Coating applied on one side and edges			Coating applied on both sides and edges			Coating applied on both sides and edges		
Specimen No.	I	II	III	I	II	III	I	II	III
Initial T, °C	20	21	19	21	20	21	20	22	19
Time of reaching T = 100 °C (min)	5	6	5.5	6.5	6.75	7	3.25	4	3.5
Time of reaching T = 200 °C (min)	9.3	10.5	10.2	10.2	10.7	11	4.8	6.1	5.6
Time of reaching T = 250 °C (min)	11.9	13.2	12.4	12.8	13.7	14.2	6.8	8.3	7.5
Time of reaching T = 300 °C (min)	13.5	14.7	14.2	18	18.7	19.5	12	13.3	12.8
Time of reaching T = 500 °C (min)	38.3	37	32.7	49	47.1	51.4	36.5	39	38.3



a



b

Fig. 6. Fire protective coating after exposure to high temperature: a – thickness of the formed coating h_1 is 22 mm; b – a piece of foam cut out of the specimen

Such behaviour can be explained by the fact that the fire protective coating applied on the unexposed side also starts to intumesce, although such intumescence is weak and the layer is not subject to charring or destruction. The obtained results have demonstrated that the increase in fire resistance time is significant in the case of one-side heating and full coating of the steel plate. Thus, the test results are influenced not only by different principles of heating, but also by the coated area of specimens. In order to better assess the thermoinsulative properties of different fire protective coatings was choosen one side steel plate covering and heating from covered side.

Fig. 6, a, shows the effect of heating on fire protective coating. From Fig. 6, b, it is evident that the coating is made of two layers: top (white) layer is non-consolidated, whereas the lower layer is sufficiently consolidated. This can be explained by the fact that the top layer is fully burnt-out (oxidised) and has only mineral components left in it, whereas the grey layer is not fully burnt-out and is partially charred.

The linear dependency model has been chosen for data processing [28, 29]. The coefficient of correlation $R = 0.917$, coefficient of determination $R^2 = 0.8426$, allowed tolerance $S_e = 4.997$ min. The following equation of the regression line has been obtained.

$$y = 1.2667x + 0.3364\ . \qquad (3)$$

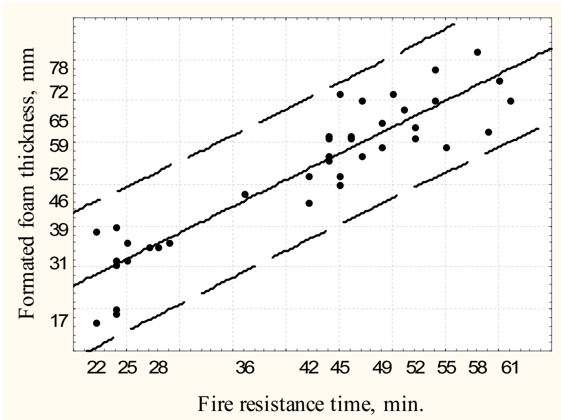


Fig. 7. Results of the fire protective coatings one-side heating tests

In Fig. 7 we can see the effect of the formed layer of foam on the time of reaching the critical temperature under identical test conditions and using the same thickness of coating. After heating fourteen different samples of fire protective coatings, the thickness of the formed foam layer is within the range of 17 mm – 82 mm and fire resistance time was from 22 minutes up to 61 minutes. Such a wide range can be explained only by the existence of effective components in the given coatings.

4. CONCLUSIONS

The pattern of the fire protective coating application and the selected method of test have influence on the results of tests of fire protective coatings where one-side and volumetric methods of heating are used. Volumetric method of heating is more strict, because exposed to fire is a larger area of specimen so the fire resistance is less than specimens of one-side heated tests. For small scale testing it is usefull to start testing from steel plate test to know primary values of fire protective coating characteristics.

The thickness sweeled foam of fire protective coating work as termoinsulation layer and is directly proportional to the fire resistance of the steel plate. Influence of thickness coating to thermoinsulative properties is not directly proportional, because not all layers of coating become foam at the same time. Most efficient thickness of fire protective coating is up to 1 mm.

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