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BEHAVIOUR OF AN INTUMESCING SYSTEM SUBJECTED TO DIFFERENT HEATING CONDITIONS

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ABSTRACT

Previous studies have indicated that the expansion of fire seals and fire stoppers will be dependent on the heat exposure. Standardized methods for classification includes exposure to a rapid heat exposure of the product that is tested, but this might not always be the worst case scenario for the product. In this paper a series of tests are presented in order to study how a graphite based intumescing system, intended for cavities, reacts when subjected to fire conditions different form that in the standardised method EN 1366-4. Two different experimental setups, a cone calorimeter at Lund University and a small-scale furnace at SP Fire Technology, were used in the tests.

The start of expansion reaction in the tested fire stopper was around 180°C. The start of reaction temperature is rather independent of the incident heat flux, while expansion rate is clearly dependent on the incident heat flux. Furthermore, the studied fire stopper performed well in the small-scale furnace because the graphite expanded enough to give the same protection, although it is preheated or wet, as when subjected to a heat exposure similar to ISO 834. These results are good but they are only considered valid for the studied system and for the given situation. Future research is needed in order to study how other types of intumescing fire protection systems work when subjected to realistic fire environments.

INTRODUCTION

Intumescing systems can be used in fire seals around fire doors, in dampers around plastic pipes, in cavities or to close small ventilation openings. In a fire it is intended that a material in the fire stopper will char and expand as a result of the heat exposure. The material increases in volume and decreases in density and the expansion can be 4-10 times the original thickness. The expanded material will have a low thermal conductivity, which reduces the heat transfer. There are a number of materials that exhibit intumescent behaviour for example: Ammonium Phosphate, Hydrated Sodium Silicate and Intercalated Graphite.

Fire stoppers are included in a variety of products tested according to standards like ISO 834 or ASTM E119 to be able to be classified with a fire resistance rating. Also the function of the product in a standardised wall setup can be tested according to the linear joint seals standard, EN 1366-4. When testing according to this standard a rapid heating of the product that is tested is achieved by applying a minimum positive pressure of 15 Pa when mounted in a vertical construction or during a horizontal test a positive pressure of 20 ±3 Pa 100 mm below the lowest point of the construction is prescribed. This positive pressure gives a rapid flow of hot gasses from the furnace heated with the standard fire curve which leads to a fast activation of the intumescing system. In a real fire the heating rate that the fire stopper is subjected to can differ very much depending on the type of fire. A slowly growing fire that e.g. starts in wooden materials will gradually increase the temperature in the fire stopper, while in a fast growing fire, that for example starts in flammable liquids or upholstered furniture, can a rapid temperature increase occur in the material. In most cases the classification test
will provide a worst-case scenario but cases where the foaming behaviour is sensible to the speed of activation may exist.

A special application of intumescent systems is to use them to prevent fire spread from the outside of a building to cavities or the building attic through ventilation openings. Exterior fires caused by arson cause a lot of damage when the fire spreads into the construction or attic. In several cases this type of fire has caused total destruction of schools and kindergartens in Sweden during the last couple of years. There are several approaches that can be taken in order to prevent and/or mitigate the consequences of this kind of fire and such technical measures have been identified and studied in a research project at Lund University. Real scale fire experiments have been preformed as a part of that research project. Several detection systems were tested in that experiments along with a graphite based intumescing system fitted in a ventilation opening in the eave. However, the systems did not close ventilation openings to the attic when it was slowly heated by a moderate fire (50-250 kW), placed near the façade wall. This implies that some intumescing systems might not behave as well in actual fires as when heated in accordance to more rapid heating at the standard fire curve, like ISO 834. In a previous study by Adl-Zarrabi both graphite based and sodium silicate based intumescent fires seals where subjected to different heating conditions. The purpose was to study if the rate of temperature increase affected the expansion of the materials. It was found that the expansion ratio for the graphite-based samples were 25% lower for a heating rate of 5°C/min compared with 10°C/min, this difference was not found for the sodium silicate based samples. Adl-Zarrabi also studied how water content affects the expansion of fire seals and it was seen that the expansion was reduced with up to 50% for some types of expanded graphite. These results indicate that the performance of graphite based intumescing systems can be dependent on the temperature increase and moisture content. But since only single material samples were tested and not an entire system it is not possible to know how such a systems will perform when applied in actual building elements and exposed to a realistic fire environment, where temperature, pressure and turbulence conditions might be different.

The purpose of this study is to investigate if the performance of a graphite based intumescing fire stopper for cavities will be affected when subjected to a more realistic fire environment than the heat exposure stated in standardized for classification. The fire stopper consists of a strip of graphite with a PVA polymeric binder that is held in place by a metallic net that have been tested according to the linear joint seals standard, EN 1366-4 with good results.

**METHOD**

Two experimental setups; a cone calorimeter according to ISO 5660 at Lund University and a small-scale furnace at SP Fire Technology, SP Fire 119, have been used to study the performance of the intumescent system.

**Cone calorimeter**
The cone calorimeter was used in the first setup in order to investigate how a strip of graphite used in the fire stopper responds to different heat fluxes. Four different heat fluxes were used; 10, 15, 20 and 25 kW/m². The sample was placed on a fibre silicate board and placed in the sample holder with retainer frame. The area below the sample was filled with ceramic wool. No wire grid to restrain the sample was used. The temperature at to the sample surface was measured with a 0,26 mm thermocouple (Type K) placed in the centre of the sample. Each test lasted for 8 minutes. The sample was video recorded and the time to reaction was estimated visually. The tests were conducted during one day and repeated a couple of days later. The experiment had two purposes; the first was to gain an understanding of how the graphite behaves when subjected to different heating conditions and if it influences the expansion of the material. The second purpose was to see if there is a single critical temperature at which the tested graphite starts to expand.

**Small-scale furnace**
A small-scale furnace was used in the second experimental setup. The fire stopper system was placed in a slot (0.3 m long and 0.04 m wide) between blocks of light weight cellular concrete on top
of the furnace (see Figure 1) in order to give a representation of a vertical cavity in an eave or similar construction. The temperature in the furnace was controlled and kept at different pre-defined heating conditions according to Table 1. Eight different tests were performed and duration of each test was mainly one hour.

Table 1: Description of the tests conducted in the furnace experiments.

<table>
<thead>
<tr>
<th>Test</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Standard time temperature fire curve with no cavity vent in place.</td>
</tr>
<tr>
<td>2</td>
<td>Standard time temperature fire curve with cavity vent in place.</td>
</tr>
<tr>
<td>3</td>
<td>Temperature increase of 10 °C/min during 50 min, then a more rapid increase (40 °C/min)</td>
</tr>
<tr>
<td>4</td>
<td>Temperature increase of 4 °C/min during 40 min, then a more rapid increase (approximately according to the standard time temperature fire curve)</td>
</tr>
<tr>
<td>5</td>
<td>Steady temperature of 150°C during 40 min, then more rapid increase (approximately according to the standard time temperature fire curve)</td>
</tr>
<tr>
<td>6</td>
<td>Steady temperature of 180°C during 40 min, then more rapid increase (approximately according to the standard time temperature fire curve)</td>
</tr>
<tr>
<td>7</td>
<td>The cavity vent was placed in water for approximately 70 hours. The graphite gained 16% in weight. Then it was subjected to I standard time temperature fire curve in the model furnace.</td>
</tr>
<tr>
<td>8</td>
<td>After 5 minutes of pre heating with temperature less than the foaming temperature a rapid temperature and pressure rise was achieved. This test was designed to see if it was possible to blow away the sealing during the foaming process.</td>
</tr>
</tbody>
</table>

The purpose of the different tests was to subject the fire stopper to both realistic and extreme heat exposures in another sense than the thermal exposure and pressure specified in tests according to the linear joint seals standard, EN 1366-4 \(^5\) (the thermal exposure in this standard is the standard time temperature fire curve). Parameters to regulate the exposure were the temperature in the furnace and the static pressure. The latter was governing the flow in the cavity where the fire stopper was mounted.

Figure 1: Experimental setup in small-scale furnace experiments.
Two thermocouples, mounted to the metallic net, and a plate thermometer (PT_2 in Figure 1) were used to study the temperature increase on the unexposed side of the fire stopper (marked X in Figure 1). Two plate thermometers (PT_1 and PT_3 in Figure 1) and a thermocouple were used to control and monitor the temperature in the furnace and two more thermocouples were attached to the metallic net on the exposed side of the system. Visual observations of the expansion of the fire stopper were conducted during the measurements.

Small pieces of wood and cotton were placed on the unexposed side of the fire stopper in order to study the possibility to ignite material on the unexposed side of the system.

RESULTS

Cone calorimeter
There was a great difference in how much the graphite expanded when exposed to different heat fluxes. But, as the graphite expanded it also came closer to the cone in the calorimeter, which means that the incident heat flux increased. In the tests with 25 kW/m² the vertical expansion was limited after a while because the conical heater obstructed the graphite. The final expansions for three of the tests after eight minutes of exposure are shown in Figure 2.

![Figure 2: Expansion for test with 10, 15 and 20 kW/m².](image)

The measured temperatures for each on of the tests in the cone calorimeter are presented in Figure 3.

![Figure 3: Measured temperature at surface of the strip for different heat fluxes.](image)

The temperature measurement is rather unreliable, for the higher heat fluxes due to the poor repeatability, as can be seen in Figure 3. However, the repeatability of the lowest fluxes (10 kW/m² and 15 kW/m²) is considered to be good.

The thermocouple was not properly fixed during experiment 20_1, this caused the thermocouple to
measure the temperature under the sample and this is probably the reason for the low measured temperature in experiment 20_1. It is not relevant to study the temperature after the graphite starts to expand because the thermocouple will be embedded in the graphite. It can be seen in Figure 3 that the temperature levels of around 200°C in many of the experiments, this is due to that the thermocouple is embedded in the graphite.

Table 2: Time to reaction of the samples and estimate total incident heat at reaction time.

<table>
<thead>
<tr>
<th>Incident heat flux (kW/m²)</th>
<th>Time to start of reaction (s)</th>
<th>Incident heat (kJ/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Test 1</td>
<td>Test 2</td>
</tr>
<tr>
<td>10</td>
<td>105</td>
<td>105</td>
</tr>
<tr>
<td>15</td>
<td>75</td>
<td>60</td>
</tr>
<tr>
<td>20</td>
<td>55</td>
<td>45</td>
</tr>
<tr>
<td>25</td>
<td>40</td>
<td>30</td>
</tr>
</tbody>
</table>

It is not possible to see that the estimate total incident energy at the reaction time differs between the experiments. The second test with 25 kW/m² (25_2) deviates somewhat, but if it is excluded: the total incident energy at the reaction time is within the range 900-1100 kJ/m². It is known that the incident heat towards the sample is not equal to the absorbed heat by the sample but it gives an indication of the amount of heat needed and shows some consistency of the data. A similar parameter has also been used to investigate ignition behaviour of materials 11.

Table 3: Measured temperature at start of reaction.

<table>
<thead>
<tr>
<th>Incident heat flux (kW/m²)</th>
<th>Temperature at reaction time (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Test 1</td>
</tr>
<tr>
<td>10</td>
<td>136</td>
</tr>
<tr>
<td>15</td>
<td>120</td>
</tr>
<tr>
<td>20</td>
<td>66</td>
</tr>
<tr>
<td>25</td>
<td>128</td>
</tr>
</tbody>
</table>

The temperature at the start of reaction varies between 120-140°C except in experiment 20_1, in which the thermocouple was not fixed correctly.

**Furnace experiments**

The results of the furnace experiments are presented with the plate thermometer 12 measurements, because plate thermometers can in this experimental setup be seen as a standardised small object with low thermal diffusivity where the surface temperature is accurately measured. This together with the fact that it is direction dependent in the same way as a flat surface and that a boundary layer more representative for larger surfaces is created is the reason why plate thermometers are superior to small thermocouples when regulating thermal exposure of large objects in fire resistance furnaces.

Test 1 – Standard fire curve, no fire stopper in place
This test was performed as a reference test. The pieces of wood and cotton ignited.

Test 2 – Standard fire curve, fire stopper in place
The pressure in the furnace was raised from 0 to 5 Pa after 5 minutes and 20 minutes later it was raised further to 10 Pa. When the pressure in the furnace was raised to 5 Pa the fire stopper expanded quickly, the opening was sealed and the temperature on the unexposed side did not exceed 100°C.
Figure 4: Plate thermometer temperature on exposed and unexposed side of fire stopper in test 2

Test 3 – Temperature increase of 10 °C/min during 50 min, then a more rapid increase
The pressure was kept around 5 Pa after approximately 10 minutes.

Figure 5: Plate thermometer temperature on exposed and unexposed side of fire stopper in test 3

When the temperature reaches 200 °C in the furnace the temperature on the unexposed side stabilises around 150-180°C and later drops. This indicates that the fire stopper has expanded and that limited amounts of hot gas pass through the vent. It was noted in the visual observation that the fire stopper did close partially after 25 minutes, but was not entirely closed until the after 50 minutes when the furnace temperature was rapidly increased.

Test 4 – Temperature increase of 4 °C min during 40 min, then a more rapid increase
The pressure in the furnace was raised from negative values to 1-2 Pa after 5 minutes, which led to hot gases passing though the vent and temperatures on the unexposed side to follow the furnace temperature (see Figure 6).
Figure 6: Plate thermometer temperature on exposed and unexposed side of fire stopper in test 4.

After 40 minutes the temperature in the furnace is increased approximately according to ISO834 and at the same time the pressure rose to 15-20 Pa. It is clear from Figure 6 that this causes the fire stopper to expand and close the opening since the measured temperature on the unexposed side drops. The opening was entirely closed after about 20 seconds after the rapid temperature increase according to the visual observations.

Test 5 – Steady temperature of 150°C during 40 min, then more rapid increase
The pressure in the furnace was raised from negative values to around 1 Pa after 5 minutes.

Figure 7: Plate thermometer temperature on exposed and unexposed side of fire stopper in test 5.

The temperature on the unexposed side was steady and 40-50°C below the furnace temperature, but after 40 minutes the furnace temperature was increased, similar as in test 5. The pressure in the furnace increased to 15-20 Pa and temperature on the unexposed side followed the furnace temperature up to 200°C, and then dropped because of the expansion of the fire stopper. The very rapid temperature increase and pressure caused the small wood pieces on the unexposed side to char a little before the fire stopper closed the opening.

Test 6 – Steady temperature of 180°C during 40 min, then more rapid increase
The results from test 5 are very similar to test 4 (see Figure 8).
The temperature on the unexposed side of the fire stopper did not reach the same temperatures as in test 5 and the small wood pieces on the unexposed side did not char.

Test 7 – High moisture content in fire stopper
The graphite strip had an additional 16 % moisture content compared with the normal moisture balance and was subjected to the standard time temperature fire curve in the small-scale furnace. After 5 minutes the pressure was increased from around 0 to 15 Pa.

When the pressure was raised hot gas started to flow through the opening and the fire stopper expanded. The performance of the fire stopper was not very different from the tests with dry graphite (test 2).

Test 8 – High pressure in the furnace.
The pressure in the furnace was raised from zero to around 30 Pa after 5 minutes at the same time as a rapid temperature increase to try to blow away the system during the foaming process.
Figure 10: Plate thermometer on exposed and unexposed side of fire stopper in test 8.

When the pressure and temperature was increased, the fire stopper expanded and sealed the opening in around 20 seconds. The fire stopper did stay in place and the high pressure did not affect its foaming.

DISCUSSION

The temperature measurements in the cone calorimeter experiments gives a hint of the surface temperature of the sample, even though it is difficult to measure surface temperatures with a thermocouple as they are more difficult to attach and receive radiation from the conical heater. A better indication of the reaction temperature is retrieved from the small-scale furnace experiment. From several of the tests (see Figure 7 and Figure 8) it is obvious that the graphite expands around 180°C. This is somewhat higher than the gas temperature measured with thermocouples in the cone calorimeter, which is considered reasonable.

The cone calorimeter tests indicate that the reaction temperature is independent of the incident heat flux since the measured temperatures are similar in all the successfully performed tests. However, the final expansion of the graphite is clearly dependent on the incident heat flux because of the higher heat wave into the sample. The graphite will expand several orders of magnitude when exposed to fluxes that could ignite construction materials and this is of course a prerequisite if the graphite is going to be used to protect construction materials as was shown in earlier publications.\(^1^3\)

The tests in the small-scale furnace were designed in order to subject the graphite to temperature conditions that could occur in an actual fire. The temperature was kept close to reaction temperature, estimated from the cone calorimeter experiment, in order to see if it would affect the performance of the fire seal negatively when the temperature was raised rapidly. The temperature was raised slowly in two tests since Adl-Zarrabi\(^1^)\) found that the heating rate affects the expansion of expanded graphite. One test was designed that the graphite had high moisture content, which also affects the expansion according to Adl-Zarrabi. Even though this previous research indicates that the material will behave different for different heating conditions it cannot be seen in the small-scale furnace experiment that this has any implication on the performance of the tested system for cavities. In this case the graphite expanded enough to give the same protection, although it is preheated or wet, as when subjected to ISO 834. However, this does not mean that it cannot be of importance for other types of intumescent system or situations.

Standardized methods like ISO 834 or ASTM E119 are needed to classify products and the classification does provide a high stress on the product that is tested. But, the heating curves used in such standards are not representative for natural fires. For example, an EI60 rating according to ISO
834 does not reveal anything about how the rated product will behave in an actual fire, it merely says that the product fulfils ISO 834 for integrity (E) and insulation (I) for 60 minutes. In a real fire the product can last of 2 hours or maybe fail after 30 minutes. During the last decades there has been a paradigm in fire protection of buildings with the introduction of performance-based design in several building codes around the world. In this sense the performance of individual building products in actual fires will be of great importance because a fundamental part of performance-based design is to analyse the fire protection in the building in regard to a probable worst-case or realistic fire. The focus of this paper has been to see how a classified product behaves in a more realistic fire environment. Further research in this area is considered necessary and especially when it comes to different types of intumescent systems.

CONCLUSION

The performance of a graphite based intumescent system used for cavities has been studied in this paper. A series of tests, in two experimental setups, are presented in order to see how a graphite based fire stopper performs when subjected to realistic fire conditions. The tested type of system will react at around 180°C and the reaction temperature is rather independent of the incident heat flux, while the rate of expansion is clearly dependent on the incident heat flux. Furthermore, the studied system performed well in the small-scale furnace because the graphite expanded enough to give the same protection, although it was preheated or wet, as when subjected to the standard time temperature fire curve. These results are good but they are only considered valid for the studied system and the given situation. Future research is needed in order to study how other types of systems works when subjected to realistic fire conditions.

REFERENCES

10 SP Fire 119, *Fire test of building constructions in a small-scale furnace* (in Swedish), issue 5, 2012