

# **Influence de la flamme de kérosène sur le comportement au feu et les propriétés mécaniques en flexion de stratifiés hybrides à matrice thermoplastique PEEK**

## *Influence of kerosene flame on the fire behavior and the flexural mechanical properties of hybrid PEEK thermoplastic composite laminates*

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### **Résumé**

Le travail s'intéresse à étudier l'influence d'une flamme kérosène sur le comportement résiduel en flexion (essais de flexion trois points) des stratifiés hybrides à matrice Polyéther éther cétone (PEEK) renforcées par des fibres carbone/verre. L'effet d'une agression thermique (116 kW/m<sup>2</sup> et 1100 °C) sur l'intégrité structurelle des composites est examiné en fonction du temps d'exposition (300 – 600 – 900 s) et de la position de l'éprouvette d'essai à la flamme. La discussion des mécanismes d'endommagement induit par la flamme et le changement mécanique s'appuie sur l'analyse fractographique des échantillons (tomographe et microscope). Lorsque les stratifiés sont soumis à une flamme, le gradient de température se traduit par des mécanismes d'endommagement hétérogènes dans le plan et dans l'épaisseur du stratifié. Une réduction significative de la rigidité en flexion (-65%) et de la résistance en flexion (-85%) est observée après une exposition de 900 s, par rapport aux éprouvettes vierges. L'effet d'une exposition prolongée (de 600 s à 900 s) est modéré sur le comportement en flexion car la barrière thermique formée par un délaminage important contribue à protéger les plis proches de la surface arrière.

### **Abstract**

The present work investigates the influence of kerosene flame exposure on the residual flexural behavior (three-point bending tests) of hybrid quasi-isotropic composite laminates consisting of carbon/glass fibers and a PEEK thermoplastic matrix. The effect of a kerosene flame exposure (116 kW/m<sup>2</sup> and 1100 °C by a burner bench), on the composites structural integrity is examined as a function of exposure time (300 – 600 – 900 s) and distance to fire. The discussions on fire- and mechanically-induced damage mechanisms are supported by fractographic analysis of specimens (tomography and microscopy). The kerosene flame exposure involves in-plane and through-thickness temperature gradients within the plate specimen. A significant reduction in the flexural stiffness (-65%) and flexural strength (-85%) occurs after a 900 s flame exposure, as the structural integrity of fire exposure central zone is significantly affected, compared to virgin specimens. The effect of prolonged exposure time (ranging from 600 s to 900 s) is moderate on flexural behavior as the barrier formed by an extensive thermally-induced delamination contributes to relatively preserve the plies close to the back surface.

**Mots Clés :** thermoplastique PEEK ; flamme de kérosène ; essais de flexion ; pyrolyse ; fibres de carbone ; fibres de verre  
**Keywords:** thermoplastic PEEK; kerosene flame; bending test; pyrolysis; carbon fibers; glass fibers

## **1. Introduction**

There has been a growing demand lately to use high performance thermoplastic-based (TP) composites in aeronautics field [1]. However, regarding the high flammability of composites and stringent fire safety regulations, the wide use of most TPs in many applications is limited by their mechanical properties under severe heat exposure [2], such as the flame exposure of aircraft engines. To date, most attention has been given to the thermosetting (TS) matrix laminates because their widespread usage in aerospace structure, while far less studies focus on the mechanical behavior of TP-based composites under fire conditions for aeronautics [1-3].

After fire exposure, it is a critical safety issue to determine the residual mechanical properties since the loss in stiffness, strength and creep resistance could cause the ultimately failure of composite structures [1]. However, the progress towards the assessment of the residual mechanical properties of thermally degraded composites under critical service conditions has been slow [2]. While there are far less references dealing with TP-based composites [1-3]. The present work thus aims at investigating the influence of fire exposure conditions (116 kW/m<sup>2</sup> and 1150 °C kerosene flame [3]), on thermal degradation, decomposition and the residual flexural behavior of hybrid quasi-isotropic carbon/glass fibers reinforced PolyEther Ether Ketone (PEEK) TP matrix composite laminates (noted as CG/PEEK).

## 2. Materials and experimental set-up

### 2.1 Materials and specimens

The composite material used in this study is CG/PEEK, which consists of PEEK thermoplastic matrix reinforced with a continuous carbon fiber fabric (5-harness satin weave). The specimens have two outer plies of glass fabric (5-harness satin weave), which are used to provide corrosion and electrical protection. The laminates samples (150\*100 mm), as shown in Fig. 1 (a), have 16 plies with a quasi-isotropic layup [(0/90)<sub>G</sub>, (0/90), (±45), (0/90), (±45), (0/90), (±45), (0/90)]<sub>s</sub>. Their average thickness is about 4.5 mm. The characteristic temperatures of PEEK matrix are given in Tab. 1.

$T_{\text{glass transition}} / \text{°C}$	$T_{\text{melting}} / \text{°C}$	$T_{\text{decomposition onset}} / \text{°C}$
143	350	583

Tab. 1. Characteristic temperatures of CG/PEEK laminates

### 2.2 Fire exposure conditions

In this study, a kerosene burner bench [3] is used to provide fire exposure environment, as shown in Fig. 1, which is a domestic device (Cuenod manufacturer). The air to fuel ratio has been selected at 0.85 of the stoichiometric value, in order to obtain the similar flame condition as described in the standard (116 kW/m<sup>2</sup> and 1150 °C) at the sample location. The sample holder is presented in Fig. 1 (c), the protective shield is also used to limit the fire exposure area of the specimen and prevent edge effects from occurring. In accordance with critical service conditions [1], a maximum 900 s exposure to a 1150 °C kerosene flame was considered. The temperature at the back surface is monitored by means of an infrared camera (Fig. 1 (b)). Three sets of specimens have been tested in the following configurations: after a 300 – 600 – 900 s fire exposure.

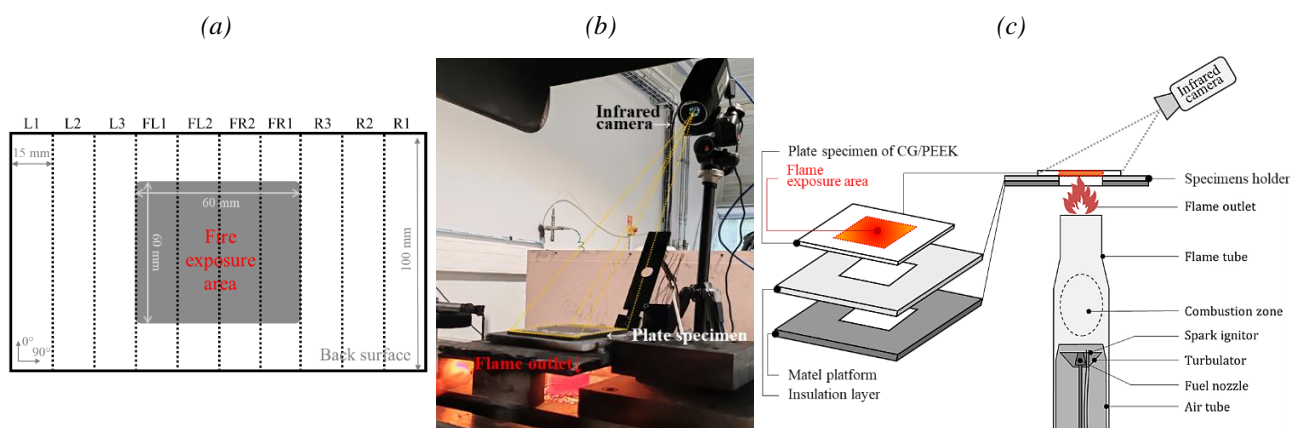


Fig. 1. Specimen and experimental set-up: (a) schema of plate specimen (dotted line represents the slicing positions by waterjet after fire exposure) – (b) kerosene burner bench – (c) schema of fire exposure condition

### 2.3 Mechanical characterization of residual flexural properties and fractographic analysis

After kerosene flame exposure, the specimens are cut by waterjet into 10 bending specimens (15\*100 mm each), as shown in Fig. 1 (a). Three-point bending tests were further conducted to evaluate the residual flexural properties, using a MTS 810 servo-hydraulic testing machine in displacement-controlled mode (2 mm/min) at Room Temperature (RT). The flexural mechanical properties were further determined according to the ASTM standards [4].

Fractographic analysis has been carried out by means of microscopic and tomographic observations using a numerical optical microscope Keyence VHX-5000 and a micro-CT RX Solutions Easy Tom 150 respectively to build 3D pictures of damaged areas. The analysis was conducted on specimens after kerosene flame exposure as well as after mechanical loading, to investigate both fire- and mechanically induced deformation and damage mechanisms.

## 3. Results and discussion

### 3.1 Kerosene flame behavior

Using the infrared camera, the temperature evolution at the back surface under fire exposure up to 900 s is monitored, as shown in Fig. 2. From Fig. 2 (a), after a 200 s of fire exposure, the temperature reaches the plateau of PEEK matrix melting temperature (around 350°C). Then, the jump of temperature is mainly due to the ignition of pyrolysis gases within the specimen [1]. Eventually, the back-surface temperature stabilizes after 300 s, reaching a level of 425°C. As a result of a one-sided fire exposure, the through-thickness and in-plane temperature gradients cause heterogeneous thermal degradation (delamination, porosity and char). As expected, the degraded area becomes larger as the exposure times increases on the back surfaces, as shown in Fig. 2 (a). In the center of the specimen, thermally induced degradation is the most severe. Carbonaceous char has formed along with the PEEK matrix pyrolysis. Pyrolysis is completed on the exposed surface as shown by bare glass fibers.

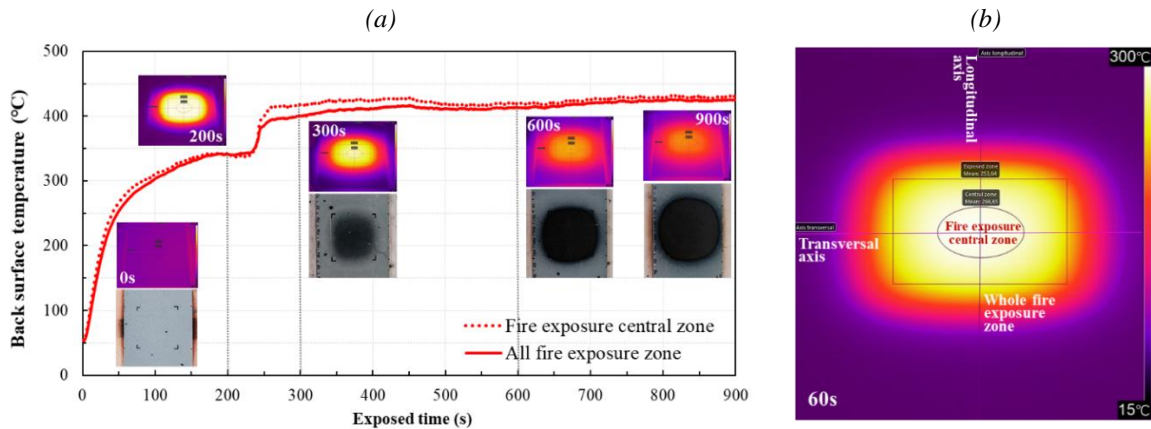


Fig. 2. Temperature measurement: (a) evolution of temperature and thermal degradation – (b) zones of measurement

Through the thickness, the laminates after flame exposure could be divided into two well-defined areas: the thermal degraded area and the relatively preserved area, as shown in Fig. 3 (a). In the first zone, the polymer content is negligible since the matrix has completely decomposed, then the matrix in the decomposition zone is partially decomposed (char and pyrolysis gases [2]). As fire-exposure time increases, the first zone extends progressively towards the unexposed surface. After a 900 s exposure, the damaged area has reached about 2/3 of the laminates thickness. Compared to the 600 s case, a prolonged exposure time seems to have a limited influence of damage extension. This is because the fast temperature increase, resulting in the thermal expansion of matrix polymer and the thermal contraction of the carbon fiber [1], leads to fiber – matrix debonding and delamination, which usually acts as barriers for heat conduction [2].

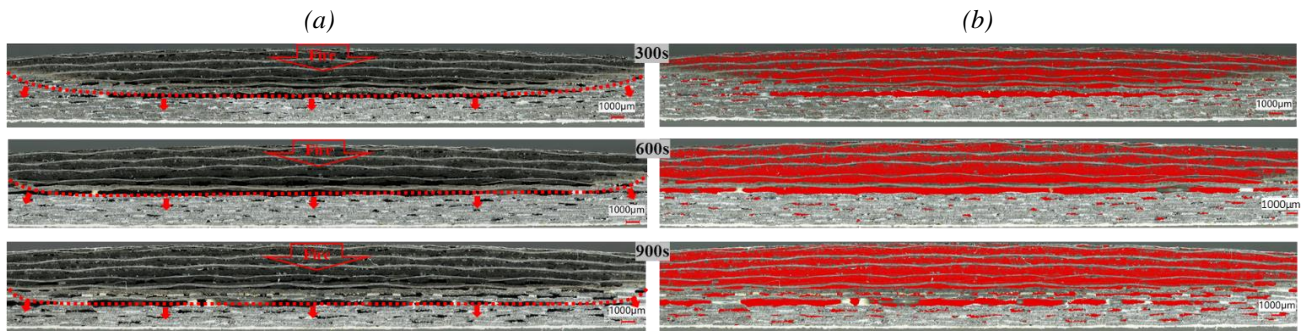


Fig. 3. Through-thickness microscopic observations of cross sections FL1 after different kerosene flame exposure time: (a) thermal degradation extension – (b) porosity (in red) distribution within the material

After thermal aggression, thermal decomposition of the PEEK matrix results in the formation of pyrolysis gases within the laminates, coming along with the mass loss and the formation of porosities in the laminate. The porosity volume fraction is defined by the percentage of porosities of matrix over the whole specimen volume. The porosity volume fraction is given by the following equation (Eq.1):

$$\rho_p = \frac{V_f - V_i}{V_f} + \frac{V_l}{V_f} \quad (\text{Eq. 1})$$

Where  $V_i$  and  $V_f$  is initial and final volume respectively, and  $V_l$  is the increase in void volume induced by mass loss, which is calculated by the mass loss and the density of PEEK. From Fig. 4, likewise microscopic observations, the mass loss and porosity volume fraction growth tend to stabilize after 600 s. And the porosity content is mainly located in the fire exposure area (FL1-2 and FR1-2) and specimens nearby (L3 and R3). The higher mass loss can be interpreted as the consequence of severe localized degradation. For the bending specimens on the edges of large specimens (L1-2 and R1-2), the mass loss ratio is close to 0% and porosity mainly results from the thermal expansion of the fibers and the matrix (induced by heat conduction from central zone).

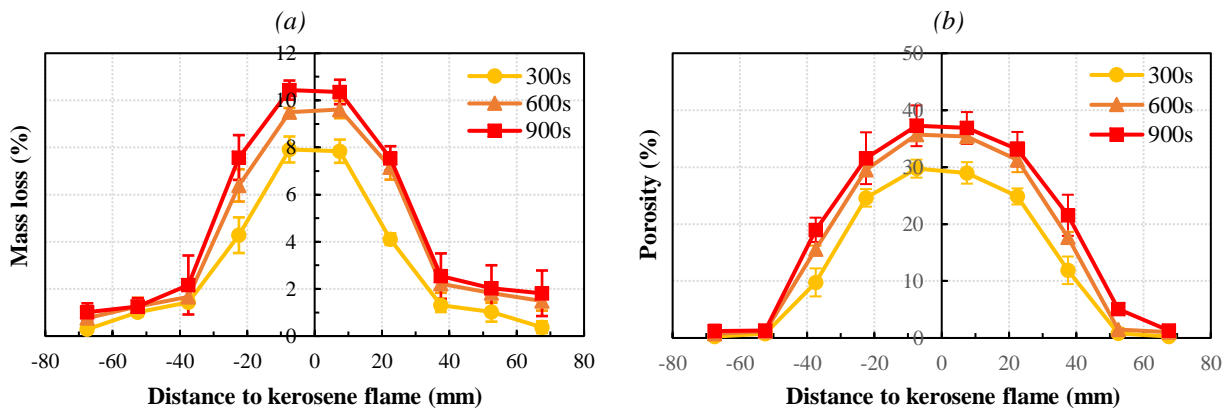


Fig. 4. Mass loss and porosity after different kerosene flame exposure time: (a) mass loss ratio – (b) porosity volume ratio

### 3.2 Residual flexural behavior

Depending on the location of the bending specimen (directly waterjet cut from the plate exposed to fire) and the exposure time, the macroscopic mechanical response significantly differs from one specimen to another, as shown in Fig. 5. Regardless the exposure time, it appears that the specimens far from kerosene flame (specimens L1-2-3 and R1-2-3) are not seemingly degraded for all fire testing conditions. The macroscopic response is similar to the one observed in the virgin state (no prior fire exposure), while the fracture appears earlier than the virgin one. The flexural response shifts to an elastic quasi-brittle behavior as the distance to kerosene flame decreases (specimens FL1 and FR1). Specimen FL2 and FR2 seem much degraded with an elastic domain interrupted at 0.7% strain.

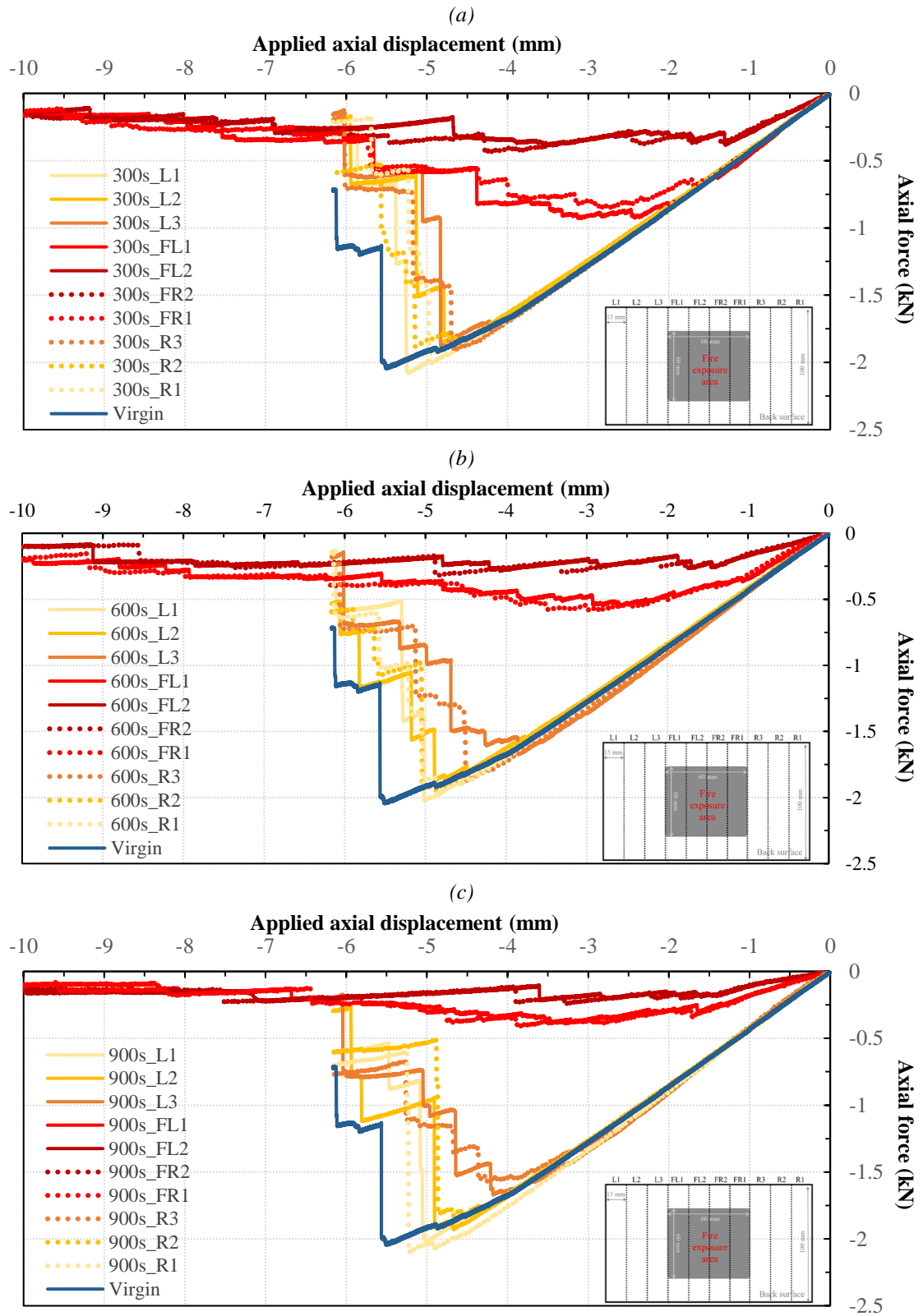


Fig. 5. Flexural responses of CG/PEEK laminates on different positions after different kerosene flame exposure times: (a) 300 s – (b) 600 s – (c) 900 s

The influence of in-plane and through-the-thickness temperature gradients on the decrease in the flexural properties is shown on Fig. 6. In specimens L1-2-3 and R1-2-3, the decreases in both flexural stiffness and strength are moderate (less than 15%), confirming that thermal degradation is limited in these specimens. In the most degraded state (specimens FL1-2 and FR1-2 under fire exposure), the

flexural stiffness dramatically decreases by more than 65% and the axial strength loses around 85% of its initial value (as-received state). Regarding the influence of fire exposure time, both the flexural stiffness and strength tend to be stable after 600 s. A prolonged exposure time (ranging from 600 s to 900 s) seems to have limited influence on flexural properties as damages slightly propagate through-the-thickness, as the char layer acts as a thermal insulation layer with reduced thermal conductivity to prevent the extension of thermally-induced damages.

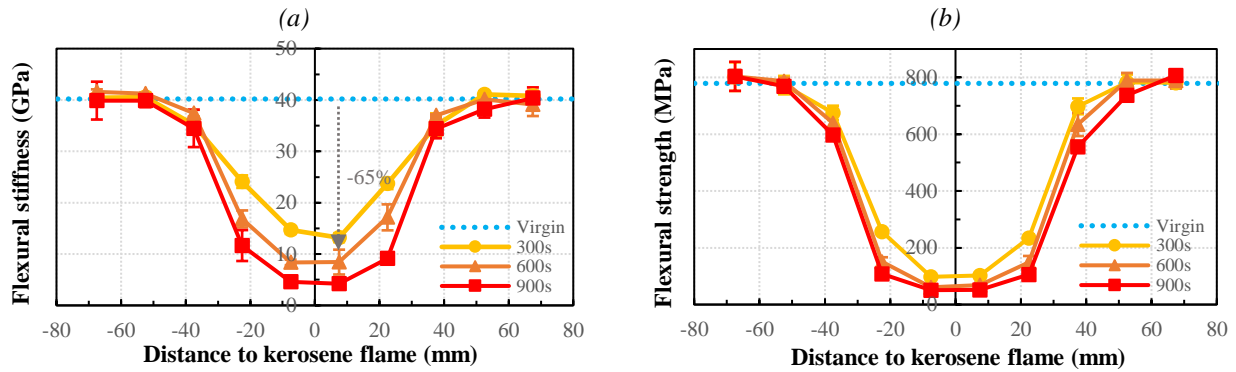


Fig. 6. Changes in the flexural properties of post-fire specimens: (a) flexural stiffness – (b) flexural strength

Tomographic observations (Fig. 7) of failed bending specimens are now discussed along with the residual mechanical behavior. When the laminates are exposed to a kerosene flame, the heterogeneous thermal degradation through-the-thickness results in atypical failure mechanisms under mechanical loading. As shown in Fig. 5, the failure mechanisms of post-fire laminates with different exposure times are generally characterized by the gradual breakage of  $0^\circ$  plies of less damaged region. From Fig. 7 (b), the bending load borne by  $0^\circ$  fibers is different in these two regions due to the heterogeneous thermally-induced damages. It seems that the load is mostly taken up by  $0^\circ$  plies of less damaged region. Depending on the thermal decomposition of each ply, the bearing capabilities of the plies is gradually degraded. As exposure time increases, the gradual failure of most degraded specimens subjected to a flexural loading comes along with fiber/matrix debonding and delamination.

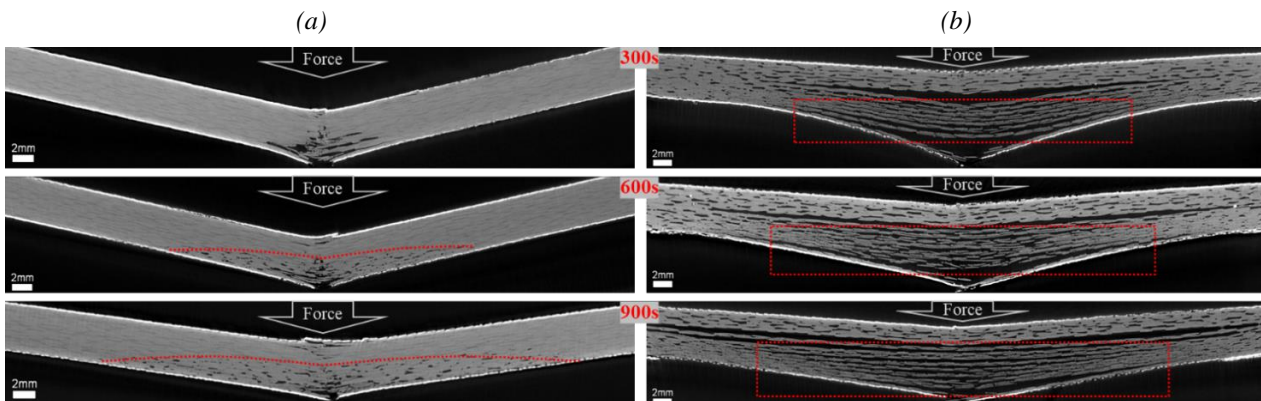


Fig. 7. Tomographic observations of failure in bending of the CG/PEEK laminates after exposed to kerosene flame for different exposure times on different positions: (a) cross section L2 – (b) cross section FL2

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