COMPRESSIVE BEHAVIOR UNDER FIRE EXPOSURE OF CARBON FIBERS POLYPHENYLENE SULFIDE COMPOSITES FOR AERONAUTICAL APPLICATIONS

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Abstract

The influence of fire exposure on compressive behaviors of carbon fibers woven-ply Polyphenylene Sulfide (PPS) has been investigated for aeronautical applications. Compression tests under one-sided heat flux or after prior fire exposure, as well as creep test, have been performed. For heat fluxes ranging from 20 to 50kW/m^2, prior fire exposure is highly detrimental to compressive mechanical properties, as the strength and stiffness decrease by -80% and -60% respectively (vs -30% and -35% in tension). Fire-exposure results in gradually increasing damages within tested laminates, as PPS matrix thermal decomposition leaves intra- and inter-laminar voids which leads to more or less extensive delamination depending on fire testing conditions. 2D Digital Image Correlation technique, C-scan inspections and microscopic analyses have been performed to investigate the deformation and damage mechanisms. In compression tests after fire exposure, local buckling and delamination associated with the failure of 0° fibers appears to be the primary failure mechanism, whereas global plastic buckling is the main damage observed during combined tests.

1. Introduction

With the increasing use of fiber reinforced composites in critical applications such as aeronautics, understanding the behavior of these material under extreme conditions like fire exposure has become of utmost importance to ensure a complete passenger safety. Fire makes the material mechanical properties weaker and consequently the structure resistance. Due to the rapid reaching of its low glass transition temperature (few seconds), the matrix loses its properties in rush and the entire load is carried by the fibers until failure [1]. Thus, in order to determine the materials providing the best post-fire behavior, different composites can be considered [2]. Among them, thermosetting-based composites (denoted TS) have been extensively used in aeronautics for the past 40 years. Indeed, most of the studies available in the literature on the composites fire behavior deal with carbon/Epoxy composites [1,3-4] and E-Glass/Vinylester composite [5-7]. Therefore, the behaviors of TS-based composites during fire exposure [1,3-11] or after fire exposure [12] are well established in the literature. Recently, thermoplastics (denoted TP) received considerable attention as matrix constituents in structural composites because they present several advantages compared to thermosets [13-15], such as toughness, impact resistance, or manufacturing process time. When it comes to fire resistance, high performance TP-based composites (e.g. PEEK, PPS) are expected to have better mechanical properties than TS-based composites (e.g. Epoxy, Vinylester). Previous studies showed that after fire exposure, a smaller decrease is observed in the tensile properties of PPS-based laminates as compared to Epoxy-based ones [13-16]. This has been supposed to be linked to char formation, one of the main transformations induced by fire, resulting from

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the thermal decomposition of the organic resin. Indeed, the char composition appears to be different in these materials as the TP resins have different chemical nature and the thermal degradation does not give the same residue [14]. Actually, the effect of thermal degradation and char formation on mechanical properties are only partially understood. Concerning the fire exposure behavior of TP-based composites, very few studies are available in the literature and they are focused on models describing the mechanical response of the material [17-18]. Thus, the failure mechanisms need more investigations to be clearly understood. In other words, the issue of the mechanical behaviors of TP-based composites during or after fire exposure is still an open question. This work addresses this question concerning C/PPS composites under compressive loading, which corresponds to the most severe conditions in case of fire exposure. In a first section the considered material and the experimental approach are described in details. Then results are discussed concerning post-fire as well as combined tests where mechanical loading is superimposed to fire exposure.

2. Material and methods

2.1. Materials and specimens

The composite materials studied in this work are 7 plies carbon fabric-reinforced PPS prepreg laminate plates [13]. The semi-crystalline PPS resin (Fortron 0214) is supplied by the Ticona Company. The woven-ply prepreg supplied by the SOFICAR Company, consists of 5-harness satin weave carbon fiber fabrics (T300 3K 5HS), with a mass fraction of fibers of 58%. The prepreg plates are hot pressed according to the following lay-up: [(0/90), (±45), (0/90), (±45), (0/90), (±45), (0/90)]. The glass transition temperature T_g of C/PPS is 98°C and its melting temperature T_m is 280°C [15].

2.2. Experimental set-up

2.2.1 Fire exposure tests

To reproduce the effect of fire, many studies have used the radiant heater of a cone calorimeter to burn laminate specimens [1,12,14-16]. Due to this heating method, the heat flux and the heating conditions are controlled and repeatable. As specimens are placed within a special fixture originally designed by Boeing [19] to prevent specimen global buckling during compressive loading (described in Paragraph 2.2.2), an initial experimental study has been conducted to evaluate and analyze the metrology of the heat flux at the surface of the specimens. A 24-holes plate has been machined, allowing the effective heat flux all over the specimens to be known and well controlled.

2.2.2 Mechanical testing

All the mechanical tests were performed using a 100 kN capacity load cell of an MTS 810 servo-hydraulic testing machine. The specimens' dimensions were 150x100x2,2mm. They were placed within a special fixture originally designed by Boeing to perform compression after impact testing (Fig.1), which incorporates adjustable side plates to accommodate for both variations in thickness and overall dimension, as well as to prevent specimen Euler buckling and allow a higher in-plane stress. In order to comply with standard Airbus AITM 1-0010 [19], all the tests (except the creep testing) were conducted with a constant crosshead speed of 0.2mm/min ($\dot{\epsilon}$ = 0.13%.min^{-1}).
In order to study prior fire exposure, samples were exposed to four different heat fluxes (20, 30, 40 and 50kW/m²) during 2 minutes and then cooled for one night. Post fire compression tests were then conducted at 120°C (service temperature in the nacelle surroundings).
For compression tests under fire exposure, the specimens were first subjected to a pre-loading of 0,15kN to ensure the homogeneity of the load at specimen boundary before applying the thermal stress. Then, they were exposed to a heat flux of 50 kW/m² during 100 seconds (time needed to reach a thermal

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equilibrium on the hot surface), and only after that the compressive loading was applied. In creep testing, the compressive load was adjusted to the set point at a rate of 0,5 kN.s-1. In order to evaluate the influence of the experimental procedure, the fire exposure was started either 40 seconds before or after the mechanical loading. To obtain a given degraded zone during test under fire (compression and creep tests), and to ease the comparison between the different experimental procedures, samples' edges were isolated in a way that only a 75x65mm zone was exposed to fire.

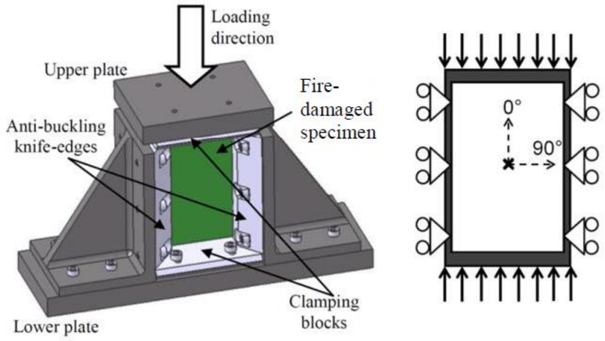

Figure 1. Compression tests: anti-buckling fixture

2.2.3 Digital Image Correlation technique

Fire-exposure leads to local damage within the laminates, resulting in singularities in the strain field on the outer surface. 2D Digital Image Correlation (DIC) technique is an appropriate tool to continuously track the effect of damage on the surface of fire-exposed laminates subjected to compressive loads. This method is based on the measurement of the displacement field between a reference time (stress-free state) and a given time of the loading (deformed state) and requires the painting of a black and white speckle at specimen surface to obtain different shades of grey. Therefore, the Green-Lagrange strain field can be derived from the 2D displacement field by means of the VIC-2D correlation software. A 3D DIC method should be used to investigate the out-of-plane displacements when buckling occurs. However, to a first approximation, the 2D DIC technique was applied during compressive loading to investigate the early deformation mechanisms, but also to detect the onset of local damage events (transverse matrix cracking, breakage of 0° fibers and onset of global buckling). Indeed, the localization of positive and negative strains is a good indicator of the onset of global plastic buckling bands at some level of the compressive loading.

2.2.4 C-scan ultrasonic inspections

Even though the qualification of the fire-induced damage (type, size and severity of damage) remains difficult, standard nondestructive evaluation methods such as ultrasonic C-scan imaging are often used, particularly when it comes to detect delamination in polymer-based composites. In the present case, the interest is to compare the fire-degraded area at laminates surface and the delaminated area through the thickness. C-scan inspections have been performed with an ultrasonic device ULTRAPAC II system (automated immersion system). Data acquisition, control and C-scan imaging was conducted by means of UTwin^{tm} software.

3. Results and discussion

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3.1. Compression tests on virgin samples

The macroscopic response is elastic ductile until it reaches a maximum stress equal to 120 MPa (Fig. 2a), corresponding to the onset of failure in the zone above the anti-buckling knife and under the upper plate (Fig. 1). The observation of laminates surface (Fig. 2b) reveals that failure is associated with the matrix transverse cracking and the breakage of 0° oriented fibers. It also appears that failure is gradual as transverse cracks propagate and fibers breakage results in the redistribution of the compressive load to the unbroken fibers at their vicinity.

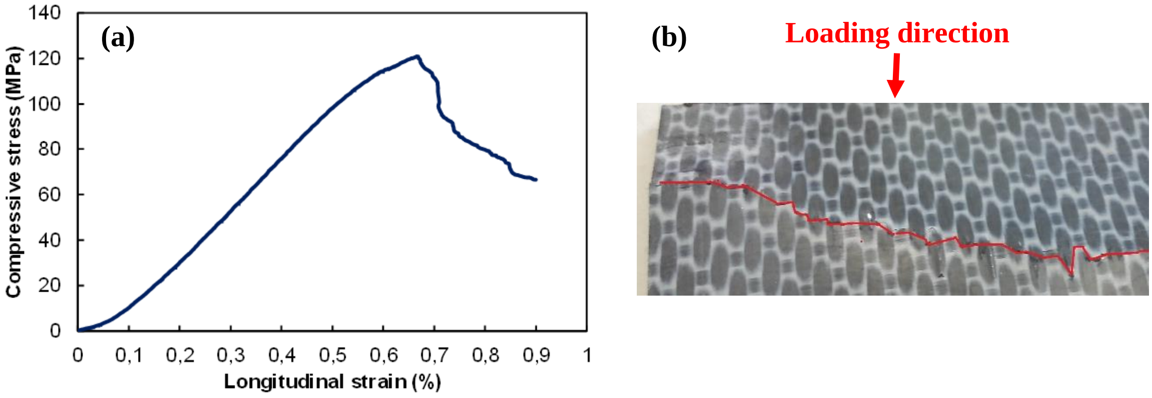

Figure 2. Compressive behavior of virgin C/PPS (**a**) Stress-strain curve **(b)** Surface observation

3.2. Thermal exposure

3.2.1 Temperature evolution

Thermocouples have been placed on the fire exposed and unexposed surfaces in order to monitor the material temperature evolution and the temperature gradient between the two surfaces. The temperature gradient evolves linearly with the heat flux (from 86°C for a heat flux of 30kW/m² to 230°C for 50kW/m²), suggesting that the higher the heat flux is, the lower the thermal conduction is. Indeed, the thermal decomposition may contribute to isolate the lower layers of the composite by leading to the formation of an insulating layer called “char”, also known as non-fuel fraction and resulting from the thermal decomposition of organic resin and fiber. Char can also improve fire resistance by limiting the access of oxygen to the region of the composite undergoing decomposition and thereby limiting oxidation processes [12]. In TP based composites, the presence of melted resin into a fibrous network suppresses heat radiation and physical modification of the char. Thus, the char formation in thermoplastic composites also protects the lower layers of the laminates against thermal degradation.

3.2.2 Thermal degradation and fire induced mechanisms

Depending on the fire testing conditions, different thermally-induced damages were observed within the laminates. From a macroscopic point of view, the cone exposure led to a circular degraded zone whose area increases linearly with the heat flux. At low heat flux (20 kW/m²), no damage were observed through the thickness and the exposed surface was only slightly degraded. As the heat flux increased, damages extended below the hot surface through the thickness in zones termed char region, decomposition region and virgin region [12]. Theses damages were intra and inter-laminar debonding as well as extensive delamination (Fig. 3a). For C/Epoxy composites matrix cracking in the “virgin” region was also observed.

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3.3. Post fire compressive tests

3.3.1 Macroscopic response

A previous study has shown that even if virgin C/Epoxy samples show better tensile properties than virgin C/PPS samples, prior fire exposure is more detrimental on their mechanical properties at 120°C [13, 15]. An exposure to 50kW/m² during only one minute leads to a 70% drop of their axial strength compared to only a 30% loss for C/PPS samples. Considering these results, the influence of prior fire exposure on compressive properties is more dramatic for C/PPS samples, as compression is ruled by different mechanisms [15]. The macroscopic response is elastic ductile until it reaches a maximum which seems to be associated with the macroscopic plastic buckling (Fig. 3b). From the obtained results, repeatability is better for lower heat fluxes (20 and 30 kW/m²) presumably because local instabilities (such as micro-buckling) are enhanced at high heat fluxes.

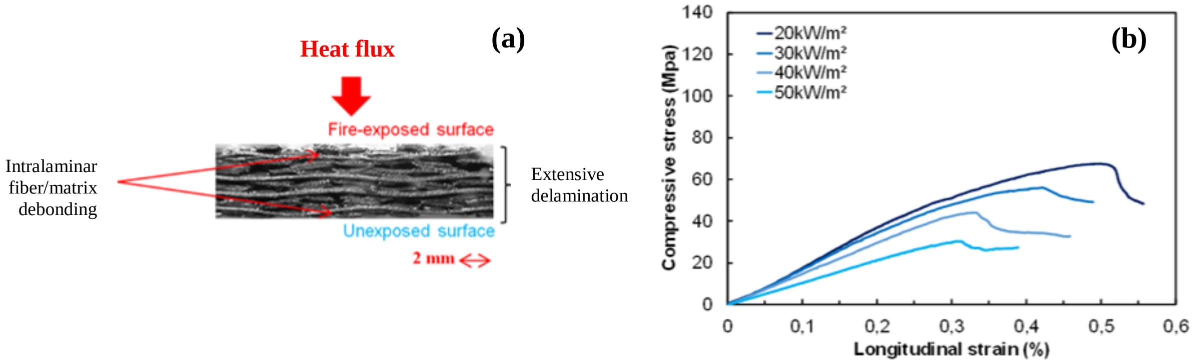

Figure 3 (a) Macroscopic observations of the fire-induced damages through the thickness in samples subjected to 50kW/m² **(b)** Compressive responses of samples subjected to a different prior fire exposure

3.3.2 Analysis of post fire deformation by 2D Digital Image Correlation

A 2D DIC technique is not fully operational to investigate the deformation mechanisms as medium to high heat fluxes induced significant out-of-plane effects. However, this technique can be helpful to detect the onset of local damage events (transverse matrix cracking, breakage of 0° fibers and onset of global buckling). At low heat flux (20 kW/m²), compressive failure primarily results from transverse matrix cracking and the 0° fibers breakage (see Fig. 4). For higher heat fluxes (30-40-50 kW/m²), the onset of global plastic buckling can be detected from the localization of positive and negative longitudinal strains.

3.3.3 Post compression C-scan inspections

The C-scan maps of specimens subjected to compressive loading after fire-exposure provide further information on fire-induced mechanisms. For each heat flux, the macroscopic damage (delamination) is similar on both fire-exposed and unexposed surfaces of laminates. As it was described above, prior fire-exposure leads to a circular damage area whose surface linearly increases as heat flux increases. Fire-induced delaminated area appears to be circular, and it is expected that compressive loading will promote further delamination. C-scan inspections performed after compressive tests clearly show that delaminated area gradually increases under the degraded surface as heat flux increases. The comparison between an approximate estimation (from macroscopic observations) of the surface degraded area after fire exposure and an estimation of the delaminated area after compression (based on C-scan analysis)

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show that they are rather well correlated, suggesting that delamination is primarily associated with thermal-degradation.

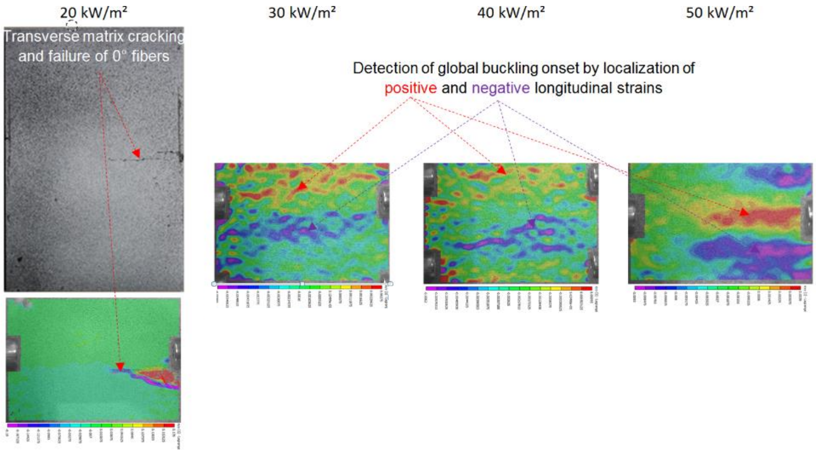

Figure 4. Green-Lagrange longitudinal strain distribution in samples subjected to compression after fire exposure.

3.4. Combined testing: compression under one sided fire exposure

The mechanical response is elastic until the material reach a maximum stress, followed by a drop in load capacity (Fig. 5a). Measurements have shown that during the compression tests, the hot face temperature reaches a plateau at 610°C, while the cold face temperature levels off to approximately 300°C. At such temperature levels, the mechanical properties in the zone exposed to fire are extremely low compared to the properties of the non-exposed zones (above the specimen edges and in the anti-buckling knifes) which are expected to bear virtually the whole load. Thus, the drop is associated with the formation of one (or several) macroscopic buckling band initiating in these non-exposed zones and propagating through the degraded zone perpendicularly to the compression load. The stress then remains stable, which can be explained by the propagation of the global buckling transversally to the loading direction (Fig. 5b).

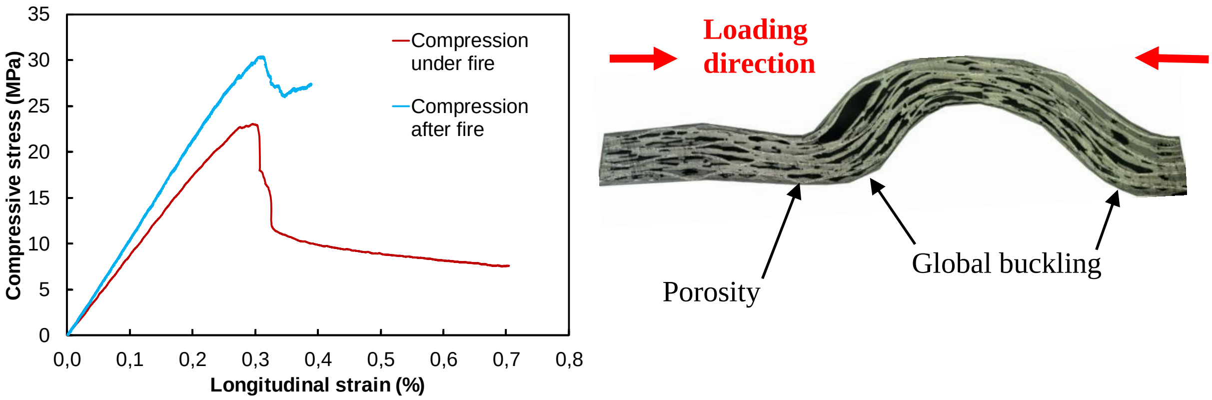

Figure 5 (a) Typical thermomechanical response of samples subjected to compressive loading during/after a fire exposure of 50 kW/m² **(b)** Post mortem observation of global buckling after compression combined to a 50kW/m² fire exposure

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3.5. Compression creep testing under one sided fire exposure

The application of the thermal stress before or after the mechanical loading is of major importance on the mechanical behavior of carbon/PPS. Although the failure mechanisms are quite similar to those observed during compression tests for both experimental procedure (Fig. 5b), the mechanical responses are significantly different (Fig. 6a). One the one hand, the measured time-to-failure is typically 5 times smaller when the samples are first exposed to fire before they are subjected to compressive loading. Indeed, the matrix is already thermally degraded where the compressive load is applied. Thus, the load transfer between fiber bundles via the matrix is impaired, resulting in a rapid propagation of local instabilities. The formation of two or more kink bands seems also to be promoted in this experimental procedure. On the other hand, when the mechanical stress is applied first, the fibers are already in a compression state when they are exposed to fire, and the matrix thermal degradation has a limited effect on the mechanical response. The influence of an insulation mask (as described in Paragraph 2.2.2) has also been studied (Fig 6b). Time-to-failure is typically twice as low as in the non-insulated case. Further investigations are currently carried out to understand the influence of the insulation on the failure mechanisms.

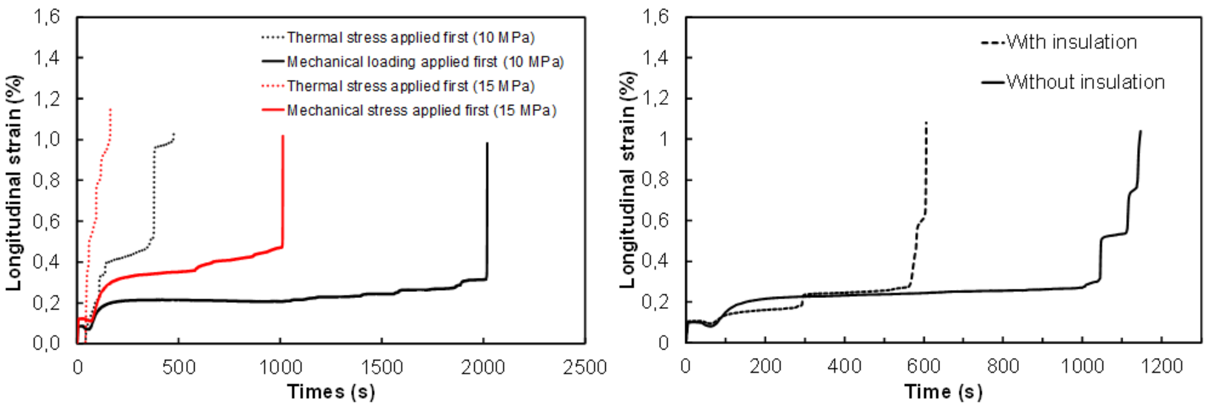

Figure 6. Compression creep testing under 50kW/m²
(a) Influence of the experimental procedure **(b)** Influence of the presence of insulation (σ = 11,4 MPa)

4. Conclusions

From the present study conducted on quasi-isotropic C/PPS laminates subjected to compressive loading and fire exposure, the following conclusions can be drawn:

- With respect to the values of unexposed specimens, a severe fire-exposure contributes to a significant decrease in laminates longitudinal stiffness (-60%) and strength (-80%).
- Prior fire-exposure results in gradually increasing damages within tested laminates, as PPS matrix thermal decomposition leaves intra- and inter-laminar voids which lead to more or less extensive delamination
- The onset of global plastic buckling can be detected by means of a 2D DIC technique from the localization of positive and negative longitudinal strains.
- The delaminated areas are rather well correlated with the surface fire-degraded areas, suggesting that delamination is primarily associated with thermal-degradation.
- Under superimposed fire exposure and mechanical loading (quasi-static or creep tests), global plastic buckling appears to be the primary failure mechanism under compression. This differs from the observations of post-fire failure mode: in this case, it corresponds to local buckling and delamination associated to the propagation of transverse cracks (failure of 0° fibers).

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