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Fire Resistance of Aeronautical Composite Materials under Reduced Pressure Conditions

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Abstract

Safety aboard aircraft is one of the main concerns for aircraft manufacturers and airline companies. The use of composites, for weight reduction and energy saving purposes, causes the fire scenarios to evolve. In all fire configurations, the fuselage must be a safe barrier to flame penetration into the cabin and maintain the fly home capacity of the plane by avoiding an uncontrollable fire situation.

The objective of this work is to determine the resistance to fire of new aeronautical composite materials in various representative conditions of fire, particularly in reduced pressure medium, i.e. simulating an in-flight engine fire.

1 Context and objectives

1.1 Introduction

Fire safety is considered as a major research topic to enhance the technology aiming at the improvement of safety for passengers and crew. The statistical studies of accidents involving fire show that in most of cases fire is due to oven, but, also to the fire propagation into the aircraft cabin caused by the combustion of fuel resulting from fuel leakage, tank damage, material or electronic default during in-flight or post-crash fire scenarios.

For conventional aircraft, the barrier to an external fire is the fuselage (Figure 1) made of an aluminium skin, thermo-acoustic insulation, air layer and cabin wall.

In the new generation of aircraft (A350 or B787 families), significant operational cost savings are gained from lower weight, corrosion resistance, and less maintenance due to improve fatigue strength compared to conventional aircraft structure. Then, the designers have increased widely the use of flammable polymer materials for interior decorative panels and equipment. More recently, composite materials (carbon fibres embedded in thermoset or thermoplastic resins) replaced also metallic elements in the structure. For example, they represent 53% in mass in an Airbus 350.

But, as aluminium alloys melt around 660°C, polymers burn due to the resin pyrolysing from low temperature (≈380°C) that adds an important calorific power. Nevertheless, they are authorized by the aeronautic regulation if they can resist to a flame attack consecutive to an engine, electrical or mechanical trouble, to ensure a safe fire barrier for passengers and crew in cases of external fire and to protect the flying equipment (wings, cowling, empennage, flaps, rudder winglet, slats, spoiler, stabilizer, elevator, landing gear, fuel tank, power plant, etc.) for a safe fly home and emergency landing. For all these reasons, the development of new safety approaches to understand the fire behaviour of these materials is crucial.

A postcrash fire is the configuration where passengers and crew have the larger probability to survive if the crash is not too severe. Fires can also occur in low pressure environment associated with high altitude locations (local pressure is up to 35% less than at the see level - at 10 km altitude the local ambient pressure is 0,26 atm) and inside sub-ambient pressurized compartments of a cruising aircraft. Then, burning characteristics and rates play an important role in characterizing the flammability of solid material as well as the local oxygen concentration [1]. Pressure and oxygen concentration can act directly on the chemical reaction in gaseous phase, but also on the thermal composite degradation: this is the specific fire scenario studied in this work .

1.2 Context of aeronautic material certification

To get a flight agreement, the structure materials are submitted to standard passed/failed burnthrough tests (Figure 2, Figure 3) [2, 3] accurately defined by FAA[1] [4, 5, 6, 7, 8, 9, 10, 11, 12]. Full-scale, medium-scale and lab-scale tests

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[1] FAA Federal Aviation Administration

are conducted to investigate ways of improving the fire burnthrough resistance of aircraft fuselages to the impact of a flame.

Marker [6] performed full-scale fuselage burnthrough tests to access the insulating properties of materials. From a data analysis of full size fire tests or accidents, FAA defined thermal and aerodynamical conditions for the gas temperature and heat flux at the material surface representative of generic cabin, cargo, engine and post-crash fires. It performed lab-scale tests involving the Bunsen-burner flammability device [8] with working conditions specified by AC 20-135 [U.S. Department of Transportation, 1990]. Later on, Lyon [9] developed a research programme to qualify fire safe material assemblies for commercial aircraft, but these materials were mainly interior materials and not the structural ones.

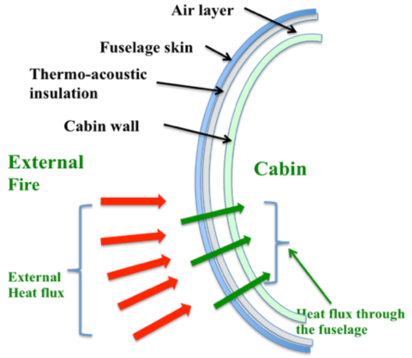

Figure 1 : Fire barrier to flame penetration with an aluminium or composite skin

Figure 2 : DGA : (LeNévé, 2010). Developed Standard Test

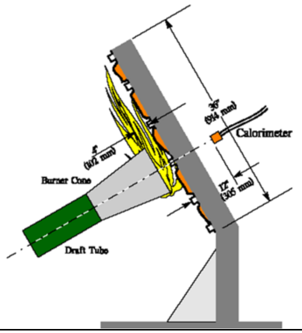

Figure 3 : FAA apparatus : (1)AC 20-135; (2)AC 20-107B

Among the set of tests, the Burnthrough Test (BT) is the most widely used to quantify the fire resistance of structural materials. It allows the determination of the BurnThrough Time (BTT) of the material defined as the time needed for the flame to penetrate the fuselage and spread inside the aircraft cabin in conditions representative of a cabin, engine or post-crash fires. The BTT must be always at least greater than the conventional evacuation time of two minutes defined by the regulator, then, the minimum required BTT was fixed to four minutes.

For the two main aeronautical types of external fire, the heat fluxes q and gas temperature at the material surface are calibrated to:

- engine fire (AC 20-135 norm), the incident heat flux at the material surface is fixed to 106 kW/m^2 with a gas temperature of 1093 ±83°C;
- post-crash fire (AC 20-107B norm), the incident heat flux at the material surface is fixed to 182 kW/m^2 with a gas temperature of 1160 to 1200 ±93°C.

To improve fire safety, researches and tests must be performed to develop, characterise and understand in a better way the behaviour of aeronautic materials during fire to enhance the fire barrier efficiency, even in reduced pressure, environment partly simulating fire in altitude.

1.3 Objectives of this work

The first objective of this work, supported by the European Community, has engineer and industrial purposes. It consists in measuring the fire resistance of composites for aircraft structure to determine their flammability and burning properties together with a material ranking.

In a first step a lab-scale setup allowing was designed and built to determine the material behaviour during a flame attack by the measurement of the BTT of composite samples to qualify and measure the fire performances of the polymers, in thermal and aerodynamic conditions representative of real fires defined by the regulator.

In a second step eight aeronautical thermosets (epoxy resin embedded carbon fibres or phenolic resin with glass fibres) and thermoplast composite (PEEK resin reinforced by carbon fibres) materials were charactering.

Due to a serious lack of knowledge on the composite thermal behaviour in ambient reduced pressure, in this work, the focus is particularly put on the effects of this parameter on the fire development. Two composites are studied: the first one, referenced as AcF2, is a thermoset composite (epoxy resin/carbon fibres), the second one, referenced as AcF6, is a thermoplast composite (PEEK resin/carbon fibres). This approach is complementary to studies provided with apparatus more conventional such as cone calorimeters, pyrolysis combustion flow calorimeter [13, 14, 15]. On the other hand, ignition and burning of polymer or liquid fuel in elevated pressure are widely studied. As an example, we report a study performed in the present set-up [16].

The second and complementary objective is more academic. It is focused on the understanding of the phenomena involved during the degradation of composites submitted to a strong heat fluxes at its surface. The study should determine if the composite pyrolysis rate is controlled either by a kinetic or a diffusional regime corresponding to a simple thermal pyrolysis of the material, an exothermal heterogeneous reaction at its surface, or an heat transfer from a from the flame to the composite surface.

A new database of fire resistance of aeronautic composites for aircraft structure will be available for engineers and modellers purposes.

2 The experimental apparatus

An original home-made laboratory scale device was designed and drawn from the standard FAA BurnThrough Test apparatus. The materials can be submitted to a calibrated flame impinging on its surface in order to determine their fire resistance in thermal and aerodynamical conditions similar to those of regular FAA tests. The two main parameters are the heat flux and the gas temperature at the composite surface, the other ones are the composition of the material, the sample thickness, the ambient pressure and oxygen mass fraction, and the burner-sample distance. The developed device is well adapted to allow the reproduce inflight engine and post-crash fires.

2.1 The original test facility: the Pprime device

The Pprime device [17, 18], latter used on [19], consists of a generator of a jet of hot premixed propane/air combustion products impinging the surface of the material in full-scale fire conditions. The FAA standard BurnThrough Test was mimic using a small-scale gas burner. A homogeneous global heat flux and gas temperature at the external material surface (AC 20-135 and AC 20-107B specifications) impinges the exposed material sample surface. The sample dimensions are reduced to 0.05x0.05 m^2 for cost reasons that allows the multiplication of tests.
The combustion products at the exit of the combustion zone are diluted before the burner output with an adjustable O_2/N_2 mixture to generate a given thermal impact at the sample surface. The system (Figure 4, Figure 5, Figure 6) is designed to operate both in vertical or horizontal positions, with an excess of oxygen (simulation of flame ventilation) or in reduced pressure (simulation of fire in altitude).
Before and after each set of tests, a calibration of the flow is performed to ensure that the thermal impact at the sample surface is within specifications. Calibration tests for heat fluxes are performed with a water-cooled total heat flux meter (Custom-made model from Captec Enterprise, France – Range 0-200kW/m^2). The value of the heat flux is cross-checked by measurements with an in-house heat transfer copper tube exchanger [20]. A good agreement was always obtained between the two methods for all selected conditions.
The device allows mainly to follow, during the tests, the evolution of the Mass Loss Rate (MLR) of the composite and the temperature on its two sides. This leads, according to the flight configuration, to the determination of the BurnThrough Time (BTT, time for the flame to drill the material) which is generally considered by the regulators as a key parameter.
To ensure a homogeneous heat flux at the sample surface, the flow aerodynamic (mean velocity field, turbulence, symmetry, buoyancy effects on the hot jet) at the burner output is beforehand characterized by PIV. The results have shown a well organised flow structure between the burner and the impinging stagnation plane.
Nevertheless, although the developed system provides reliable information on the fire resistance of materials, standard FAA tests will be always required by the regulators for a material certification.

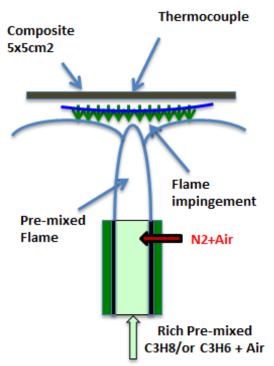

Figure 4 : Schematic of Pprime lab-scale BurnThrough Test facility

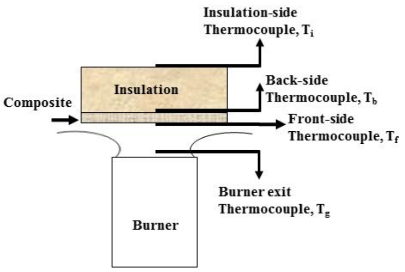

Figure 5 : Scheme of the material stack for BurnThrough Tests and thermocouple locations

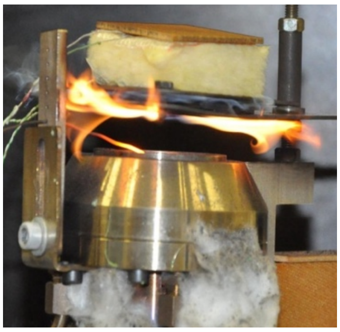

Figure 6 : Behaviour of the stack of materials at lab-scale BurnThrough Test facility

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2.2 Burner working conditions

The test conditions are adjusted for thermal output powers of the burner varying from 3.5 to 6.0 kW and a constant gas mass flow rate of combustion products of ≈ 0.0028 kg/s (130 lit/min, Reynolds number ≈ 6000). The complement to the flow rate up to 0.0028 kg/s is provided by the injection, by a downstream orifice, of nitrogen, or nitrogen/oxygen mixture. Then, it can be evaluated the influence of the combustion of the material degradation products that allows the burning of the excess of reactants in post combustion. The required flow stretching at the wall (≈ 430 s^{-1}), the total heat flux (convective heat transfer coefficient) and the gas temperature at the material surface are reached by adjusting the air and propane mass flow rates. The distance between the burner and the sample can be adjusted from 0.017 m to 0.052 m. Although this configuration results in a much smaller flame thermal power than standard FAA tests, on the impingement area, the required heat flux and temperature levels can always be reached.

2.3 The sample holder

The schematic and snapshots of the BurnThrough Test facility are shown in Figure 4 to Figure 6. As illustrated, the sample, $0.05\times0.05 m^2$ surface and 0.002 m or 0.004 m thickness, is trapped inside the holder between two 0.002 m thick mild steel sheets. A window of 0.035 m diameter (the diameter is identical to the burner tube exit) is drilled in the holder for the impingement of the hot flux on the sample surface. The small thickness of the material allows neglecting the 3D effects on the thermal diffusivity inside it.

The sample and its holder are mounted on a weight sensor. The holder is motorized to keep it in place (in front of the flow of combustion products) exactly at the starting time of the test, particularly for trials inside an enclosure.

To impose a representative and similar thermal boundary during all tests, a thermo-acoustic blanket (≈ 0.025 m thickness) is located on the not exposed sample backside. Moreover, this arrangement mimics the insulating effects of an aircraft fuselage assembly (Figure 1 and Figure 5).

2.4 Diagnostic methods

Mass flow meters and thermocouples are used to check the burner working conditions during the characterisation of BTT in order to deduce flammability and burning properties of the composites.

Mass evolution of the sample : a weight sensor (full-scale equals to 0.3 kg with accuracy of ≈ 0.06 g) measures the evolution of the Mass Loss (ML) of the sample. The time derivative of the ML signal provides the material Mass Loss Rate (MLR). A sample weighting before and after each test gives the Total Mass Loss (TML) allowing both to check the consistency with TML obtained from the time-integration of the weight signal ML, and the mass of the unburn resin.

Temperature evolution at the sample sides : Temperatures are measured on both sides of the sample with K-type thermocouples ($0.5\ 10^{-3}$ m diameter, coated to avoid short-circuit with the carbon fibres) glued on each side. During the test, glue and material burn. The validity of the thermocouple signal directly exposed to the flame can obviously be discussed (influence of the contact of the temperature welding against the sample surface).

Video recordings are also simultaneously performed.

Temperatures and Mass Loss Measurements are performed during independent trials to avoid a potential aerodynamic perturbation of the thermocouple wires during the Mass Loss measurements.

2.5 The reduced pressure enclosure test facility

The interest of the development of tests in reduced pressure is mainly focused on the understanding of the chemical kinetic on the material pyrolysis. Other works, performed in reduced pressure allows the simulation of flame spread over a solid (or liquid) fuel in micro-gravity [21] for spacecraft system [22].

In order to perform tests in reduced pressure for a partial simulation of altitude, the device (Figure 7) is placed inside a pressurized metallic chamber (internal diameter 0.345 m, height 0.68m, ≈130 litres) equipped with Pyrex windows. An air mass flow rate of 400l/min is introduced from the bottom of the chamber to swap the enclosure and to warranty a constant ambient air temperature and gas composition around the impinging jet. A pressure regulated vacuum pomp controls the vessel pressure between 0.1 MPa (≈ 1 atm-sea level) to 0.03 MPa (≈ 0.3 atm ≈ 30 000 feet). The studied pressures are 0.1, 0.07, 0.05 and 0.03 MPa. The ambient low temperature in altitude (up to -50°C) could not be reproduced. A heat exchanger cools the exhaust gases before pumping them in order to avoid the decrease of the thermodynamic efficiency and the live time of the vacuum pump.

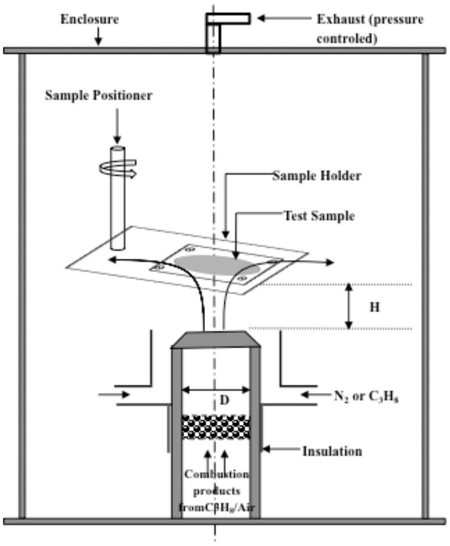

Figure 7 : Behaviour of the stack of materials at lab-scale BurnThrough test facility

Figure 8 : The pressured chamber

The flow conditions at the impinging surface (heat flux q and temperature) are beforehand measured and calibrated for all selected pressures. For two flame conditions (q equals to 75 and 106kW/m^2), Figure 9 presents only a limited decrease of the heat flux at the impinging plane which can be attributed to the variation of the molecular diffusion coefficient with pressure. No experimental correction was performed and during this work, the burner characteristics are supposed to be invariant to the pressure.

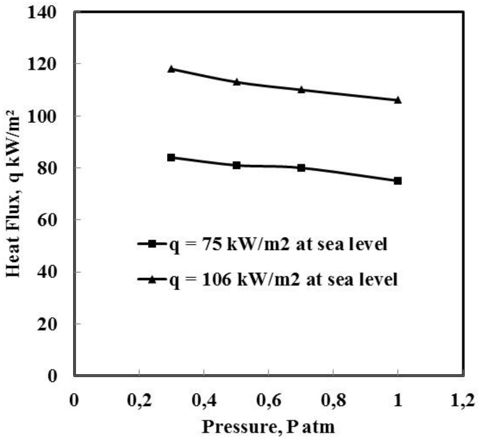

Figure 9: Influence of the pressure P on the heat flux at the sample surface for 106 and 75 kW/m^2

2.6 Experimental data base

The Pprime device allows the determination of the flammability and burning properties of the composite under full scale fire specificities from the measurement of the sample **Mass Loss (ML)** and **temperatures** (T_{front} , T_b) at front and backside of the sample. A processing of these signals provides the **Burnthrough Time (BTT)**, the material **Mass Loss Rate (MLR)** – *time derivative of the ML* or **burning rate** - which is proportional to the **Heat Release Rate** (HRR), the **pyrolysis and ignition times t_{ign}** (*the derivative of the MLR becomes infinite*) , the **off-gassing time** (time of the beginning of the degradation at the sample backside), the **Peak of MLR (PMLR),** the **total Burning time**, **mean burning time.** The definition of these variables are reported on Figure 10.

These parameters allow the evaluation, for full scale flame conditions, of a dimensional parameter for fire growth (**FIGRA (Fire Growth Rate** [23]) defined below from t_{ign} and the square of HRR, allowing a ranking of the fire resistance of the material, etc.. .

$$FIGRA = \left(\frac{\dot{Q}_{\max}}{t_{\max}}\right)_{SBI} \propto \frac{PHRR^2}{t_{ign}}$$

where Q'_{max} is the maximum of heat release.

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To conclude, the working parameters of the tests are the material composition and thickness, the incident heat flux q, the gas temperature T_g, the burner-sample distance (flow stretching), the ambient pressure, the mass fraction of oxygen in the hot jet and the impinging angle (results not presented here).
The obtained database was compared to results obtained by cone calorimeter tests for lower thermal conditions of heat fluxes at the surface. The agreement are good.

3 Experimental results

3.1 Generic behaviour of the composite submitted to a strong external heat flux

To validate the test device, preliminary BurnThrough tests were performed with aeronautical aluminium samples under the FAA specifications. The results show than the BTT values obtained with the present device (≈60s) are consistent to those obtained with standard BT tests [24, 25] that can qualify the developed set-up. To work out the BTT device and check the reproducibility of the results, for cost reason, the first trials are performed with black PMMA samples.
Then, the fire performances of thermoset composites (carbon fibres embedded in an epoxy resin matrix, glass fibres embedded in a phenolic resin) and thermoplasts (carbon fibres embedded in PEEK resin) could be investigated.

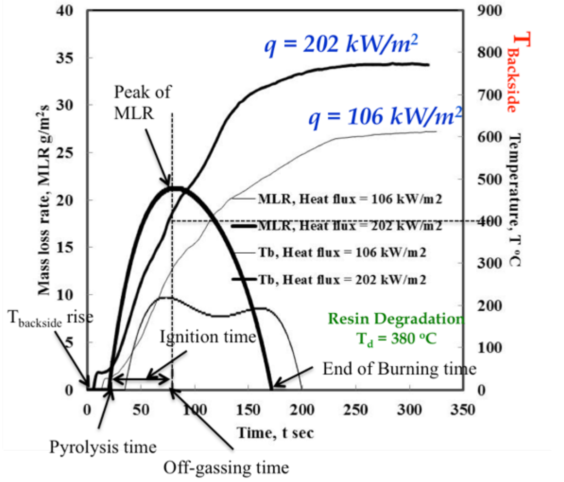


Figure 10: Generic evolution of the MLR of a composite sample (thermoset) under thermal condition AC 20-107B

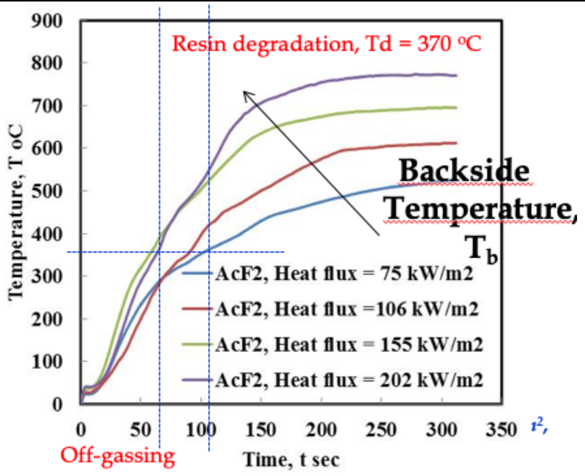


Figure 11 : Backside temperature evolution function of the incident heat flux

The first result shows that, under the aerodynamic and thermal conditions representative of post-crash (AC 20-107B norm) or engine fire (AC 20-135 norm), for all studied aeronautic materials, the sample is never burnthrough during the first 15 minutes of the tests while regulation requires only a passed test after four minutes. As well, all studied composites (both thermosets and thermoplasts) pass the BT test: they present all a better fire resistance than aluminium slabs (BTT≈ 60s) of equivalent thickness. It can be deduced, a priori, that composites can be introduced for aircraft structure. Consequently, although they burn, the composites provide an efficient firewall to the flame penetration for standard flame attack. This phenomenon can be explained by the role plays, after the pyrolysis of the resin, by the carbon fibres to re-radiate the incident heat flux; the net conductive heat flux penetrating through the composite decreases, and finally, BTT increases.

On Figure 10, the timing of the phenomena such as the ignition, pyrolysis, burning, off-gases are reported to defined these variables.

According to the heat fluxes q, on Figure 10, two shapes of evolutions of MLR (Mass Loss Rate) for AcF2 (thermoset composite) are observed:

For q=220 kW/m^2
After the pyrolysis delay where the resin is heated up to its degradation temperature (the time of heat penetration $t_{penetration}$ in the composite is 2,4 s from the beginning of the front face heating), MLR, controlled by the value of the heat flux at the front side of the sample, grows up to its single maximum, the peak of Mass Loss rate (PMLR) which is proportional to the maximum (PHRR) of Heat Release Rate (HRR). At this moment, the backside temperature T_b

of the sample (i.e. 350-380°C for epoxy resin) reaches its degradation one (Figure 11). MLR decreases function of the residual resin availability. The backside surface of the material is pyrolysed, the material becomes permeable to degradation products. Black smokes are emitted inside the cabin with a real potential of poisoning occupants and a potential hazard of propagating the fire inside. This time, called **off-gassing time**, is around 1 or 2 minutes, that to say the same order of magnitude than the aluminium BTT. Then, it can be considered that, although the material is not drilled, the degradation products are emitted from the backside. Nevertheless, this risk for passengers seems limited due to the airproof of thermo-acoustic blankets and cabin wall. MLR decreases with the degradation of the residual resin.

For q=106 kW/m^2

For lower incident heat fluxes at the wall (106 kW/m^2), we observe a change in the MLR evolution with the presence of two MLR peaks. The first increase of MLR is observed up to a first maximum (PMLR) with a value strongly smaller those for q=220kW/m^2. Then, MLR decays before to increase again up to a second PMLR followed by the pyrolysis of the remaining resin. These phenomena are attributed to the formation of a char protective layer during the first rise of MLR which re-radiates a part of the incident heat flux and decreases the net heat flux q_{net}. The heat penetration to the sample is partly blocked and the degradation of the resin decreases. Then, the char burns and the resin continue its pyrolysis up to the second PMLR.

This interpretation, previously proposed for wood or PMMA and also from experimental results and numerical model for polymer materials [26] are adapted here for the composite burning.

3.2 Burning rate evolution as a function of the incident heat flux at the wall in atmospheric pressure

Figure 12 reports the MLR evolution for four incident heat fluxes q at the sample surface. The two MLR behaviours are observed, two MLR peaks for the higher heat fluxes, two smaller for the two lower q values. These two types of evolution can be correlated with the backside temperature T_b evolution (Figure 11) which decreases with q slowing down the pyrolysis rate.

Complementary information can be extracted from the MLR evolutions with q. The different characteristic times ($t_{pyrolysis}$, $t_{ignition}$, t_{PMLR}, $t_{off-gasing}$) increase with q, and obviously, the total burning time (time integration of MLR) is lengthen in order to allow the complete burning of the resin.

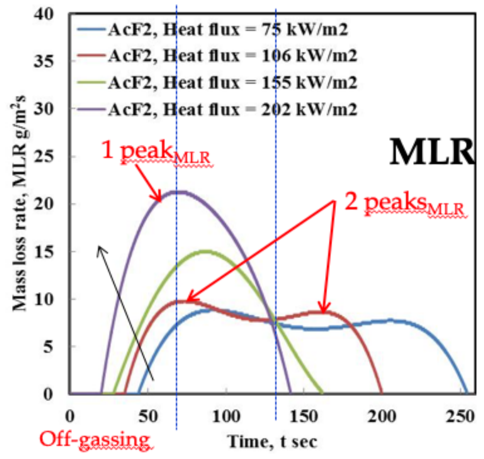


Figure 12 : MLR evolution of AcF2 for 4 values of the incident heat flux

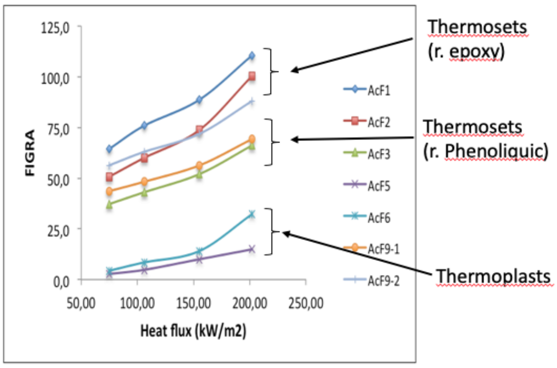


Figure 13: Variation of the FIGRA parameter with the incident heat flux for all studied composites (AcF1 to AcF3: thermosets (epoxy resin, carbon fibres); AcF5-AcF6: thermoplasts (PEEK resin, carbon fibres); AcF9-i: thermoset (phenolic, glass fibres)

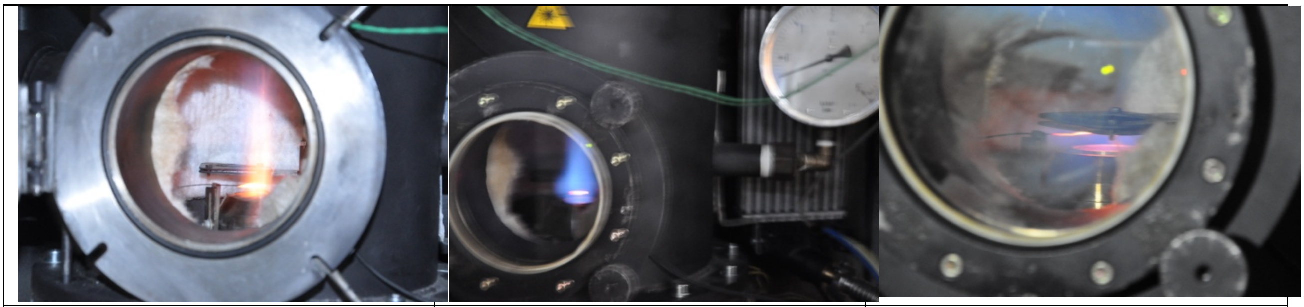
Finally, the variation of the FIGRA parameter with the incident heat flux q is drawn on Figure 13 allowing a ranking of the considered aeronautic composites in terms of q. The thermosets (epoxy resin/carbon fibres) have a lower fire resistance although they pass the burnthrough tests, but the thermoplasts have better fire performances.

3.3 Behaviour of polymer degradation in reduced pressure

Few works have been developed to investigate the effects of ambient pressure on piloted ignition of solid fuels on the ignition and flame spread of a polymer to evaluate the effect of altitude on the fire hazard on building [27]. Such pressure effects were also investigated in the fire configuration of spacecraft (reduced pressure, increased oxygen concentration). It was searched here the effect of pressure on the ignition time [28, 29] of a slab of polymer. In these works, no interpretation seems available for a direct effect of pressure on the solid degradation, as well, to estimate the modifications of the fire resistance in altitude.

With our device, tests are now performed in reduced pressure conditions (0.03 to 0.1 MPa), but at an enclosure temperature of around 40°C. Two composites were investigated AcF2 (thermoset) and AcF6 (thermoplast).

3.3.1 AcF2 and AcF6 composite behaviour in reduced pressure



a/ P=0.1 MPa | b/ P=0.07 MPa | c/ P=0.3 MPa

Figure 14: Flame visualisations for pressure = 0.1, 0.07 and 0.03 MPa (AcF2 composite, $q= 106kW/m^2$)

Figure 14 clearly shows that the soot concentration decreases when pressure is decreasing, leading to a modification of the flame-solid heat transfer. For $q=106kW/m^2$ and P=0.03 MPa (engine fire condition), the red emission of the thermocouple wires attests of high temperature at its surface while the flame is blue (OH radical emission).

The observation of samples (Figure 15) before and after thermal impact (4 min of test, thickness 4mm) shows an important delamination of the composite material and a thickening of the sample in reduced pressure conditions. The composite is swelled between the fibre plies [30].

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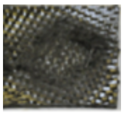
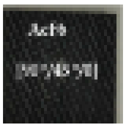
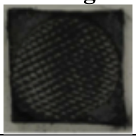
<table>
<tr><th rowspan="2">No.</th><th rowspan="2">Sample Specification</th><th colspan="2">Thermoset Composite sample
Matrix: epoxy resin
Carbon fibres</th><th colspan="2">Thermoplast composite sample
Matrix: PolyEtherEtherKetone resin
carbon fibres</th></tr>
<tr><td>AcF2 before test
</td><td>AcF2 after burning
</td><td>AcF6 before test
</td><td>AcF6 after burning
</td></tr>
<tr><td>1</td><td>Fire barrier</td><td colspan="4">Woven laminate</td></tr>
<tr><td>2</td><td>Layup</td><td colspan="2">[90°/0 °] Carbon/Epoxy</td><td colspan="2">[90°/45°/0°] Carbon/PEEK</td></tr>
<tr><td>3</td><td>Ply nb/thickness</td><td colspan="2">22 for 0.004 m</td><td colspan="2">16 for 0.004 m</td></tr>
<tr><td colspan="6">Figure 15 : Characteristics of the studied composites</td></tr>
</table>

As the holder constrains the edges of the sample, the swelling is mostly at the sample centre. This phenomenon is amplified when pressure is reduced for all composites, AcF2 and AcF6. It attests of the quasi-impermeability of the carbon fibres and residual resin curtain with the formation of gas pockets. The thermal characterisation of these composites shows that the swelling decreases the thermal diffusivity, hence, enhances le flame blocking effect and decreases the resin pyrolysis rate.

During the tests, the thickening can reach more than five times the original thickness at 0.03 Mpa. The thickness variations were not measured being related to the diameter of the exposed sample (window size) and not a characteristic property of the material.

Characterisation of the influence of pressure on AcF2 thermoset composite

Figure 16 shows the effect of reduced ambient pressure P (0.1; 0.07; 0.05; and 0.03 MPa) on the MLR evolutions at engine fire conditions 106 kW/m^2; T_{gas}=960°C) on the AcF2 pyrolysis and burning. Again, the sample burnthrough was never observed on the studied pressure range.

When pressure decreases, MLR varies from the two peaks regime to a single peak (the trend of soot formation is reduced) leading to a parallel decrease of $t_{pyrolysis}$, $t_{ignition}$, t_{PMLR}, $t_{off\text{-}gasing}$, $t_{burning}$: then, the fire resistance seems to be raise in altitude.

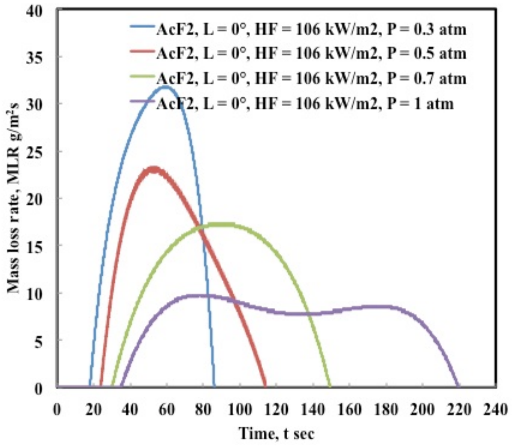


Figure 16: Evolution of MLR for 4 enclosure pressures - Thermoset composite AcF2

Test conditions:
Heat flux at the sample surface : q=106kW/m^2
O_2 mass fraction = 0

All other parameters stay equivalent

3.3.2 Characterisation of the influence of ambient pressure for the thermoplast composite AcF6

Aryl poly ether ketone (PEEK) is an engineering thermoplastic with excellent thermal, chemical and mechanical properties allowing an use in a variety of high performance applications with light weight, high thermal stability polymer [31]. They are tested to check if their fire resistances can be also identified during tests in reduced pressure when submitted to high heat flux.

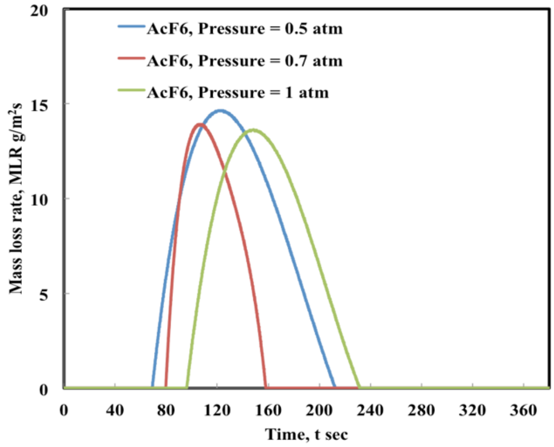


Figure 17: Evolution of MLR for 4 enclosure pressures - Thermoplast composite AcF6

Test conditions:
Heat flux at the sample surface : q=106kW/m^2
O_2 mass fraction = 0

All other parameters stay equivalent

For a thermoplast composite (AcF6), Figure 17 presents the same shape of MLR evolutions for different enclosure pressures. But, it was observed a light reducing of the fire performances of the material when the pressure decreases compared to thermosets. The regime with two MLR peaks (the protection of the sample by a char layer) was never obtained. The variations all the characteristics burning times follow the same trends than for thermosets. To conclude, the pressure influence seems to be smaller for thermoplast than for thermosets.

3.3.3 Comparison of AcF2 and AcF6 behaviours

This paragraph compares the fire resistance properties for thermoset (AcF2) and thermoplast (AcF6).

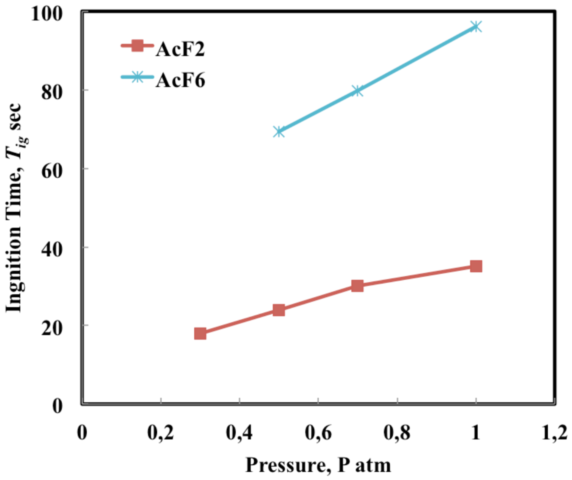


Figure 18: Ignition time vs pressure - Composite materials AcF2 (Thermoset) and AcF6 (thermoplast)

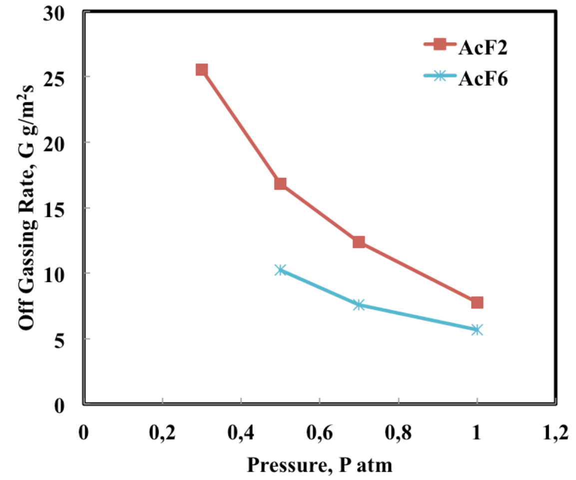


Figure 19: Off gasing time - vs pressure - Composite materials AcF2 (Thermoset) and AcF6 (thermoplast)

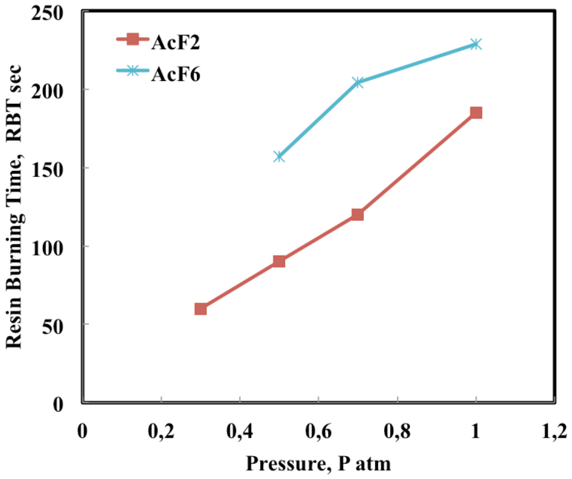


Figure 20: Variation of the resin burning time vs pressure - Composite materials AcF2 (Thermoset) and AcF6 (thermoplast)

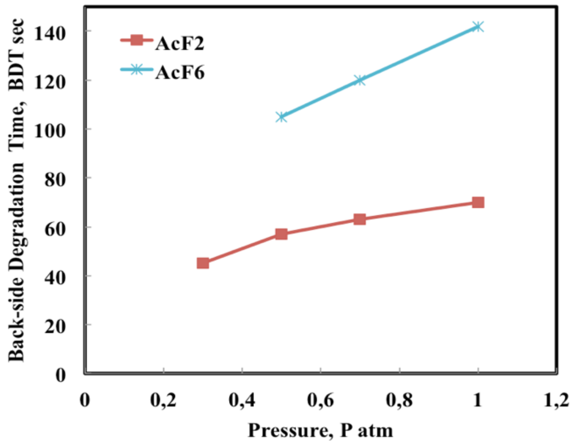


Figure 21: Variation of the backside degradation time with pressure - Composite materials AcF2 (Thermoset) and AcF6 (thermoplast)

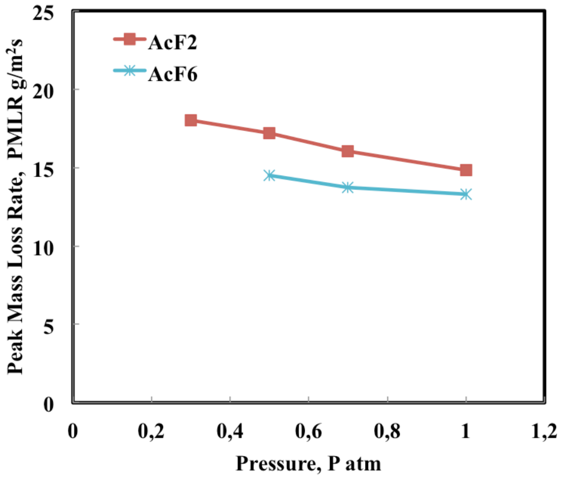


Figure 22: Variation of PMLR with pressure, Composite materials AcF2 and AcF6

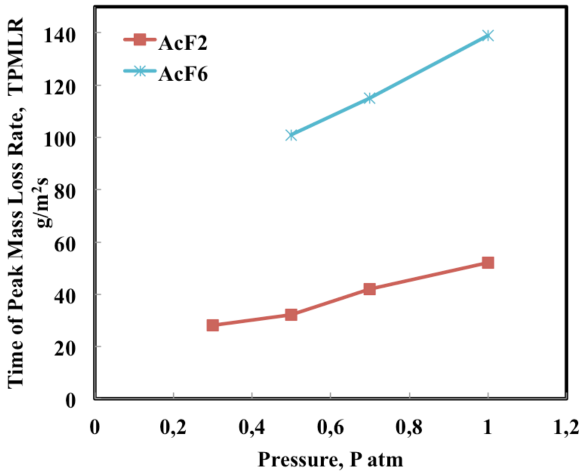


Figure 23:Variation of the time of the peak of MLR with pressure - Composite materials AcF2 and AcF6

A synthesis of the burning properties of the two kinds of composites (Figure 18 to Figure 23) is drawn on Figure 24. All results are consistent with the previous interpretations namely the same trends are reproduced.

T_{ign} (Figure 18) is a critical parameter which is essential to understand the processes acting inside a burning material to predict ignition and growth of fires. The fire resistance is larger for thermoplastic composites than for thermosets. It drastically increases with pressure, particularly for these last ones. Consequently, the risk of ignition and of the burning rate of composites in reduced pressure are globally increased in comparison to the hazard at the ground level. It must be reminded that the tests are performed close to ambient temperature without taking into account the flight conditions (external temperature around -50°C, shear stress,...). The change in the pressure effects with the composite types should be interpreted.

	AcF2 -> AcF6	P↑
Ignition time	↑	↑
Off-gassing time	↓	↓
Resin burning time	↑	↑
Back-side degradation time	↑	↑
Peak of MLR	↓	↓
Time of the peak of MLR	↑	↑

Figure 24: Variation of properties with P for AcF2 and AcF6

4 Interpretation of the results and resin degradation regime

For a decrease of the ambient pressure, the experimental results show an increase of the composite material burning rate, this is to say, a reduction of their fire resistance. This behaviour contradicts all chemical kinetic behaviour and theories where the chemical kinetic accelerates with pressure. To explain both these trends and the different degradation behaviours between thermoset and thermoplastic composites, additional tests were performed, at normal pressure and temperature, but for different ambient oxygen mass fractions.

Three types of hypothesis can be considered for the material degradation:

- a chemical reaction in the gaseous phase between the resin degradation products and oxygen close to the stagnation plane;
- an heterogeneous oxidative degradation at the gas/solid interface;
- a direct physical effect of the ambient pressure on the blowing of pyrolysis products.

4.1 The chemical effect in gaseous phase

This first hypothesis states that the pressure acts on the chemical reaction, in gaseous phase, between the fuel degradation products and the oxygen available close to the gas/sample interface. When oxygen is added to the hot impinging flow (inert combustion products), it can be considered that a diffusion flame is stabilized in the reactant mixing zone that increases the gas temperature. Then, the heat transfer towards the composite surface is increased, increasing its burning rate (MLR). But, the results, both for thermosets and thermoplastics (Figure 16 and Figure 17), contradict this tendency by showing a contrary variation.

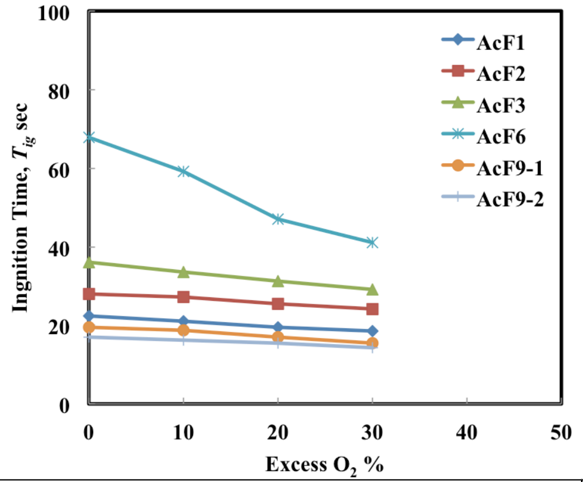


Figure 25: Ignition times of all composites for excess mass fraction of oxygen

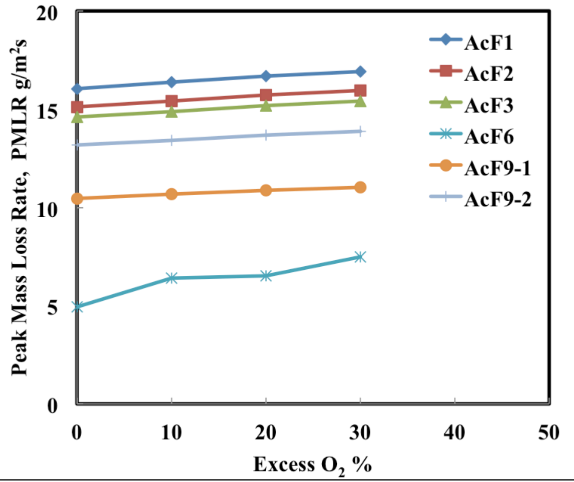


Figure 26: Peak Mass Loss Rate of all composites for excess mass fraction of oxygen

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Given that the pressure, generally, does not act on the diffusion processes and that the heat released in the flame has a upward effect on the heat transfer to the solid, the observed opposed MLR variations with pressure are not recovered.
To validate this interpretation, in this work, tests were performed with oxygen rich impinging flow. Figure 25 and Figure 26 report the variations of the ignition time t_{ign} and the peak intensity of MLR (PMLR) - parameters involved in the FIGRA number - with an excess of oxygen. These results show that, mainly for thermosets, but also for thermoplastics, t_{ign} and PMLR stay practically independent of the oxygen mass fraction, while a great dependence with ambient pressure is observed: the pyrolysis rate of these composites is not controlled by a chemical process of oxidation [22].
It can be concluded that the burning rate of the composite is not controlled by a chemical kinetic in gaseous phase.

4.2 The pyrolysis rate of composites

During the thermal degradation of a solid (or liquid) fuel, the gasification rate generally follows a pyrolysis law which controls the mass lost rate m" ($kg/m^2/s$). It takes the well-known form:

$$m^{"} = A\,exp\left[-\frac{E}{RTw}\right]$$

where A, E, R and T_w are the standard parameters of this Arrhenius law. No influence of the reactant concentrations (or reactant partial pressures) is considered i.e. the global reaction rate is of order 0 for reactants (or partial pressures). Accordingly, this pyrolysis law cannot predict a direct variation of MLR (m") with the oxygen concentration at the wall, or ambient pressure: the composite burning rate is not controlled by the reactions of oxidation at the composite surface.

In this work, we never observed an increase of the burning rate (or MLR) with the oxygen concentration, but, rather, an opposite variation. Consequently, it can be concluded that the heat released by the heterogeneous exothermal reactions at the pyrolysis surface, if they exist, does not modify the energy balance at the composite interface, then the burning rate.

To conclude, no chemical effect, either in the gaseous phase or heterogeneous at the composite surface, can explain the negative variation of MLR with pressure.

4.3 The physical effect

To interpret the observed effect of pressure on the degradation rate of thermosets, but of lesser intensity for thermoplastics, it must be admitted that the pressure acts as a strength opposed to the blowing of the pyrolysis gases at the solid-gas interface, and related to the saturation vapour pressure: weaker is the blowing (MLR) is weak, more important is the pressure effect. In these conditions, it must be admitted that the composite burning rate is widely influenced by the blocking effect of the external pressure on the material pyrolysis, without any action of the chemical process involved.
This conclusion was verified for all the studied composites, even for a change in the MLR evolutions (one or two peaks of MLR) due to soot deposition and burning at the different fibre plies (carbon curtains).
To interpret the slab swelling at low ambient pressure, it can be concluded that the pyrolysis rate is increased after the delamination of the sample during the test.
To conclude:

- The material burning rate increases as pressure is reduced due to the direct influence of ambient pressure on the blowing rate of the degradation products,
- As the resin pyrolysis rate is enhanced, an inflating of the space between the fibre plies is observed, and finally a thickening of the slab.

5 Conclusion

Safety aboard aircraft requires a complete knowledge of the fire resistance of materials when they are submitted to a strong external heat flux. In all cases, the fuselage and aircraft skin, must be safe barriers to the flame penetration into the cabin to protect the passengers and crew and to allow a safe flight home.
Given that, in new generation of aircraft, composites materials replace more and more aluminium alloys, then tests must be performed to prove that these new flammable materials present at least equivalent or better fire resistance than metallic elements by passing regular tests.
An original device was designed to determine the impact of a strong heat flux on the pyrolysis of aeronautical composite materials. To do so, the raising variation of the Mass Loss Rate (MLR) of the composite with the incident heat flux was quantified allowing the determination of the main flammability and burning properties of the studied polymers. However, the main original point of this study consisted in measuring and understanding the effects of the

ambient pressure (altitude) on the pyrolysis rate of the composite in thermal conditions representative of full scale aeronautic fires.

The results show that the composite burning rate increases as the ambient pressure is decreased: the composite fire resistance is then decreasing and the fire hazard growing after ignition. This result is contrary to all theories considering a chemical effect on the composite pyrolysis.

As a second point, by performing tests for different oxygen mass fractions, it was confirmed that no significant chemical effects able to control the material degradation was identified. In these conditions, the dependence of the composite burning rate on the ambient pressure seems to be rather related to the direct influence on the degradation products blowing rate or on its vapour pressure at the composite surface.

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7 References

[1] Archibal Tewarson, Nonmetallic Material Flammability in Oxygen Entiched Atmosphères, Journal of Fire Sciences, Vol. 18. May-June 2000.

[2] Cherry, R., and K. Warren. 1999. Fuselage burnthrough protection for increased post-crash occupant survivability: safety benefits analysis based on past accidents. FAA Office of Aviation Research. Washington DC. DOT/FAA/AR-99/57.

[3] Cherry, R. 2009. Burnthrough resistance of fuselage. EASA service contract number EASA 2008.C19. Prepared for: European Aviation Safety Agency.

[4] Dodd, D.C., C.R. Jenkins, M A. Snell.1996. Burnthrough assessment study. CAA Paper 96002 Civil Aviation Authority. London.

[5] Dodd, D.C., C.T.M. Hall, J. Pollard, M.A. Snell. 1994. Burnthrough resistance of fuselages: Initial findings. CAA paper 94002. London

[6] Marker, T.R. 1999. Full-scale test evaluation of aircraft fuel fire burnthrough resistance improvements. FAA Office of Aviation Research. DOT/FAA/AR-98/52.

[7] Marker, T.R. 2000. Development of improved flammability criteria for aircraft thermal acoustic insulation. Document Fire Safety Section AAR-422. FAA Office of Aviation Research. Washington DC.

[8] Cahill, P. 1986. An investigation of the FAA vertical Bunsen burner flammability test method. FAA Office of Aviation Research. Washington DC. DOT/FAA/CT-8/22.

[9] Lyon, R.E. 1997. Fire-resistant materials: research overview. FAA Office of Aviation Research. Washington DC. DOT/FAA/AR-97/99.

[10] Horner, A. 2000. *Aircraft materials fire test handbook.* FAA Office of Aviation Research. Washington DC. DOT/FAA/AR-00/12.

[11] Le Neve, S. 2010. Fire Behaviour of structural composite materials. In: *Proceedings of the* International Aircraft Fire and Cabin Safety Research.

[12] Webster, H. 1994. Fuselage burnthrough from large exterior fuel fires. FAA Office of Aviation Research. Washington DC. DOT/FAA/CT-90/10.

[13] Bernhard Schartel, Kristin H. Pawlowski, Richard E. Lyon, Pyrolysis Combustion Flow Calorimeter. A tool to assess flame retarded PC/ABS Materials?. Thermochimica Acta, 462, (2007) 1-14.

[14] Richard N. Walters, Natallia Safronava, R.ichard, E. Lyon, Combustion and Flame, 162 (2015) 855-863.A microscale combustion calorimeter study of gas phase combustion of polymers,

[15] Rodolphe Sonnier, Belkacem Otazaghine, Laurent Ferry, José-Marie Lopez-Cuesta, Study of Combustion Efficiency of Polymers using a pyrolysis-Combustion Flow Calorimeter, Com. and Flame, 160, (2013) 2182-2193

[16] Jean-Michel Jean-Michel Most, Michel Champion, Effects of pressure and oxygen concentration on a stabilized premixed, ASPACC 2017, Sydney.

[17] Jean-Michel Most, Sachin Dahikar and Michel Champion, Development of a new laboratory scale burnthrough apparatus for aeronautical composites, 13th ICCEU, Oct. 2-5, 2016, Taipei, Taiwan.

[18] Jean-Michel Most, Michel Champion, For a more sustainable and safe aviation, Int. Symposium on Sustainable Aviation (ISSA 2018), 9-11th July, Roma.

[19] E. Schuhler, A. Coppalle, B. Vieille, J. Yon, Y. Carpier, Behaviour of Aeronautical Composite to Flame : A Comparative study of Thermoset and thermoplastic-based laminate, Polymer Degradation and Stability, 152, (2018) 105-115.

DOI: 10.13009/EUCASS2019-193

[20] Horner, A. 2000. Aircraft materials fire test handbook. FAA Office of Aviation Research. Washington DC. DOT/FAA/AR-00/12.

[21] Maria Thomsen, Carlos Fernandez-Pello, David L. Urban, Gary A. Ruff, Sandra L. Olson, On simulating concurrent flame spread in reduced gravity by reducing ambient pressure, Proceedings of the Combustion Institute 37 (2019) 3793-3800.

[22] Mariusz Zarzecki, James G. Quintiere, Richard E. Lyon, Tobias Rossmann, Francisco J. Diez, The effect of pressure and oxygen concentration on the combustion of PMMA, Combustion and Flame 160, (2013) 1519-1530.

[23] B. Sundström, Development of a European Fire Classification System for Building Products, phd Thesis, Lund, 2007

[24] Le Neve, S. 2010. Fire Behaviour of structural composite materials. In: Proceedings of the International Aircraft Fire and Cabin Safety Research.

[25] Jean François Petit, A. Mansuet.1998. Development of small-scale burnthrough test equipment. In: Proceedings of the International Aircraft Fire and Cabin Safety Research.

[26] Florian Kempel, Bernhard Schartel, Gregory T. Linteris, Stanislav I. Stoliarov, Richard E. Lyon, Prediction of the Mass Loss Rate of Polymer Materials: Impact of residue Formation,Comb. and Flame, 159 (2012) 2974-2984.

[27] Dai Jiakun, Michael A. Delichatsios, Yang Lizhong, Piloted ignition of solid fuels at low ambient pressure and varying igniter location, Proceedings of the Combustion Institute 34 (2013) 2497-2503.

[28] Sonia Fereres, Chris Lautenberger, Carlos Fernandez-Pello, David Urban, Gary Ruff, Mass Flux at Ignition in reduced pressure environments, Comb. anf Flame, 158 (2011), 1301-1306.

[29] Maria Thomsen, Carlos Fernandez-Pello, David L. Urban, Gary A. Ruff, Sandra L. Orson, On simulating concurrent flame spread in reduced gravity by reducing ambient pressure, Proceedings of the Combustion Institute, 37 (2019), 3793-3800.

[30] M. A. Maaroufi, Y. Carpier, B. Vieille, L. Gilles, A. Coppalle, F. Barbe, Post-Fire compressive behaviour of carbon fibers woven-ply Polyphenylene Sulfite Laminates for aeronautical applications, Composites Part B, 119 (2017) 101-1130.

[31] Parina Patel, T. Richard Hull, Richard E. Lyon, Stanislav I. Stoliarov, Richerd N. Walters, Sean Crowley, Natallia Salfronava, Investigation of the thermal decomposition and flammability of PEEK and its carbon and glass-fibre composites, Polymer Degradation and Stability, 96, (2011), 12-22.