



Correlation between post fire behavior and microstructure degradation of aeronautical polymer composites



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ABSTRACT

The influence of fire conditions on the thermo-physical properties of carbon fibers woven-ply PPS and epoxy has been investigated. More specifically, this work was aimed at comparing the changes on laminates meso- and microstructure due to increasing heat fluxes, and depending on matrix nature. The residual tensile mechanical properties were compared considering the specific thermal decomposition of thermosetting- and thermoplastic-based composites, with a particular attention paid to the char formation along with the mass loss. For the range of heat fluxes studied (up to 50 kW/m²), the post-fire tensile mechanical properties of PPS-based laminates are significantly higher than those of C/Epoxy laminates (about twice higher at 50 kW/m²), not due to the char yield which is similar in the studied materials (about 80%), but rather to a better residual cohesion of the fibrous reinforcement at both microscale (within fiber bundles) and mesoscale (intra and inter-ply).

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1. Introduction

1.1. Background and literature review

Since the beginning of aeronautic industry, constructors have been looking for low weight and robustness in composites. These factors associated with equal performances and cost reduction contribute to the replacement of thermoset (TS) composites by thermoplastic (TP) composites. When compared with TS polymers, thermoplastics present several advantages for aircraft structures. They absorb much less moisture with less consequential reduction of the mechanical properties at elevated temperature. They exhibit a higher toughness, a better interlaminar strength and resistance to impact. Because no chemical reaction is required, TP polymers have a very short processing time with ease of shaping and storage. However, the behavior under fire is clearly the Achilles' heel of polymer matrix composites (PMCs), though the fire-resistance of high-performance TP composites is generally better with excellent ignition times, even at high fluxes [1]. The behavior of PMCs under fire conditions has been the subject of many investigations concerning the fire-induced degradation and the residual post-fire mechanical properties [2]. The major types of fire damage suffered

by laminates are char formation, softening and degradation of the matrix and fibers, delamination and matrix cracking. Char can improve fire resistance by limiting the access of oxygen from the atmosphere to the region of the composite undergoing decomposition, which again can slow the combustion rate [3]. High-performance thermoplastics (TP), such as Polyphenylene Sulfide (PPS) and Polyether ether ketone (PEEK) yield high amounts of char provided the heat flux is high enough to lead to the onset of pyrolysis [1]. Thermal degradation processes which occur within PPS laminates have been studied by Montaudo et al. [4]. Condensation is of prime importance in residual char formation [5]. The amount of char formed depends on the chemical nature of the polymer matrix and (if present) organic fibers [6]. The amount of retained char is determined by the temperature, exposure time and type of resin, with less than ~10% of the original weight of polymers such as many epoxies reduced to char whereas aromatic-based and TP resins yield substantially higher amounts of char. The char yield (mass fraction of char) of C/PSS is typically 60%, which is due to the high density of aromatic ring compounds in the molecular chain, and which is comparable with phenolic resin [1,7,8]. Depending on fire conditions, epoxy-based composites usually have relatively low post-fire properties [9–12], as they can be severely degraded after fire exposure. Investigations have been carried out for enhancing the fire properties of epoxy composites [13–18]. More specifically, it may consist in using flame-retardant thermoplastic film or carbon fiber-reinforced thermoplastic as

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interlayers within epoxy-composites without compromising the structural performance. There are many other methods to enhance the flame-retardant properties of polymer matrix composites including additive compounds blended into the polymer matrix, heat-resistant coatings, or sophisticated methods that involve chemical modification of the matrix or heat-induced intumescence of the composite surface [6].

Several authors have identified the changes in the microstructure of the fire-damaged composites [19,20] using Scanning Electron Microscopy (SEM), while the amount of retained char can be evaluated using thermogravimetric analysis (TGA). Until recently, little was known about the correlation between microstructure and structural properties of TP-based composites in fire, and most attention has been given to determining the damage experienced by thermoset (TS) matrix laminates because of their usage in aerospace structures. Mouritz and Gibson proposed a detailed review of the fire behavior of polymer-based composites [6]. Particularly, it is well established that the reduction to the post-fire mechanical properties of TS laminates is due to thermal degradation and damage caused by fire. According to their conclusions, fire damage extends below the hot surface in zones known as the char region, decomposition region, and virgin region. It is observed that TP-based composites generally have higher decomposition temperatures, yield high amounts of char, and are less susceptible to delamination cracking than TS laminates. As a result, TP-based composites usually have higher post-fire properties [21], but the thermophysical properties and the changes in microstructure of thermally degraded TP-based composites are still not well understood.

That is the reason why an initial work was done to compare the influence of fire on the post fire thermo-mechanical behavior of two different fiber-reinforced polymers for aeronautics purposes [22]: TS-based (Epoxy) composite vs high-performance TP-based (PPS) composite, consisting of the same 5-harness satin weave carbon fiber fabrics. By means of a cone heater, three different heat fluxes (20–35–50 kW/m²) have been applied to the materials for different exposure times (1–2–5 min). Compared with C/PPS laminates, the mechanical properties of C/Epoxy laminates are higher in the virgin state (no prior fire exposure). The analysis of the post fire tensile properties shows that prior severe fire exposures are more detrimental to C/Epoxy than to C/PPS laminates. Although the PPS matrix behavior is highly ductile at a test temperature higher than glass transition temperature, it clearly appears that the decrease in the tensile properties laminates of PPS-based composites is much slower than the one observed in C/Epoxy laminates subjected to severe prior fire conditions. It is assumed that provided the heat flux is high enough to lead to the outset of pyrolysis, PPS-based composites yield higher amounts of char, whose formation retains the structural integrity of fire-damaged composites. The question is therefore to verify this assumption.

1.2. Objectives of the work

It is expected that melting and resolidification of the thermo-plastic matrix due to fire exposure influence the post-fire properties by changing the meso- and micro-structures, hence justifying that high-performance TP-based laminates maintain a high degree of retention of residual properties [22]. The present work was aimed at investigating the influence of different fire conditions on laminates meso- and micro-structure (using SEM analysis), the thermo-physical properties and the thermal decomposition (by means of thermogravimetric analysis) of PPS (TP) and Epoxy (TS)-based composites for aeronautical purposes (e.g. at service temperatures higher than T_g). One of the key points of the present study is an estimation of the changes in the tensile properties

along with the char yield in laminates consisting of different matrix systems subjected to different fire conditions.

2. Materials and method

2.1. Materials and specimens

The composite materials studied in this work consist of 7 plies carbon fabric-reinforced PPS or epoxy prepreg laminate plates [22]. The semi-crystalline PPS resin (Fortron 0214) and the epoxy resin (914) are respectively supplied by the Ticona and by the Hexcel Companies. The woven-ply prepreg, supplied by the SOFICAR Company, consists of 5-harness satin weave carbon fiber fabrics (T300 3K 5HS). The mass fraction of fibers is 58% in both materials. The prepreg plates are hot pressed according to a Quasi-Isotropic lay-up. The average thickness (calculated from five measurements each) of consolidated specimens is virtually constant in both materials: 2.22 ± 0.2 mm in C/PPS laminates and 2.20 ± 0.1 mm in C/Epoxy laminates. The porosity content of laminates as manufactured with hot bonding process is 0.52% [23].

2.2. Experimental set-up and methods

Thermal decomposition was investigated by thermogravimetric analysis (TGA) in different atmospheres using a Netzsch TGA 209 device. Parallelepipedic samples of 10 mg (± 2 mg) were cut from areas of laminates exposed to fire. Measurements were performed under oxygen and nitrogen with a constant heating rate of 10 K/min and flow rate of 35 ml/min. The thermal degradation of the polymer and subsequent mass loss can be observed more specifically under a nitrogen atmosphere, as the degradation of carbon fibers is limited in inert atmospheres. The experimental set up of fire exposure tests is described in [22]. Four different heat fluxes (20–30–40–50 kW/m²) have been applied to the materials for the same exposure time (120 s). After fire-exposure, all the specimens were cooled in the air for one night. The “virgin” state chosen for comparison purposes represents the state of specimens exposed to no prior fire. Five specimens were tested in each configuration. In order to explain the damage mechanisms, an analysis based on fractography observations was performed using an optical microscope Olympus. SEM investigations were also performed on LEO 1530 microscope.

3. Results and discussion

3.1. Thermogravimetric analysis

In order to investigate the thermal degradation kinetics of polymers at a constant heating rate, TGA tests have been performed. TGA curves represent the weight loss measured with increasing temperature or time, and can be used to quantify the weight loss of polymer matrix converted into char [24]. During a TGA test under an inert atmosphere (N₂), the material burns in a manner similar to a charring material, in that the carbon fibers comprise most of its mass [25]. In epoxy-based laminates, the composite burns primarily from the vaporization of its epoxy resin. A Differential Scanning Calorimetry test performed on C/PPS laminates has shown that $T_g = 98$ °C, $T_m = 280$ °C, and its crystallinity rate is equal to about 30%. In C/Epoxy laminates (amorphous polymer), T_g is equal to 190 °C. At glass transition temperature, the viscous softening of the polymer matrix can be observed [9]. As temperature increases, the pyrolysis of the matrix leads to its thermal decomposition [2,6]. The reduction in the post-fire properties is due to chain scission reactions leading to decomposition and vaporization of the polymer matrix phase. Char, also known as

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