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An efficient bi-layer intumescent paint metal laminate fire barrier for various substrates: Extension to other application

Laura Geoffroy[a], Fabienne Samyn[a], Maude Jimenez[a,b] and Serge Bourbigot[a,b]

[a]Univ. Lille, CNRS, ENSCL, UMR 8207, Unité Matériaux et Transformations (UMET), Lille, France; [b]Institut Universitaire de France (IUF), UMET, Lille, France

ABSTRACT

Different types of passive fireproofing materials exist such as intumescent paints. Our approach was to modify the design the material instead of changing the formulations. By combining two concepts namely intumescence and delamination, and adjustable design, new effective fire barrier was developed to protect composites. It was evaluated using a burn-through fire scenario (heat flux of $116\,kW/m^2$ and temperature of flame of $1100\,°C$). The fire barrier revealed to provide fire protection to the composite and stabilized the temperature at the backside of the composite plate under $200\,°C$. Characterisations (cross-section observations, expansion measurements, etc.) were carried out on the samples and a mechanism of action was proposed.
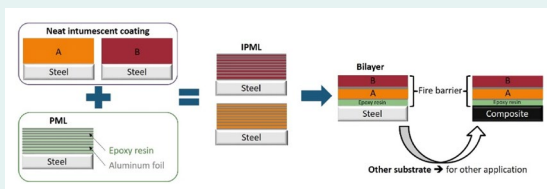
GRAPHICAL ABSTRACT

Introduction

In many applications, materials must meet stringent fire safety standards. Indeed, materials such as steel, aluminium or polymer based composite materials have to be protected against fire to avoid losing their structural properties. For example, when an unprotected steel structure is exposed to fire, due to its high heat conductivity, the temperature at the backside of the steel increases rapidly. With the increase in temperature, the mechanical properties of steel change, and thus steel begins to loose most of its structural properties between 470 and 550 °C (Alongi, Han, & Bourbigot, 2015; Dai, Wang, & Bailey, 2009; Mariappan, 2016; Petersen, Dam-Johansen, Català, & Kiil, 2013; Puri & Khanna, 2017). Moreover, in the case of polymer-based composite materials, their compressive and flexural load-bearing capacity decreases above their glass transition temperature. In addition, they start decomposing at 300–350 °C or even earlier (depending on the composite materials considered) releasing flammable volatiles feeding fire (Mouritz, Feih, Kandare, & Gibson, 2013; Mouritz et al., 2009). Therefore, to comply with more demanding standards (e.g., aeronautics), other passive fireproofing solutions, i.e., insulating systems designed to decrease heat transfer from a fire to the structure being protected, must be further developed. Among all passive fireproofing materials, cementitious products (Weil, 2011), fibrous materials (Didane, Giraud, Devaux, & Lemort, 2012; Didane, Giraud, Devaux, Lemort, & Capon, 2012), composites or fire protective panels (Mouritz & Gibson, 2006; Wang, 2016), ceramic coatings (Weil, 2011), or intumescent materials (Alongi et al., 2015; Dai et al., 2009; Mariappan, 2016; Petersen et al., 2013; Puri & Khanna, 2017) can be cited as examples.

The interest of intumescent paints to fire protect different kinds of substrates is particularly well-known and used. These paints form an expanded porous carbonaceous coating, which act as a fire barrier to protect a substrate (Alongi et al., 2015). However, even if intumescent paint is a mature technology, it becomes almost impossible to improve their fire protective performances using conventional chemistry.

Some works report an interesting strategy which consists in modifying the design and combining various materials to reach superior fire protection. Polymer Metal Laminates (Christke, Gibson, Grigoriou, & Mouritz, 2016; Timme, Trappe, Korzen, & Schartel, 2017), namely hybrid composite materials composed of a metallic and polymeric part, are part of this strategy. They also offer the versatility in their design. These materials were developed to obtain lightweight structures in many fields, notably in aeronautics and revealed high mechanical and fire protective properties (Christke et al., 2016; Timme et al., 2017). For the fire protection, delamination (located between the polymeric and the metallic

part) creates gas filled cavity, delaying the heat propagation in the material and thus protecting it (Christke et al., 2016).

Combining both concepts, i.e., intumescence (Alongi et al., 2015) and delamination (Christke et al., 2016), novel fireproof multi-materials were developed to reach high fire protection capable of responding to aeronautical constraints. The influence of the number of layers, the type or combination of paints were investigated, leading to two different types of design: 10 layers Intumescent Polymer Metal Laminate (IPML) (Geoffroy, Samyn, Jimenez, & Bourbigot, 2018) and bilayers intumescent paint metal laminate (Geoffroy, Samyn, Jimenez, & Bourbigot, 2020). Based on that (Geoffroy et al., 2020), it was demonstrated that two layers composed of two aluminium foils and two intumescent paints of different chemistries exhibited an outstanding fire resistance at the burn-through test. Indeed, thanks to two delamination (located between the aluminium foils and the coating) occurring during the fire exposure, and the intumescence coming from the two intumescent paints, heat diffusion was slowed down and revealed superior fire protection (Geoffroy et al., 2020).

In this context, bilayer metal laminate with the overlay of two different intumescent coatings revealed superior fire protection, especially at the beginning of the fire testing. The aim of this paper was to evaluate whether this fire-resistant barrier developed on steel can be used on other substrates for other applications. Therefore, composites (epoxy resin reinforced with carbon fibres) were evaluated as substrate. The targeted application is materials and components used in aircraft in engines and APU installations and in areas adjacent to fire zones as defined in ISO 2685. As for example, such composite is many used in aeronautics application like A350 and A380 or for structural aspect. After characterisation, the fire protective performances of the fire barrier were evaluated on the composite. Cross-section of residue observations, expansion and mass loss measurements were carried out to investigate the mechanism of action.

Materials and methods

Materials

$100 \times 100\,mm^2$ composite plates with a thickness of 3 mm and composed of carbon fibres and epoxy resin (with a volume fraction of fibres equal to 55%), were used as a substrate, and were purchased from Goodfellow (Huntingdon, Cambridge, United Kingdom). In addition to that, $100 \times 100 \times 3\,mm^3$ steel plates (grade XC40, Couzon Aciers Service, Saint Chamond, France) were used as an alternative substrate for comparison purposes. All of the steel plates were sandblasted (Normfinish, Jean Brel

SA, Stains, France) to improve the coating adhesion, using a white aluminium oxide (Guyson, Chambly, France) (particle size around 355–500 µm), at a 5 bars pressure. Acetone (VWR, Radnor, Pennsylania, United States), was used to clean substrates prior to use. 30 µm thick aluminium foils (Carl Roth, Karlsruhe, Germany) with 99% of purity were applied to elaborate metal laminate samples. Two intumescent coatings hereafter called A and B (proprietary formulation) were studied, and described in Table 1. A and B are conventional commercial epoxy and acrylic based coatings, respectively, containing classical intumescent agents and designed to pass hydrocarbon (UL 1709) and cellulosic (ISO 834) fire scenarios, respectively.

Samples elaboration process

Table 2 describes the name and composition of all samples studied in this work.

Bilayer Metal Laminate structures processing (AlA + AlB) was carried out in several steps (Figure 1). Aluminium foils were first cut into 100×100 mm² squares. Then, an aluminium foil was glued onto a substrate (a sandblasted steel plate or a composite plate, depending on the system studied S-AlA + AlB or Composite-AlA + AlB, respectively) using almost 1 g of an epoxy resin (DGEBA, purchased from Sigma-Aldrich, St Louis, Missouri, United Stated, reference 31185) cross-linked with Diethylenetriamine (purchased from Sigma-Aldrich, St Louis, Missouri, United States, reference D93856), with a ratio of 100:11.7. A curing step

Table 1. Intumescent coatings characteristics.

Paint	Targeted fire scenario	Type of paint	Type of intumescence
A	UL 1709	Epoxy, bi-component (Jimenez, Duquesne, & Bourbigot, 2006a, 2006b, 2006c, Jimenez et al., 2016)	Chemical
B	ISO 834	Acrylic, mono-component	Chemical

Table 2. Name and composition of sample studied.

Samples	Substrate	Epoxy glued	Metal foil	Coating A	Metal foil	Coating B
S-AlA + AlB	Steel	Yes	Aluminium	Yes	Aluminium	Yes
Composite	Composite	–	–	–	–	–
Composite-A	Composite	–	–	Yes	–	–
Composite-B	Composite	–	–	–	–	Yes
Composite AlA + AlB	Composite	Yes	Aluminium	Yes	Aluminium	Yes

was occurred for 48 h at room temperature, to fix the first aluminium foil onto a substrate. After that, a controlled mass of intumescent coating A was manually applied onto aluminium foil glued onto a substrate, using a film applicator. A second aluminium foil was added on top of the first paint layer, and the system was cured for 24 h at room temperature. Finally, a controlled mass of intumescent coating B was applied on the previous aluminium layer and the whole system was cured for 48 h at room temperature. Using this process, two intumescent bilayer metal laminates were prepared and composed of two aluminium foils and two layers of coating A and B (Figure 1). The only difference is that steel plate is used as a substrate for S-AlA + AlB while a composite plate is used for Composite-AlA + AlB.

To provide an accurate comparison between the bilayer intumescent paint metal laminate systems and monolayers of intumescent coatings, some additional samples were prepared, namely Composite-A and Composite-B. Composite plates were covered using a film applicator, with respectively, coating A (Composite-A) and coating B (Coating-B) (Table 2). Uncoated composite plate was also used as a reference for each fire test.

Moreover, it is noteworthy that the global intumescent coating mass deposited on the substrate was kept constant and corresponds to 20 g in order to be able to compare the different designs.
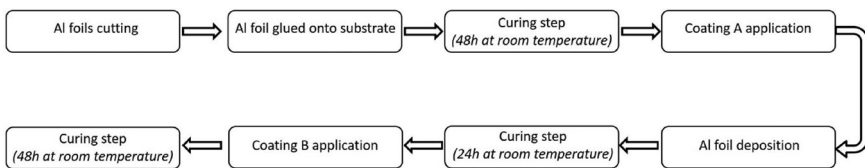

Figure 1. Bilayer metal laminate structure processing.

Fire testing

Burn-through test

The selected small-scale burn-through fire test (fully described elsewhere, in reference (Tranchard et al., 2015)) was developed based on two aeronautical certification fire tests: ISO2685:1998(E) and FAR25.856(b):2003. It consists in exposing the samples to a $116 \, kW/m^2$ heat flux, using a propane torch, as illustrated in Figure 2. Samples were fixed between 10 mm thick insulating panels from FINAL Advanced Materials (Calsil) and attached using four screws. This test, carried out without any ventilation (to avoid the influence of the convection), is divided into two
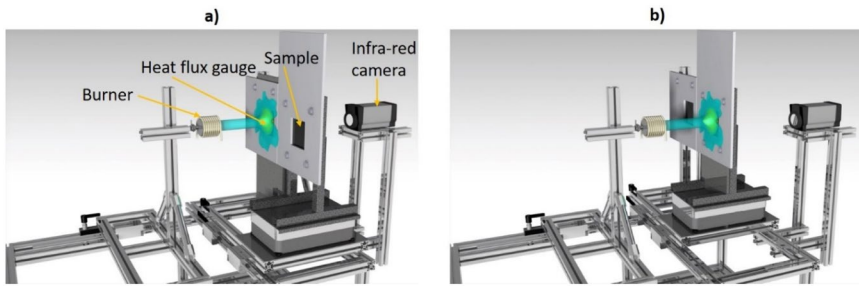
Figure 2. Burn-through test (a) calibration of propane flame and (b) sample fire exposure.

steps. The propane flame is first calibrated for 5 min to ensure that the heat flux was constant and equal to 116 kW/m² (corresponding to a flame temperature of ~1100 °C) Figure 2a). Then, the flame is switched on the sample and the sample is fire exposed for 30 min. At the end of the fire test, the sample is cooled down to the room temperature (Figure 2b).

The weight and thickness (using a ruler placed vertically) of all samples were measured before and after the torch test to calculate mass loss and intumescent expansion, respectively.

Temperature versus time measurements

For steel plate as substrate. To compare the resistance to fire of the samples deposited on steel plate, temperature versus time profiles were measured during the fire test. K-type thermocouples (TC SA, Dardilly, France) welded onto the middle of the backside of the steel plates were used. Agilent 34970 A data logger (Keysight Technologies, Santa Rosa, California, United States) was used to record all data. Each experiment was done in triplicate to check the repeatability.

For composite plate as substrate. For sample using composite plates as substrate, the temperature versus time profiles cannot be registered using a K-type thermocouple as it cannot be welded onto the plate. Therefore, during the fire testing, temperature changes of these samples were measured using an infrared camera (FLIR thermovision A40, Wilsonville, Oregon, United States), placed at a fixed distance from the backside of the steel plate. All sample backsides were previously spray-coated with a heat resistant mat black paint (Jelt noir mat 700 °C supplied by ITWPC Spraytec, Asnières-sur-Seine, France), ensuring that they all had the same surface emissivity (0.92). All the IR measurements were recorded using Thermacam research professional 2.0. software (FLIR system, Wilsonville,

Oregon, United States), and computed using Research IR software (FLIR system, Wilsonville, Oregon, United States). As above, the experiments were done in triplicate.

Characterisations

Mass loss measurements

The mass of the samples before (m_{before}) and after 30 min of fire exposure ($m_{after\ 30}$) were measured using a scale. The mass loss (ML) is calculated as the ratio between the difference of mass loss after and before fire testing to mass before fire testing, as illustrated by this equation: ($ML = (m_{after\ 30} - m_{before})/m_{before}$).

Expansion measurements

A numerical calliper and thickness gauge were used to measure the thicknesses of the samples before (e_{before}) and after 30 min of fire exposure ($e_{after\ 30}$). The expansion (E) was determined by the ratio between the difference of thicknesses after and before fire testing to thickness before fire testing ($E = (e_{after\ 30} - e_{before})/e_{before}$).

Optical microscopy

Before fire testing, optical microscopy observations were carried out on cross sections using a microscope VHX-1000 HDR (High Dynamic Range), Keyence (Osaka, Japan). To prepare the cross sections, each sample was put in liquid nitrogen for 5 min and then cut using a blade and a hammer. Using this method, a brittle fracture was obtained without affecting the integrity of the sample. Samples obtained were then embedded in an epoxy resin, which was then cured for 48 h at room temperature, and finally polished (up to 1/4 μm) using silicon carbide disks (ESCIL, Chassieu, France). It is noteworthy that the cross-section observations were done without substrate.

After fire testing, the cross-section of the residues was also cut (using a thin cutter blade) and observed visually in order to see if delamination phenomenon occurs.

Thermal analyses

Thermogravimetric analyses (TGA) were carried out on composite plate, using a TG 209 F1 Libra supplied by Netzsch (Selb, Germany). The thermograms were recorded in the 40–800 °C temperature range, with a heating rate of 10 °C/min under nitrogen flow (40 mL min^{-1}). The TGA

under nitrogen was chosen because no thermo-oxidation occurs during the fire test because of an oxygen depletion zone between the flame and the decomposing material.

Thermal conductivity

Thermal conductivity was measured at room temperature by a hot disk thermal constant analyzer (Hot Disk TPS 2500S, Thermoconcept, Bordeaux, France), which is a transient plane source technique (Gustafsson, 1991). The sensor (warmth emitter) is mold in the middle of two samples ($25 \times 25 \times 6\,mm^3$) to ensure a good contact during the experiment. The axial and radial conductivity measurements were carried out by applying a power of 0.06 W for 10 s for composite plate. The experiment was repeated four times to check the repeatability of the measurement. The presented results are the average of the obtained values.

Results and discussion

Characterizations before fire testing

Cross-section picture of Composite-A, Composite-B and Composite-AlA + AlB was observed using optical microscopy (Figure 3). To examine the cross-section, the coatings were not applied on substrate because of the optical microscopy sample preparation. Moreover, it is noteworthy that the cross-section pictures of S-AlA + AlB (not shown) are similar to those of Composite-AlA + AlB, the only difference is the substrate on which AlA + AlB was coated. Figure 3(c) reveals that no bubble or void can be detected between coating A, coating B and aluminium foils. The adhesion between layers looks homogeneous. The coating A and coating B thicknesses are similar for each sample and the averages correspond to 740 μm and 650 μm, respectively.

Table 3 gathers the quantitative values of all samples studied. For each sample, same mass of coating, 19 g, was deposed on substrate, and thus same total thickness is obtained, around 5 mm. All samples look
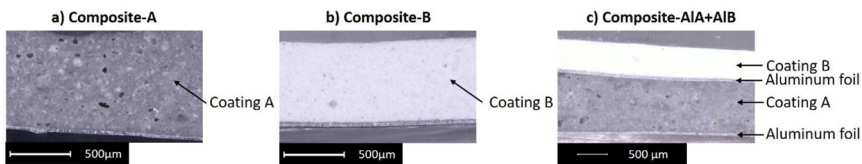

Figure 3. Cross section optical microscopy analysis before fire testing of: (a) Composite-A, (b) Composite-B and (c) Composite-AlA + AlB.

Table 3. Quantitative characterization of all samples studied before fire testing.

Samples	Mass of coating A (g)	Mass of coating B (g)	Mass of substrate (g)	Total mass (g)	Total thickness (mm)
S-AlA + AlB	9.39	10.22	233.81	253.15	5.08
Composite	–	–	55.5	55.5	3
Composite-A	18.55	–	54.86	68.73	5
Composite-B	–	20.27	53.19	73.1	5.1
Composite AlA + AlB	9.24	10.41	56.89	76.1	5

homogeneous, the only difference between them is the substrate mass, because of the high density of steel compared to composite (mass of the composite material is divided per 4.3 compared to steel).

Fire testing

Temperature as a function of time was shown in Figure 4 and illustrates the influence of the substrate modification (with the substitution of steel by composite plate). In addition, the temperature reached after 30 min fire testing and the slope of the curves at different time intervals during fire test were gathered in Table 4. The temperature at the backside of the virgin composite plate dramatically increases from the beginning of the fire test (with 234 °C/min as a starting slope for 1 min), and reaches its steady state temperature at 328 °C, after 5 min fire test (Figure 4 and Table 4).

For the composite plate coated with intumescent paint A (Composite-A), the temperature rapidly increases for 2 min, with a slope of 90 °C/min (which is 2.60 times lower than virgin composite). From 2 min to 5 min, it raises with a slope of 32 °C/min and reaches its steady state temperature at 288 °C (12% lower than for virgin composite), after 5 min fire exposure. Similar trend was observed for Composite-B until 2 min fire test., i.e., a fast temperature rise with a slope of 106 °C/min (which is 2.21 times lower than the slope obtained for virgin composite). Then, a stabilization after 2 min fire exposure is observed and temperature reaches 217 °C, −34% lower than the temperature reached for the virgin composite. The deposition of neat intumescent coatings (A and B) on composite reduces slightly the temperature rise and significantly the steady state temperature.

Regarding Composite-AlA + AlB, a strong reduction of temperature rise is observed compared to neat composite. Compared to S-AlA + AlB, similar temperature rise was measured but the steady state temperature reached at the end of the test is much higher (it makes sense because of the high
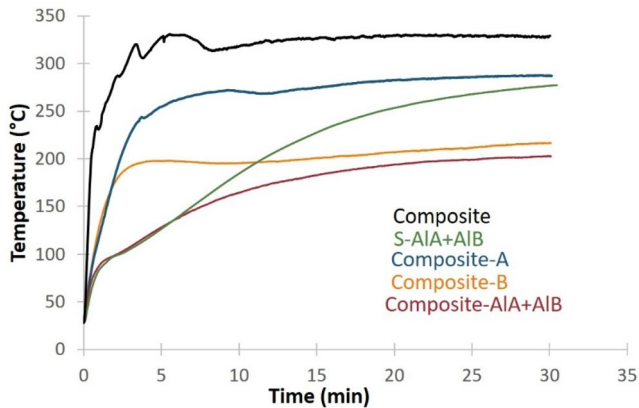
Figure 4. Fire behaviour of S-AlA + AlB and of systems deposited on composite substrate.

Table 4. Rate of temperature and temperature reached during fire test for each sample studied.

Samples	Rate of change of temperature (°C/min): from 0 s to 1 min	Reduction Against composite	Rate of change of temperature (°C/min): from 2 to 4 min	Reduction Against composite	Temperature after 30 min fire test (°C)	% Reduction of temperature after 30 min fire test Against composite
S-AlA + AlB	62		9		278	
Composite	234		29		328	
Composite-A	90	÷ 2.60	32	× 1.10	288	−12
Composite-B	106	÷ 2.21	8	÷ 3.625	217	−34
Composite-AlA + AlB	59	÷ 3.97	10	÷ 2.90	203	−38

The bold values correspond to the slope of the temperature vs time curves at different times: 0 s to 1 min, 2 to 4 min an after 30 min.

thermal conductivity of steel). Similar slopes (59 °C/min from the beginning to 1 min, and 10 °C/min from 2 min to 4 min) are obtained. After 6 min of fire test, the temperature measured at the backside of S-AlA + AlB still increases whereas a steady state for Composite-AlA + AlB is observed. As a consequence, after 30 min fire test, the temperature reached for Composite-AlA + AlB is 203 °C, 38% and 27% lower than temperature reached for neat Composite plate and S-AlA + AlB, respectively. Based on these results, the fire barrier (AlA + AlB) exhibits high fire resistance.

Mechanism investigation

To explain the fire performance of samples gathered in Figure 4, the residues at the end of fire test were collected and analyzed (Table 2 and

Figure 6). At the same time, the mass loss and expansion of each sample was determined (Table 5) after 30 min fire exposure.

For the virgin composite, delamination of the substrate was observed at the end of the fire exposure (Figure 6a). The epoxy resin looks also decomposed associated to 32% mass loss. Note this mass loss is the same as that measured by TGA at high temperature (Figure 5). An expansion of 132% was also measured after fire testing (Table 5).

Figure 6(c) and Figure 6(d) shows the residue obtained for Composite-A and Composite-B, respectively. No delamination between substrate and intumescent residue occurs during the test. However, in the case of Composite-A, delamination was observed between carbon fibres and the composite: the substrate starts to delaminate, while it does not happen in the case of Composite-B. This difference can be explained by the temperature reached after 30 min fire exposure which is 70 °C higher for Composite-A (288 °C) compared to Composite-B (217 °C). It is noteworthy that the temperature reached at the backside of Composite-A (288 °C) is below the epoxy resin decomposition temperature (360 °C) measured by TGA (Figure 5). This difference can be explained by two aspects. On one hand, the low thermal conductivity of composite should involve a large temperature gradient inside the material during the test.
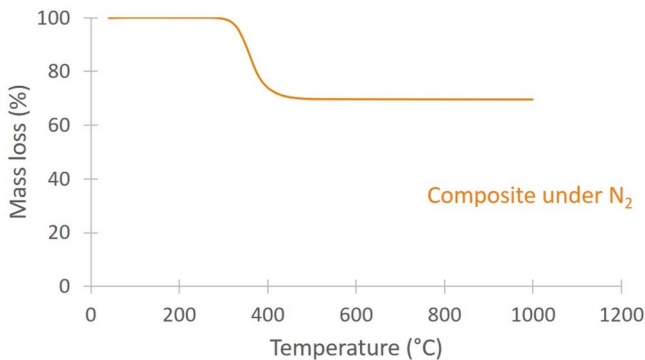

Figure 5. TGA of composite under N_2.

Table 5. Mass loss and expansion after 30 min fire exposure for each sample studied.

Samples	Mass loss after 30 fire exposure (%)	Expansion after 30 min fire exposure (%)
Composite	32	132
S-AIA + AIB	3	610
Composite-A	11	170
Composite-B	26	529
Composite AIA + AIB	6	640

Indeed, the axial and radial thermal conductivity of composite were measured equal to 0.61 W/(m K) ± 0.03 W/(m K) and 2.12 W/(m K) ± 0.09 W/(m K), respectively. Therefore, even if the backside temperature of the Composite-A (288 °C) is lower than the decomposition temperature of the composite measured by TGA (360 °C see in Figure 5), carbon fibres delamination can be observed because of the decomposition of epoxy resin in the layers close to the interface coating/composite. On the other hand, according to Tranchard et al. (2015) and Mouritz and Gibson (2006), the delamination effects can appear in the virgin part of material, before the beginning of the decomposition of the material. Indeed, due to the thermal expansion coefficient difference between carbon fibres and epoxy resin, it creates delamination upon heating in the first plies.

Furthermore, a char (carbonaceous residue) was observed after fire testing with an expansion equal at 170% and 529%, respectively. This expansion difference of the paint is related to the chemical composition of the intumescent coating (Geoffroy et al., 2020).

Regarding Composite-AlA + AlB, delamination occurs between coating A and the two aluminium foils (Figure 6e) similar to that observed for S-AlA + AlB (Figure 6b). This observation could explain the same low temperature rise at the beginning of the fire testing. Indeed, delamination in the bilayer improves the fire protection performances creating a heat sink and hence, delaying the heat propagation in the system (Geoffroy et al., 2020). Moreover, due to delamination and also to the intumescent coatings swelling, the expansion after 30 min fire test of S-AlA + AlB and Composite-AlA + AlB reaches 610% and 640% with a low mass loss equal at 3% and 6% (Table 3), respectively.

Furthermore, the substrate of Composite-AlA + AlB remains intact, no delamination or epoxy degradation occurs during the fire exposure. This observation is consistent with the low temperature reached in the steady state (203 °C) (Table 5). It evidences also that the thermal barrier behaves in the same way whatever the substrate used and it suggests the
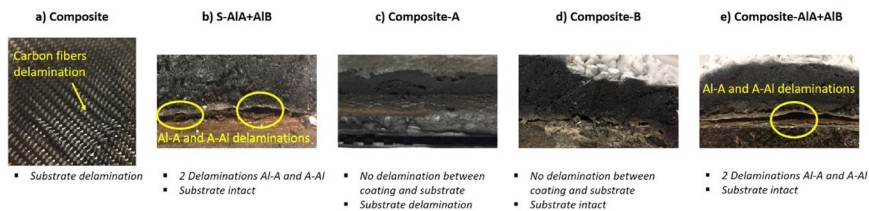

Figure 6. Residues aspect after 30 min fire exposure for (a) Composite, (b) S-AlA + A lB, (c) Composite-A, (d) Compaosite-B, (e) Composite-AlA + A lB.

delaminating intumescent fire barrier may be applied each of substrate needed to be protected.

To summarize, at the beginning of the fire test, the similar fire behaviour between S-AlA + AlB and Composite-AlA + AlB can be explained by the use of fire barrier design and coatings (two aluminium foils with coatings A and B), and thus the occurrence of same phenomenon (delamination between both aluminium foils and coating A and intumescence of coating A and B). Then, the heat diffuses through the system to the substrate and differs according to the thermal conductivity of the substrate. As there is no decomposition of the composite substrate during the test (temperature at the interface fire barrier substrate below the decomposition temperature of the composite), the temperature changes are governed only by the thermal conductivity of the composite which is almost 100 times lower than for steel (ratio about 0.6 (Joven, Das, Ahmed, Van, & Ie, 2012) to 68 W/(m K) (Akni, 2015), respectively). The temperature in the stationary state is therefore much lower than in the case of steel. The fire-resistant barrier created in this work can thus be used to protect other substrates, and provide a fireproofing protection for other applications.

Conclusion

Substituting steel by polymeric composite, a benefit was identified: the total mass of the sample was reduced per 4.3 and thus lighter materials were elaborated. Moreover, after 30 min fire exposure, the temperature at the backside of composite plate does not exceed 200 °C. These excellent fire protective properties can be explained by (i) delamination located between coating A and both aluminium foils, which delays the heat spread in the system at the beginning of the test, (ii) the swelling resulting from the intumescent phenomena, (iii) the higher protective performance of composite compared to steel, composites having a lower heat conductivity. The same fire protection is measured for steel with the difference that the steady state temperature is higher than that of composite based materials because of the high heat conductivity of steel. This work highlights that the fire barrier developed is very efficient, flexible and can protect diverse substrates (including organic ones) against fire for various other applications such as aeronautic or structural engineering fields.

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