Compressive properties of single-filament carbon fibres

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Compressive properties of mesophase pitch-based carbon fibres (NT-20, NT-40 and NT-60) were measured using the tensile recoil test and the elastica loop test. The NT-40 fibre with a 400 GPa tensile modulus showed a smaller loop compressive yield strain and a larger recoil compressive strength compared to these values obtained from the longitudinal compression test on its unidirectional composites. Further, the recoil compressive strength of this fibre was higher than that of PAN-based carbon fibre with a corresponding modulus. Under the ideal conditions in the tensile recoil test, the strain energy was conserved before and after recoil, and the initial tensile stress and the recoil compressive stress do not coincide when **fibre** stress-strain behaviour is non-linear, and the non-linearity in compression and in tension is different. The difference between the composite compressive strength and the recoil compressive strength of NT-40 was quantitatively explained by taking account of the fibre compressive stress-strain non-linear relation. The difference between the loop compressive yield strain and the composite compressive strain to failure was also explained by this non-linearity.

1. Introduction

Carbon fibre reinforced composites have been extensively used in many fields because of their high specific modulus and strength, and their use continues to increase. Recently, mesophase pitch-based carbon fibres are finding applications due to their higher specific moduli and high electrical conductivity. Although the tensile strength of pitch-based carbon fibres have recently been improved significantly $\lceil 1-4 \rceil$, they need more improvement in the composite compressive strength. Despite significant research activity, the single-fibre compressive properties are not fully investigated, though the longitudinal compressive property dictates the use of composites in many structural applications. This is mainly due to difficulties in measuring single-fibre compressive mechanical behaviour. Various issues related to the compression behaviour of high-performance fibres were discussed in a recent paper [5].

Several methods have been used to estimate the single-fibre compressive strength [5]. Some in current use are (i) the critical fibre length method, (ii) the compression test of single-fibre composites, (iii) the bending beam test, (iv) the elastica loop test and (v) the tensile recoil test. In the critical fibre length method, a single filament embedded in a resin matrix is compressed and the lengths of broken filament pieces are

measured. A fibre compressive strength is estimated using the average length of broken fibres and the yield shear strength at the fibre-matrix interface [6]. In the second method the compressive strain (rather than the length of the broken fibre pieces) of a single fibre embedded in a matrix is measured by optically monitoring the fibre failure [7]. In the bending beam test [8, 9] a fibre placed on a beam is adhesively bonded to the beam and the compressive strain to failure in the fibre is monitored while progressively bending the beam. In the last two methods, the product of the strain to failure and the tensile modulus is regarded as the fibre compressive strength. The compressive strengths determined from these three methods may be influenced by factors such as matrix properties, fibre-matrix interface and specimen fabrication because a fibre is embedded in a matrix or bonded to the beam.

Different from the previous three methods, both the elastica loop test and the tensile recoil test use a single filament without any matrix, and they can measure the compressive properties of fibres, which are free from the influence of the matrix. In the elastica loop test, the onset strain of non-Hookean behaviour is measured by observing the shape of a filament loop $[10-12]$. This onset strain is considered to indicate the compressive yield strain for PAN-based carbon fibre [13],

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and the compressive strength is calculated as the product of the compressive yield strain and the tensile modulus. In the tensile recoil test, compression is imposed on a fibre by the recoil force after controlled tensile stress [14-19]. Recoil compressive stress is assumed to be the same as initial tensile stress in magnitude [14].

In the present study, the single-fibre compressive properties are measured and then the difference between the single-fibre compressive strength and the composite compressive strength is discussed. The main focus of this paper is on mesophase pitch-based carbon fibre; however, several PAN-based carbon fibres are also included for comparative analysis.

2. Experimental procedure

2.1. Carbon **fibres**

The carbon fibres used in this study, along with their diameters and tensile properties, are listed in Table I. The mechanical properties of PAN-based carbon fibres are based on manufacturers' catalogue data.

2.2. Elastica loop test

Fibre compressive yield strains were measured by the elastica loop test [10, 11, 13]. In theory, the ratio of major to minor axes of a filament loop stays a constant value of 1.34 as long as the fibre behaves elastically, and this ratio sharply increases when the fibre deviates from elastic behaviour. This deviation from elasticity is taken as the yield point in compression. The compressive yield strain in the fibre, ε , is obtained from the fibre diameter (d) and the length of the loop minor axis (D) at which loop's major to minor axis ratio deviates from a constant value of 1.34. This strain is given by the following equation [10]:

$$
\varepsilon = 1.07d/D \tag{1}
$$

The product of the fibre tensile modulus and the loop strain was taken as the compressive strength of the fibre.

A filament loop was placed in light oil between two glass slides. The loop was successively deformed by pulling on both ends of the filament, and photographs were taken using an optical microscope. The major

TABLE I Mechanical properties of various carbon fibres

Fibre	Diameter (μm)	Tensile strength (GPa)	Tensile modulus (GPa)
PAN			
$HTA-7a$	6.9	3.6	230
$T-300b$	6.9	3.6	230
$M-40b$	6.5	2.7	390
$M-46b$	6.4	2.4	450
Mesophase pitch			
$NT-20^\circ$	10.0	2.8	201
$NT-40^\circ$	9.5	3.5	400
$NT-60^\circ$	9.4	3.0	595

Fibres from ^aToho, ^bToray and °Nippon Steel Corp.

and minor axes were measured from the photographs, and the ratio of the major to minor axis was plotted as a function of strain at the loop head. In each case five to nine fibres were tested. The fibre diameter was measured by an optical microscope. Fig. 1 shows a typical loop test result for NT-40 carbon fibre: This figure shows that the ratio of major to minor axis increases sharply when the strain at the head is over 0.5%. Therefore, for this fibre the compressive yield strain is estimated as 0.5%.

2.3. Tensile recoil test

In the tensile recoil test $\lceil 14-19 \rceil$, a single fibre is subjected to a static tensile stress of predetermined magnitude and then cut at its centre. Two halves of the fibre are allowed to snap back or recoil, and thus compressive waves are generated in the fibre. If this stress amplitude of the compressive wave exceeds the fibre's compressive strength, it fails in compression during the recoil. By performing this test with progressively increasing tensile stresses, a threshold stress can be located at which recoil compressive damage is observed, and a fibre compressive strength is obtained.

Fibres of 1 in. (25 mm) gauge length were mounted on cardboard tabs using Quick-GelTM glue (a cyanoacrylate adhesive from Loctite Corp.) With the tab arms cut away, the fibre was loaded in tension on an Instron Model 1125 using a 500 g load cell and a fullscale load limit of 50 g. At a predetermined tensile load, the fibre was cut at its centre with an electric spark and the two halves recovered for examination. Thirty to forty fibres were tested to obtain the compressive strength. An average of the stress values where recoil failure is never observed and the value where recoil failure is always observed was taken as the recoil compressive strength of the fibre [15].

3. Results and discussion

3.1. **Fibre compressive** strength

In Table lI, the fibre compressive strengths from the elastica loop and the tensile recoil tests are shown along with those from the unidirectional composite compression data. The fibre compressive strengths

Figure 1 Elastica loop test result of an NT-40 fibre.

TABLE II Compressive properties of various carbon fibres

"Composite compressive strength normalized to 100% fibre was taken as fibre compressive strength.

b Manufacturer's catalogue data.

From Tomioka [3] and Sato *et at.* [4],

a Unpublished data.

from both the loop test and the composite data show that PAN-based carbon fibres exhibit higher compressive strengths than mesophase pitch-based carbon fibres for the same modulus, and that the compressive strengths decrease with increasing tensile modulus. However, the compressive strengths from the loop test are higher than those from the composite data.

The recoil compressive strength of one mesophase pitch-based carbon fibre, NT-40, is higher than that of PAN-based carbon fibre, M-40 with a corresponding modulus. To our knowledge this is a new observation, and such compressive behaviour for other mesophase pitch-based carbon fibres has not previously been reported [15, 17, 18, 20].

3.2. The elastica loop test

In order to compare the compressive strength values from three methods, the loop and recoil compressive strengths are plotted as a function of compressive strength from composite data in Fig. 2. This figure shows that the fibre compressive strengths from the loop test are higher than those estimated from the composite data. In Fig. 3, the loop compressive yield strains versus composite compressive strains to failure are plotted. Except for NT-40 fibre, the loop yield strains are slightly higher than the composite compressive strains. On the other hand, NT-40 fibre has a lower loop strain value than the composite compressive strain value.

The loop test is equivalent to a very short gauge length test, and also, the composite compressive strength depends on both fibre compressive strength and composite buckling strength, and is determined by the weaker of the two [13]. If the composite fails in a buckling mode then the fibre compressive strength from composite data would be lower than its true strength [5, 13, 21]. Further, depending on the strain, the compressive modulus can be significantly lower than the tensile modulus for carbon fibres $[22-24]$, and the loop compressive strengths calculated using fibre tensile modulus are overestimated. These factors may explain why the compressive strengths from the loop test are higher than those from the composite test, but they cannot explain why the loop compressive yield strain of NT-40 is lower than the composite compressive strain.

The elastica loop test has often been used to determine the strength, stress-strain behaviour and failure

Figure 2 Fibre recoil and loop compressive strength as a function of fibre compressive strength calculated from composite data. PAN: (\Box) loop, (\blacksquare) recoil. Mesophase pitch: (\bigcirc) loop, (\lozenge) recoil.

Compressive strain from composite data (%)

Figure 3 Fibre yield strain from loop test as a function of fibre compressive strain from composite data.

modes in carbon fibres. Jones and Johnson [12], DaSilva and Johnson [25] and Tsushima [26] have measured the strength from the loop method. All three groups concluded that when the loop was taken to fracture, the strain in the loop represented tensile failure strain. On the other hand, the loop deviation from elasticity as shown in Fig. 1 which precedes the loop fracture indicates the presence of some form of non-Hookean behaviour. On the basis of non-linearity in the tensile stress-strain curve (hardening), Tsushima showed that the neutral axis in bending moves over to the tensile side. However, this movement of the neutral axis was considered to be small. If the non-linearity in the stress-strain curve is larger on the compression side and the material is softening, then the movement of the neutral axis to the tension side can be greater. Recent work on compressive and tensile stress-strain measurement by Kubomura and Tsuji [22, 23] indicates that in pitch-based carbon fibres, especially NT-40, the non-linearity in compression is much greater than in tension and the material is softening, while in PAN-based carbon fibres the non-linearity is reported to be relatively small [22-24]. Based on these results, it is argued that the deviation from elasticity in NT-40 in Fig. 1 must be due to the strong non-linearity in compression, which will explain why the onset strain of loop deviation from elasticity in this fibre is smaller than the composite compressive strain (which is the compressive strain to failure).

3.3. The tensile recoil test

A comparison of the fibre compressive strength from recoil and composite data, for the limited number of fibres in this study, in Fig. 2 suggests that the pitchbased carbon fibres have equal or higher recoil compressive strength values, while for the PAN-based carbon fibres the recoil compressive strengths are equal to or lower than those calculated from the composite data.

Dobb, *et al.* [17, 18] proposed two mechanisms, namely buckling failure for PAN-based carbon fibres and shear failure for mesophase pitch-based carbon fibres, in the tensile recoil test. Crastro and Kumar [15] also reported that the recoil failure of PAN-based carbon fibres occurred in the buckling mode, and that grease-coating of fibres reduced the recoil buckling. The recoil compressive strengths of grease-coated fibres were higher than the compressive strengths calculated from composite data. The buckling failure is considered to cause a reduction in the recoil compressive strength of PAN-based carbon fibres.

According to the Allen's recoil test analyses [14] the initial strain energy is converted to kinetic energy, and then the fibre kinetic energy is transformed back into compressive strain energy. In these processes the strain energy is conserved, lf, different from Allen's assumption, the fibre stress-strain relation is nonlinear and the non-linearities in tension and in compression are different, the stresses in tension and in compression are not equal.

There have been attempts to study the stress-strain relationship of carbon fibres. Hughes [27] reported

that carbon fibres showed non-linear behaviour in tension; the tensile modulus of PAN-based carbon fibres slightly increased with increasing strain. Arsenovic *et al.* [28] also reported an increase in modulus in pitch-based carbon fibres with an increase in tensile strain. Crasto and Kim [24] studied the compression properties of AS4-epoxy composites (AS4 is a PANbased carbon fibre with tensile modulus of 240 GPa), and found that the compression moduli of those composites decreased with increasing strain. Further Tsuji and Kubomura [22, 23] reported that longitudinal compression tests of unidirectional composites of high-modulus mesophase pitch-based carbon fibres showed strong modulus reductions with increasing strains, as shown in Fig. 4 which is reproduced from their work. A compressive non-linear behaviour for high-modulus mesophase pitch-based carbon fibres was also suggested by a study of bending beam tests by Miwa *et al.* [29]. The magnitude of the composite compression non-linearity of high-modulus mesophase pitch-based carbon fibres is much larger than that of the tensile non-linearity [27] and that of the composite compression non-linearity of PAN-based carbon fibres [24].

The question arises whether the non-linearity in the composite compression stress-strain relation is truly a fibre property. Fibre bending or waviness in the composite can also lead to non-linear behaviour [30]. However, the magnitude of non-linearity in the present discussion is not associated with plastic deformation which was clearly indicated by the work of Kubomura and Tsuji [23]. They observed that the softening non-linearity of unidirectional composites in compression was recoverable; when the load was removed before coupon failure no appreciable damage was observed, suggesting that this was a fibre property associated with some kind of buckling of the fibre as a whole or the inside of the fibre.

We calculated the magnitude of recoil compressive stress under strain energy conservation conditions by using the non-linear composite compression property in Fig. 4. To simplify the calculations, the compressive modulus $E_c(\varepsilon)$ at compressive strain ε would be represented by the equation

$$
E_c(\varepsilon) = E_c(0) \qquad \text{if } \varepsilon < \varepsilon_0
$$

= $E_c(0) \exp[-f(\varepsilon - \varepsilon_0)]$ if $\varepsilon > \varepsilon_0$ (2)

Figure 4 $E_c(\varepsilon)/E_c(0)$ as a function of compressive strain. $E_c(\varepsilon)$ and E_c (0) are compressive moduli at strains ε and 0, respectively [23]. (O) NT-20, (\square) NT-40.

where $E_c(0)$ is the compressive modulus at zero strain, f is the non-linear factor and ε_0 is the onset strain of compression non-linear behaviour. The factor f represents the degree of non-linear behaviour. From Fig. 4 values of f and ε_0 are estimated to be 100 and 0.003, respectively, for NT-20, and 360 and 0.0005 for NT-40. The calculated compressive moduli using Equation 2 are shown by the solid lines in Fig. 4. Equation 2 parameters for NT-60 are almost the same as those for NT-40, but the compressive strain to failure of NT-60 is much smaller than that of NT-40.

Fig. 5 shows the stress-strain energy curve for NT-40 calculated using Equation 2. Since the non-linearity in the tensile stress-strain relation was much smaller, it was neglected. From this figure, it is seen that the tensile stress of 1.9 GPa, which is the recoil compressive strength of NT-40, corresponds to a compressive stress of 1.2 GPa under the strain energy conservation condition. Thus, the fibre compressive strength estimated from the recoil test based on the strain energy conservation condition is 1.2 GPa for NT-40. The recoil compressive strengths corrected for the non-linearity in NT-20 and NT-60 are 1.5 and 0.7 GPa, respectively. For PAN-based carbon fibres, the recoil compressive strength based on the strain energy conservation condition is nearly the same as the compressive strength obtained from the initial tensile stress, because the magnitude of non-linearity of the PAN-based carbon fibre stress-strain relation is much smaller than that of mesophase pitch-based carbon fibres.

The corrected and uncorrected recoil compressive strengths are plotted against the fibre compressive strengths estimated from the composites in Fig. 6. It is observed that for the pitch-based fibres the corrected values agree well with those estimated from the composites. The difference between the composite compressive strength and the recoil compressive strength of NT-40 can be quantitatively explained by using the non-linear strain-stress relation observed in the composite test for mesophase pitch-based carbon fibres. This also strongly suggests that the longitudinal nonlinearity in unidirectional composites of mesophase

Figure 5 Stress-strain energy relationship of NT-40. The + and signs of stress represent tensile and compressive stresses, respectively.

Figure 6 Corrected and uncorrected fibre recoil compressive strength as a function of fibre compressive strength from composite data. (\blacksquare) PAN, (\bigcirc) mesophase pitch (uncorrected), (\spadesuit) mesophase pitch (corrected).

pitch-based carbon fibres in compression is truly a fibre property.

The corrected recoil compressive strength of NT-60 is nearly the same as the uncorrected strength, though NT-60 could have the same degree of non-linearity as NT-40 does. This is because NT-60 failed at a low stress level and the non-linear effect was much less significant compared with NT-40. The previous results [15, 17, 18] show that the recoil compressive strengths of mesophase pitch-based carbon fibres are not higher than the fibre composite compressive strengths and than the recoil compressive strengths of PAN-based carbon fibres with the same modulus. This can be explained by the same reasoning as for the recoil compressive strength of NT-60.

The tensile recoil test estimates properly the compressive work of fracture but not the fibre compressive strength when the fibre stress–strain relation deviates from linearity. The recoil test results in Table II suggest that the compressive work of fracture of NT-40 is higher than that of PAN-based carbon fibre with the same modulus.

4. Conclusions

The compressive mechanical properties of mesophase pitch-based and PAN-based carbon fibres have been studied by the elastica loop test and the tensile recoil test. One mesophase pitch-based carbon fibre (NT-40) exhibited a smaller compressive yield strain measured by the elastica loop test than the compressive strain to failure from the unidirectional composite test. On the other hand, this fibre has a higher recoil compressive strength (1.9 GPa) than the compressive strength estimated from the unidirectional composite test. This recoil compressive strength is higher than that of PAN-based carbon fibre of the same tensile modulus, though the NT-40 composite compressive strength is smaller than that of PAN-based carbon fibre.

It has been demonstrated that in the tensile recoil test the strain energy is conserved, and that the initial tensile stress and the recoil compressive stress will not be the same in magnitude if the stress-strain relations in tensile and/or in compression are non-linear. This has quantitatively been demonstrated in a number of pitch-based carbon fibres. For PAN-based carbon fibres the recoil compressive stress is nearly the same as the tensile stress, because their non-linearity is not so strong. Further, this non-linearity of mesophase pitch-based carbon fibre has well explained the difference between the loop compressive yield strain and the composite strain to failure. The tensile recoil test result suggests that the compressive work of fracture of NT-40 is higher than that of PAN-based carbon fibre, though the compressive strength of NT-40 is lower.

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