Axial tensile and compressive properties of high-performance polymeric fibres

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(Received 23 July 1990; accepted 14 October 1990)

Axial tensile moduli, compressive moduli and compressive strengths of rigid-rod poly(*p*-phenylene benzobisoxazole) and stiff-chain poly(*p*-phenylene terephthalamide), Kevlar™, fibres were measured with a Tecam Micro-tensile Testing Machine. This machine was configured to allow for testing of single fibres in direct tension and compression at extremely small gauge lengths. The appropriate gauge lengths were estimated based on the discussion of Euler buckling and non-uniform stress distribution in anisotropic materials. The measured tensile and compressive moduli were analysed for corrections to machine compliance and possible gauge length error. The corrected compressive moduli were slightly lower than the corrected tensile moduli, probably due to fibre misalignment under compression and the fibrillar nature of the fibres. The fibre axial compressive strengths measured by direct compression were comparable to those measured from recoil and composite tests but lower than those from cantilever beam and elastica loop tests.

(Keywords: tensile modulus; compressive modulus; compressive strength; polymeric fibre; rigid-rod polymer; poly(*p*-phenylene benzobisoxazole); stiff-chain polymer; poly(*p*-phenylene terephthalamide); Kevlar™ fibre; direct compression)

INTRODUCTION

Rigid-rod heterocyclic aromatic polymers have shown superior thermal and thermo-oxidative stability. These polymers can be processed into fibres with nearly perfect uniaxial orientation, resulting in excellent tensile properties[1,2]. Their relatively low density elicits potential applications in advanced composites as ultra-lightweight structural materials. However, these fibres suffer from a relatively poor axial compressive strength, a necessary property for aerospace structural composite applications.

In recent years, considerable research effort has been directed towards improving the compressive strength of rigid-rod polymeric fibres. They have involved chemical modifications by incorporating the rigid-rod backbones with bulky pendant groups to disrupt the molecular packing order[3], with different crosslinking moieties[4,5], and with pseudo-ladder structures through intramolecular hydrogen bonds[6]. At the early stage of development, these polymers were prepared and processed only in small quantities that prohibited composite fabrication for mechanical evaluations. With this restriction, it is desirable to have a test method that can characterize the fibre axial compressive properties that are translatable to composite compressive properties. Such a method would speed the development of new fibres as well as their composites.

Composite mechanical properties are determined by the compositions and the properties of the fibre and the matrix based on the rule of mixtures. In many cases, fibre properties become the dominant factor in determining the ultimate composite properties. A number of techniques were explored to test single fibres for axial compressive strength, such as the elastica loop test[7], cantilever beam method[8], tensile recoil test[9] and single fibre composite technique[10]. However, none of these techniques yielded results consistent with the data derived from composites[11]. It is the objective of this study to demonstrate the feasibility of measuring fibre axial compressive strengths by direct compression of single fibres in a Tecam Micro-tensile Testing Machine.

EXPERIMENTAL

Materials

Three high-performance polymeric fibres were studied. The rigid-rod poly(*p*-phenylene benzobisoxazole) (PBO) fibre was from the Dow Chemical Company and the two stiff-chain poly(*p*-phenylene terephthalamide) fibres, Kevlar™ 29 and Kevlar 49, were from the E. I. DuPont Chemical Company. All the fibres were used as received. Fibre diameter was determined using an optical microscope with filar eyepiece. Every fibre specimen, prior to being mounted in the Tecam Micro-tensile Testing Machine (MTM), was carefully examined for existing flaws. A thermoplastic adhesive, 1,5-diphenylcarbazide, was used to adhere each end of the fibre specimen to a quartz anvil with a small soldering iron. The specimen could be repositioned to improve the alignment by remelting the adhesive.

Tecam Micro-tensile Testing Machine

The MTM is an opto-mechanical device designed for

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0032-3861/92/010100-06

measuring tensile properties of whiskers and fibres. It has a pair of sample mounting anvils. The left anvil is adjustable in three dimensions to facilitate sample positioning and alignment. The right anvil is connected to the left mirror of an optical system, which is composed of two mirrors for strain determination. Each mirror reflects an image into a telescope mounted on the side of the MTM. In measurement, a null position was first achieved by aligning the reflected image from the right mirror to that from the left before any loads were applied. The gauge length of the fibre was then determined using the micrometer attached to the travelling microscope positioned over the sample anvils. The tensile (or compressive) load was applied to the fibre by rotating the load micrometer, which imposed a moment to the right sample anvil via a torque rod system. This caused the right anvil to translate away from (or towards) the left anvil, forcing the mirror reflections to separate. Realigning the images by rotating the displacement micrometer yielded the fibre displacement due to the applied load. This loading and realigning process was continued until the fibre failed. The load increment was determined by a noticeable separation of the reflections in the telescope. Fibre failure was readily identified when the mirror images could no longer be realigned due to a sudden excessive deformation. Accumulated data on load and displacement were used to construct a complete stress and strain relationship.

RESULTS AND DISCUSSION

The MTM was designed for tensile measurements of single fibres. To demonstrate that this machine is also capable of measuring fibre axial compressive properties by direct compression, three high-performance polymeric fibres were intensively tested in both tension and compression. It is well known that a slender column tends to buckle under axial compressive loading. In the Euler buckling process, when a critical compressive load is exceeded, a modest increase in the load results in a dramatic increase in the strain, leading to the buckling failure of the column. The critical compressive load (P_{cr}) for buckling a column with built-in end boundary conditions relates to the length and the shape of the column, and is governed by the equation:

$$P_{cr} = 4\pi^2 EI/L^2 \qquad (1)$$

where E is longitudinal Young's modulus, L is column length and I is the moment of inertia. For a cylindrical column the moment of inertia is $\pi D^4/64$, where D is diameter. In this study, equation (1) was used as a guideline to estimate the maximum allowable, or the upper bound (UB), gauge lengths for PBO and Kevlar fibres, so the fibres would undergo a compressive failure before suffering from an Euler buckling. These upper bounds were calculated by substituting the fibre axial compressive strengths (CS) derived from composite compressive tests and the reported fibre tensile moduli into equation (1). Therefore, the calculated upper bound is actually the gauge length at which a fibre will buckle at its axial compressive strength. The calculated upper bounds and the parameters used for the calculation are presented in *Table 1*. The maximum allowable gauge lengths were estimated to be 0.87 mm for PBO fibre, 0.28 mm for Kevlar 29 and 0.34 mm for Kevlar 49.

The St. Venant's principle describes that the stress field exerted in a material from an external force becomes uniform over some distance from the points of application. In practice this distance was taken as one lateral dimension. However, recent experimental[12,13] and theoretical developments[14] showed that this common practice is not true for anisotropic materials, in which the stress non-uniformity may persist over much greater distances than in isotropic materials. Horgan[14] derived the lower bound of the stress decay length in anisotropic materials by establishing an exponential decay inequality for the strain energy contained in that region. For transversely isotropic circular cylinders the critical decay length (L_{cr}) is of the form:

$$L_{cr} = R(E/E_T)^{1/2} \qquad (2)$$

where R is the radius of the cylinder and E_T is the transverse Young's modulus. Equation (2) was used to determine the minimum allowable, or the lower bound (LB), fibre gauge lengths so the fibres would not fail prematurely due to a stress concentration. The calculated lower bounds and the transverse compressive moduli[15] of PBO and Kevlar fibres are also reported in *Table 1*, where the transverse Young's modulus used for PBO fibre was only an estimate[8]. The estimated minimum allowable gauge lengths were 0.180 mm for PBO fibre, 0.064 mm for Kevlar 29 and 0.078 mm for Kevlar 49.

According to the principle of continuum mechanics, the tensile modulus and compressive modulus must be the same for a linear material at zero deformation. This is demonstrated in *Figure 1* with Kevlar 49 as an example, where the fibre experienced a deformation cycle of tension and compression. The tensile stress (σ_t) was in a linear relationship with the tensile strain (ε_t) up to 260 MPa, while the compressive stress (σ_c) showed a yielding against the compressive strain (ε_c) at about 140 MPa. The stress–strain curve showed a small deflection of the null position after cycling. This deflection was about 0.13 μm corresponding to a 0.03% strain uncertainty for

Table 1 Determination of the upper and lower bounds of gauge lengths

Fibre	D (μm)	E (GPa)[9]	CS (MPa)[9]	E_T (GPa)	UB (mm)	LB (mm)
PBO	17.5	200	200	0.50[8]	0.87	0.180
Kevlar 29	12.5	80	400	0.77[15]	0.28	0.064
Kevlar 49	12.0	130	400	0.76[15]	0.34	0.078

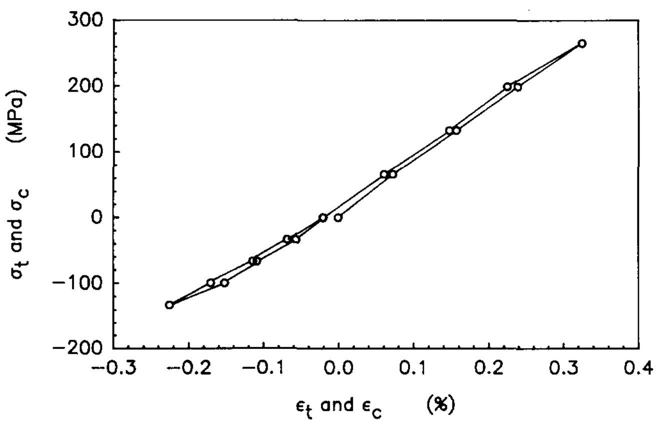

Figure 1 Apparent stress–strain relationship for Kevlar 49 undergoing a deformation cycle of tension and compression

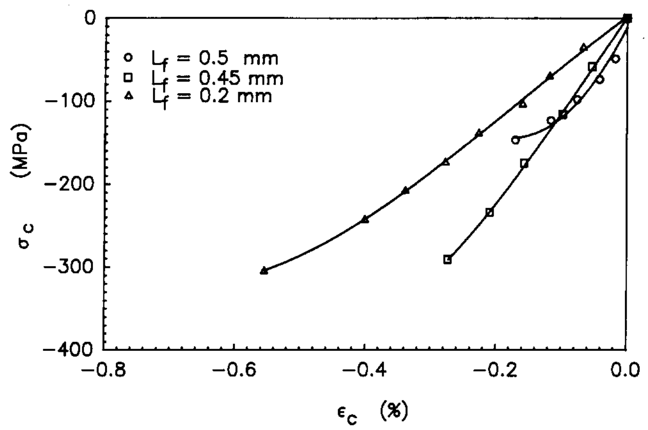

Figure 2 Apparent compressive stress–strain relationship for PBO fibre at various gauge lengths

the fibre 0.44 mm long. However, the tensile and compressive moduli at zero deformation were indeed identical within experimental error.

The relationship between the compressive stress and strain of PBO fibre and its dependence on gauge length are shown in *Figure 2*. At a gauge length of 0.5 mm, the fibre initially showed a higher modulus, but the fibre buckled with increasing compressive load, resulting in a lower compressive strength. This buckling gauge length is smaller than the predicted 0.87 mm from equation (1) and may relate to the fibrillar nature of rigid-rod polymeric fibres. In such fibres, Euler buckling may, in fact, start at fibril level and then cascade to the whole fibre. A detailed discussion on the buckling of rigid-rod polymeric fibres was given elsewhere[16]. At gauge lengths of 0.45 and 0.2 mm, respectively, the fibre showed a linear relationship between the compressive stress and strain almost to the failure point. The compressive strengths measured at these two gauge lengths were comparable, about 300 MPa, suggesting that the stress non-uniformity has little or no effect on the compressive strength of PBO fibre between these two gauge lengths. The non-linearity near the compressive failure point does not agree with the linear stress–strain relationship observed in tensile measurements using an Instron Universal Test Machine. It is conceivable that an intensified misalignment for a fibre under axial compression and the yield of loosely bundled fibrils could both result in such a non-linearity.

The apparent compressive modulus, $(E_c)_{app}$, was found to be strongly dependent on gauge length due to the effect of machine compliance, since the gauge length was so small. The equation that describes the stress (σ) in relation to the apparent modulus (E_{app}) and apparent strain may be expressed as:

$$\sigma = E_{app}(l_f + l_m)/L_f$$

where l_f is the deformation of the fibre, l_m is the deformation contributed by the machine and adhesive, etc., and L_f is fibre gauge length. This equation may be rearranged to give a linear dependence of the reciprocal apparent modulus on the reciprocal gauge length as:

$$\frac{1}{E_{app}} = \frac{1}{E_f} + \left(\frac{l_m}{\sigma}\right)\left(\frac{1}{L_f}\right) \tag{3}$$

where E_f is the true or the corrected fibre modulus. A plot of such a relationship is presented in *Figure 3* for PBO fibre at gauge lengths from 0.19 to 0.5 mm. The fibres that had gauge lengths longer than 0.47 mm or shorter than 0.20 mm showed lower apparent compressive moduli. The former is due to the Euler buckling of the fibre and the latter is probably due to a number of factors such as an underestimated gauge length and a stress concentration (for the fibre was very close to the lower-bound gauge length). An underestimated gauge length causes an overestimated strain and, thereby, an underestimated modulus. By extrapolating the reciprocal apparent compressive modulus to zero reciprocal gauge length using the data for the gauge lengths between 0.20 and 0.47 mm, a corrected compressive modulus of 220 GPa was obtained for PBO fibre.

The apparent fibre gauge length was measured from the distance between the two adhesive edges on either side of the sample anvils, but the effective gauge length may penetrate into the adhesive. This penetration depth may be very small, but it could be significant in affecting the apparent modulus when the fibre gauge length is small. Assuming the apparent gauge length, $(L_f)_{app}$, is the true gauge length (L_f) less a small depth (d) into the adhesive, the stress, the apparent modulus and fibre gauge length may be correlated by the equation:

$$\sigma = E_{app}\left(\frac{l_f + l_m}{L_f - d}\right)$$

This equation may be transformed into an expression similar to equation (3) as:

$$\frac{1}{E_{app}} = \frac{1}{E_f} + \left(\frac{d}{E_f} + \frac{l_m}{\sigma}\right)\left(\frac{1}{L_f - d}\right) \tag{4}$$

Based on equation (4), if d is constant, an underestimated fibre gauge length would only affect the slope of reciprocal E_{app} *versus* reciprocal $(L_f)_{app}$, while the corrected fibre modulus remains unchanged. There are other factors, such as misalignment and interfacing problems, that may affect the apparent modulus in various degrees at small gauge lengths. The low apparent compressive moduli observed for PBO fibre at gauge lengths smaller than 0.20 mm might be due to a synergistic effect of these factors.

The apparent tensile moduli, $(E_t)_{app}$, of PBO fibre are plotted against gauge length from 0.35 to 4.75 mm in *Figure 4* according to equation (3). The corrected tensile modulus by linear regression was about 295 GPa, significantly higher than the corrected compressive modulus. The same set of data was plotted in *Figure 5*

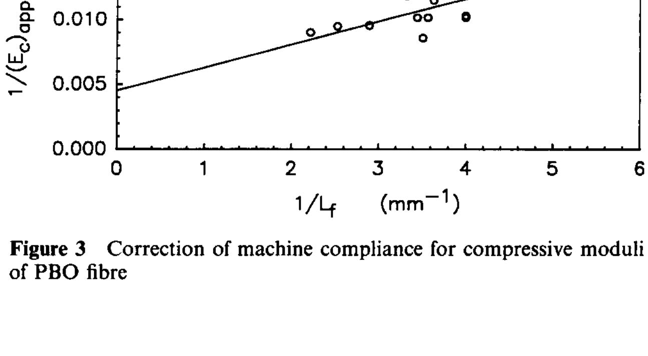

Figure 3 Correction of machine compliance for compressive moduli of PBO fibre

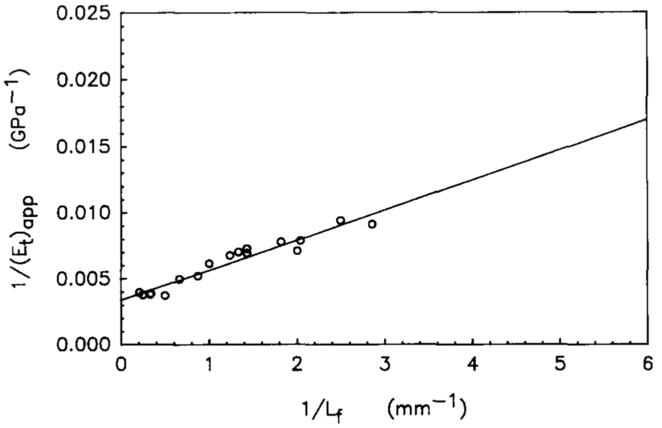

Figure 4 Correction of machine compliance for tensile moduli of PBO fibre

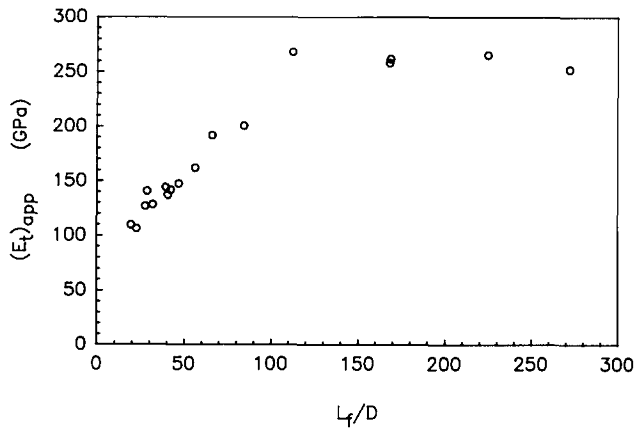

Figure 5 Apparent tensile moduli as a function of fibre aspect ratio

as a function of apparent tensile moduli against the fibre aspect ratio of gauge length over diameter (L_f/D). The data exhibit a trend that the apparent moduli initially increase with the aspect ratio and then approach an asymptote of about 260 GPa at an aspect ratio of about 100. In the plateau region, the gauge length dependence of modulus can no longer be described by equation (3). If the data in the plateau region were excluded from linear regression, the corrected tensile modulus turned out to be 245 GPa, fairly close to the corrected compressive modulus.

Figures 6 and *7* present the relationship between the reciprocal $(E_c)_{app}$ and the reciprocal L_f for Kevlar 29 and Kevlar 49, respectively. The fibres did not buckle up to 0.4 mm for Kevlar 29 and up to 0.5 mm for Kevlar 49. The two fibres failed compressively at 210 and 290 MPa, respectively, significantly lower than the reported 400 MPa from the composite compressive tests. If 210 and 290 MPa were substituted into equation (1), the Euler buckling lengths for Kevlar 29 and Kevlar 49 were estimated to be 0.4 mm, in agreement with the experimental observations. The corrected compressive moduli for Kevlar 29 and Kevlar 49 were 76 and 103 GPa, respectively. *Figures 8* and *9* show that the corrected tensile moduli are 96 and 115 GPa, respectively, for the two Kevlar fibres. Kevlar fibres also showed the asymptotic tensile moduli as demonstrated by PBO fibre in *Figure 5*. The asymptotic values, 95 GPa for Kevlar 29 and 110 GPa for Kevlar 49, were reached also at the aspect ratio of about 100. These asymptotic moduli were very close to their corresponding corrected tensile moduli.

The corrected tensile and compressive moduli and the effects of machine compliance in both tension and compression are listed in *Table 2*. For all three fibres, the tensile moduli are slightly higher than the compressive moduli. As discussed earlier for PBO fibre, this may be attributed to the intensified misalignment under compression and the fibrillar nature of the fibres. When a fibre is stretched, its alignment is enhanced with the

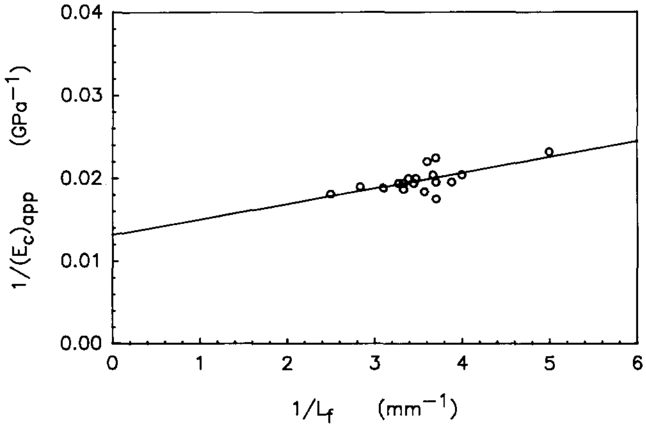

Figure 6 Correction of machine compliance for compressive moduli of Kevlar 29

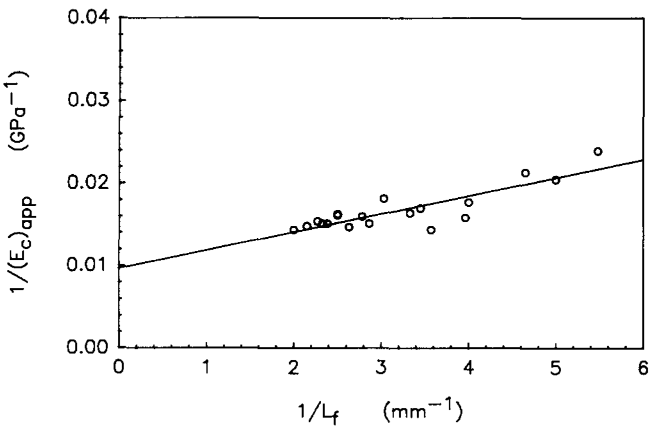

Figure 7 Correction of machine compliance for compressive moduli of Kevlar 49

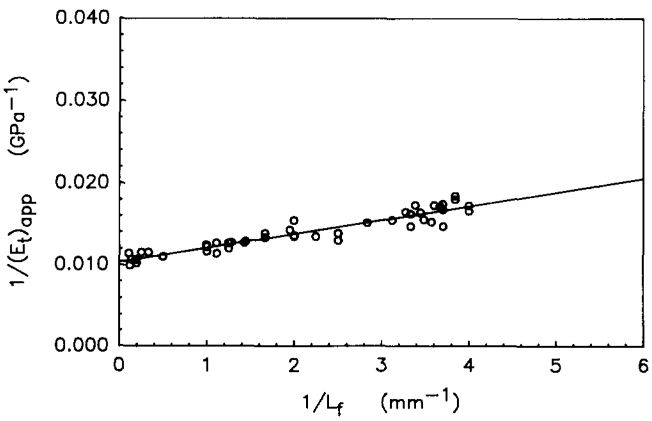

Figure 8 Correction of machine compliance for tensile moduli of Kevlar 29

Table 2 Correct tensile and compressive moduli of BPO and Kevlar fibres

Fibre	E_t (GPa)	l_m/σ_t (μm GPa^{-1})	l_m/F_t (μm N^{-1})	E_c (GPa)	l_m/σ_c (μm GPa^{-1})	l_m/F_c (μm N^{-1})
PBO	295 245[a]	2.27 1.90[a]	9.46 7.92[a]	222	1.77	7.38
Kevlar 29	96	1.70	13.8	76	1.89	15.4
Kevlar 49	115	1.89	16.7	103	2.20	19.5

[a] Corrected values with the data in plateau region excluded from linear regression

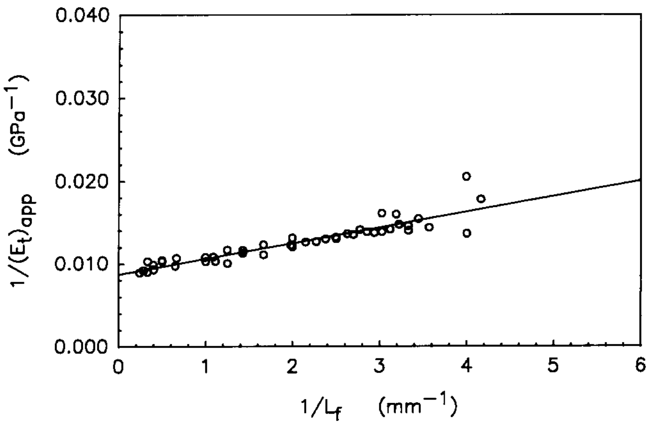

Figure 9 Correction of machine compliance for tensile moduli of Kevlar 49

tension, but when a fibre is axially compressed, its alignment is difficult to control. The misalignment effect becomes more prominent as the gauge length gets shorter. It was reported that both PBO[1,2] and Kevlar fibres[17,18] have fibrillar or microfibrillar structures. Those loosely bonded microfibrils may locally yield or buckle at a very small load compared with the axial compressive strength, giving rise to a lower compressive modulus than tensile modulus. The slopes, l_m/σ, as represented in equation (3) were comparable for all three fibres in both tension and compression. To characterize the machine compliance correctly, the deflection of the machine should be based on the total force, F_t in tension and F_c in compression, applied to the machine rather than the stress applied to the fibre. However, the values of l_m/F_t and l_m/F_c for Kevlar fibres were found to be about twice as high as that for PBO fibre. It suggests that the machine compliance is not the only contributor to the deformation l_m. In fact, other contributions such as the deformation of adhesive may be included as well. These data should not be analysed in any greater detail, because the variables involved are numerous.

Fibre axial compressive strengths measured by direct compression of single fibres are compared with the data measured from other techniques[10,12,13] in *Table 3*. Contrary to the data reported from composite tests, the compressive strength of PBO fibre is slightly higher than that of Kevlar fibres from direct compression of single fibres using the MTM. *Table 3* lists the compressive strengths cited from a number of sources, but for the comparison of various techniques discussions were made on the data measured from the same fibre. For PBO fibre, the compressive strengths obtained from direct compression and cantilever beam method are comparable to that from the composite test, while the data

Table 3 Comparison of fibre compressive strengths (MPa) measured from various techniques

Fibre	Loop[20]	Beam[20]	Recoil[9]	Composite[9]	MTM
PBO	680[9] 430–470	 270–320	200	200 280–350[19]	 295±35
Kevlar 29	500	590	350	400	210±12
Kevlar 49	670 740–790[9]	690	365	400–450	290±35

from the loop test are higher. For Kevlar fibres the compressive strengths measured from direct compression are lower than but comparable to the data from composite and recoil tests, and significantly lower than the data from the loop test and cantilever beam method. In general, the MTM data from direct compression of single fibres translate fairly well to the data from the composite test, indicating that the fibre compressive strength can be measured by direct compression. However, it should be noted that all these measurements were made on polymeric fibres, whose compressive strengths are relatively low. More extensive demonstrations with various types of fibre are needed for the implementation of this direct compression technique.

ACKNOWLEDGEMENT

This work was sponsored in part by US Air Force Contract No. F33615-87-C-5241.

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