Experimental characterization of the thermal behavior of intumescent paints

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Abstract: Fire protection of exposed structures through intumescent paints, which is based on the creation of a cellular foam cushion which develops upon the substrate when high temperatures are reached, may be rather effective. Unfortunately, physical properties of intumescent paints after the chemical reaction has occurred are not widely known, which makes impossible to carry out reliable fire-resistance evaluations for the coated structure. In this context, a first-approach experimental investigation of both the thermal conductivity and mass density of the foam cushion is performed, and the main results obtained are presented.

Keywords: Experimental investigation; Intumescent paints; Thermal internal conductivity; Mass density.

1 Introduction

The protection of exposed structures against fire may effectively be achieved through intumescent painting, which is common in construction applications [1,2]. In fact, as the coating thickness is very small, both the dimensions and the aspect of the structures remains substantially unchanged, which is of primary importance for buildings of high architectural value.

The flame-retardant working principle of the intumescent paints is to create a multi-cellular foam cushion when subjected to high temperatures, in the typical range between 300°C and 350°C, so as to prevent heat from penetrating into the substrate and flames from spreading along the surface [3–7].

Main drawback of such materials is the almost complete lack of data on their thermal properties. Actually, although experimental measurements of several thermo-mechanical properties have been carried out quite recently [8,9], data on thermal internal conductivity, whose values are indeed required for a reliable analytical computation of the fire resistance of the painted structure [10,11], are not readily available.

In this framework, the main results of a first-approach experimental investigation carried out through a test facility specifically designed for the measurement of the thermal internal conductivity of the multi-cellular foam at the end of the chemical intumescent process, are here presented and discussed.

In addition, mass density and weight loss data obtained through a ponderal method are also reported.

2 Experimental equipment

The experimental equipment consists mainly of a cylindrical electric oven, and a cylindrical test section with an embedded electric heater, mounted vertically in the middle of the oven. The external surface of the test section is coated by the intumescent paint to be investigated. The oven has the role of raising the temperature around the test section in order to allow the cellular foam cushion to develop on its outer surface. The electric heater inside the test section is used for establishing a given heat transfer rate across the foam cushion after its formation, so as to allow for the evaluation of its average thermal conductivity.

The oven, with internal diameter of 38 cm and height of 44 cm, is powered through a variable auto-transformer connected to a stabilized a-c supply unit. The power supplied is measured through a 0.5-class instrument. Air temperature inside the oven, which in the following of the paper we will refer to as ambient temperature, is measured at different locations by three K-type thermocouples, whose junctions are properly shielded to avoid radiative interactions.

The test section consists of a steel tube with inside diameter of 22.2 mm, outside diameter of 40 mm, and length of 410 mm. An electric heater consisting of a 0.5-mm dia. nickel-chrome wire rolled around a threaded ceramic cylinder with outside diameter of 15.2 mm, threading step of 1 mm, and overall length of the threaded portion of 380 mm, is mounted inside the steel tube. The gap between the heater and the inner surface of the tube is filled with magnesium oxide condensed powder for insulation. Both ends of the tube are sealed through fireproof concrete. Electric power is supplied through an a-c/d-c rectifier fed by a variable auto-transformer connected to a stabilized a-c supply unit. The power supplied is calculated as the product of the squared current flow by the electric resistance of the nickel-chrome wire. The current flow is determined by measuring the voltage drop across a 0.01Ω calibrated resistor in series with the heater. The voltage drop across the calibrated resistor is read to $6^{1/2}$ significant figures through an Agilent 34970A data-acquisition unit equipped with a 20-channel multiplexer Hewlett-Packard 349020A. The electric resistance of the wire has been measured at a wide number of temperatures in the range between 20°C and 600°C by a Wheatstone bridge with accuracies of ±0.01%, displaying variations less than 0.5% with respect to the mean value, which is due to the small temperature coefficient of nickel-chrome, i.e., nearly 0.0003°C^{-1}. The wall temperature of the test section is measured through eight K-type thermocouples with an outside diameter of 1 mm, a stainless steel sheath, and a mineral powder insulation. The thermocouples are soldered inside grooves milled axially along the outer surface of the tube with an angle interval of 45°. Four thermo-couple junctions are located midway between the ends of the tube, whilst the remaining two pairs are located near the two ends of the tube.

The reference junctions of all the thermocouples are maintained at the reference temperature of 298K±0.1K in a thermostatic bath. The thermocouple voltages are read through the same meter Agilent 34970A mentioned above.

Fig. 1. Electric oven and test section inside.

A picture of the electric oven with the test section mounted in its interior after the intumescence process has occurred is shown in Fig. 1.

3 Experimental procedure

The experimental procedure starts with the application of the intumescent paint upon the test section. The weight of the coating is calculated as the difference between the weights of the test section after and before the painting process, measured through an electronic balance with capacity of 3 kg and accuracy of 0.01 g. The corresponding surface mass density of the intumescent paint is denoted as δ.

The second step is the creation of a multicellular foam cushion, compact and well adherent to the outer surface of the test section. To this end, the intumescent process must proceed gradually from the outer layer of paint up to reaching the innermost layer in contact with the steel tube, which is obtained by supplying a constant power P to the oven, whilst maintaining the electric heater of the test section switched off. What happens is described in Fig. 2, where the time-distributions of the mean ambient temperature inside the oven T_O and the mean surface temperature of the test section T_W, as well as their difference, are represented. It may be seen that, as soon as T_W reaches a definite value T_S, the intumescent process starts, which is reflected by a significant slope decrease. This is due to the foam insulation effect, and to the fact that most of the heat is used for the endothermic intumescent reaction. The formation of the cellular foam cushion ends at T_E, over which the only insulation effect governs the achievement of the thermal equilibrium between the test section and the oven.

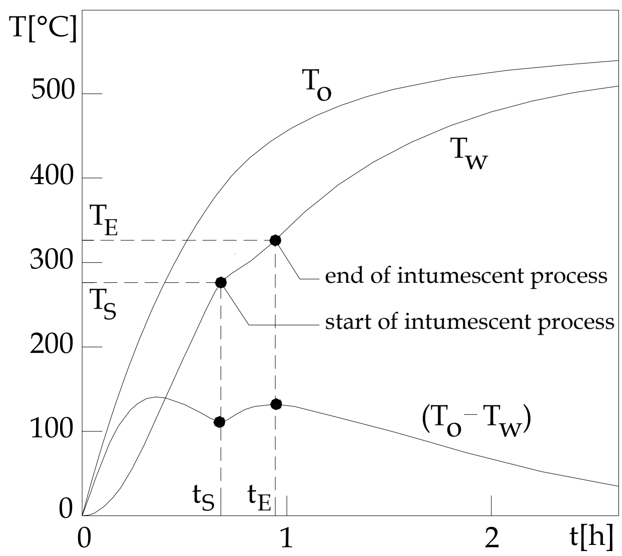

Fig. 2. Time-evolutions of T_O, T_W and $(T_O - T_W)$.

However, the time-evolution of T_W depends directly on the evolution of T_O, which, on its turn, depends on the power supplied to the oven. This means that, once the surface mass density of the intumescent paint δ is assigned, the time required by the chemical reaction to take place decreases as the ambient temperature inside the oven increases faster, i.e., as the power supplied to the oven is higher, which may have non negligible effects on the multi-cellurar structure of the foam, and then on its insulation properties and average volume mass density. In this regard, the speed of the temperature raise may be characterized by the following parameter:

$$U = \frac{T_E - T_S}{t_E - t_S} \qquad (1)$$

where t_E and t_S are the times at which the average surface temperature of the steel tube is equal to T_E and T_S, respectively.

As third step of the investigation procedure, a constant power W is supplied to the heater embedded in the test section, whilst maintaining the electric oven switched on, which implies a more or less pronounced increase in the surface temperature of the test section over the ambient temperature. Once the steady-state is achieved, i.e., the temperature difference $(T_W - T_O)$ does not change anymore, the average thermal conductivity of the foam cushion k_F is evaluated through the following relationship:

$$W = \frac{2\pi R_{ml} L (T_W - T_O)}{\dfrac{R_F - R}{k_F} + \dfrac{R_{ml}}{R_F h}} \qquad (2)$$

with

$$R_{ml} = (R_F - R)/\ln(R_F / R) \qquad (3)$$

where h is the coefficient of heat transfer at the outer surface of the foam cushion, R and L are the radius and the length of the steel tube of the test section, and R_F is the mean radius of the outer surface of the foam cushion. Since, as expected, the thickness of the cellular foam at the end of the intumescent process is not uniform, R_F is calculated as the average of 360 local measurements executed through a micrometric sensor device at 20 different heights with a 20 mm uniform spacing, and at 18 different angular positions with a 20° uniform spacing, with a ±0.7 mm accuracy. As far as the heat transfer coefficient h is concerned, several preliminary measurements have been carried out for the bare test section, i.e., for the steel tube without intumescent painting, which has allowed us to obtain its average value as a function of the power supplied to the heater and the ambient temperature inside the oven:

$$h = \frac{W}{2\pi R L (T_W - T_O)} \qquad (4)$$

Finally, the average volume mass density of the foam cushion ρ_F is also calculated:

$$\rho_F = \frac{M_P - M}{\pi (R_F^2 - R^2) L} \qquad (5)$$

where M_P is the weight of the test section with the foam cushion completely developed on it, and M is the weight of the bare test section, both measured through the aforementioned electronic balance. It is worth pointing out that the weight M_P of the painted test section measured after the intumescent process has occurred is generally well lower than the weight $(M_P)^0$ measured before the chemical reaction. This is due to the considerable amount of gaseous products leaving the foaming structure as it develops, which may be quantified through the percent weight loss ML = $[(M_P)^0 - M_P]/(M_P)^0$.

4 Experimental results and discussion

Experiments are performed for a wide number of combinations of (a) the surface mass density of the intumescent paint δ in the range between 500 g/m^2 and 2000 g/m^2, and (b) the speed parameter U in the range between 5 °C/min and 30 °C/min, which is modified by simply modifying the power P supplied to the oven. The corresponding ambient temperatures obtained inside the oven at the steady-state are included in the range between 400°C and 500°C. The power W supplied to the heater embedded in the test section after the cellular foam cushion is created, is chosen so as to obtain a temperature difference $(T_W - T_O)$ around 25°C.

The distributions of k_F, ρ_F and ML vs. U, for different values of δ, are reported in Figs. 3, 4 and 5, respectively, where non-negligible dispersions of data may be noticed. The distributions of the corresponding average values vs. δ are reported in Figs. 6, 7 and 8, respectively.

The following considerations may be done:

a) the percent weight loss at the end of the intumescent process, of the order of 35% ± 5%, does not depend significantly on both U and δ;
b) the average volume mass density of the cellular foam, of the order of 10÷15 kg/m^3, which is approximately one-half of that typical for porous insulating materials, decreases with increasing the surface mass density of the intumescent paint before the chemical reaction occurs;

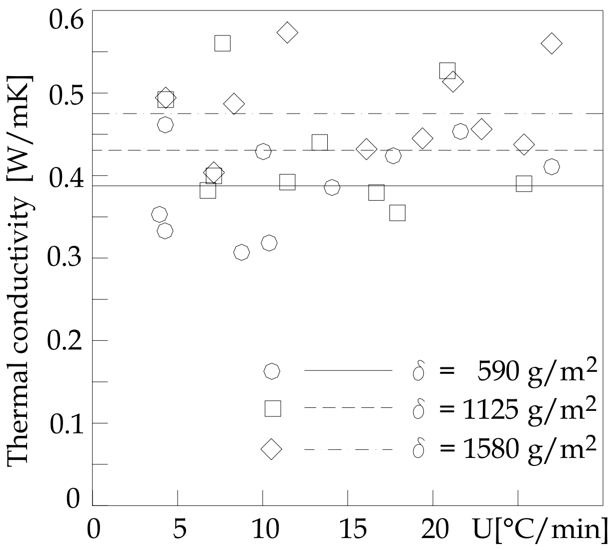

Fig. 3. Distributions of k_F vs. U for different values of the surface mass density δ.

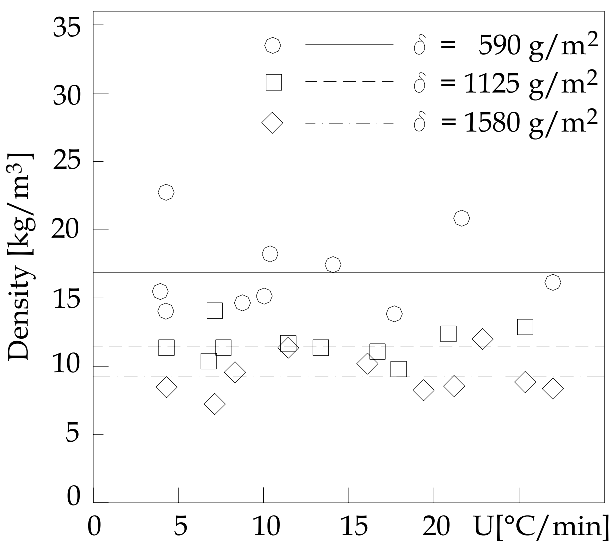

Fig. 4. Distributions of ρ_F vs. U for different values of the surface mass density δ.

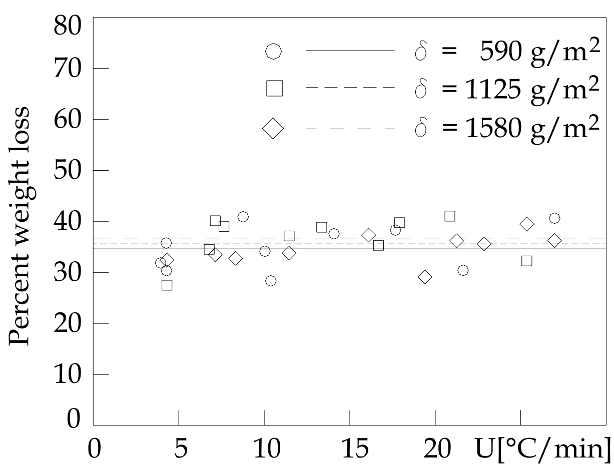

Fig.5. Distributions of ML vs.U for different values of the surface mass density δ.

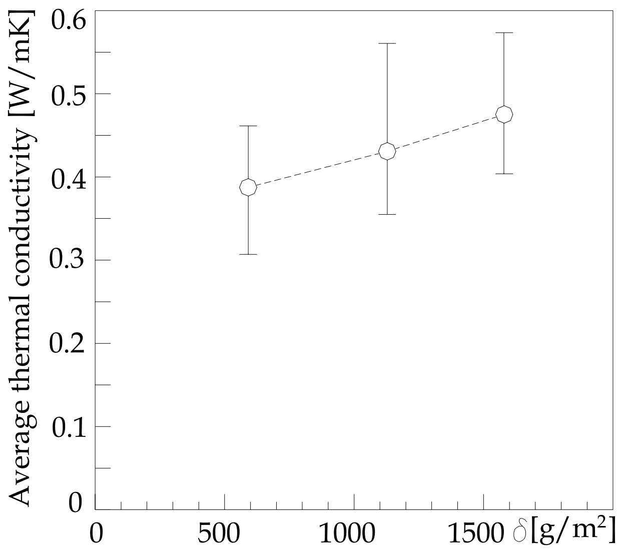

Fig. 6. Distribution of the average thermal internal conductivity vs. the surface mass density δ.

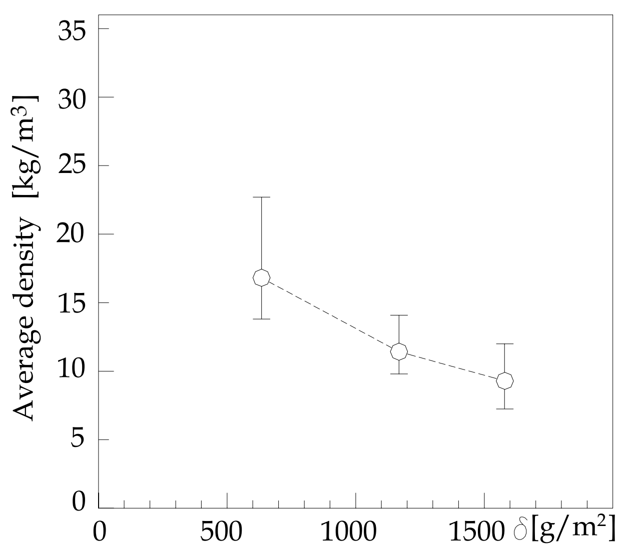

Fig. 7. Distribution of the average volume mass density vs. the surface mass density δ.

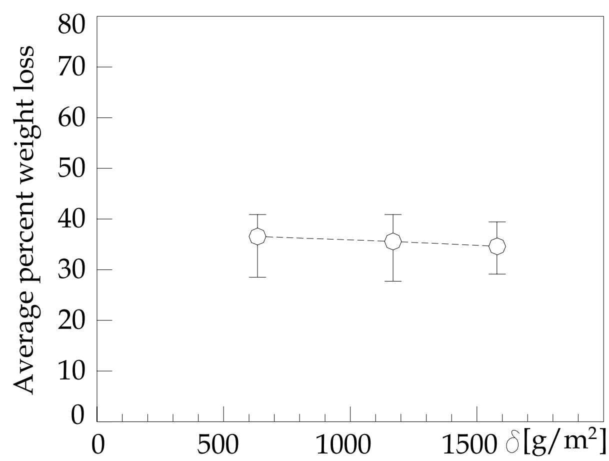

Fig. 8. Distribution of the average percent weight loss vs. the surface mass density δ.

c) the average thermal conductivity of the cellular foam, of the order of 0.35÷0.45 W/mK, which is typical for porous insulating materials in the experimental range of temperatures, increases slightly with increasing the surface mass density of the intumescent paint before the chemical reaction takes place.

5 Concluding remarks

The variability of the values displayed by the physical properties of the cellular foam cushion, with deviations up to ±35% from their mean value, might be ascribed to the inherent variability in the occurrence of the intumescent process. Indeed, the creation of the multi-cellular structure originating from the gas production which takes place as the chemical reaction occurs, is not at all completely deterministic, which may partly justify the different properties of the final product.

On the other hand, both the hypotheses that heat is transferred "radially" across the foam cushion, which is assumed for the use of equation (2), and that the coefficient of heat transfer for the bare test section applies also to the case of the foam cushion, may not be completely met in experiments.

However, the present first-approach results may be usefully used as a basis for future work on this same subject, that we deem worth being performed in the context of the necessity to carry out reliable predictions on the fire resistance of structures covered by intumescent paints.

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