# **Post-fire flexural properties of fibre-reinforced polyester, epoxy and phenolic composites**

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The effect of fire damage on the flexural properties of fibre-reinforced polymer (FRP) composites is investigated. The FRP composites studied contained glass, carbon or Kevlar fibres with a polyester, epoxy or phenolic resin matrix. Artificial fire tests were performed on the composites using a cone calorimeter. The residual flexural modulus and strength of the burnt composites were determined at room temperature after the fire tests. The post-fire flexural properties of all the composites decreased rapidly with increasing heating time. Even the properties of the fibre-reinforced phenolic materials were severely degraded despite their low flammability and excellent fire resistance. The flexural properties of the phenolic-based composites were reduced due to thermal degradation and cracking of the resin matrix. In comparison, the properties of the polyester- and epoxy-based composites were reduced by combustion of the resin and formation of delamination cracks. A model is presented for determining the post-fire flexural properties of FRP composites with good accuracy. -<sup>C</sup> *2002 Kluwer Academic Publishers*

## **1. Introduction**

Fibre-reinforced polymer (FRP) composites are used in aircraft, marine craft, high performance automobiles, civil structures and sporting goods. The most commonly used FRP composite is glass/polyester because of its moderate cost, good strength and excellent corrosion resistance. Composites requiring higher stiffness, strength and fatigue endurance than glass/polyester are usually made of carbon/epoxy, glass/epoxy or Kevlar/epoxy. These materials are used in aircraft structures such as wing panels and fuselage sections as well as in racing yachts, powerboats, and body panels to racing cars.

While composites are used in a wide variety of applications, there is concern about their poor fire resistance and high flammability. When polyester- and epoxy-based composites are exposed to a hot fire they quickly ignite and then release large amounts of heat, smoke and fumes. A further problem is that the mechanical properties of these composites can be severely degraded during a fire from combustion and thermal softening of the polymer matrix. Even after the fire has been extinguished, studies by Pering *et al.* [1], Sorathia *et al.* [2], and Mouritz and Mathys [3–5] have shown that the post-fire mechanical properties of heavily burnt composites can be much lower than the original property values. The poor fire resistance of most types of FRP composites has limited their use in applications where fire is a major concern, such as on submarines and off-shore oil drilling platforms.

Various techniques have been developed to improve the fire resistance of composites. Resins with fire-retardant additives such as aluminium trihydrate, antimony trioxide or zinc borate can reduce the flammability of FRP composites when exposed to low-tomedium intensity fires. However, the drawbacks are that fire-retardant resins are expensive and some additives can decompose into toxic gases when the resin matrix burns. Another technique to improve the fire resistance is to coat the composite with a thermal barrier material such as an intumescent mat or ceramic fibre blanket. Some thermal barrier materials are highly effective in delaying the onset of combustion and minimising the loss to the mechanical properties of composites caused by a fire [3, 6]. Another technique for improving the fire resistance is to use phenolic resin as the matrix material in FRP composites. Phenolic-based composites have longer ignition times and lower flame spread, heat release and smoke production rates than polyester- and epoxy-based composites [6–9]. However, the effect of fire on the mechanical properties of phenolic-based composites has not been extensively studied [2, 4].

The purpose of this study is to assess the effect of fire damage on the flexural properties of a variety of FRP composites containing glass, carbon or Kevlar fibres with a polyester, epoxy or phenolic resin matrix. These materials encompass the board range of engineering composites used in structural applications. Artificial fire tests are performed using the radiant heater to a cone calorimeter, which causes heat damage to composites that is similar to the damage caused by a real fire. The

composites are exposed to the heat flux of 50 kW/m<sup>2</sup>, which is about the heat intensity of a room fire [2, 10], for times ranging from 15 to 240 seconds. After the fire tests, the residual flexural modulus and strength of the composites are determined at room temperature. This information is used to identify those composites that have the highest mechanical properties after a fire. The data is also used to further validate a model recently proposed by Mouritz and Mathys [3, 5] for determining the post-fire flexural properties of FRP composites.

### **2. Model for the post-fire flexural properties of FRP composites**

When FRP composites are exposed to a hot fire for a sufficient time the resin matrix is thermally degraded and pyrolysized. This can cause heat damage in the forms of charring and delamination cracking. Charring is defined as the thermal degradation process in which the resin matrix is combusted and degraded to a carbonaceous residual material. Fire research by Mouritz and Mathys [3–5] show that when FRP composites are exposed to uniform heating over one surface then the fire damage extends through the material along an even, well-defined combustion front as shown schematically in Fig. 1. This figure shows that a composite that is burnt part-way through basically consists of a char layer and an underlying unburnt layer.

Mouritz and Mathys [3–5] have also found that the mechanical properties of the char layer are much lower than the properties of the unburnt composite layer. In most materials the mechanical properties of the char layer are negligible whereas the properties of the unburnt layer are similar to the original properties of the composite before fire testing. Therefore, a partially burnt composite can be considered as a two-layer material with one layer having been severely degraded by fire while the second layer has the properties of an unburnt composite. A major simplifying assumption with this analysis is that no damage occurs in the unburnt composite, such as overheating of the resin matrix or heat-induced cracking. It will be described later that some damage does occur, although it does not significantly degrade the flexural properties of the unburnt portion of the composite.

When a long, slender beam of uniform properties is loaded symmetrically in the four point bending configuration (shown in Fig. 2) then the stress in any layer of



*Figure 1* Schematic of a fire-damaged composite represented as a twolayer material with one layer being the char region and the second layer is the unburnt region. Shown is an imaginary line indicating the neutral bending axis of the composite beam.



*Figure 2* Schematic of four-point flexural loading on a fire-damaged composite.

fibres at the distant *y* from the neutral axis is determined by:

$$
\sigma = \frac{Ey}{R} = \frac{My}{I} \tag{1}
$$

where  $E$  is the bending modulus and  $R$  is the bending radius. *M* is the bending moment that is calculated by:

$$
M = \frac{PL}{8} \tag{2}
$$

where  $P$  is the applied force and  $L$  is the length of the support span. In Equation 1, *I* is the second moment of area which equals:

$$
I = \frac{bd^3}{12} \tag{3}
$$

where *b* and *d* are the width and depth of the beam.

When a two-layer material, such as a partially burnt composite, is loaded in quarter-point bending (as shown in Fig. 2) then Equation 1 can be re-written in the form:

$$
\sigma = \frac{M \cdot (d - d_{\rm n})}{R} \tag{4}
$$

where  $d_n$  is the neutral axis of the beam. The bending moment, *M*, of the fire-damaged beam can be determined by solving:

$$
M = \int \sigma \cdot b \cdot y \cdot dy = \frac{E_0 b}{R} \int_0^{d - d_n} y^2 \cdot dy
$$
  
+  $\frac{E_0 b}{R} \int_0^{d_n - d_c} y^2 \cdot dy + \frac{E_0 b}{R} \int_{d_n - d_c}^{d_n} y^2 \cdot dy;$   
 $E_c > 0; d_c > 0$  (5)

where  $d_c$  is the thickness of the char layer, and  $E_o$  and  $E_c$  are the bending modulus of the unburnt and char layers, respectively.

Solving Equation 5 yields:

$$
M = \frac{\sigma \cdot b}{3} (d - d_n)^2 + \frac{\sigma \cdot b}{3} (d_n - d_c)^2 + \frac{\sigma \cdot b}{3} \cdot \frac{E_c}{E_o} \frac{[d_n^3 - (d_n - d_c)^3]}{(d - d_n)}
$$
(6)

where the neutral axis is determined by:

$$
d_{\rm n} = \frac{E_{\rm o}d^2 - d_{\rm c}^2(E_{\rm o} - E_{\rm c})}{2E_{\rm o}d + 2E_{\rm c}d_{\rm c} - 2E_{\rm o}d_{\rm c}}\tag{7}
$$

The failure load is then determined by substituting Equation 6 into Equation 4 to give:

$$
P = \frac{8}{L} \left\{ \frac{\sigma \cdot b}{3} \cdot \frac{(d - d_n)^3 + (d_n - d_c)^3}{(d - d_n)} + \frac{\sigma \cdot b}{3} \cdot \frac{E_c}{E_0} \cdot \frac{[d_n^3 - (d_n - d_c)^3]}{(d - d_n)} \right\}; \quad E_c > 0 \quad (8)
$$

For the case where the flexural properties of the char layer are negligible, then Equation 8 is reduced to:

$$
P = \frac{8}{L} \left\{ \frac{\sigma \cdot b}{3} \cdot \frac{(d - d_n)^3 + (d_n - d_c)^3}{(d - d_n)} \right\}; \quad E_c = 0
$$
\n(9)

The apparent flexural strength of a fire-damaged material is then calculated using:

$$
\sigma = \frac{3PL}{4bd^2} \tag{10}
$$

by rearranging Equation 9 with  $d_c = 0$  and  $d_n = d/2$ .

The apparent flexural modulus of a fire-damaged composite is calculated using:

$$
E = \left\{ \frac{4(d - d_n)^3 + 4(d_n - d_c)^3}{d^3} + 4\frac{E_c}{E_o} \cdot \frac{[d_n^3 - (d_n - d_c)^3]}{d^3} \right\} \cdot E_o; \quad E_c > 0 \quad (11)
$$

For the case where the flexural properties of the char layer are negligible, then this equation reduces to:

TABLE I Composition of polyester-based composites

$$
E = \left\{ \frac{4(d - d_n)^3 + 4(d_n - d_c)^3}{d^3} \right\} \cdot E_o; \quad E_c = 0 \quad (12)
$$

#### **3. Materials and experimental techniques** 3.1. Composite materials

The composites used in the study are grouped as polyester-, epoxy- or phenolic-based materials. The composition of the polyester-based composites is described in Table I. The resin matrix used in all these composites is an isophthalic polyester (Synolite 0288-T-1) made by Dulux Australia. The polyester resin is cured by mixing with 2.1 parts per hundred (pph) (by weight) of 6% cobalt (II) 2-ethylhexanoate solution and 1.7 pph of 25% methyl ethyl ketone peroxide solution. The polyester-based composites were manufactured using the wet hand lay-up process, and then cured at room temperature for several weeks before testing.

A description of the composites with the epoxy resin matrix is given in Table II. The resin used in the carbon/ epoxy composite is a PR500 epoxy supplied by 3M. This composite was fabricated using vacuum-assisted resin transfer moulding. The resin was drawn into the mould at 150◦C, and after fabrication the composite was post-cured at 170◦C for 1 hour. The two types of glass/epoxy, on the other hand, contained a resin made of an Araldite M epoxide mixed with 15 pph of HY951 hardener (100% triethylene tetramine). Both the Araldite M and HY951 were supplied by Ciba-Geigy Australia Ltd. The two glass/epoxy composites were laminated using the wet lay-up process and then cured at room temperature for several weeks.

The composites with the phenolic resin matrix are described in Table III. The resin is a low temperature acid-

Composite	Fibre type	Fibre lay-up	Number of plies	Fibre volume fraction	Thickness
Woven glass/polyester	1400 $g/m2$ plain woven roving E-glass fabric (type DF1400) made by Colan Industries Pty. Ltd.	[0/90]		0.34	5.1 mm
Chopped glass/polyester	450 g/m <sup>2</sup> chopped strand mat E-glass (type PM200) made by ACI Fibreglass Pty Ltd.	Isotropic		0.17	$5.5 \text{ mm}$
Kevlar/polyester	$325$ g/m <sup>2</sup> plain woven roving Kevlar-49 fabric made by Clark-Schwebel Pty Ltd.	$[0/90]_s$	14	0.86	4.4 mm

TABLE II Composition of epoxy-based composites

Composite	Fibre type	Fibre lay-up	Number of plies	Fibre volume fraction	Thickness
Carbon/epoxy	195 $g/m^2$ T650 plain woven roving carbon fabric (type W46282) made by Amoco Polymers, Inc.	$[0/+45/-45/90]$	32	0.79	$4.9 \text{ mm}$
Woven glass/epoxy	1400 $g/m2$ plain woven roving E-glass fabric (type DF1400) made by Colan Industries Pty. Ltd.	$[0/90]_s$		0.33	7.4 mm
Chopped glass/epoxy	450 $g/m2$ chopped strand mat E-glass (type) PM200) made by ACI Fibreglass Pty Ltd.	Isotropic		0.14	$6.7$ mm

TABLE III Composition of phenolic-based composites





(b)

*Figure 3* (a) General view of the cone calorimeter. (b) A close-up view showing a burning composite specimen in the fire test chamber.

cure resole phenolic (Resinox $\mathscr{B}$  1916) mixed with 7 pph hardener (Resinox AH1964F). The Huntsman Chemical Company Australia Ltd. manufactured the phenolic resin and hardener. The three phenolic-based composites were made using the wet hand lay-up process and then cured at room temperature for about 24 hours, followed by a post-cure at 60◦C for 1 hour and 80◦C for two hours.

## 3.2. Fire testing

Artificial fire tests were performed on the composites using a Stanton Redcroft cone calorimeter, which is shown in Fig. 3. The composites were placed horizontal to an electric radiant heat source and exposed to an incident heat flux of 50 kW/m<sup>2</sup> for different times up to four minutes. Temperatures generated in the cone calorimeter when operated at the heat flux of  $50 \text{ kW/m}^2$ are shown in Fig. 4. The surface temperature of the composite rises rapidly with time up to ∼700◦C after four minutes.

A spark igniter located between the heat source and specimen was used to ignite combustible gases released from the composites. A large flame was produced when the composites ignited, as seen in Fig. 3b. The heating time needed to produce a stable, continuous flame is known as the time-to-ignition, and these times were



*Figure 4* Time-temperature profile at a composite surface for the heat flux of  $50 \text{ kW/m}^2$ .

measured for the different composites during fire testing. After testing, any flames were extinguished and the composites were cooled to room temperature before the flexural properties were determined.

#### 3.3. Flexural testing

The flexural modulus and strength of the composites were determined at room temperature using the four-point bend method in ASTM D790M. The specimens were loaded using an Instron 5500 machine at a cross-head speed of about 5 mm/min in 1/4-point loading. The flexural properties of four specimens of each composite were determined after testing in the cone calorimeter. The surface of the composite specimen that has been exposed to the heat source was placed against the two load points in the four-point bend test, as shown schematically in Fig. 2. Because of this, the heat-damaged surface of the composite was subject to a bending-induced compressive stress.

The flexural modulus  $(E_B)$  of the specimens was calculated using:

$$
E_{\rm B} = \frac{0.17L^3m}{bd^3} \tag{13}
$$

where *m* is the slope of the tangent of the initial straightline portion of the load deflection curve. The flexural strength was determined using Equation 10.

#### 3.4. Microstructural analysis

Fire damage to the composites was investigated after testing in the cone calorimeter using scanning electron microscopy (SEM) and ultrasonics. Polished sections of the fire-damaged composites were examined using an SEM operated in the secondary and back-scattered imaging modes. The size and depth of delamination cracks in the composites were measured using a Panametrics 0.5 MHz ultrasonic transducer and a Krautkrämer-Branson USD15 (Version 4.00.02) ultrasonic dialog flaw detector operated in the pulseecho mode.

#### **4. Results and discussion**

#### 4.1. Ignition and fire damage

The ignition times and fire-induced damage for the polyester- and epoxy-based composites were similar, and these differed significantly from the phenolic-based

materials. The polyester- and epoxy-based composites ignited shortly after being exposed to the heat source in the cone calorimeter, and then burned with a large flame for the duration of the fire tests. The phenolicbased materials, on the other hand, smouldered for a long period before eventually igniting at times that were much longer. The time-to-ignition values for the composites tested at the heat flux of  $50 \text{ kW/m}^2$  are given in Fig. 5. It is seen the polyester- and epoxy-based composites began burning in less than one minute whereas the phenolic laminates took between 2.5 to 7.5 minutes to ignite. The short ignition times of the polyester and epoxy composites is due in part to the low combustion temperature of the resin matrix, which is between 300 and 400 $^{\circ}$ C [11, 12]. The heating profile of the fire



*Figure 5* Ignition times for the (a) polyester-, (b) epoxy- and (c) phenolic-based composites exposed to the heat flux of 50 kW/m2.



#### $5 \text{ mm}$

*Figure 6* A cross-section view of a composite after fire testing. The top surface of the specimen was exposed to the heat source, and the discoloured (black) layer is the char region.

tests in Fig. 4 shows that the composite surface reaches 300–400◦C within 30–60 seconds, and this rapid heating is responsible for the short ignition times for the polyester- and epoxy-based materials.

The longer ignition times for the phenolic-based composites is due to the higher decomposition temperature and the lower release rate of combustible volatiles during heating. St John *et al.* [13] have studied the thermal decomposition of phenolic composites, and found that the main chemical network structure of the resin remains reasonably stable until the temperature rises above  $\sim$ 550°C. It is seen in Fig. 4 that the composites do not reach this temperature until after two minutes, and this is part of the reason for the phenolic composites having a long ignition time. Ignition is further delayed because combustible gases are released by thermal decomposition of the phenolic matrix at a slower rate than polyester or epoxy resins. As a result, a longer heating time is needed for the phenolic-based composites before the gas concentration reaches the critical level required for ignition to occur in the cone calorimeter.

The precise temperature at which thermal decomposition of the polyester-, epoxy- and phenolic-based composites occurs depends on a number of factors such as the cross-linking density and composition of the resin matrix, the cure process, and the type of fibre reinforcement [12, 13]. It is seen in Fig. 5 that the type of reinforcement affects the ignition times. For example, the ignition times for the composites reinforced with woven glass fibres are longer than for the laminates containing chopped glass fibres when the resin matrix is the same. Brown and Mathys [9] have reported a similar result, and this difference is attributed to the early ignition of volatiles generated from the thermal decomposition of additional binding agents used in the preparation of chopped glass mats. As another example of the influence of fibre reinforcement, the ignition times for the Kevlar fibre composites are shorter than for the materials reinforced with glass or carbon fibre. The short times may be due in part to thermal decomposition of the Kevlar reinforcement at a relatively low temperature. Thermal stability studies by Brown and colleagues [14–16] and Penn and Larsen [17] show that Kevlar fibres begin to decompose at relatively low temperature  $(150-250°C)$  with the formation of combustible gases that contribute to early ignition, whereas the other fibre types are stable to much higher temperatures.

Fire damage to the composites was investigated after testing in the cone calorimeter through visual inspection, ultrasonics and scanning electron microscopy. A feature common to all the composites was the formation of a discoloured (black) layer below the heated surface, as seen in Fig. 6. This layer began to form in the polyester- and epoxy-based composites at ignition whereas the phenolic materials began to discolour well before the ignition of the resin. The discoloured layer, which in this paper is called the 'char region', occurs as a well-defined layer that can be clearly distinguished from the lower layer of unburnt composite. In this way the fire-damaged composites appear as two-layer materials with one layer being the char and the other layer the unburnt laminate, as illustrated in Fig. 1.

The physical structure of the char layer in the polyester- and epoxy-based composites was different to the structure of the char in the phenolic materials. The resin matrix within the charred polyester- and epoxybased composites is almost completely consumed during the fire tests, leaving exposed fibres as shown in Fig. 7. As a result, the flexural modulus and strength of the char layer for the composites is very low. The resin in the char layer to the phenolic composites, on the other hand, is not consumed because of the low flammability of the matrix. However, the fact that the phenolic matrix within the char layer is discoloured suggests it is heavily degraded. Mouritz and Mathys [4] report that the flexural stiffness and strength of charred phenolic composite is very low due to embrittlement of the resin matrix. The thickness of the char layer increased with heating time for all the composites. For example, Fig. 8 shows that the char layer thickness increased steadily with time for the polyester-, epoxy- and phenolic-based composites containing chopped glass fibres. The other composites showed similar trends to these materials.

The appearance of the polyester-, epoxy- and phenolic-based composites within the unburnt layer below the char layer was similar. Small delamination cracks were detected by pulse-echo ultrasonics close to the interface between the char and unburnt layers.

## 4.2. Post-fire flexural properties

The flexural modulus and strength of all the composites is degraded by fire, including the phenolic-based materials that have excellent fire resistant properties. Fig. 9 shows the effect of heating time on the post-fire flexural properties for the different composites containing



*Figure 7* Microstructure to the char in the polyester- and epoxy-based composites that consists of exposed fibres.



*Figure 8* Effect of heating time on the thickness of the char layer in the chopped glass composites.

chopped glass fibres. The flexural properties remain unchanged for only a short time  $\left( < 30 \text{ seconds} \right)$ , and then drop rapidly with increasing heating time. The reduction to the flexural properties starts to occur at about the same time that the composites begin to char. Numerous studies [1–5] attribute the reduction to the post-fire mechanical properties of composites to damage caused by the fire, particularly the char damage. It is seen in Fig. 9 that the flexural properties decrease at the same time as the thickness of the char layer increases (Fig. 8), and this supports the proposition that char damage is mainly responsible for the degradation to the mechanical properties. The post-fire flexural properties of the other materials studied in this paper drop with increasing heating time in a similar manner to that shown in Fig. 9 for the chopped glass composites.

The curves in Fig. 9 show the theoretical reduction to the post-fire flexural properties determined using



*Figure 9* Effect of heating time on the post-fire flexural properties of the chopped glass composites with a (a&b) polyester, (c&d) epoxy and (e&f) phenolic resin matrix. The error bars represent two standard deviations in the measured post-fire properties. The curves show the predicted post-fire properties determined using the model.

the model described earlier (ie. Equations 10 and 12). These curves were easily calculated by simply measuring the flexural modulus and strength of the original composite and the thickness of the char layer after the fire test. The char layer thickness can be measured using a variety of methods, and in this study it was determined using pulse-echo ultrasonics. It is seen that there is good agreement between the measured and theoretical post-fire flexural properties for the chopped glass composites, and the model was found to be equally accurate for determining the post-fire properties of the other composite materials.

The results shown in Figs 8 and 9 indicate that the main cause for the deterioration to the flexural properties is thermal degradation of the resin matrix. Fig. 10 shows the effect of increasing amount of char damage  $(d_c/d)$  on the normalised flexural modulus  $(E/E_0)$ and strength ( $\sigma/\sigma_o$ ) of the composites. The normalised flexural modulus is the post-fire flexural modulus (*E*) divided by the original modulus of the composite  $(E_0)$ .



*Figure 10* Effect of the amount of charddamage  $(d_c/d)$  on the normalised post-fire flexural (a) modulus and (b) strength of the composites.

Similarly, the normalised flexural strength is the postfire strength ( $\sigma$ ) divided by the original strength ( $\sigma_0$ ). Despite the large amount of scatter in the results, the overall trend is that the post-fire flexural properties of all the composites drop rapidly with increased amount of char damage. It is seen in Fig. 10 that relatively small amounts of char cause a large reduction to the properties. For example, when only 10% of a composite has been charred (ie.  $d/d_c = 0.10$ ) the flexural properties drop by 20–30%, and when 25% of a composite is char (ie.  $d/d_c = 0.25$ ) the properties fall by 50–60%. This is because the flexural properties are sensitive to small changes to the effective load-bearing thickness of the composite, as indicated by Equations 9 and 12.

Fig. 11 compares the theoretical against the measured post-fire flexural modulus and strength values for all the composites. The straight line has a slope of unity, and therefore the closer the data points are to the line the better is the agreement between the theoretical and measured properties. The insets to the figure show the cluster of relatively low flexural properties in greater detail. With the exception of a few out-lying data points, there is excellent agreement between the theoretical and measured post-fire properties. This proves that the post-fire flexural properties of FRP composites can be accurately determined using the models that require knowledge of



*Figure 11* Comparison of the theoretical and measured post-fire flexural (a) modulus and (b) strength values of the composites.

three easily measured parameters, namely the original modulus and strength of the material and the thickness of the char.

#### **5. Conclusions**

This study has shown that the flexural properties of polyester-, epoxy- and phenolic-based composites can be severely degraded by fire. The post-fire properties of thin composite materials drop rapidly with increasing heating time, and therefore even relatively small amounts of fire damage can cause a large reduction to the properties. The rapid reduction to the properties of polyester- and epoxy-based composites is due mainly to combustion of the resin matrix. The combustion process degrades the composite to a char that has no flexural stiffness or strength.

A significant finding is that the post-fire flexural properties of phenolic-based composites are similar to the post-fire properties of polyester- and epoxy-based composites. This is despite the better fire resistance and lower flammability of phenolic composites. It appears that the high temperature generated in a hot fire is sufficient to cause severe thermal degradation of the phenolic matrix without the composite burning. Thermal degradation causes the flexural properties of phenolicbased composites to drop rapidly with increasing heating time. It is proven that the post-fire flexural modulus and strength of FRP composites can usually be predicted with good accuracy using the model presented in this paper.

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