Mechanical behaviour of rigid rod polymer fibres: 1. Measurement of axial compressive and transverse tensile properties

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Rigid rod polymer fibres have low axial compressive and transverse tensile strengths which can be measured by several methods. The tensile recoil method is tedious but a new fibre cutting device which simplifies and improves the method has been developed. Also a new method to test the transverse strength index of single fibres has been devised. The index is found to be similar among a variety of fibres suggesting that such properties depend more upon interfibrillar morphology than upon interchain properties. The same device can also perform three point bending tests on single fibres. This permits the determination of the fibre compressive modulus which for rigid rod polymer fibres appears to be considerably less than the tensile modulus.

(Keywords: mechanical behaviour; fibres; rigid rod; tensile properties)

INTRODUCTION

Rigid rod polymer fibres have low axial compressive strengths; several methods are available to measure this[1,2]. All of the methods mark failure by the onset of visible kink-band formation. The methods include the elastica loop test[3], matrix shrinkage[2] and beam bending[2,4]. The compressive strength is calculated from the product of the tensile modulus and the critical strain for kinking. It is assumed that the fibre behaves in a linear elastic fashion to compressive failure and that the tensile and compressive moduli are identical. These assumptions cause substantial uncertainty so a more direct measurement of single fibre axial compressive strength is desirable. The tensile recoil test developed by Allen is such a method[5]. Reflected elastic recoil stresses created from tensile failure cause compressive damage in the fibre. The compressive strength is determined by fracturing a number of tensile specimens at different stress levels to find the minimum value which just initiates kink-band formation. This requires tensile failure at different stress levels and several cutting techniques have been developed for the purpose; they include spot etching, heat cutting, prior localized mechanical damage, and scissor cutting[5]. Reproducibility in the first three is poor and scissor cutting induces undesirable increases in the applied stress because of the shearing action. We have developed a new device for symmetrical cutting of the fibre during recoil testing which gives a more accurate assessment of the axial compressive strength.

The transverse strength of rigid rod polymer fibres is also very low. To our knowledge, no methods are available to measure this with single fibres. Therefore, we developed a test in which an opening mode crack is propagated axially in such fibres. The crack initiation force provides a measure of a transverse mechanical property of a fibre. A variation of the device permits three point bending tests on single fibres, which enables the calculation of the flexural modulus. Since the flexural modulus is a combination of the compressive and tensile moduli, the compressive modulus can be calculated if the tensile modulus is known.

EXPERIMENTAL

Tensile recoil testing

The analysis of the tensile recoil test has been presented elsewhere[5]. The most difficult part of the test is finding a suitable method to cause tensile failure in the fibres. If breaking is not done with great care, large increases (spikes) in the applied load will occur. If the spikes are too large, the test is invalid because the exact stress state in the fibre becomes unknown. Although some researchers have found that surgical scissors can provide reasonable reproducibility, problems exist with this technique. The blades cut by a shearing action, and as shown schematically in *Figure 1*, this imposes a twist on the fibre causing an increase in the applied load. Another problem arises when the blades do not cut symmetrically: both blades do not come in contact with the fibre at the same time. The fibre is displaced laterally, as shown in *Figure 2*, which also causes a spike in the load. Both of these effects are more pronounced as the fibre modulus increases.

To remedy these problems we made a device named FI-RE-CUT (fibre-recoil-cutter), which is shown in *Figure 3*. It employs scalpel blades mounted on blocks which are supported by linear bearings. The blades remain coplanar, avoiding any shearing action. The blocks are connected to a drive rod with opposing left- and right-hand threads; when the rod is rotated it brings the blades together smoothly at a uniform rate.

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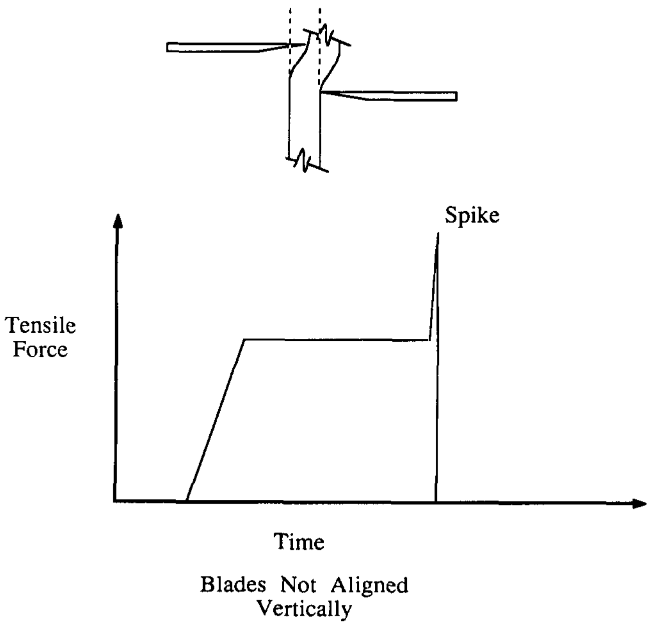

Figure 1 Schematic of spike in load during tensile recoil testing caused by shearing action of scissors

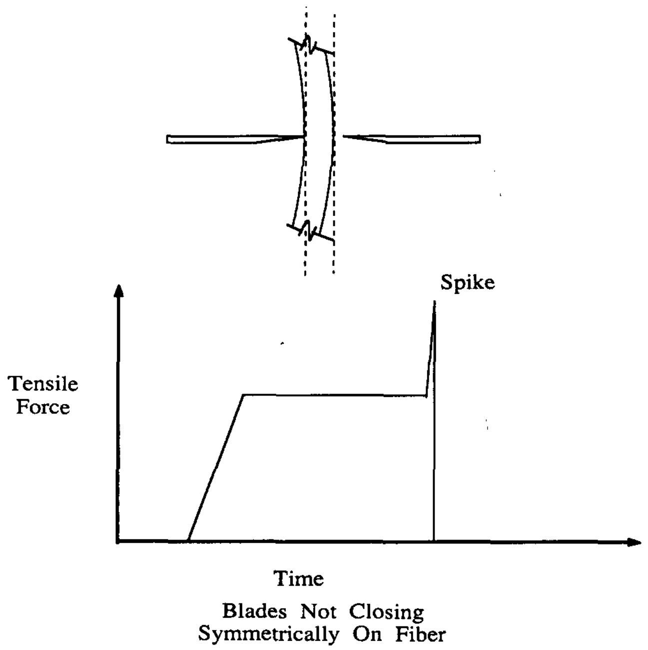

Figure 2 Schematic of spike in load during tensile recoil testing caused by unsymmetrical cutting

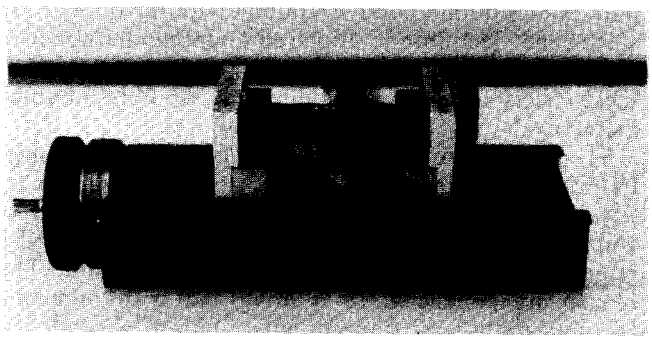

Figure 3 FI-RE-CUT device

The entire device is mounted on a micrometer substage which facilitates precision centring of the fibre between the blades and prevents unsymmetrical cutting. We have found that using FI-RE-CUT our success rate for correctly cutting fibres is nearly 100% for Kevlar 49 and about 80% for PBO and other stiff experimental fibres, compared with about 80 and 30%, respectively, using other methods. (No results of our recoil testing are presented since it has already been shown that it measures axial compressive strengths in good agreement with composite data[5].)

Transverse testing

The poor lateral integrity of rigid rod polymer fibres makes them susceptible to damage from handling. We observed that a fibre of circular cross-section could easily be flattened with tweezers or other instruments. Consequently, if the end is split, the force required to propagate an axial crack could give some measure of the transverse strength. This procedure is shown in *Figure 4* and, experimentally, we have made such specimens with a micromanipulator (*Figure 5*). The loads involved in the test are extremely small and difficult to measure. To determine the critical crack initiating force we constructed an instrument which operates with dead weights (*Figure 6*). The operation is simple: one ligament of the notched fibre is placed in a fixed grip and the other in a movable grip. The latter is supported by a gas bearing which eliminates friction effects. Attached to the movable grip is a cable running over a gas bearing supported shaft. The cable ends at a bucket in which weights are placed.

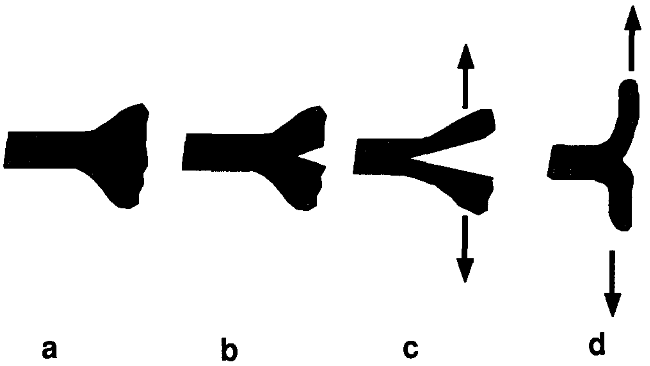

Figure 4 Schematic of lateral splitting test of single fibre. (a) Flattening of fibre end; (b) creating notch in flattened portion; (c) pulling ligaments apart; (d) propagating crack

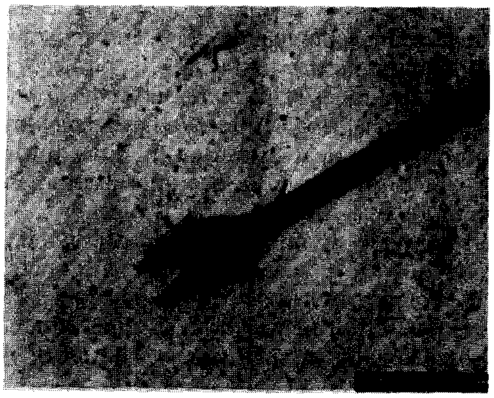

Figure 5 Optical micrograph of single fibre which has been flattened and then had a notch created in it using a micromanipulator

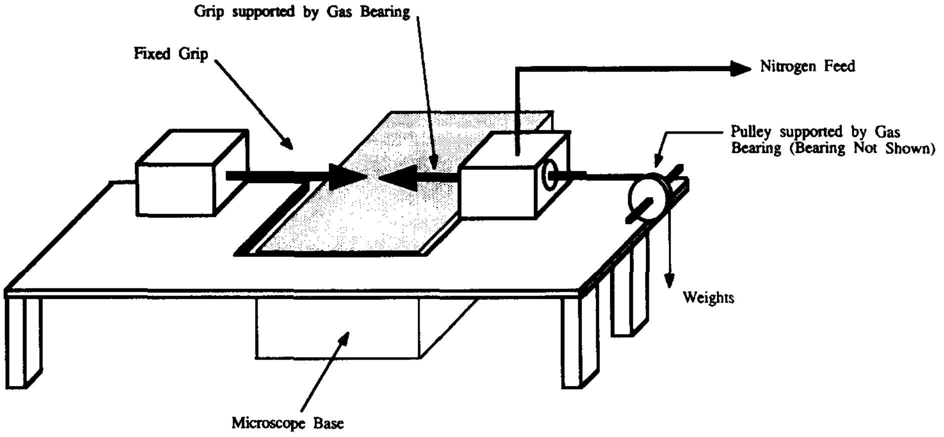

Figure 6 Schematic diagram of device used for transverse testing and three point bending

(Since the loads required for crack initiation are in the milligram range, the gas bearings are critical: frictional forces in conventional bearings easily exceed the loads of the test.) To balance the system before testing, the entire device is slightly elevated at the right end causing the movable grip to displace to the left. Weights are added until the movable grip is in a neutral position then the fibre is inserted into the grips and more weights are added until the fibre splits. The entire test procedure is observed with a stereo microscope and a video screen. The incremental weights are measured on a chemical balance which is inexpensive, accurate and easy to use. Their value, divided by the diameter of the fibre, provides a number to represent the lateral integrity of the fibre: the opening mode axial crack initiating force, normalized by diameter.

Three point bending

This apparatus also can be used for three point bending tests. The fixed grip is replaced by a fibre support block (*Figure 7*). A hooked probe is attached to the movable grip. A fibre is placed on the platform with the hooked probe beneath it. (The platform is made of aluminium and the probes of stainless steel, both of which have thin nylon coatings to minimize friction.) When weights are added to the bucket the hooked probe loads the fibre at its midpoint. Deflections are measured with an optical microscope and kept small so that linear behaviour occurs. Other researchers have conducted bending tests on much larger monofilaments but measured only the deflection; to our knowledge, this is the first time that measurements both of load and deflection have been made[6,7].

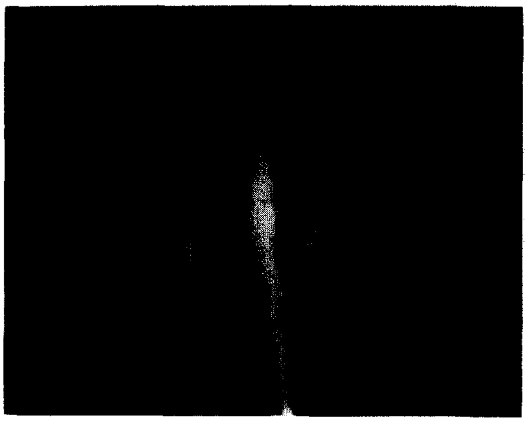

Figure 7 SEM micrograph of support device for three point bending

RESULTS

Results for transverse splitting of Spectra (Allied Signal), Kevlar 49 (E. I. DuPont de Nemours) and PBO (Dow Chemical) fibres are presented in *Table 1*. The critical load to produce the mode I crack in the axial direction, $P_{\mathrm{CRI,A}}$ is normalized by the fibre diameter. Although this is not the transverse tensile strength, it does provide some quantitative information on the lateral integrity of the fibre.

The results show that Kevlar 49 and PBO-1 split at about the same force while Spectra splits at a much lower value. If the splitting force is controlled by interchain attraction, then the Kevlar 49, a polyamide with substantial hydrogen bonding, should require a higher force than the PBO. Since the results are very similar, it appears that interfibrillar attractions dominate this behaviour. The results also show that the splitting load is independent of fibre diameter.

Table 1 Transverse test results

Fibre designation	Fibre diameter [μm]	$P_{\mathrm{CRI,A}}$/Diameter [N m^{-1} (lb in^{-1})]
Spectra	35	349 (1.99)
Kevlar 49	12	519 (2.96)
PBO-1	24	491 (2.80)

A representative scanning electron micrograph of a split PBO fibre is shown in *Figure 8*. The fibrillar structure on the interior is very evident so the technique has also been used to study the morphology of fibres.

A rather low transverse strength is necessary for the successful performance of this test. Fibres with less anisotropy, such as nylon, have been tried and their higher resistance to crack propagation causes the ligaments to break before the axial crack propagates. Thus the usefulness of the test may be limited.

Three point bending

The deflection, δ, for a simply supported beam loaded at its centre, is:

$$\delta = \frac{PL^3}{48E_f I}$$

Figure 8 SEM micrograph of single fibre tested in transverse testing device

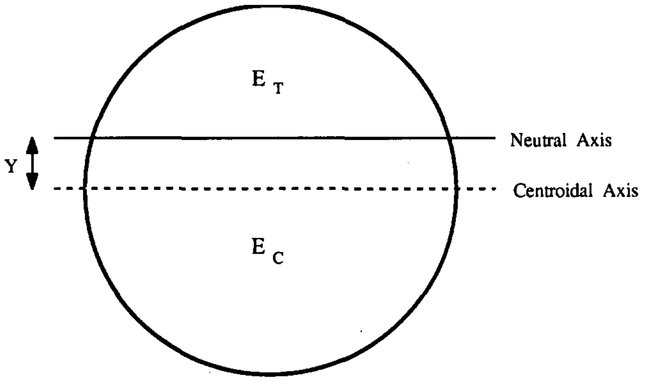

Figure 9 Schematic diagram of the positions of the neutral and centroidal axis in a material of circular cross-section with a compressive modulus less than the tensile modulus

where P is the applied load, L the span, E_f the flexural modulus and I is the moment of inertia about the neutral axis. The moment of inertia for a circular cross-section about its neutral axis is:

$$I = \frac{\pi d^4}{64} + Ay^2$$

where d is the diameter, A the area and y is the distance from the neutral axis to the centroidal axis (*Figure 9*). For an isotropic material, the flexural modulus is the same as the tensile and compressive moduli and the neutral and centroidal axes coincide. With an anisotropic material the three moduli are different and the neutral axis shifts toward the direction of the higher modulus segment.

Using the three point bending device, we measure the flexural rigidity, $(EI)_f$, for single fibres from the slope of a load–deflection curve (*Figure 10*). The flexural rigidity of the fibre is the sum of the rigidities of the tensile and compressive sections:

$$(EI)_f = E_T I_T + E_C I_C$$

The algebra to solve this simple equation for E_C, the unknown compressive modulus, is quite complex because of the circular cross-section of the fibre. In fact, for simplicity we approximated the cross-section with a square of equal area to that of the circle, since an analysis showed little error is involved.

The results for Kevlar 49 and PBO with a span of 950 μm are shown in *Table 2*; each entry represents the average of at least 10 tests.

For all fibres the compressive modulus is less than the tensile modulus. This is to be expected because of the highly fibrillar microstructure of these materials. In simple terms, the fibrils will react very differently to a push or pull, in contrast to the response of an isotropic material.

A serious source of error in the bending test is in the measurement of the fibre diameter and any departure from a truly circular cross-section. Since the moment of inertia calculation for a circle or a square requires the fourth power of diameter, a ± 1 μm variation for a 14 μm average diameter fibre can produce a $\pm 35\%$ spread in the calculated value of E_C. If the absolute value of E_C is low, the absolute spread may be negligible; if not, it can be appreciable. To minimize the error, we measure the diameter of the fibres with the scanning electron microscope, and discard any specimens with non-circular cross-sections: a cumbersome process.

Two other factors should be mentioned: in the linear portion of the bending load–deflection plot, it is possible to calculate the maximum compressive strain on the surface of the fibre. With a central deflection of about 40 μm, this strain is 0.12–0.16%, depending on fibre diameter and as far as we have been able to observe, the linear region is reversible: the behaviour appears to be Hookean. Also, because of the very large span-to-depth ratio of the test, the shear component of the total deflection is negligible, even if very low values of the shear modulus are used. Thus, in Kevlar 49 we estimate the error from this source to be no more than a few per cent.

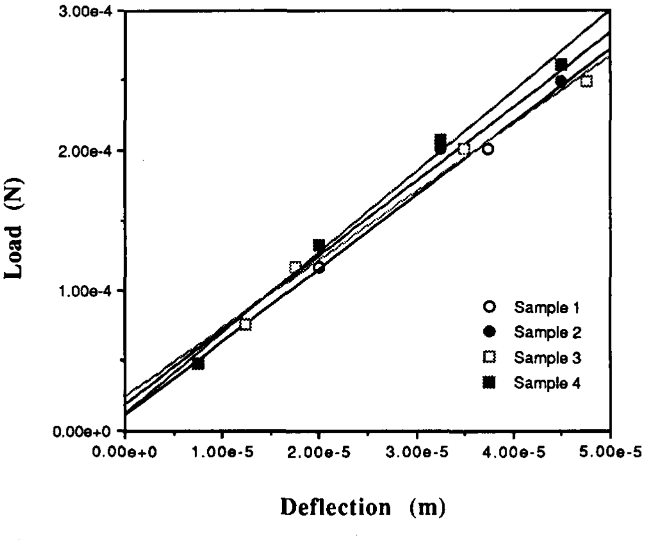

Figure 10 Load *versus* deflection curves for three point bending on single Kevlar 49 fibres

Table 2 Three point bending results

Fibre	Measured flexural rigidity [N m^2 (lb in^2)] ($\times 10^9$)	Assumed tensile modulus [GPa (Msi)]	Calculated compressive modulus [GPa (Msi)]
Kevlar 49	0.104 (36)	124 (18)	90 (13)
PBO-1	0.543 (189)	276 (40)	41 (6)
PBO-2	0.786 (273)	276 (40)	241 (35)

Current estimates of compressive modulus for rigid rod polymer fibres are 70–90% of the values for the tensile modulus, found from unidirectional composite tests[8] or from Raman frequency measurements on single fibre composites[9]. The latter was not a measurement of modulus since no determination of stress was made, though the data presented were convincing. With respect to the unidirectional composite tests, they more representatively average over the length and number of fibres than any direct test on an unbonded single fibre. They may be a better indication of actual fibre performance in a composite. In a new procedure such as reported here, the possibility of undiscovered errors must be recognized.

Some other interesting three point bending experiments are currently underway: by loading a fibre to the point of kink-band formation we have observed that some exhibit pronounced non-linear behaviour before kinking. This suggests that some complicated deformation mechanisms are operating. Also we are experimenting with shorter spans which will produce significant shear deformations; it may be possible to determine the shear modulus directly in this manner.

CONCLUSIONS

The tensile recoil test is an effective method for compressive strength measurements of high performance fibres if load spikes can be avoided when the sample is cut. A device for symmetrical uniform cutting has been developed and it produces good results.

The force required to propagate an opening mode crack along the axial direction of a single fibre can be measured. Such experiments with PBO and Kevlar 49 fibres suggest that this force depends on interfibrillar rather than interchain attractions.

The compressive modulus of single fibres can be determined from a three point bending test. The compressive moduli of Kevlar 49 and PBO fibres are both less than their tensile moduli.

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REFERENCES

1 Greenwood, J. M. and Rose, P. G. *J. Mater. Sci.* 1974, **9**, 1809
2 DeTeresa, S. J., Allen, S. R., Farris, R. J. and Porter, R. S. *J. Mater. Sci.* 1984, **19**, 57
3 Sinclair, D. J. *J. Appl. Phys.* 1950, **21**, 380
4 DeTeresa, S. J., Porter, R. S. and Farris, R. J. *J. Mater. Sci.* 1988, **23**, 1886
5 Allen, S. R. *J. Mater. Sci.* 1987, **22**, 853
6 Dmitruk, L. N., Gureeva, N. N. and Shelyubskii, V. I. *Zavodskaya Lab.* 1978, **44**, 1284
7 Guthrie, J. C., Morton, D. H. and Oliver, P. H. *J. Text. Inst.* 1954, **45**, T912
8 Kumar, S. in 'Materials Science and Engineering of Rigid Rod Polymers' (Eds W. W. Adams, R. K. Eby and D. E. McLemore), Vol. 134, MRS Symp. Proc., Pittsburgh, PA, 1989
9 Young, R. J. and Ang, P. P. Final Report, US contract AFOSR-88-0148, May 1990