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Factors controlling the strength of carbon fibres in tension

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ABSTRACT

We have investigated the fracture mechanisms of different types of carbon fibres, in terms of skin-core differences in single fibres, flaw size and fracture toughness. The fibre strength distribution was measured precisely using the fragmentation test for single-fibre composites. The failure probability for intermediate/high modulus types fibres was found to be constant with fibre strength in the range 2–4 GPa, but in contrast the strength scatter for high modulus type fibres was reduced. The fracture toughness of the carbon fibres, determined by introducing notches with lengths in range 60–200 nm, was found to be about 1.1 MPa $m^{1/2}$. The average flaw size of the carbon fibres increased with increasing fibre modulus, suggesting that the crack growth of surface flaws on the tens-of-nm scale occurred. This appears to be the main reason for the reduction in tensile strength during the carbonisation treatment.

1. Introduction

Polyacrylonitrile (PAN)-based carbon fibres play an important role in producing light-weight structural materials. In order to optimise and improve fibre properties, it is important to evaluate the tensile strength of the fibres accurately and to understand the factors that control this strength. A number of studies have already been undertaken on the characterisation of fibre strength distributions using different types of Weibull distribution; the two-parameter Weibull distribution [1–4], the bimodal Weibull distribution [5] and the Weibull of Weibull model [6]. The fibre strength can also be estimated using the Griffith theory in terms of fracture toughness [7,8] and flaw size [1,9]. In order to gain a full understanding of fibre fracture behaviour it is also necessary to know the dependence of the tensile strength of the fibres upon nanostructure. This is a result of the potential complex relationship between the strength, fracture toughness, flaw size distribution and structure for different types of fibres.

This study deals with the changes in tensile fracture behaviour of carbon fibres during the carbonisation treatment. In this present work, the tensile strengths were measured for a series of carbon fibres from the same precursors, treated at different maximum carbonisation temperatures to produce fibres with different Young's modulus. The effect of the internal nanostructure upon fibre strength was investigated to give an insight into methods of improving this strength. Although single-fibre tensile tests (SFT) are widely used for the determination of fibre strength, for short gauge lengths there is a problem that the average strength is underestimated because the actual gauge length is longer than the nominal one due to end effects as a result of slippage of the fibres and adhesive in the gripping region. Accordingly, we have utilised the single-fibre-composite test (SFC) to determine fibre strength. The relationship between fragment length and tensile elongation of the fibre at failure has been analysed previously for the SFC [10]. In this present study we have used Raman spectroscopy to determine the stress in the fibres. Additionally, a direct measurement technique of the fracture toughness has been developed by introducing a notch into a fibre through the use of a focused ion beam [11,12]. By considering the fracture toughness and the flaw size distribution, we have been able to determine the key structural factors that control the fibre tensile strength.

2. Experimental

2.1. Materials

In order to follow the dependence of the mechanical properties of the carbon fibres upon nanostructure, we prepared six different types of PAN-based carbon fibres, with Young's moduli in the range 50–500 GPa (Table 1). These PAN-based carbon fibres were all supplied by TORAY Industries, Inc. The crystallite thickness, L_c of carbon fibres from the same precursors, treated at different maximum carbonisation temperatures were thickened by the carbonisation treatment (Table 1). With the exception of the 700 °C

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Table 1
Summary of the structure and properties of the PAN-based carbon fibres. The fibres, except for M30S, were all experimental fibres. x °C indicates that the fibres were carbonised at a maximum temperature of x °C. L_c and π_{002} are crystallite thickness and orientation parameter determined by X-ray diffraction [14], respectively.

Fibres	Tensile modulus (GPa)	Density (g/cm^3)	Diameter (μm)	L_c (nm)	π_{002}
700 °C	55	1.57	6.6	1.3	0.815
1000 °C	240	1.78	5.8	1.4	0.822
T800G	295	1.80	5.5	2.0	0.821
M30S	295	1.73	5.6	2.4	0.845
M40S	380	1.80	5.4	3.7	0.883
M50S	480	1.90	5.3	5.4	0.918

fibre, the samples were the same as those reported in a previous study [13]. The properties of the 700 °C bulk fibre and its components; the crystallites and amorphous carbon are shown in Table 2 along with similar information for the other fibres. The structure of the bulk fibres was characterised using synchrotron radiation and the properties of the crystallites and amorphous carbon were calculated using the Mori–Tanaka method as explained in our previous study [13]. e_1^f is the axial elastic constant of the bulk carbon fibres and g^f is the axial shear modulus of the bulk carbon fibres. The crystallinity indicates the total volume fraction of the crystallites. $\langle\sigma_{11}\rangle$ and $\langle\gamma_{11}\rangle$ are the stress and the total strain of the components; the crystallites, representing I and amorphous carbon, representing M for the application of unit stress (1 GPa), respectively [13]. In this present analysis, we have assumed that the crystallite modulus of the 700 °C fibre is 605 GPa based upon its Raman G band initial shift rate and Eqs. (11) and (12) in the previous paper [13]. The Young's moduli of the amorphous carbon within the fibres are 40–220 GPa, which is reasonable by considering that the Young's modulus of ultra-thin amorphous carbon films were mechanically characterised to be 178–211 GPa [23].

2.2. Methods

2.2.1. Single fibre composite fragmentation test

The specimens were prepared using a 2 mm thick poly(methyl methacrylate) (PMMA) beam and single fibres were embedded on the surface of each beam, all parallel to the axis of applied load. A PMMA was dissolved in chloroform, and the solution was applied over the single fibres on the beam. A thin layer (~50 μm) of PMMA was then coated on top of the beam after complete removal of chloroform. The PMMA beam was deformed in 4-point bending and the strain monitored using a strain gauge attached to the beam top surface. The strain was increased stepwise by 0.2%, and for each straining step, numbers of fibre breaks was evaluated over the central 10 mm of the fibres. A total of 15 single fibre composites were tested for each of the carbon fibres. The fibre breaks for each single fibre are ~10, and the number of total breaks for the Weibull plots from the fragmentation tests is ~150 for each type fibres, which is enough number for the evaluation of the Weibull moduli. The applied stress was calculated from the relationship between the Raman G band and applied strain, based on the linear behaviour between a Raman G band and applied stress [15,16].

Well-defined Raman spectra could be obtained from the carbon fibres through the PMMA coating using a low-power He–Ne laser (1.96 eV and <1 mW at the sample) in a Renishaw 2000 spectrometer.

2.2.2. Single-fibre tensile test

The tensile strength of T800G fibres was also investigated using a single-fibre tensile test. Single fibres were mounted on paper cards and the paper cards placed between the grips of a universal tensile testing machine (Tensilon RTF-1210; A&D Ltd., Japan). The edges of the cards were cut and the load-elongation curves were recorded. Gauge lengths of 5, 10, 25 and 50 mm were used with an initial strain rate of 0.02 min^{-1}. A total of 50 specimens were tested for each gauge length. The fibre diameters and the flaw sizes on the fracture surfaces were measured using a scanning electron microscope (SEM S-4800; Hitachi, Japan).

2.2.3. Fracture toughness measurements

A notch was introduced on one side surface of a carbon fibre for a measurement of the fracture toughness, using a FIB system (Strata 400S; FEI). A Ga^+ ion beam was produced from an ion source and accelerated using a 30 kV accelerating voltage and a probe current of 9.7 pA. Fig. 1 shows a SEM picture of the notched fibre before and after fracture. The radius of curvature of the notch tip was controlled approximately 40 nm (30–50 nm). The notched carbon fibres were tested in tension in water, following a tensile test method [1] for carbon single-fibre that avoids secondary damage of the fibre. The tensile test for the notched monofilament fibres was carried out at a crosshead speed of 0.2 mm/s at room temperature in a universal tensile testing machine (Tensilon RTF-1210; A&D Ltd., Japan). After the tensile tests, the fracture surfaces were examined by SEM (S-4800; Hitachi, Japan) to determine the fracture initiation points and measure the notch depths.

2.2.4. The stress–strain curves for the carbon fibres

In order to obtain accurate Young's modulus values for the fibres, the non-linearity of the stress–strain (S–S) curves for the carbon fibres must be taken into account. A number of studies have already been undertaken on the S–S curves of carbon fibres; estimated (a) from the S–S curves of unidirectional carbon fibre composites [17], (b) from sonic modulus of unidirectional carbon fibre composites [18], (c) from the S–S curves of SFT [19] and Raman

Table 2
Calculated properties of the bulk carbon fibres and their components; the crystallites and amorphous material for the application of unit stress. e_1^f is the axial elastic constant of the bulk carbon fibres and g^f is the axial shear modulus of the bulk carbon fibres. $\langle\sigma_{11}\rangle$ is the stress and $\langle\gamma_{11}\rangle$ is the total strain in the direction of the fibre axis [13].

Fibres	e_1^f (GPa)	g^f (GPa)	Amorphous modulus (GPa)	Crystallinity	$\langle\sigma_{11}\rangle_I$ (GPa)	$\langle\sigma_{11}\rangle_M$ (GPa)	$\langle\gamma_{11}\rangle_I$	$\langle\gamma_{11}\rangle_M$
700 °C	122	5.4	41	0.575	1.11	0.85	0.0015	0.0021
1000 °C	333	24.9	159	0.458	1.22	0.82	0.0031	0.0052
T800G	434	27.1	200	0.508	1.18	0.81	0.0028	0.0041
M30S	454	22.0	186	0.551	1.19	0.76	0.0029	0.0041
M40S	579	18.4	211	0.633	1.18	0.68	0.0025	0.0033
M50S	739	11.2	212	0.770	1.12	0.60	0.0021	0.0029