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Residual mechanical properties of a carbon/epoxy wound composite exposed to fire

A. BENELFELLAH^a, T.H.Y. QUACH^a, D. HALM^a, T. ROGAUME^a, D. BERTHEAU^a

a. Institut Pprime UPR 3346 – CNRS – ENSMA – Université de Poitiers – Département Physique et Mécanique des Matériaux – BP40109, 86961 FUTUROSCOPE CHASSENEUIL CEDEX, France.
abelkibir.benelfellah@ensma.fr, thi-hai-yen.quach@ensma.fr, damien.halm@ensma.fr,
thomas.rogaume@univ-poitiers.fr, denis.bertheau@ensma.fr

Résumé:

L'hydrogène devrait être un vecteur d'énergie très précieux pour le 21^{ème} siècle comme il devrait répondre aux préoccupations économiques principales de la société. Pour exploiter les avantages de cette source d'énergie à grande échelle, des recherches et développements technologiques sont nécessaires pour sécuriser le stockage. Ainsi, des études sur le comportement thermomécanique des matériaux utilisés pour le stockage de l'hydrogène sont nécessaires. L'utilisation de composite époxy / fibre de carbone est largement répandue en raison de son faible poids, et de ses bonnes propriétés mécaniques. Afin de reproduire les conditions d'un incendie, l'agression thermique est effectuée en utilisant un cône calorimètre (ISO 5660). L'échantillon composite est exposé à des flux thermiques homogènes, de 20 à 60 kW / m². L'exposition au feu et l'arrêt à des moments différents ont pour but d'étudier l'influence de l'énergie thermique (différents flux de chaleur et durées d'exposition) sur les propriétés mécaniques résiduelles en traction.

Les résultats obtenus montrent que l'épaisseur de résidu (charbon) des échantillons est proportionnelle à l'énergie appliquée sur les échantillons et la durée de l'inflammation. La rigidité et la force ultime diminuent lorsque la durée d'exposition au cône calorimètre augmente. Une relation proportionnelle entre la contrainte à la rupture de l'échantillon exposé et l'épaisseur non carbonisée est également observée.

Abstract:

Hydrogen is expected to be a highly valuable energy carrier for the 21st century as it should participate in answering main society and economical concerns. To exploit the benefits of hydrogen at large scale, further research and technological developments are required in order to secure the storage. Thus, studies on the thermal-mechanical behaviour of the materials used for the storage of hydrogen are necessary. Actually, the use of epoxy/carbon fibre composite is developed widely because of its low weight, and its good mechanical properties. The thermal aggression representing the one of a fire is performed by using a cone calorimeter apparatus (ISO 5660). The composite sample is exposed to homogeneous heat fluxes, from 20 to 60kW/m². The fire exposure is stopped at different times in order to study the influence of the thermal energy (different heat fluxes and exposure durations) on the residual mechanical tensile properties.

The results obtained show that the residue thickness of the samples is proportional to the applied energy on the samples and the inflammation duration. The stiffness and the ultimate strength decrease when the duration of exposure to the cone calorimeter increases. A proportional relationship between the ultimate stress of the exposed sample and the non-charred thickness is also observed.

Key words: Composite materials, thermo-mechanical behaviour, fire exposure.

1 Introduction

Carbon fibres possess very high specific mechanical properties, in particular, stiffness and strength, which make them attractive as reinforcing components in the composite materials. To take full advantage of these properties, it is necessary to combine them with a matrix material, such as polymer, that ensures the cohesion of the material, protects the fibres and transfers stress effectively to them [1] [2]. The matrix also stabilizes the fibre in compression, contributes to the resistance to damage due to impact by exhibiting plastic deformation, and provides out-of-plane properties to the laminates [2][3]. Epoxy is the preferred choice as the matrix for CFs due to their good impregnation and adhesion to fibre reinforcement [4]. Carbon fibre-reinforced epoxy (EP/CF) composites have been widely used in many areas, including aerospace, automobile, marine, military, etc., due to their outstanding properties, such as high strength, high modulus and light weight [2][5-9]. It results in excellent mechanical performances, chemical and electrical resistance and low shrinkage during cure [4][10]. This type of composites can be used in winding processes in order to manufacture hydrogen storage cylinders. Type IV pressure vessels have demonstrated promising results: these cylinders are made of a polymeric liner (for the tightness), metallic bosses (for the connection to fuel cells, for example), and a filament wound composite shell which ensures the mechanical strength [11]. To exploit the benefits of hydrogen at large scale, further researches and technological developments are required in order to secure the storage. For example, Gentileau et al. [11] studied the influence of temperature on storage. Berro Ramirez et al. [12] developed a damage model to accurately simulate burst modes and pressure. Wakayama et al. [13] dealt with impact on filament wound tanks, ... This work is dealing with the thermal-mechanical properties of epoxy/carbon fibre composites. The influence of a fire or a heating source on the residual tensile mechanical behaviour is studied. The thermal aggressions are performed by using a cone calorimeter apparatus, at homogeneous heat fluxes, from 20 to 60kW/m². During those tests, the fire exposure is stopped at different times in order to study the influence of the thermal energy (different heat fluxes and exposure durations) on the residual mechanical properties. In this work, two principal issues are examined. Firstly, the thermal impact by cone calorimeter on the composite samples will be studied. The relationship between the residue thickness and energy exposure (and duration of inflammation) will be also analysed. Secondly, the influence of the thermal impact on the mechanical residual strength of composite will be presented.

2 Material and samples

The composite material at stake here is composed of T700S carbon fibres in an epoxy matrix. In order to study the properties of samples representative of the hydrogen storage cylinders, parallelepipedic specimens of dimensions 300×25×5mm³ are cut from wound cylinders. Microscopic observations allow to measure the volumetric ratio of fibres and the volumetric ratio of porosities of the composite. Many authors show the influence of the fibre orientation on the mechanical strength [14-17]. In this work, different fibre orientations (with respect to the cylinder axis) have been studied: 90°, ± 45° and a quasi-isotropic sequence (±12°/90° /±45°/90°) noted EC90, EC45 and ECiso respectively.

3 Fire exposure of composite samples

The thermal aggression is performed by using a cone calorimeter apparatus (ISO 5660). The specimens have been put in a specimen holder in aluminum with a top plate in steel (only an area about $100 \times 25 \text{ mm}^2$ in the middle of the specimen is exposed to the cone calorimeter) and an insulating layer under the sample in refractory material (Figure 1). Then they have been exposed to a radiant cone under a constant heat flux, possibly ranging from 20 to 60 kW/m^2 . During these tests, the fire exposure is stopped at different times depending on the type of the sample. For each condition (fiber orientation, flux value, exposure duration), three samples have been tested for repeatability.

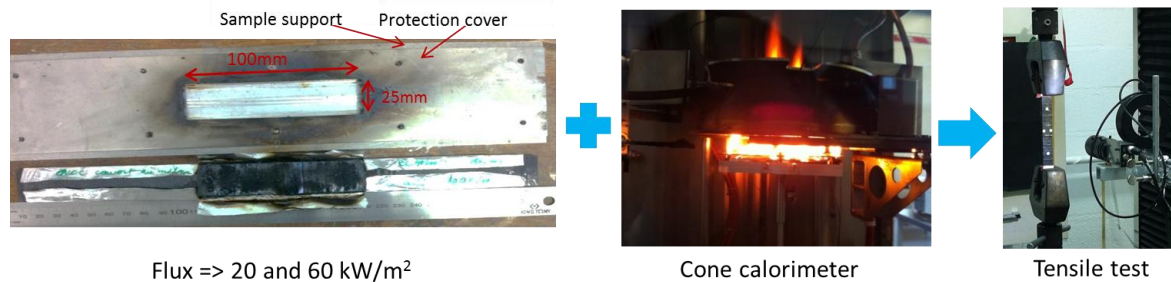


Figure 1 : Cone calorimeter and tensile machine instrumented with optical tracking

After fire exposure, if the thermal energy brought by the cone calorimeter is sufficient, a char (or residue) layer appears on the exposed sample surface. This char thickness is noted d_1 , whereas the “virgin” composite (whose aspect is similar to that of non-exposed material) is d_2 . Figure 2 shows an example of the EC90 sample after 130s exposure under a heat flux of 35 kW/m^2 . The residue thickness is observed and measured for different exposure energy level by optical microscopy apparatus (LAICA MZ95), only for the ignited samples.

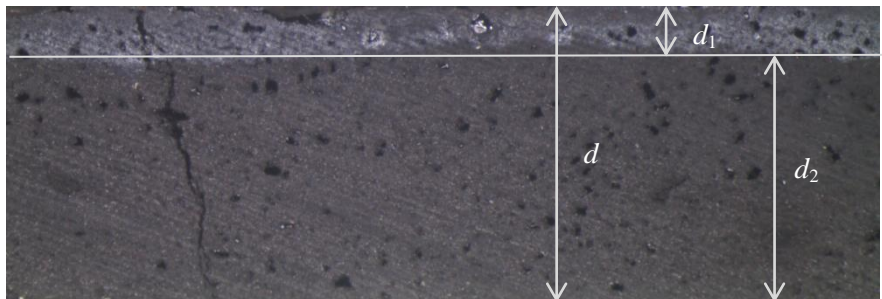


Figure 2 : EC90 sample after 130s exposure under a heat flux of 35 kW/m^2 (d : sample thickness, d_1 : char thickness, d_2 : “virgin” composite thickness).

The evolution of the residue thickness vs. the thermal energy for the three types of samples is compared in Figure 3. Each test is performed at a given value of heat flux (35 kW/m^2). A general linear trend can be observed. The following conclusions can be drawn:

1. There is a threshold (in terms of energy) below which no char appears (3.5 MJ for the EC90 sample and 5MJ for the samples EC45 and ECiso).
2. This threshold difference between EC90 and EC45/iso samples is due to the heterogeneity of the resin distribution in EC90 sample (the resin density is higher near the surface) and to its degradation by oxidation even when there is no char. This influence of resin oxidation is less marked in EC45 and ECiso samples (the fibres play a more important role in these samples).
3. Once the char appeared, its propagation kinetics is almost identical for all orientations.

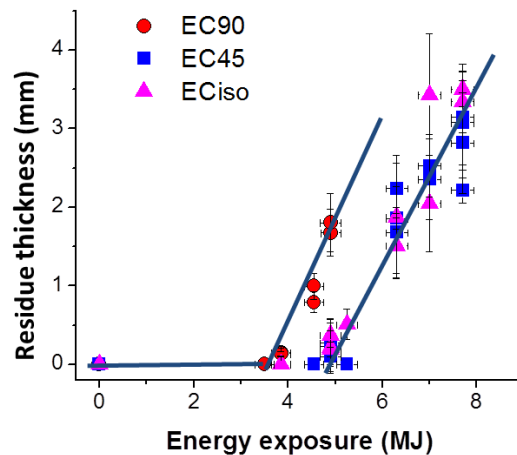


Figure 3 : Residue thickness vs. thermal energy for the EC90, EC45, ECiso samples (all tests are performed with the same heat flux of 35kW/m^2).

It is important to note that the presence of the flame at the surface of the samples (due to the ignition of the volatile products emitted during the thermal decomposition and combined with oxygen) adds a significant heat source: therefore, for the ignited samples, the residue layer growth is the result of two combined causes, the cone calorimeter exposure and the flame radiation.

4 Mechanical behaviour: stress-strain curve and fracture mode

To determine the mechanical properties (in particular stiffness and strength) of the exposed samples, tensile tests up to fracture have been performed using an INSTRON 4505 machine with self-locking grips. The maximum capacity of this device is 100kN. The crosshead speed is 1mm/mn for all tests. Each test is instrumented with optical tracking to measure strain without contact (Figure 1).

For all exposed samples, the exposure duration (or, equivalently, the energy input) clearly affects the tensile strength as well as the stiffness: the longer the exposure duration, the lower the strength and the stiffness. Figure 4-(A) represents the tensile stress - strain curve for the samples EC90. The fracture mode of this sample is a complete fibre/matrix debonding as shown in Figure 5-A. Strength undergoes a sharp drop when the sample has been subjected to the cone calorimeter, whereas the stiffness remains relatively low and less affected by the fire exposure.

Figure 4-(B) shows the tensile stress - strain curve for the samples EC45. A strong nonlinearity is observed which is due to more progressive damage mechanisms (matrix microcracking, irreversible sliding between plies...). This type of damage evolves until rupture by delamination due to microcracking between the plies as showed in Figure 5-B.

Figure 4-(C) displays the tensile stress - strain curve for the samples ECiso. Three stages can be observed: first, an elastic linear evolution, then a slightly nonlinear part probably due to microcracking and finally a complete delamination as showed in Figure 5-C.

For all tensile tests (except EC90), the stiffness decrease appears only when inflammation occurs. The case of EC90 samples is less speaking because of the very low stiffness of the resin.

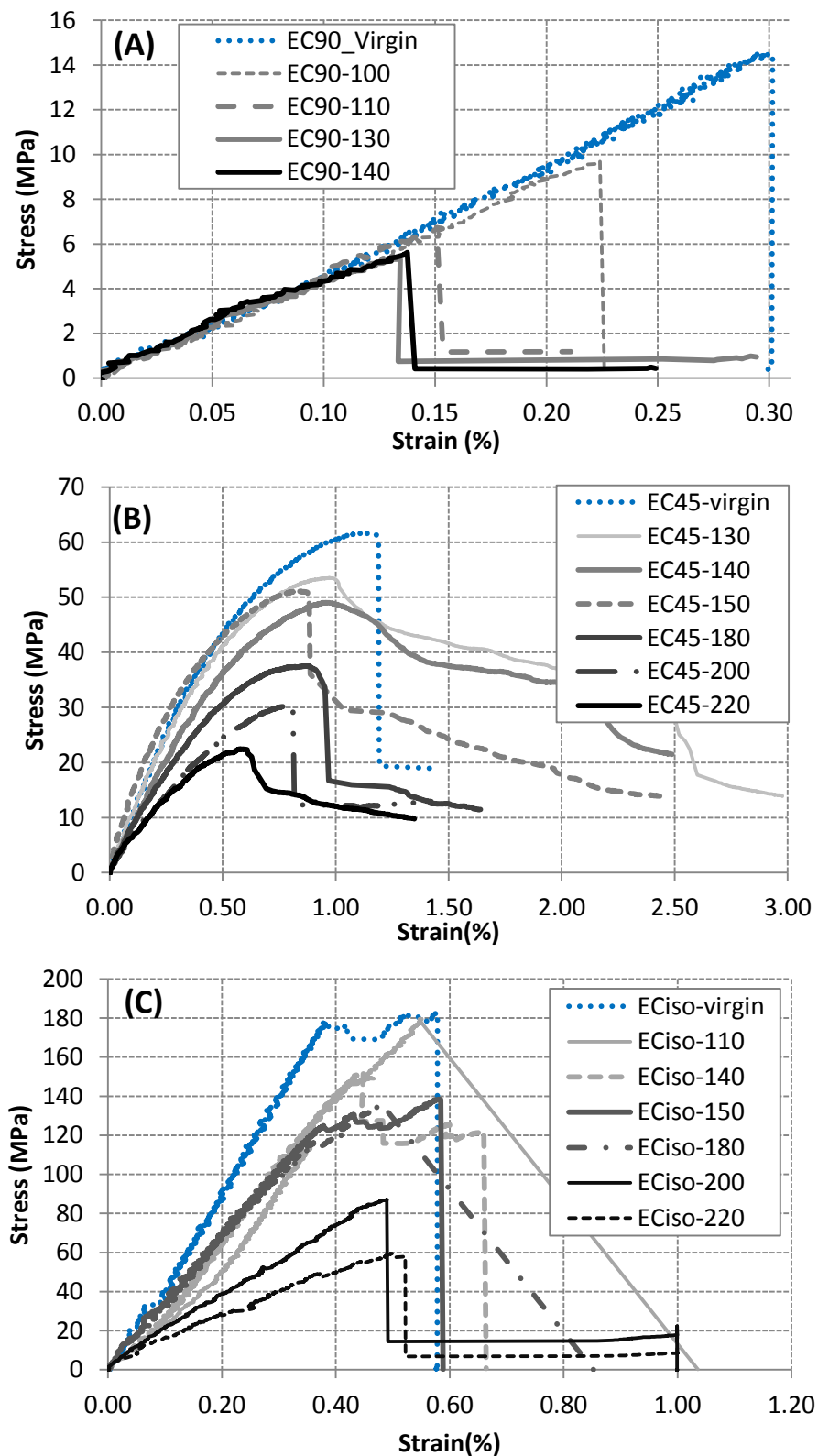


Figure 4 : Tensile stress-strain curves of samples (EC90 (A), EC45 (B) and ECiso (C)) first exposed to a given heat flux of 35kW/m^2 . The numbers (100,110,...) represent the fire exposure duration in seconds and “virgin” the non-exposed sample.

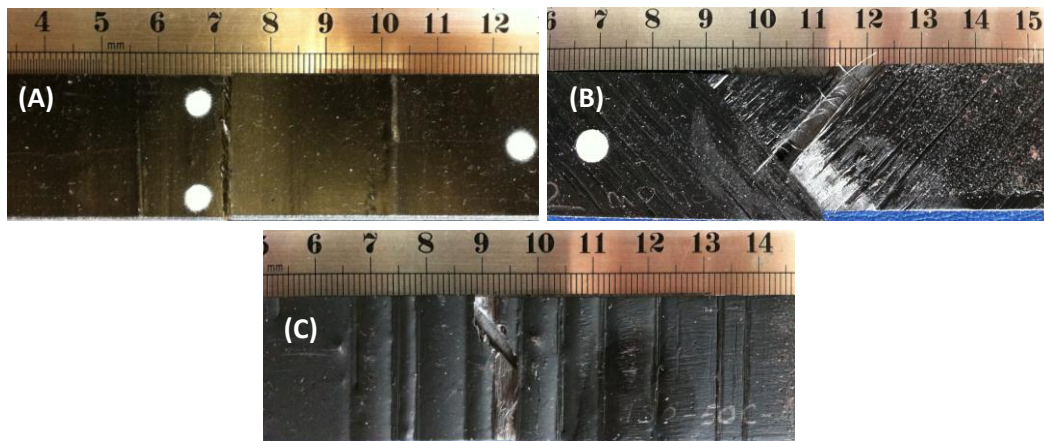


Figure 5 : Damage modes: fibre-matrix debonding for EC90 (A), sliding between plies for EC45 (B) and delamination for ECiso (C).

To compare more easily the residual mechanical properties for the different types of samples, Figure 6 plots the normalized mechanical strength, defined as the ratio 'maximum stress of the exposed sample / maximum stress of the virgin sample' as a function of the incident energy. Three conclusions can be drawn:

1. The thermal energy is the main driving force of the degradation for all exposed sample.
2. For all samples (EC90, EC45, ECiso) two slopes are observed; the first one before inflammation and the second one, more significant, after ignition. This shows the effect of inflammation on the evolution of the maximum stress.
3. Two sets of curves are observed: whereas the EC45 and ECiso curves are similar (very little effect of thermal energy before inflammation), the EC90 curve highlights the rapid degradation of the resin even before inflammation.

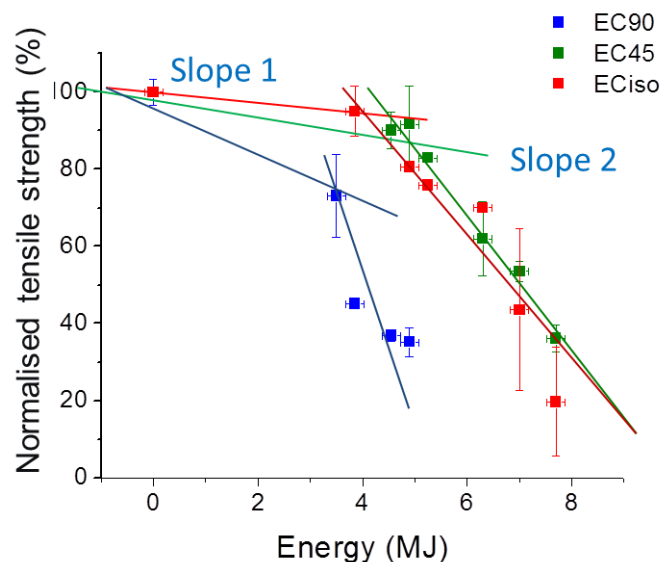


Figure 6 : Normalised tensile stress (%) vs. energy exposure (incident flux of 35kW/m²) for the EC90, EC45, and ECiso; slope 1 before inflammation, Slope2 after inflammation

5 Conclusion

The thermo-mechanical properties of a composite epoxy/carbon fibre have been studied. Two different types of strength reduction are observed depending on whether an ignition process occurred or not. Before ignition, the mechanical strength of exposed samples decreases very slightly and more sharply after ignition. The energy brought by the cone calorimeter is the principal parameter which leads to mechanical strength decrease.

For a given energy level, two groups are observed. A very little influence of the heat flux on the mechanical strength is observed as long as ignition does not occur, whereas mechanical strength drops after inflammation.

For the EC90 samples, the distribution of the resin and its oxidation has been found to have an influence on the degradation kinetics.

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