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A novel method for measuring direct compressive properties of carbon fibres using a micro-mechanical compression tester

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A novel method has been developed for measuring direct compressive properties such as strength and elasticity of a series of mesophase-pitch-based and PAN-based carbon fibres about 10 μm in diameter by uniaxial and transverse compression tests using a micro-mechanical tester. The fibres were shaped into cylindrical specimens, with their size ratio of length to diameter kept at about 2 to 3, by separating them from a "thin film" made by polishing the cut faces of a strand of carbon fibres with epoxy resin as a matrix. Individual cylindrical specimens were stood up or laid down on a glass plate without any fixer for the measurements of axial and transverse compression properties of fibres, respectively. The fibres exhibited non-linear elasticity, with the compressive modulus decreasing with compressive deformation. The direct axial compressive strengths of pitch-based carbon fibres were found to be marginally lower than the indirect ones, whereas there was no significant difference between the two strength values for PAN-based fibres. The pitch-based fibres exhibited smaller average values of axial compressive strength than the PAN-based fibres. The transverse compressive strength, which decreases with an increase in elasticity of carbon fibres, exhibited a considerably lower average value than that of the axial compressive strength. Further, the axial compressive strength was found to be smaller than the direct tensile strength for the fibres.

1. Introduction

Carbon fibres are mainly used as reinforcements in composite materials such as carbon fibre reinforced plastics (CFRP), carbon–carbon (C/C) composite, carbon fibre reinforced materials (CFRM) and carbon fibre reinforced cement (CFRC) due to the benefits of their high (specific) strength and (specific) modulus. Such composites have been utilized in the production industries not only of aerospace and/or aircraft but also of advanced functional materials and sports/leisure goods. In composites, the function of carbon fibres is to take a portion of the stress acting on the composite, and the resin plays a big role in supporting the fibres and protecting them from the environment.

During the past few years, significant efforts have been made in the development of the mechanical properties of carbon fibres. This can lead to the appearance of carbon fibres with high performance which have a tensile strength of the order of 7 GPa and elastic moduli of the order of hundreds of gigapascals [1, 2]. It is somewhat less well known that these fibres, with few exceptions, have tensile strengths significantly larger than their compressive strengths, as estimated from the data on composites [3]. It is also an observed fact that carbon fibres in composites often fracture by some compressive action combined with weak bending forces [4]. These facts suggest the importance of characterizing the direct compressive properties of a carbon fibre itself as well as its tensile ones.

Some difficulties in investigating the failure mechanisms in carbon fibres remain for improving the compressive strengths. One of these difficulties is the lack of an accurate and reproducible method to obtain the compressive stress–strain curve for an individual fibre. The small diameter of the single fibre, approximately 10 μm, is the major obstacle. Sample preparation is time-consuming, the means for holding the fibre are limited, and proper alignment of the fibre to avoid off-axis loading which induces buckling is difficult. Also, the apparatus to perform the test must be capable of fine displacement control with fine force and displacement measurements.

There are several methods available for measuring the compressive strengths of individual fibres. Some of these are the bending beam test [5], the elastic loop method [6], the recoil method [7] and the micro-

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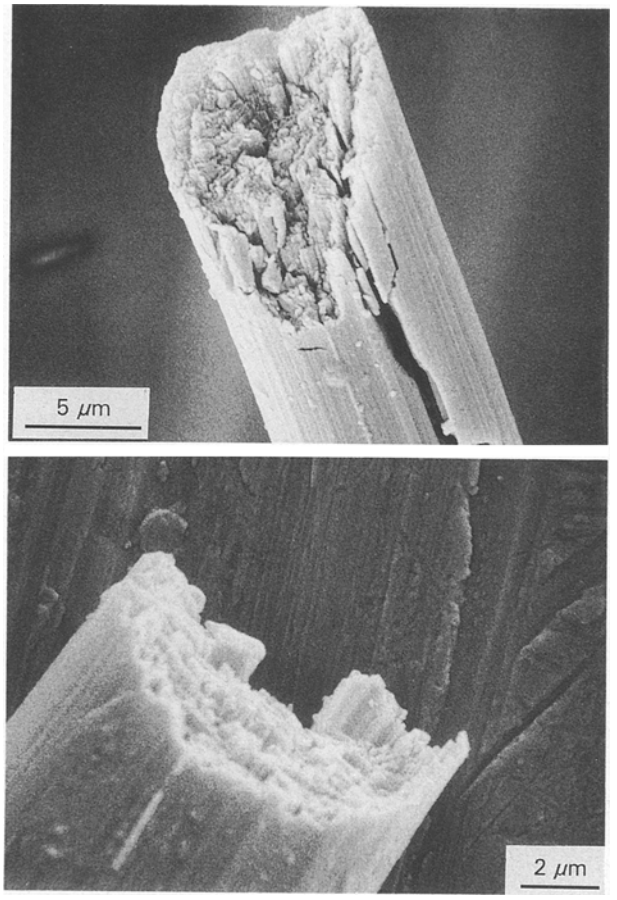

Figure 1 Broken faces of a carbon fibre (sample C).

tensile method [8]. Though each is a unique method, they can provide different information on the properties of the sample for various reasons. Macturk *et al.* [8] have recently developed a novel method for measuring the compressive stress–strain curve for individual mesophase-pitch-based graphite fibres using a micro-compression apparatus. It is mounted on an optical microscope stage, enabling one to examine the presence and orientation of any cracks in the fibre. In this method, one end of the fibre sample is adhered by an epoxy resin to a metal surface attached to a positioning element and the other end is also adhered by an epoxy resin to a fixed metal surface. However, a proper alignment of the fibre is necessary to avoid buckling.

The main purpose of this work is to establish another type of measurement method for the direct compressive properties of individual carbon fibres. This method is unique with respect to the use of a cylindrical specimen having a ratio of length to diameter for individual specimens of about 2 to 3.

2. Experimental procedure

Three types of pitch-based carbon fibre (hereafter called samples A, B, C in this paper) and one PAN-based carbon fibre (hereafter called PAN) made by Nippon Steel Corporation and Toray, respectively, were examined in this work. Their diameter, density, tensile elasticity, direct tensile strength and indirect compressive strength, as estimated from the strength of composites, are shown in Table I.

TABLE I Properties of the carbon fibres used in this work

Property	Pitch-based			PAN-based
	A	B	C	
Diameter, d (μm)	10.0	9.75	9.48	6.91
Density, ρ (g cm^{-3})	1.95	2.03	2.15	1.78
Tensile elasticity, Y (GPa)	197	392	599	235
Direct tensile strength, S_t (GPa)	3.16	3.35	3.20	3.72
Indirect compressive strength, S'_c (GPa)	1.62	1.03	0.82	2.45

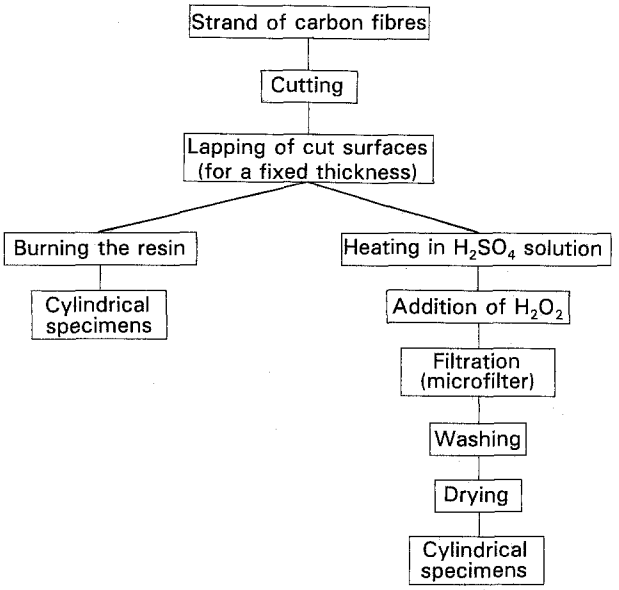

Figure 2 Preparation processes for cylindrical specimens of carbon fibre.

Fig. 1 shows typical broken end-faces of sample C due to a simple bending force. The broken end-face appears to be rather irregular, so some smoothing procedure such as polishing might be needed. Fig. 2 shows two preparation processes developed for cylindrical specimens with a ratio of length to diameter of about 2–3, starting with strands of fibres in epoxy resin. First, the strands were cut to about 5 mm length and immersed in liquid acrylic resin in a small cylindrical vial. After hardening, the cut faces of the strand were polished to form a "thin film", of which the thickness was adjusted to about 2 to 3 times the diameter of a carbon fibre. Secondly, the "thin film" was put into H_2SO_4 solution with the addition of a small amount of H_2O_2 solution at 423 K in order to dissolve the epoxy and acrylic resins. After cooling the solution, a large number of cylindrical specimens of carbon fibre were collected and washed with distilled water using a micro-filter of 1 μm pore size.

The alternative method for removing the cylindrical specimens from the "thin film" was conducted by firing epoxy resin out of the "thin film", as indicated in Fig. 2. However, the surfaces of cylindrical specimens in this firing process seemed to be somewhat changed by heating and contaminated with ashes. For this

reason, the dissolution process was adopted to remove the cylindrical specimens of carbon fibres from the "thin film" in this experimental work. Fig. 3 shows SEM photographs of the cylindrical specimens on the micro-filter together with a single specimen of sample A and a PAN sample. For the measurement of direct compressive properties in the axial direction of carbon fibres, the specimens were stood individually some distance apart on a glass plate. For the measurement of direct compressive properties in the transverse direction of carbon fibres, specimens were laid down at some interval apart on a glass plate.

The glass plate containing the cylindrical specimens was set under the loading piece of a micro-mechanical compression tester as schematically shown in Fig. 4. The tester, which is a modified micro-hardness tester (Shimadzu DUH-100), is controlled by applying an electric current in a force coil with an automatically controlled measuring unit, which is also connected with a deformation detector. The test begins by applying a current to the force coil, causing the loading level to adjust the loading piece down to the specimen on the glass plate. As soon as the loading piece touches the top of specimen, the load and the deformation of the specimen are detected automatically and the data collected by a computer through an A–D converter and processed. In the display of load and deformation of a specimen, the deformations of the glass plate and loading piece were first eliminated.

Fig. 5 shows the flow chart of the measurements of mechanical properties of carbon fibres using the micro-mechanical compression tester. Fig. 6 shows optical photographs of a specimen under compression on the glass plate. The compression tests were carried out under atmospheric conditions at 2.55 $\times 10^{-3}$ N s^{-1} loading rate. The direct compressive

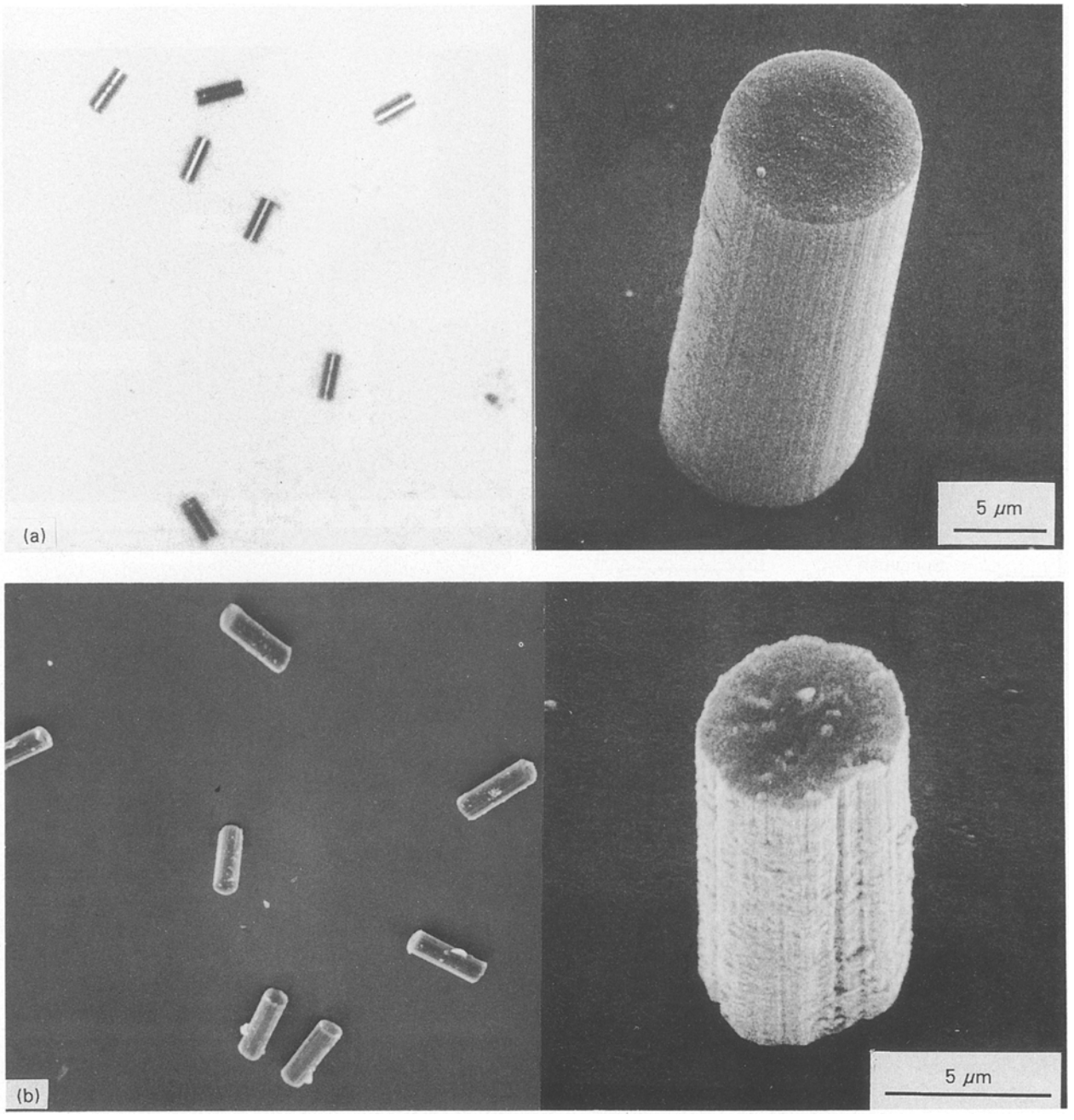

Figure 3 Cylindrical specimen(s) of carbon fibres; (a) pitch-based (sample A), (b) PAN-based.

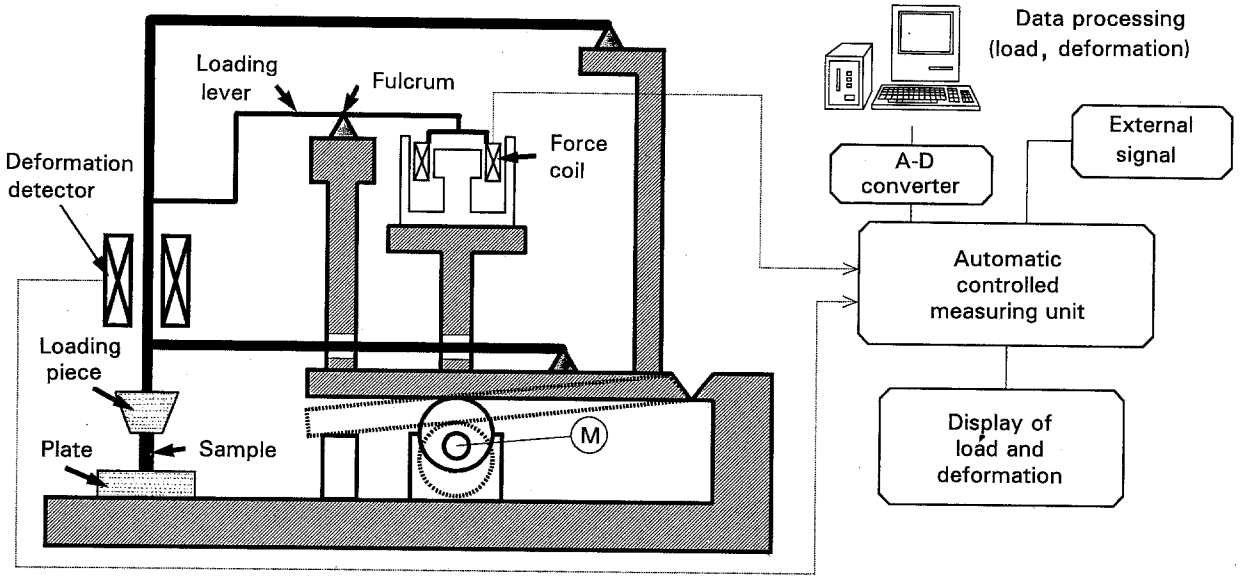

Figure 4 Schematic diagram of loading mechanism in the micro-mechanical compression tester.

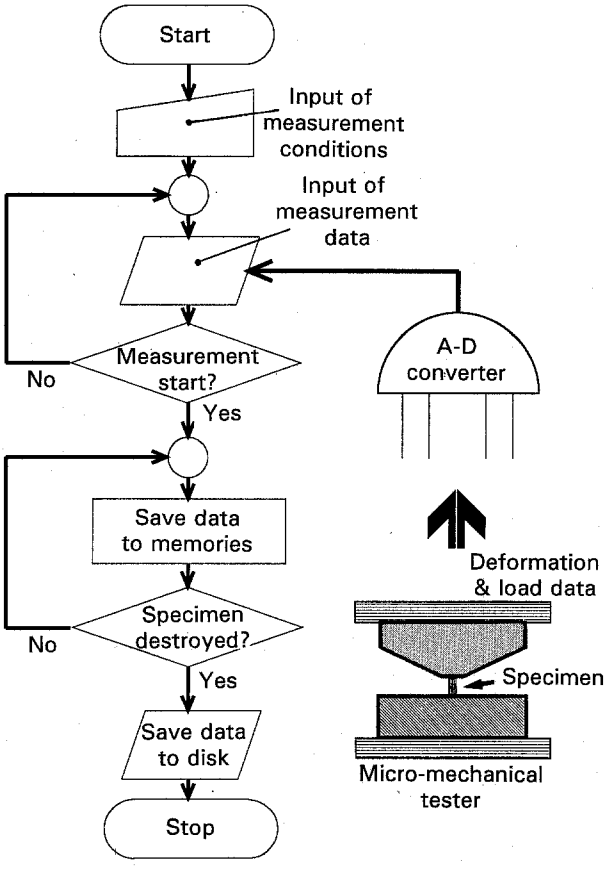

Figure 5 Flow chart of the measurements for mechanical properties of carbon fibres using the micro-mechanical compression tester.

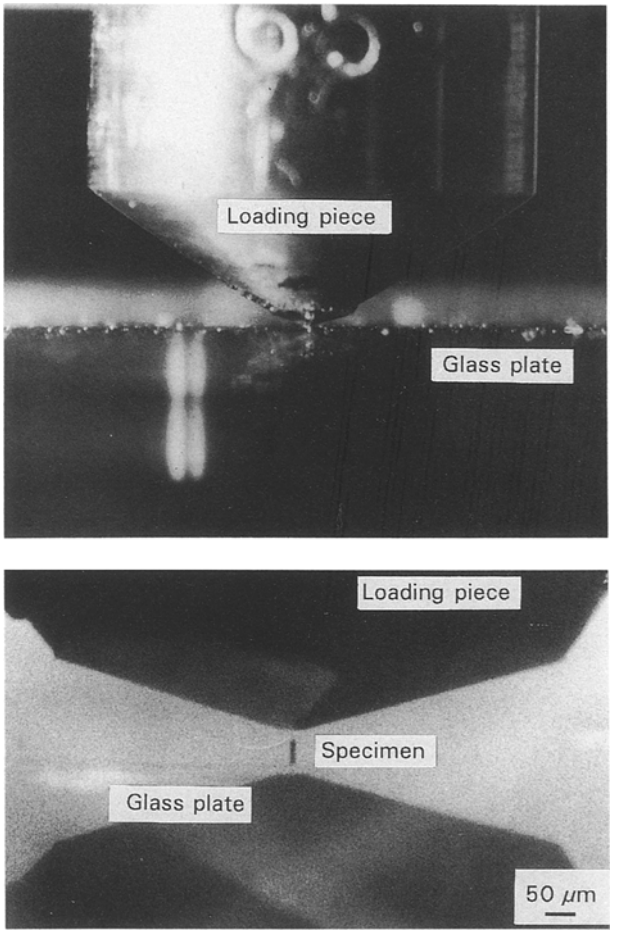

Figure 6 Compression state of a specimen on a glass plate.

strengths were defined by the following equations:

(i) Axial compressive strength, S_c (Pa):

$$S_c = \frac{P}{\pi d^2/4} \quad (1)$$

(ii) Transverse compressive strength [9], S_d (Pa):

$$S_d = \frac{2P}{\pi dl} \quad (2)$$

where d (m) is the diameter of a cylindrical specimen, l (m) its length and P (N) the fracture (maximum) load.

3. Results and discussion

3.1. Load and deformation characteristics

Fig. 7 shows typical load–deformation curves of individual specimens of sample A compressed in the axial and transverse directions. In the axial compression

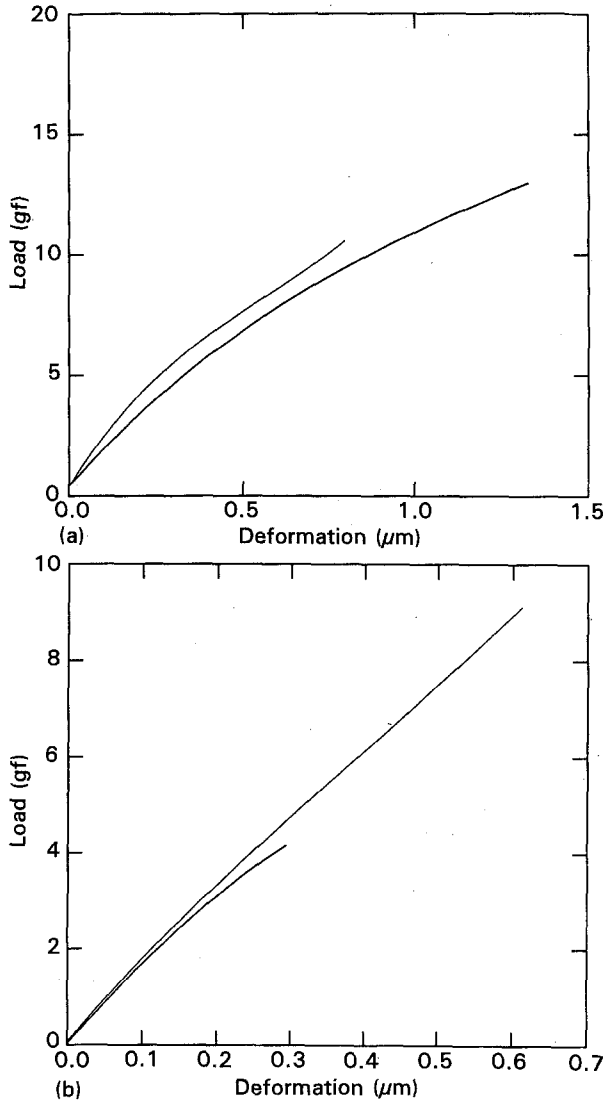

Figure 7 Load versus deformation curves for sample A: (a) axial compression, (b) transverse compression.

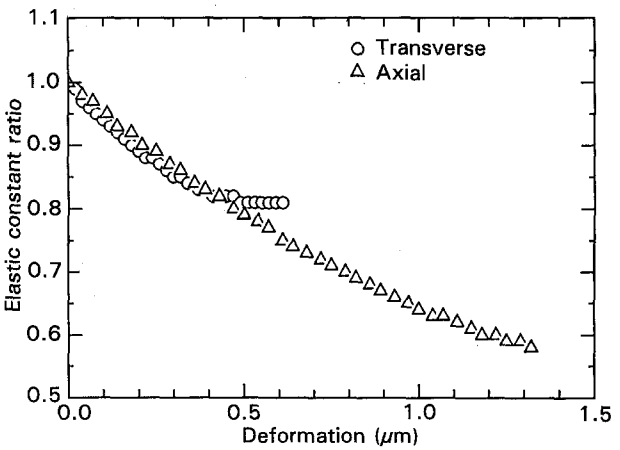

Figure 8 Elastic constant ratio of sample A in (△) axial and (○) transverse directions as a function of compressive deformation.

TABLE II Experimental results on direct axial and transverse compressive strengths of carbon fibres

Property	Pitch-based			PAN-based
	A	B	C	
Tensile elasticity, Y_t (GPa)	197	392	599	235
Axial compressive strength, S_c (GPa)	1.35	0.74	0.44	2.55
Standard deviation σ (GPa)	0.48	0.27	0.21	1.03
σ/S_c	0.36	0.36	0.48	0.40
$\varepsilon = \delta_c/l(\%)$	5	3	2	3
N	42	39	54	16
Transverse compressive strength, S_d (MPa)	160	94.3	54.9	556
Standard deviation σ (MPa)	51.3	34.8	–	171
σ/S_d	0.32	0.37	–	0.31
$\varepsilon = \delta_d/d(\%)$	8	10	5	26
N	30	6	1	20
S_c/S'_c	0.83	0.72	0.54	1.04
S_c/S_t	0.43	0.22	0.14	0.68
S_c/S_d	8.44	7.85	8.01	4.59

measurement, the load applied to the specimen is almost linearly proportional to the deformation at the beginning of loading. Subsequently, the gradient of the curve decreases gradually with an increase in applied load. The relation between load and deformation in a transverse compression test seems to be almost similar to that in an axial test. Similar compressive behaviour can be observed in the axial direction for the PAN-based carbon fibre.

Fig. 8 shows the elastic constant ratios of sample A in the axial and transverse directions as a function of compressive deformation. In this work, the elastic constant ratio is defined by the gradient of the stress–strain curve of a carbon fibre. Both elastic constant ratios decrease gradually with an increase in deformation at almost the same rate in the initial stage. Subsequently, there is a significant difference between the final ratio of elastic constants. This sort of feature in the axial and transverse compressive properties may be due to the anisotropic nature of the fibres, which indicates a difference of structure in the axial and transverse directions in pitch-based and PAN-based carbon fibres.

3.2. Direct compressive strengths

The experimental results regarding the axial and transverse compressive strengths on the three kinds of pitch-based carbon fibres together with PAN-based carbon fibres used in this study are summarized in Table II. The numbers of data are limited: from 16 to 54 and 1 to 32 for the axial and transverse compression tests, respectively.

Generally, the axial compressive strength of the pitch-based fibres is considerably smaller than that of the PAN-based fibres, although the values of direct tensile strength are relatively close. The axial compressive strength ratio of pitch-based to PAN-based fibres is approximately 53% for sample A, 29% for sample B and 17% for sample C. Thus, the higher the tensile elasticity is, the lower the axial compressive strength for pitch-based fibres becomes. The coefficient of variation in the axial compressive strength for pitch-based carbon fibres ranges from 36 to 48%. The values of final compressive elasticity for pitch-based carbon fibres, as defined by the gradient of the

stress–strain curve at fracture of a specimen, are inversely proportional to the values of tensile elasticity, (Table I). The values of final compressive elasticity correspond to about 4 to 15% of the tensile one, whilst the former value for the PAN-based carbon fibre stays at about 40% of the latter value.

Regarding the data on the transverse compressive strength of the carbon fibres, rather few specimens were measured, especially of samples B and C in pitch-based carbon fibres, because of their extremely low fracture load. The trend of transverse compressive strength is somewhat similar to that of the axial compressive strength, irrespective of the kind of carbon fibre. The strength ratios of pitch-based to PAN-based fibres are measured as approximately 0.29 for sample A, 0.17 for sample B and 0.10 for sample C. The coefficient of variation in all kinds of carbon fibres is measured in the range from 31 to 37%.

The values of axial compressive strength are much larger than those of the transverse one, by about 8 and 5 times for the pitch-based and PAN-based carbon fibres, respectively. Further, the values of axial compressive strength are considerably smaller by about 1/2.3 to 1/7.3 times those of tensile strength, S_t, for the pitch-based carbon fibres, whilst there is not much difference between the strengths for PAN-based fibres. In addition, the compressive strength ratio between the direct and indirect methods, (S_c/S'_c), ranges from 0.54 to 0.83 for the pitch-based carbon fibres, while the difference between the two strengths for the PAN-based carbon fibre is not significant within the scope of this experiment. This may be due to the difference between anisotropic and isotropic natures, derived from the processes of graphitization and/or carbonization of raw materials in the two kinds of carbon fibre.

4. Conclusions

A novel method for measuring direct compressive properties for mesophase-pitch-based and PAN-based carbon fibres with about 10 μm diameter was developed using a micro-mechanical compression tester. The cylindrical specimens used in this work were prepared by polishing the cut faces of fibres, keeping the size ratio of length to diameter at about 2 to 3. The axial and transverse compressive properties of the carbon fibre specimens were measured under normal atmospheric conditions. The following experimental results were obtained:

1. The fibres exhibited non-linear elasticity, with the compressive modulus decreasing with compressive deformation.

2. The direct axial compressive strength is lower than the indirect one by about 1/1.9 to 1/1.2 for the pitch-based carbon fibres, while the difference between the two strengths is not significant for PAN-based fibres.

3. The axial compressive strength for pitch-based fibres is considerably smaller by about 1/1.9 to 1/5.8 than that for PAN-based fibres, irrespective of their having nearly the same values of direct tensile strength. The higher the tensile elasticity is, the lower the axial compressive strength for pitch-based fibres becomes.

4. The axial compressive strength exhibits a much higher average value than the transverse compressive strength by a factor of about 5 and 8 for pitch-based and PAN-based carbon fibres, respectively.

5. There is a significant difference between the final gradients of the load versus deformation curves for the axial and transverse compression tests.

6. The axial compressive strength shows a significantly smaller average value than the tensile strength by about 1/2.3 to 1/7.3 for the pitch-based fibres and about 1/1.5 for the PAN-based ones.

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