Materials Letters
March 2008, Volume 62, Issues 6-7, Pages 1096-1099
http://dx.doi.org/10.1016/j.matlet.2007.07.071

High modulus carbon fibre composites: Correlation between transverse tensile and mode I interlaminar fracture properties

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Abstract:

This paper presents test results from mode I interlaminar fracture and transverse tensile tests on unidirectional carbon/epoxy composites used in racing yacht construction. Fibre modulus has been varied from 380 to 640 GPa. PAN fibre reinforced composite properties, delamination resistance (GIc and GIp) and transverse strain to failure, decrease as fibre modulus increases. Composites based on the highest modulus pitch fibres show higher transverse failure strains than the highest modulus PAN fibre composites. A distinct failure mechanism, crack propagation within the fibres, was observed for the pitch based reinforcement. The transverse tensile tests allowed transverse fibre modulus to be estimated as 11.5 GPa for the PAN and 9 GPa for the pitch fibres. These results indicate that more detailed study is needed if pitch based composites are to be optimised.

Keywords: Composite materials; Delamination; Deformation; Fracture; Carbon fibre

Introduction

High performance carbon fibres can be classed in three categories, high resistance (HR), intermediate modulus (IM) and high modulus (HM). The latter are often defined as those fibres with a modulus greater than 400 GPa. All these fibres are used in sports products, such as golf clubs and fishing rods, but the high modulus fibres are essentially found in aeronautical applications. However, over the last ten years the use of HM fibres has also developed for the masts of ocean racing yachts, and particularly for 60 foot Open Class multi-hulls. These structures are generally in composite sandwich, carbon composite facings on honeycomb core, of length around 30 meters and weighing around 450 kg, manufactured from prepreg either in ovens under vacuum or autoclaves.

In order to reduce weight but maintain high stiffness architects and designers progressively increased the fibre modulus. However, a number of problems in nearly all the composite masts with fibres of modulus above 500 GPa and breakage of several masts in service resulted in a limit to the fibre modulus of 450 GPa being imposed in 2003. Delamination is one of the main damage mechanisms in these structures.

The properties of composite materials reinforced by unidirectional (UD) fibres are known to be highly anisotropic with high values of stiffness and strength in the fibre direction and poor mechanical behaviour in the transverse direction. Low transverse tensile strength is a major weakness of composites. The presence of fibres results in stress concentrations and the strength is lower than that of the unreinforced matrix. For a multi-directional composite with plies in different directions the first damage (first ply failure) often corresponds to the transverse tensile strength of the plies with unidirectional reinforcement [1].

The aim of the present study is to characterize the damage resistance of composites with different fibres in order to provide data for material selection. During this work some particular differences between the PAN and pitch based fibre composites, and a correlation between transverse tensile properties and delamination resistance were noted and these are described in the present letter. Some previous studies have related transverse mechanical properties to delamination resistance [2,3] but not for such high modulus fibres.

1. Experimental procedure

1.1. Materials and preparation

Unidirectional composite materials were manufactured from 14 layers of 300 g/m² areal weight epoxy resin prepreg, from batches which had been used in mast construction. The series were cured in an oven at 110°C for 4 hours under vacuum. Four fibres were studied: M40J, M46J, and M55J, PAN fibres from Toray, and K637-12 a pitch based fibre from Mitsubishi. Their axial moduli values are 377, 436, 540 and 640 GPa respectively. Mean composite fibre volume fraction was measured to be 52%.

1.2. Test methods

The mode I tests were performed on DCB specimens according to the ISO standard [4]. Specimens were 20 mm wide, 4 mm thick, and 160 mm long, with a 40 mm long starter film. Tests were performed on an *Instron 4302* test frame with a 500 N load cell, at a crosshead loading rate of 2mm/min. Load (P), crosshead displacement (δ) and crack length (a) were recorded. At least three specimens were tested for each material. The data analysis applied is that proposed by Berry [5] :

$$C = Ka^n \quad G_{Ic} = \frac{nP\delta}{2ba}$$

Where C is the compliance δ/P, K and n are empirical constants and b is the specimen width.

Two values can be determined, at initiation from the starter film, defined using the onset of non-linearity, and during propagation. In the standard test method only the initiation values are used, as the propagation values have been shown to depend on specimen geometry. However, in the present work propagation values are also presented, as the same specimen geometry is used throughout. For mast structures the propagation behaviour measured in tests is relevant as masts are largely based on unidirectional reinforcement.

Transverse tensile tests on composites were performed on at least five parallel sided specimens of width 25 mm of each material following ISO 527. Specimen edges were polished down to 1000 grade paper. Elongation was measured using an extensometer and loading rate was 1 mm/minute.
In order to observe the damage mechanisms additional specimens were also tested on a small *Deben* tensile test machine inside a *Jeol 6460LV*. scanning electron microscope. Samples were 4x4x35 mm^3, loading rate was 1 mm/minute.

2. Results and discussion

2.1. Mode 1 delamination resistance

Crack propagation was stable in all specimens. Initiation and propagation values are given in Table 1. It may be noted that the results for each composite are quite reproducible, the coefficients of variation are below 15% in all cases and often below 5%, even though fabrication methods are not those of the aerospace industry.

			Mode I delamination		Transverse Tensile		
Fibre	Modulus (GPa)	V_F (%)	G_{Ic} (kJ/m²)	G_{Ip} (kJ/m²)	E_T (GPa)	σ (MPa)	Failure strain, (%)
M40J	377	52	0.25	0.51 (0.04)	6.37 (0.32)	30.2 (0.8)	0.45 (0.02)
M46J	436	53	0.21	0.37 (0.01)	6.15 (0.40)	24.3 (1.0)	0.40 (0.01)
M55J	540	54	0.11	0.18 (0.02)	6.68 (0.31)	14.5 (1.3)	0.21 (0.03)
Pitch K637	640	50	0.16	0.16 (0.01)	5.43 (0.33)	25.0 (0.7)	0.46 (0.04)

Table 1 : Results from mode 1 delamination and transverse tensile tests, mean values and standard deviations.

There is a significant drop in propagation values for both series as fibre modulus increases, with the highest modulus fibre composites showing very flat R-curves. The results for the M55J material are similar to those published by Allix et al. [6], who also noted a very flat R-curve compared to lower modulus fibre composites.
Fibres bridging the two crack faces are one of the causes of an increasing R-curve. This mechanism has been discussed by several authors [7-9] and it has been noted that the effect of fibre bridging on G_{Ip} values can depend on the specimen arm stiffness. This was attributed to the effect of larger crack tip opening displacement on breaking fibres in the bridging zone in more flexible specimens. In the present case the opposite effect is noted, the stiffer samples show a smaller bridged zone and lower toughness. The low failure strain of the high modulus fibres in the bridging zone may be responsible for this.

2.2. Transverse tensile properties

Table 1 shows the results from the transverse tensile tests. There is a significant influence of the fibre type on strength and a smaller dependence for stiffness. The heterogeneity of composite materials is the main cause of failure of UD plies loaded in transverse tension. Stress and strain concentrations are induced in the matrix between the fibres. De Kok has shown that a global ply strain of 1% can result in local strains greater than 5% [10]. An irregular fibre distribution accentuates this effect. As a result of these strain concentrations the transverse failure strain for UD plies is considerably lower than the longitudinal failure strain.

Many previous studies have examined the parameters which influence transverse tensile strength including matrix failure strain, interface quality, fibre distribution and diameter and fabrication parameters [11-15].

Study of the propagation of cracks by tensile loading in the SEM reveals differences in the failure mechanisms. For the composites reinforced with PAN fibres the cracks propagate in the matrix and at the fibre/matrix interface (Figure 1).

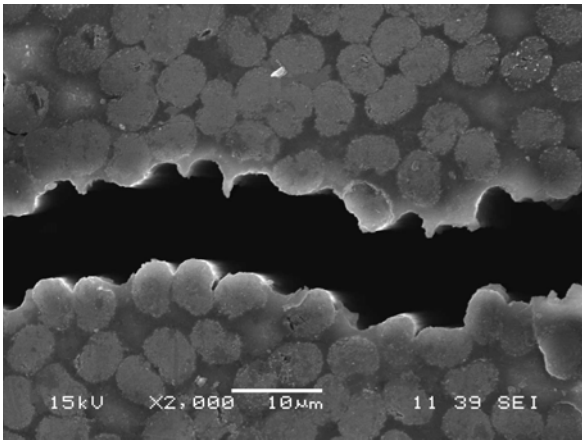

Figure 1: Transverse tensile failure in scanning electron microscope (SEM) of unidirectional M55J/epoxy composite

In the pitch fibre composites however, cracks are also noted within the fibres, Figure 2, indicating a low transverse strength of the fibres. Some fibre-matrix debonding is also noted suggesting that both mechanisms can be invoked at similar applied stresses.

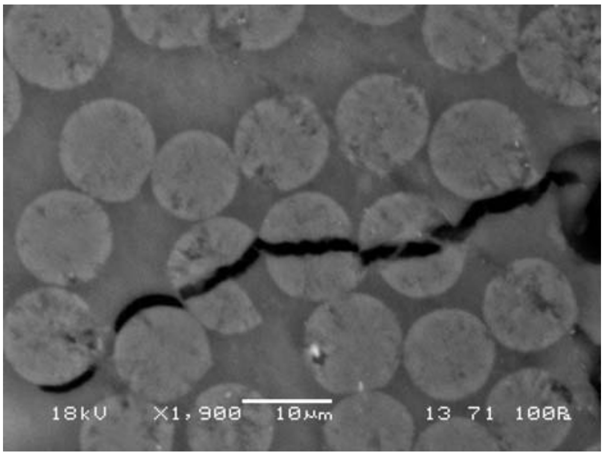

Figure 2:Transverse tensile failure in SEM of unidirectional Pitch fibre reinforced composite

These figures also indicate differences in the fibre morphology and surface areas. The morphology of these fibres varies considerably. These differences will affect local stress concentrations when transverse loading is applied. The surface areas of PAN fibres were measured and vary from 21.8 to 25.2 μm². Fibres are not perfectly circular but these areas correspond to equivalent diameters of around 5.7 μm. The pitch fibres are larger, with mean diameters around 10.5 μm. It has been noted elsewhere that the modulus of carbon fibres increases as section decreases [16].

These tests enable transverse modulus values to be estimated for the fibres. Such data are rare and difficult to obtain directly. Some values have been obtained by single fibre compression [17], by nano-indentation [18] and from angular characteristics of ultrasonic scattering [19]. An alternative is to determine them indirectly, using data from tests on unidirectional specimens[20]. This approach was retained here. Two models were applied, proposed by Halpin Tsai [21] and Uemura [22]. They are both approximate, not accounting for true fibre section, non-linear behaviour, porosity, nor residual stresses.

The transverse tensile moduli obtained using these two models are 11.5 GPa and 15 GPa for the PAN fibre composites, and 9 GPa and 11.6 GPa for those based on pitch. Miyagawa [18] showed that for T300 carbon fibres, the Halpin-Tsai provided results closest to those measured using nano-indentation and micro-Raman spectroscopy.

2.3. Correlation between interlaminar fracture energy and transverse tensile properties

Figure 3 shows the correlation between G_{Ic} values corresponding to the crack propagation plateau and transverse tensile failure strain.

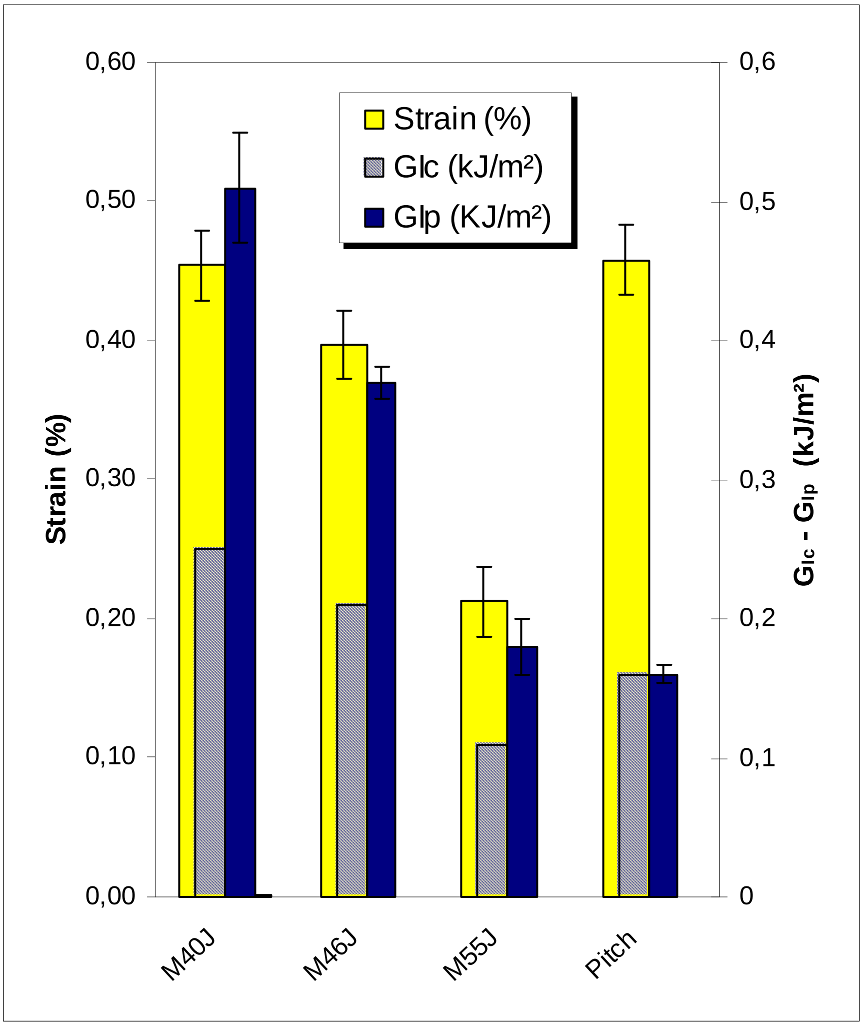

Figure 3 : Correlation between mode I interlaminar fracture energies G_{Ic} and G_{Ip} and transverse failure strain.

There is a clear trend of both strain to failure and mode I fracture energy (G_{Ic} and G_{Ip}) decreasing as PAN fibre modulus is increased from 377 GPa (M40J fibres) to 540 GPa (M55J fibres), but the pitch fibres show a different behaviour. Their G_{Ip} values are similar to those of the M55 fibre with similar modulus, but their transverse tensile failure strains are significantly higher. The latter may be related to the larger fibre diameter but more work is required to understand this difference. Differences in matrix adhesion to the different fibres may also play a role.

Conclusions

Racing yacht construction is largely based on composites reinforced with high modulus carbon fibres. The optimisation of these structures is resulting in the use of stiffer fibres but results in reduction in the critical through the thickness properties. The nature of the fibres has a strong influence on both interlaminar fracture energy and transverse tensile properties. G_{Ic} and G_{Ip} values decrease as fibre modulus increases. Transverse tensile failure strain also decreases for PAN fibres but not for pitch. A mechanism of crack propagation within the fibre is noted for the pitch based fibre composites. From transverse tensile test data the transverse fibre modulus has been estimated as 11.5 GPa for the PAN fibres and 9 GPa for those based on pitch. These results suggest that the specific nature of pitch based composites requires more detailed study if their attractive longitudinal properties are to be used effectively.

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