Latest developments in scale reduction for fire testing

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ABSTRACT

In this paper, the scaling reduction of fire tests of interest is presented. It deals with vertical fire resistance test (fire protection of steel according to UL 1709 and ISO 834), test for building products (single burning item or SBI according to EN 13823) and the environmental test procedure for airborne equipment (resistance to fire of materials in designated fire zones according to ISO 2685). It covers different types of material (composites, steel and wood) and several applications (wooden panel, composite and steel for building and carbon fiber reinforced composite for aviation).

Index Terms: fire protection, scale reduction, intumescent coating, char strength, infrared thermography

INTRODUCTION

Fire testing for evaluating the reaction and resistance to fire of materials is generally time-consuming and expensive. Nevertheless, those tests are required to evaluate materials in many sectors such as building or transportation. Due to the complexity of fire phenomenon, full-scale tests are still the main and the most credible tool for investigating fire-related issues. However, the cost of those tests significantly increases with scale. The purpose of this paper is to examine the scale reduction of different tests and to mimic fire scenarios at the small scale.

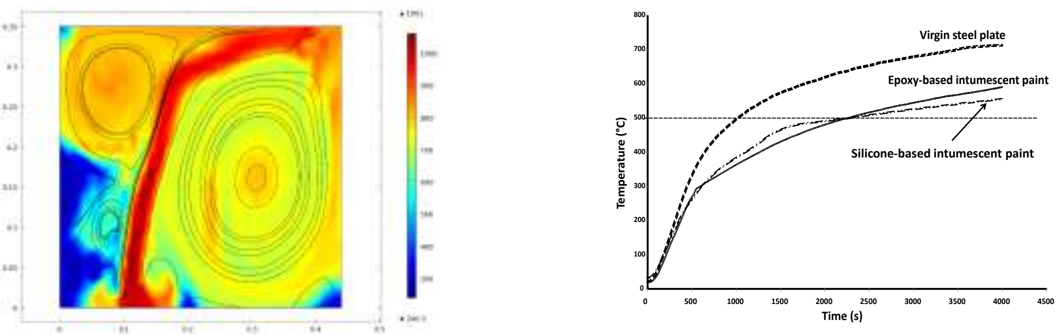
In this paper, the scaling reduction of some fire tests of interest is presented. It deals with the furnace test (UL 1709), single burning item (SBI) and the burner test (ISO 2685). It covers different types of materials (composite, wood and steel) and several applications (composite, wood and steel for building and carbon fiber reinforced composite (CFRP) for aviation). In all cases, the fire protection is provided with an intumescent coating. Intumescence is a method of passive fire protection [1]. The result of the intumescence process is a foamed cellular charred layer on the surface, which protects the underlying material from the action of the heat flux or the flame. The formation of an intumescent char is a complicated process involving several critical aspects: rheology (expansion phase, viscoelasticity of char), chemistry (charring) and thermophysics (limitation of heat and mass transfer). Typically, the ingredients of intumescence are mainly composed of an inorganic acid or a material yielding acidic species upon heating (e.g. phosphate), of a char former (e.g. pentaerythritol) and of a component that decomposes at the right temperature and at the right time to enable the blowing of the system (e.g. melamine).

FURNACE TEST (ISO 834 AND UL 1709)

The protection of metallic materials against fire has become a very important issue in the construction and petrochemical industries, as well as in the marine and military fields. Structural steel loses a significant part of its load-carrying ability when its temperature exceeds 500°C. Prevention of the structural collapse of a building is crucial to ensure the safe evacuation of people and it is a prime requirement of building regulations in many countries. We developed a small-scale furnace test to evaluate the fire performance of steel protected by intumescent coatings in cellulosic and hydrocarbon fires. This test was designed to mimic the ISO834 (or ASTME119) and the UL1709 normalized temperature/time curves, respectively related to cellulosic fire and to hydrocarbon fire (Figure 1 (a)). The lab-scale furnace exhibits an internal volume of 40 dm^3. Refractory fibers (stable up to 1300°C) cover the different faces of the furnace. The furnace was equipped with two gas burners (20 kW propane burners). A temperature probe inside the furnace regulates the temperature and a K-type thermocouple allows the furnace temperature profile to be registered. The temperature at the backside of the plate is measured as a function of time using a pyrometer (temperature measured in the center of the plate). The backside of the plate is coated with black paint (Jeltz) having a constant emissivity (0.92) and thermally resistant up to 800°C in order to get reliable temperature measurements with the pyrometer.

Silicones encompass a wide range of materials and find applications in virtually every major industry sector such as civil engineering, construction building, electrical, transportation, aerospace, textiles, and cosmetics industries. Silicones are polymers whose backbones consist of alternating Si-O bonds and have the properties to have a very low thermal conductivity, to be water and heat resistant and to evolve few toxic gases during their degradation. In addition to their unique physical/chemical properties, silicones exhibit advantageous fire behavior such as low heat release rates regardless of fire size or fire severity: they are therefore materials of choice for the development of fire retardant coatings. Recently, we developed novel silicone-based intumescent coatings for the protection of steel [2-7] and composites [8]. They exhibit higher performance than conventional intumescent paints (e.g. epoxy-based or acrylic-based intumescent paints) according to ISO 834 and UL1709 standards.

Novel intumescent paints based on silicone as polymeric matrix were applied on steel plates and were evaluated within the small-scale furnace according to ISO 834 temperature curve (Figure 1 (b)). The silicone-based intumescent paint was compared to a commercial epoxy-based intumescent paint. The failure temperature has been taken at 500°C. Uncoated steel plate reaches 500°C at 1000s while the coated steel with intumescent coatings reaches the failure temperature at times higher than 2400s. It shows that intumescence is an efficient way to protect steel since the time to failure temperature is twice longer than that with virgin steel. Our silicone paint was compared to the commercial paint and it appears that its behavior is similar as epoxy-based paint.



(a) (b)

Figure 1: (a) CFD modeling of temperature distribution (colors) and velocity field (plain line) at time = 3s in the small furnace where burners are delivering temperature/time curve according to ISO 834 ; (b) Temperature as function of time on the backside of steel plate protected or not by a silicone-based intumescent paint when undergoing ISO 834 heating in the small furnace.

A significant aspect of intumescent formulations is the mechanical strength of the intumescent char. In the conditions of a fire, char destruction can proceed not only by means of ablation and heterogeneous surface burning but also by means of an external influence such as mechanical action of the fire or convective airflows. Mechanical strength of char is generally estimated after fire testing at room temperature by empirical methods (e.g. finger touch, piston rod …) or using a rheometer as a function of temperature thanks to a method developed in our group [9-11]. Nevertheless, it is rarely measured in real dynamic conditions (fire conditions) while it is a crucial parameter determining the performance of the intumescent char. In a previous paper [12], a novel dynamic protocol for measuring the char strength of intumescent coating during fire testing was developed. It was done in the furnace fitting the ramp of temperature of a hydrocarbon fire (UL 1709). The furnace was equipped with an air-jet or N_2-jet system delivering hot air flow or nitrogen flow simulating turbulence circulating around char or fluid impacting the surface of the char. The full description of the system is shown on Figure 2.

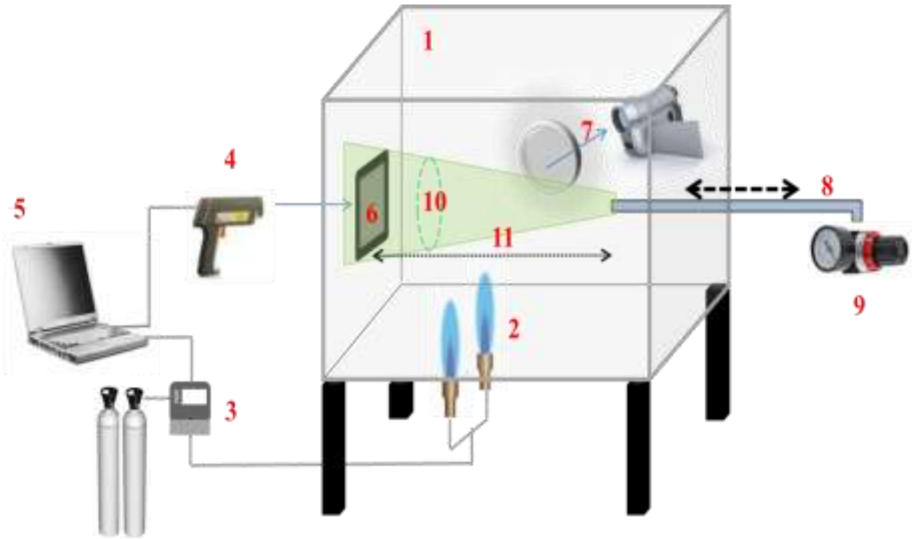


Figure 2: Schematic representation of small-scale furnace test equipped with the jet test: Refractory fibers stable up to 1300°C covers the insides of the furnace (1). Two burners (2) fed by propane have a capacity of 20kW (3). IR pyrometer (4) is used to record the temperature at the backside of the coated steel plate (10cm x 10cm x 3mm). Burner control and pyrometer readings are controlled by the attached computer (5). Coated plates (6) are sprayed with black paints of known and constant emissivity on the uncoated side and the plate is fixed in the window and sealed the sides with glass-fiber wool. Through a quartz window (7), the intumescent behavior of the char can be viewed inside the furnace during the fire test and movies can be recorded. This furnace is upgraded to carryout airjet test creating air turbulence over the fully developed char during the fire test. A provision is made to introduce a stainless steel tube (8) facing the sample which is connected to compressed air (9) inlet. This arrangement helps to provide better control over airjet velocity and cone of airjet exposure on char (10). The steel tube can be moved so as to keep fixed gap (11) between different samples as char height can vary for different samples.

The jet test permits to control gas flow /air (or N_2) current striking or flowing around the fully developed char to simulate fluid turbulence or wind perturbation during the standard UL1709 hydrocarbon fire test and to record the resistive response and the mechanical stability-integrity of char. For comparative study, epoxy-based and silicone-based coatings were evaluated according to UL1709 fire. As the char height varies among different formulations, the stainless steel tube is adjusted for each experiment to have fixed gap between char surface and tip of the tube. The jet across the direction of fire (perpendicular to direction of fire jet), slightly 'blow' the fire on char creating even harsher atmosphere around the char. Considering the available gap between tip of steel tube and surface of char air (or N_2) pressure of 1bar is found to be appropriate. This gives an optimum gas jet permitting a sustained destruction of char and providing reasonable amount of time to understand the fragmentation of char (if any). We selected 350°C as the ideal temperature to start the gas jet test as by this time char is completely developed and gives realistic time to study the events before the steel plate reaches its failure temperature of 500°C. Formulations with 4 mm coatings were selected which provide char height lying between 3 to 5cm. Three formulations (2 silicone-based coating hereafter called N1and LF7 and 1 epoxy-based coating) were selected for the study. Fire performance (time to reach 500°C) and char heights were evaluated by independent standard UL1709 fire test. Fire performances are shown in Figure 3 (a). Time to reach 500°C is highest for N1 followed by LF7 and epoxy. Char thickness measured after the fire test are in the same order as fire test, N1 (4 cm) >LF7 (3.5 cm) > epoxy (3 cm) (Figure 3 b-d).

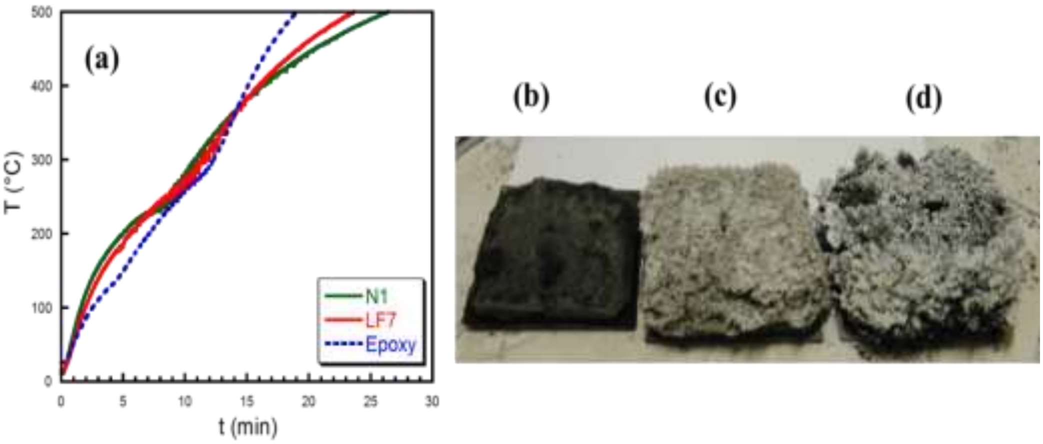


Figure 3: (a) Fire performance under standard UL1709 condition. (b-d) Comparative char heights of epoxy and silicon formulations (LF7 and N1 respectively)

In a typical gas jet procedure, standard UL1709 fire test is carried out on the coatings. At 350°C, gas valve is opened and the stainless steel tube is inserted in the furnace and gas jet is directed to the char. Quartz window allows direct vision of events and video recording the char behavior. When temperature reaches 500°C on the backside of the steel plate, jet and burners are stopped. Depending upon its strength, the withstanding and sustaining abilities of char in case of turbulence under fire exposure varies. We focused on following observations: (1) Cohesion and ablation of char: to observe char fragmentation and if so, its intensity on the time scale during jet. (2) Resistance: measuring and comparing the time taken to reach from 350°C to 500°C ($t_{350\to500°C}$) among different samples. Under identical conditions, the higher the $t_{350°C \to 500°C}$, stronger is the char.

Air was first selected to make the test. Silicone and epoxy based formulations have different destruction profiles and char strengths. A comparative T vs t profile is shown in Figure 4 (a). Epoxy char under the influence of airjet in fire test condition undergoes peculiar ablation. It is associated with slight shrinking (char starts to shrink from top corner), heterogeneous deformation, and a kind of 'melting', leading to char destruction within 2 min of airjet. Nearly half of the char is destroyed before $t_{350\to500°C}$ reaches 3.8min. Despite this, small part of the char remains glued to plate (Figure 4 (b)-(c)). Some selected snap shots are given in Figure 5.

Silicone based formulations N1 and LF7 behave quite differently from epoxy. Small fragments begin to detach early (within 1-2 min) however that does not spillover to large-scale destruction. This is usually observed at the corners and except slight erosion on surface, no significant destruction is observed even after 5 min. This char is able to resist the temperature rise of steel plate up to 8 min (LF7) and 10 min (N1) which is considerably better than epoxy chars. Char remains glued to plates in both cases (Figure 4 (b)-(c)). Room temperature observation shows that airjet makes the char porous in case of N1 and LF7 whereas larger fragmented fallen pieces of epoxy become hard. Grey-black coloration of normal char becomes pale or white after airjet test in case of silicon formulations (Figure 4 (c)). On the snapshots of Figure 5, it can be observed the silicone char remains almost intact during the whole test.

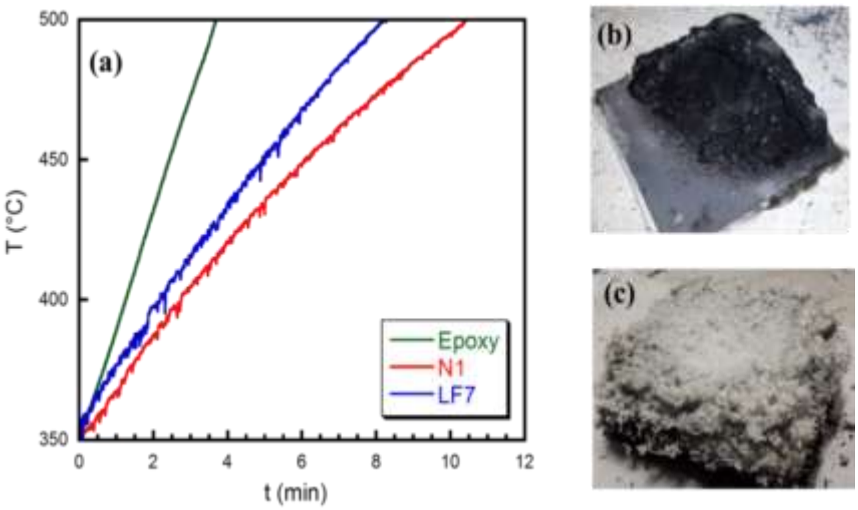


Figure 4: Evaluation and comparison of char strengths (a) plot of Temperature vs time showing relative strength of chars. (b) Epoxy char after airjet test (c) char of N1 after airjet test.

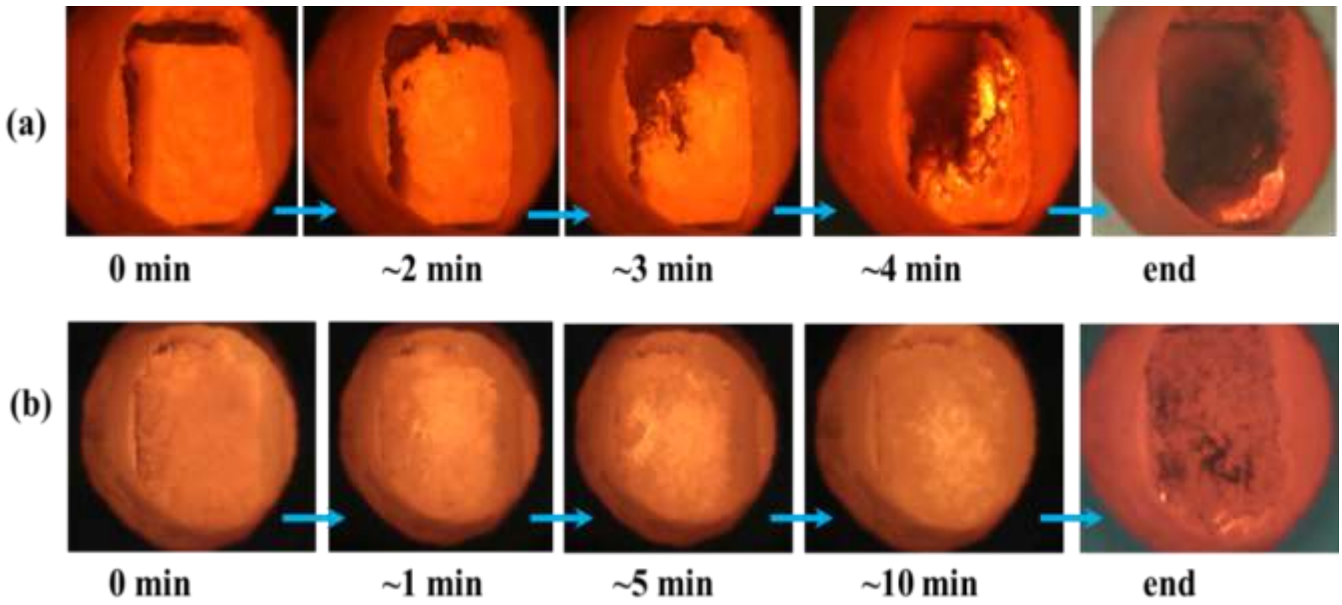
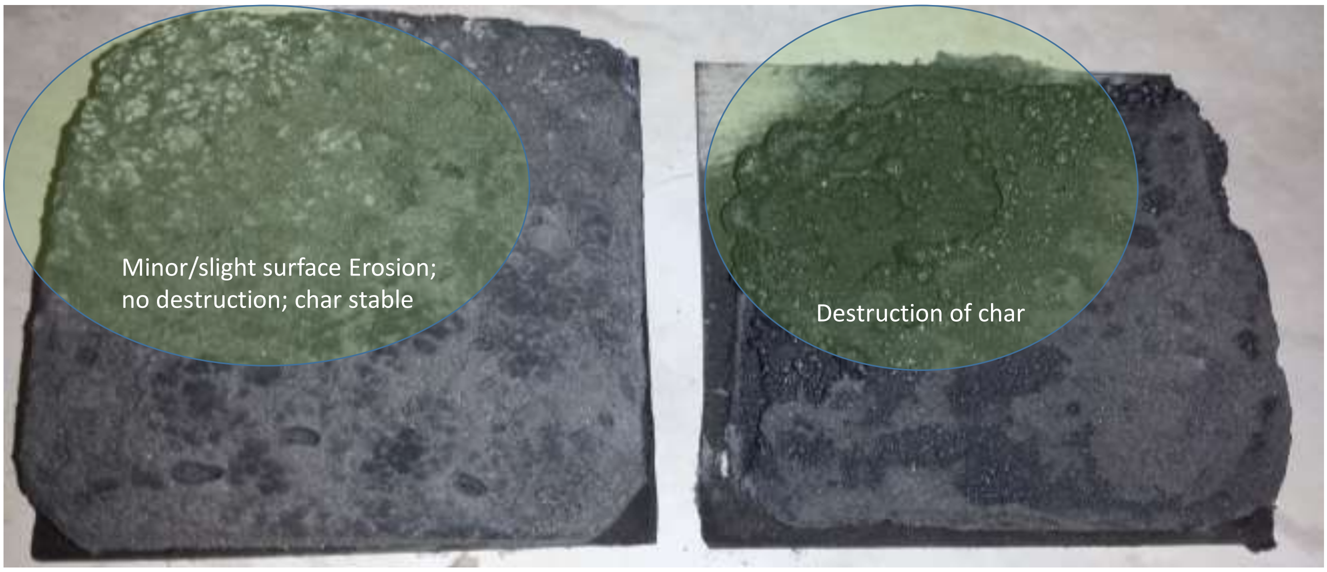


Figure 5: Snap shots at some selected intervals from video recording of events during UL1709 hydrocarbon fire test combined airjet test. (a) Events in case of epoxy coating. (b) Same in case of silicone-based coating.

Air was then substituted by nitrogen to evaluate the potential effect of oxidizing fluid (air) on intumescent coatings. In a typical furnace test, the quantity of oxygen during the test is about 4 vol%. Therefore, it makes sense to perform our gas jet test using N_2 instead of air to investigate the char strength of intumescent coating without potential additional effects of oxidation. Figure 6 and Figure 7 compare the residues after furnace tests of epoxy-based and silicone-based intumescent chars respectively. The silicone-based coating remains intact whatever the fluid employed during the test. On the contrary, the epoxy-based coating is very sensitive to oxidation and is destroyed during the airjet test. It shows the dramatic advantage to use silicone-based intumescent coating for fire protection.



(a) **(b)**

Figure 6: Char residues obtained in the furnace test (UL 1709) after airjet (a) and N_2 jet (b).

Figure 7: Char residues obtained in the furnace test (UL 1709) after airjet (a) and N_2 jet (b).

In this section, a proof-of-concept regarding evaluation of char strength made 'in-situ' was described. The study shows how a conventional protective intumescence reveals its vulnerability to external conditions (air impact) despite being judged as a good performer in the standard fire tests. The developed gas-jet test not only showed that silicone based char has better strength in protecting the steel structure but also spotlight physical integrity of char in air-turbulence. Compositional influences on char strength that are inconclusive by standard fire tests are witnessed in the air/N_2-jet test. The in situ test described in our work can be easily coupled with UL1709 test and can be performed in a standard furnace. Considering the shortcomings of current evaluation practices our novel protocol is also expected helping formulation redesign strategy and fuel investigation on theoretical aspects of char strength.

SINGLE BURNING ITEM (SBI, EUROCLASS)

The SBI test method is planned to assess the performance of building products in a (real scale) room corner scenario. In the pursuit of our efforts to make scale reduction, we have developed a SBI at 1/3 scale (Figure 8). It measures all parameters as in the large scale SBI including heat release rate (HRR), total heat release (THR), FIGRA, smoke opacity and SMOGRA. Wooden panel protected by an intumescent paint was evaluated with our bench-scale SBI. The results show that HRR is dramatically reduced when the panel is protected by the intumescent coating (Figure 9 – (a)). The intumescent charred layer develops upon burning and provide the protection (Figure 9 – (b)).

(a) **(b)**

Figure 8: (a) Mini-SBI test; (b) Cross-section of parameters measurement (smoke opacity, O_2 analyzer and pressure)

Our mini SBI was designed to be correlated with the values of the large scale SBI. It gives good prediction for the wooden panels as shown in Table 1.

Table 1: Comparison of the classification of wooden panels between the large scale SBI and the bench scale SBI

	Wooden panel (SBI)	Wooden panel (mini SBI)	Wooden panel (intumescent)
THR @600s [MJ]	15.7 (+/-2)	18.2 (+/-3)	3.2 (+/-1)
FIGRA [W/s]	440 (+/- 79)	524.3 (+/- 85)	164 (+/- 25)
TSP [m²]	47 (+/- 10)	66.8 (+/- 10)	0
SMOGRA [m²/s²]	3 (+/-1)	10.9 (+/-3)	0
Rating (Euroclass)	Ds2d0	Ds2d0	Cs1d0

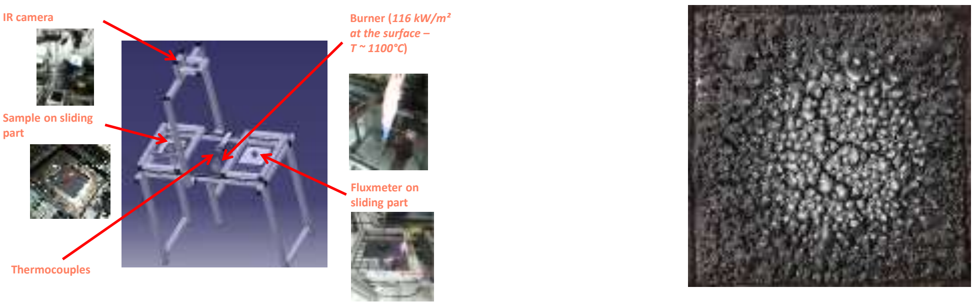
(a) (b)

Figure 9: (a) HRR as a function of time of wooden panels at the bench-scale SBI; (b) wooden panel protected by an intumescent paint burning at the bench scale SBI

BURNER TEST (ISO 2685)

Composite materials are increasingly being used in the design of aircraft. However, the recent application of carbon fiber reinforced polymer (CFRP) in aircraft structure has introduced potential fire threats and it raises concerns regarding fire safety [13, 14]. The fire resistance of components, equipment and structure located in 'fire zones' (e.g. compartments containing main engines and auxiliary power units) is conventionally evaluated according to the international standard ISO 2685. The test is established on a pass/fail basis thereby an element is considered to pass its fire test if it is capable to sustain its design function after the standard flame exposure for a specific lapse of time. In this research we are interested in the fire behavior of CRFP undergoing fire as defined in ISO 2685. Even if ISO 2685 is a medium-scale test, it is time-consuming and expensive, especially when CFRP samples must be produced for this purpose. In this section, a small-scale bench was developed to mimic ISO 2685 and to evaluate CFRPs with and without fire protection (intumescent coating).

A reduced scale bench was built up and was equipped with propane burner, insulated holder for the samples and with infrared camera and bichromatic pyrometers for measuring temperatures on the backside of the sample (Figure 10-(a)). CFRP sample prepared by Safran Company (France) was coated with a commercial intumescent paint and the temperatures measured on the backside of the sample was significantly reduced compared to virgin CFRP. As an example, temperature measured in the center of the sample is 345°C in the stationary state with intumescent coating while it reaches 485°C without intumescent coating (Figure 10-(b)).



(a) **(b)**

Figure 10: Reduced scale bench equipped with temperature rake for measuring flame temperature and with gauge for measuring heat flux impacting the sample (right); sample holder insulated from the frame (left); the two frames (right and left) are on the rail moving back and forth for calibration's purpose (a) – Intumescent coating formed after fire testing on the reduced scale ISO 2685 test (b).

An horizontal burner test was also developed to mimic a burnthrough test and to investigate the thermophysical behavior of CFRP in fire [15]. During a single test, both condensed and gas phases can be simultaneously studied measuring the temperature profile and the mass loss and studying the nature and quantity of volatile gaseous species (Figure 11). As above, the test consists of impacting a 150x150mm² sample by a propane flame at 1100°C and with a calibrated heat flux of 116kW/m².

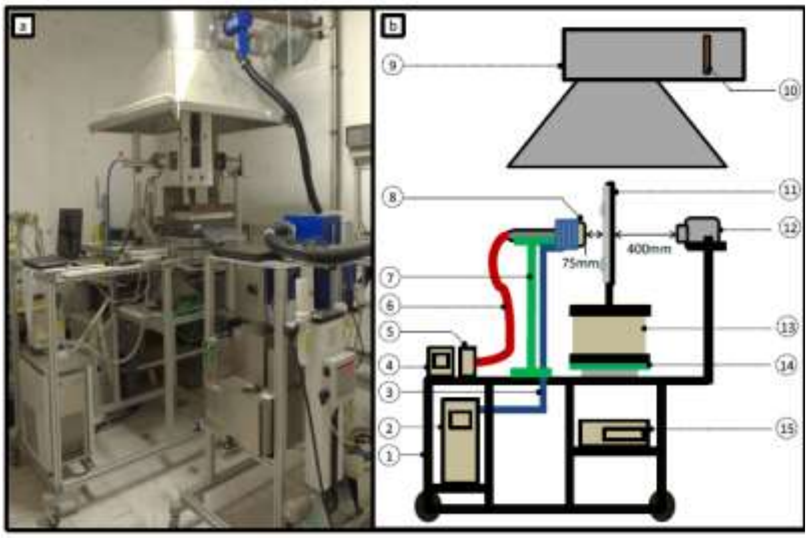


Figure 11: Picture (a) and scheme (b) of the horizontal burner test bench [8] - (1) Test bench frame, (2) Cooling thermostat, (3) Copper coil cooler, (4) Propane thermocontroller, (5) Propane flowmeter, (6) Propane gas line, (7) Burner support, (8) Propane flame burner, (9) Hood, (10) Ring sampler, (11) Coupon support, (12) Infrared (IR) camera, (13) Precision balance, (14) Balance support, (15) Data acquisition device

A commercial epoxy-based intumescent coating was evaluated for fire protecting steel with the burnthrough test. The purpose was to examine the intumescence behavior when the flame directly impinges the intumescent paint. The comparison of the temperatures as a function of time on the backside of steel plate with and without paint shows the efficiency of the intumescent protection (Figure 12): in the steady state, the temperature only reaches 290°C with the intumescent protection while the temperature is 450°C without protection.

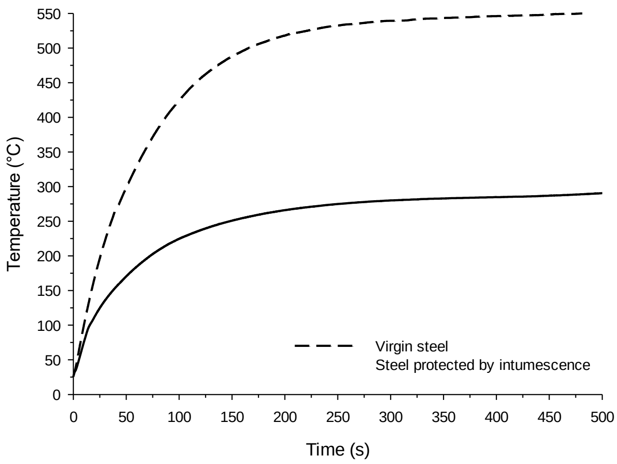


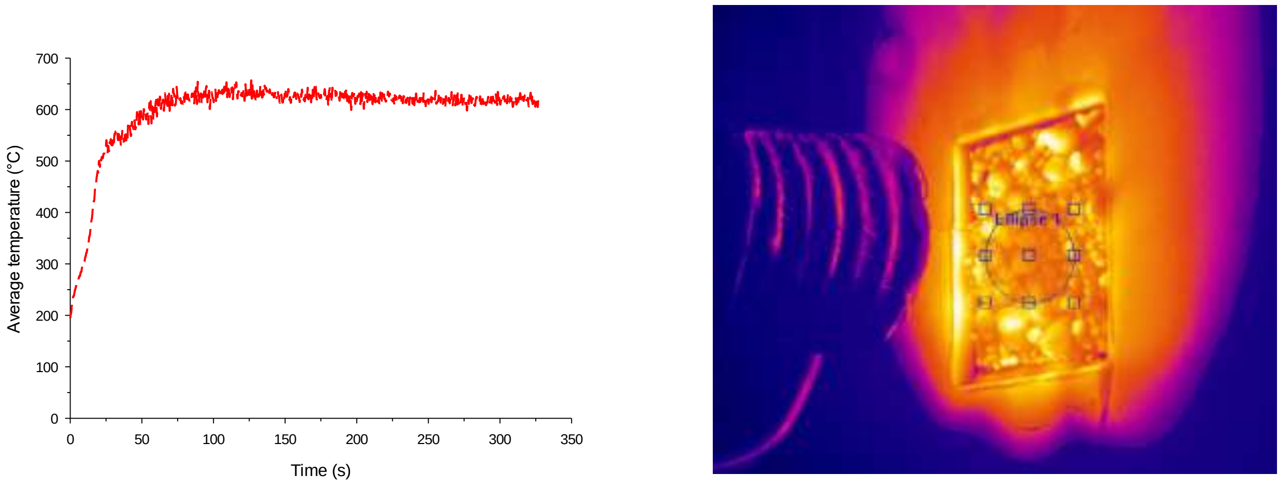
Figure 12: Temperature as a function time at the burnthrough test of steel protected by intumescent coating

Optical and infrared images (Figure 13) show the development of the intumescent coating and the flame contour during the test. Note the infrared images were obtained using specific filters to observe the flame shape (Figure 13 (b)) and the surface of the sample through the flame (Figure 13 (c)). The burner flame engulfs the intumescent coating (Figure 13 (a) and (b)) and the expansion of the intumescent coating is limited. It does not form a regular expanded char but the expansion is a distribution of small bubbles at the surface of the material (Figure 13 (a) and (c)). It is probably due to the velocity of the gas jet limiting the expansion. Nevertheless, this type of coating provides an efficient protection (see above).

(a) **(b)** **(c)**

Figure 13: Intumescent coating on steel plate during the burnthrough test (a) optical image of the test, (b) infrared image of flame contour and (c) infrared image of the intumescent surface through the flame

A crude assumption without further measurement on the emissivity of the surface (here the emissivity is assumed to be one) permits to estimate the surface temperature as a function of time of the intumescent coating during the test Figure 14. It is an average temperature measured in an elliptic zone as shown in Figure 14 (b). The temperature increases sharply when applying the burner flame and reaches a pseudo steady state temperature of 615°C. This temperature makes sense since the temperature of the flame is about 1100°C associated to a heat flux of 116 kW/m² and so, with the velocity of the jet and heat losses (radiation and convection) a temperature of about 600°C is reasonable.



(a) **(b)**

Figure 14: Average temperature at the surface of the intumescent coating during the burnthrough test (a) on the zone defined by the ellipse (b).

CONCLUSION

Scaling reduction permits to successfully build several bench-scale tests mimicking different fire scenarios and providing appropriate and reliable results. Those benches were used to examine the fire protection of several materials (steel, wooden panel and CFRP) by intumescent coatings. Intumescent coating offers highly efficient way to fire protect the substrates and the intumescence concept is therefore a method of choice in the field of fire protection.

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