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The mechanical properties and fracture behaviour of unidirectionally reinforced Nylon 6

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The tensile and shear properties of Nylon 6 polymerized *in situ* around unidirectionally aligned carbon and glass fibres have been investigated and the fracture behaviour characterized by optical and scanning electron microscopy. The tensile strengths are found to lie within the limits predicted by the law of mixtures and deviations from the predicted strengths have been correlated with fibre type and surface treatment. The shear strength values follow the same trend and an important mode of fracture in bending is shown to be the compressive failure which accompanies a yield drop in the load deflection curve. Depending upon the fibre type and the properties of the matrix this compressive damage need not lead to catastrophic failure of the composite as, in certain cases, the matrix can undergo substantial deformation before failure.

1. Introduction

The mechanical properties and the fracture behaviour of semi crystalline thermoplastics reinforced by random short fibres have been studied extensively and the mechanical properties shown to be adequate for many engineering applications. In such composite systems the fibres impart added stiffness and fracture strength. However, understanding the mechanical properties and fracture behaviour of these materials is complicated by the random orientation of the fibres [1] and the morphology changes within the matrix that may be brought about by the presence of the fibres.

In earlier work [2, 3] we have shown that by polymerizing caprolactam to polycaprolactam (Nylon 6) the nylon shows a variation in morphology, crystallinity and mechanical properties depending upon the polymerization temperature used. Also, by carrying out the polymerization *in situ* around unidirectionally aligned fibres, void free composites can be produced with good fibre alignment [4]. It has also been shown that incorporation of the fibres modifies the morphology of the nylon and that the extent of columnar crystallization around the fibres is dependent on the polymerization temperature and fibre type [5].

This paper describes studies of the mechanical properties and fracture behaviour of unidirectional carbon and glass fibre-reinforced Nylon 6 prepared by *in situ* polymerization. The tensile and shear strengths of the materials have been measured and the modes of failure examined using optical and scanning electron microscopy.

2. Experimental

2.1. Specimen preparation

The Nylon 6 was prepared by the anionic polymerization of caprolactam [2]. Before polymerization occurred the reacting solution was cast into a mould through which the preweighed fibres were positioned. The mould produced rectangular composite bars measuring 140 mm x 8 mm x 4 mm. Full details of the fabrication technique are published elsewhere [4]. Tensile strength and tensile fracture studies were carried out on curved neck specimens having a radius of approximately 55 mm and a minimum width of 4 mm, prepared from the rectangular bars. The specimen ends were tabbed with aluminium tabs

measuring 40 mm x 8 mm x 4 mm prepared according to Courtaulds specifications [6]. The tabs were bonded to the composites using an 'araldite' adhesive which was allowed to cure at room temperature for 7 days. Specimens for the three-point bond test were cut from the bars to give a range of length to depth (l/d) ratios. All specimens for mechanical testing were polished on emery paper commencing with grade 600 and finishing with grade 4/0. The final polish was achieved using alumina (0.3 μm) powder. To reduce the effects of water absorption in the nylon matrix all specimens were stored under vacuum over silica gel prior to testing.

Specimens containing several different types of carbon fibres were prepared. Full details of both the carbon and glass fibres are given in Table I.

2.2. Mechanical testing

The tensile and three-point band tests were performed at room temperature using an Instron testing machine with a cross-head speed of 2 mm min^{-1}. The bend tests were carried out on a rig to which a low power microscopy system could be attached to enable specimen deformation to be observed as the test progressed [7].

2.3. Microscopy

The deformation behaviour and fracture morphology were studied using optical microscopy of polished sections and scanning electron microscopy of specimens on which a conducting film of gold–palladium had been evaporated.

TABLE I Fibre properties*

Fibre	Type	Surface treatment	Young's modulus ($GN\,m^{-2}$)	Tensile strength ($GN\,m^{-2}$)
Courtaulds Grafil HM-S	I	Treated	345–414	1.72–2.24
Morganite Modmor I	I	Treated and untreated	382–451	1.37–2.06
Morganite Modmor II	II	Treated and untreated	245–314	2.45–3.14
Le Carbone Rigilor AC	II	Treated	190–210	1.9–2.2
Glass fibre† (Pilkington Bros.)	–	Treated	7.6	0.75–2.5

*The strength and stiffness values quoted are those obtained from manufacturers data and include the upper and lower figures listed.

†The glass fibre was treated by the manufacturer with a polyamide compatible size coating.

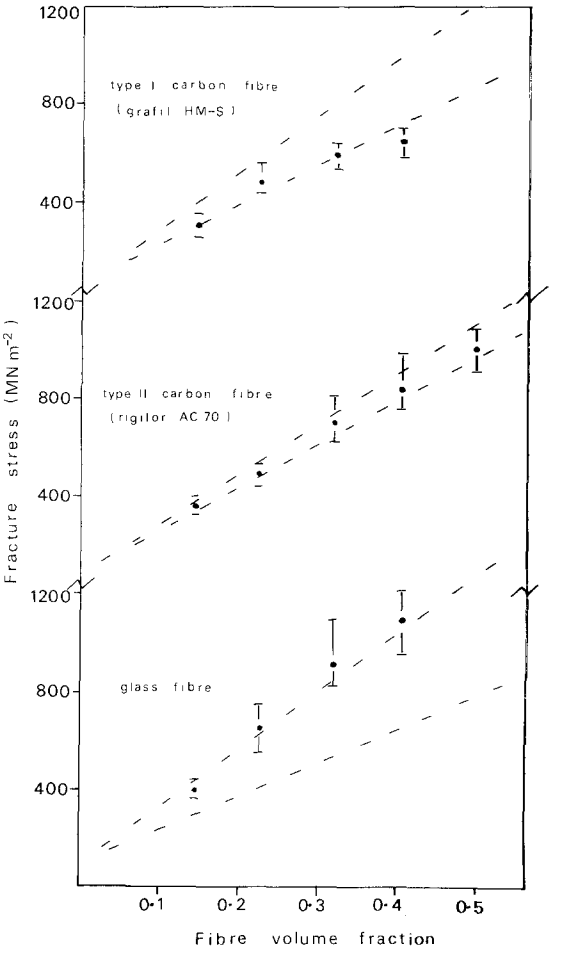

Figure 1 The variation of tensile fracture stress with fibre volume fraction for Nylon 6 composites polymerized at 453 K. The broken lines indicate the theoretical limits using the maximum and minimum values of fibre strength.

3. Results and discussion

3.1. Tensile properties

Tensile tests were carried out on composites containing a variety of different fibres with fibre volume fractions in the range of approximately 0.18 to 0.50. The tensile fracture stress of the composites containing glass fibres and types I and II carbon fibres is shown, plotted against fibre volume fraction, in Fig. 1. On this graph the experimental data is shown together with the theoretical values expected using the law of mixtures rule for predicting strength values [8]. The two dashed lines for each fibre type represent the upper and lower theoretical levels of tensile strength using the maximum and minimum value of fibre tensile strength quoted in Table I. The strength of the nylon matrix at the failure strain

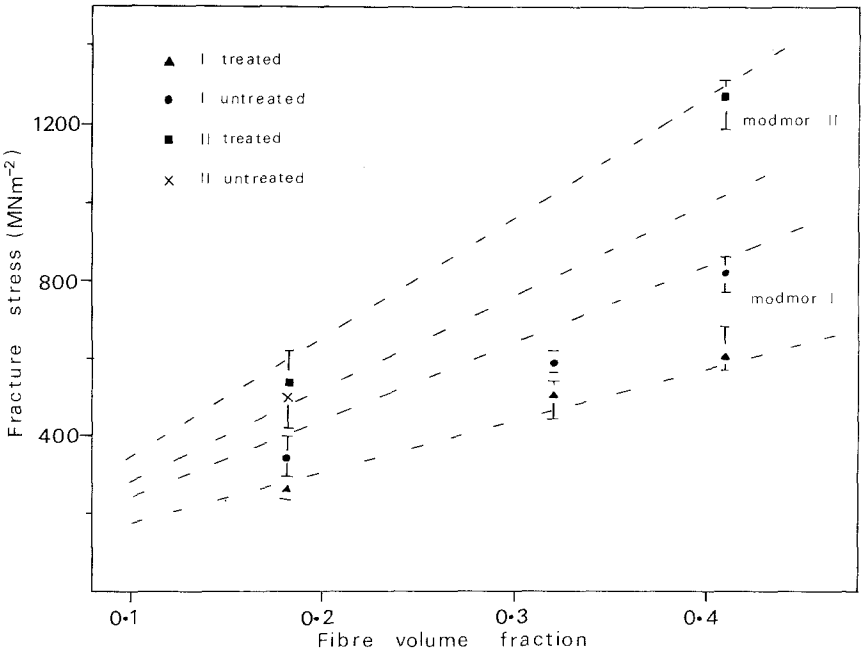

Figure 2 The effect of surface treatment on the tensile fracture strength of composites containing "Modmor" carbon fibres. The broken lines indicate the theoretical strength of the composites using the maximum and minimum fibre tensile strength.

of the composite was taken to be 60 MN m^{-2} [3]. From these results it can be seen that the fracture strength of the glass-filled material is near the upper limit of that predicted over the whole range of volume fraction, whereas the strength of the composites containing surface treated type I carbon fibres (Grafil HM–S) was near or slightly below the lower bound.

The effect of surface treatment of carbon fibres was studied in more detail using treated and untreated 'Modmor' fibres. The results of the tensile strength determinations are shown, plotted against fibre volume fraction, in Fig. 2. The upper and lower limits of the theoretical tensile strength are indicated by dashed lines. It can be seen that the strength of composites containing treated type I fibres are somewhat lower than those containing untreated fibres although both fall within the expected limits predicted by the law of mixtures. This result is reflected in a difference in the observed fracture morphology. The materials containing untreated 'Modmor I' fibres exhibited much greater fibre pull-out than the surface treated material, which were brittle and showed little pull-out [5]. The 'Modmor II' composites did not show this difference in strength due to surface treatment. However, difficulty was experienced in fabricating the composites containing untreated type II carbon fibres at fibre volume fractions greater than about 0.18. This fabrication difficulty was attributed to the poor wetting properties of these particular fibres.

3.2. Three-point bend tests

Three-point bend tests were performed on specimens having similar fibre volume fractions to those used for the tensile tests and with length to thickness ratios between approximately 3 and 15. Values of shear stress and flexure stress were calculated from the load–deflection data and were plotted against length to thickness (l/d) ratio. Typical curves obtained are presented in Fig. 3. For most samples the flexure strength was within the limits predicted by the law of mixtures although below the mean value. The shear strengths of the composites were obtained from either the constant values on the shear strength plot or the slope of the zero intercept line on the flexure strength plot. The values of shear strengths obtained are listed in Table II. In general, the composites containing glass and surface treated type II carbon fibres had the highest shear strengths and

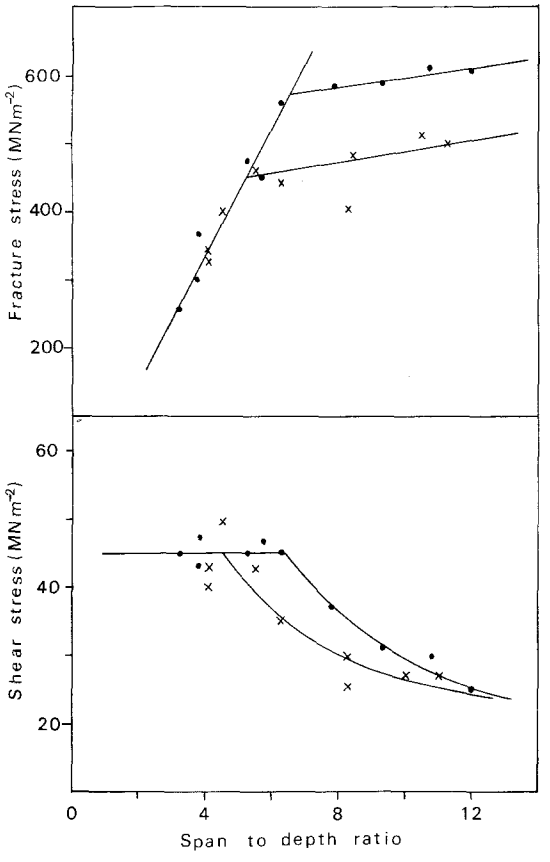

Figure 3 Plots of flexure fracture stress and shear stress in three-point bending against span to depth ratio for composites containing "Le Carbone" AC (type II) carbon fibres. Fibre volume fractions, ×, 0.28V_f; •, 0.48 V_f.

the polymerization temperature and resulting morphology and crystallinity had little effect on this value.

The shear strength values listed cannot be compared with any theoretical estimates or other results for fibre-filled Nylon 6, but they are similar in magnitude to those of Daniels *et al.* [9] who obtained values of 33 and 60 MN m^{-2} for untreated and treated 'Modmor I' fibres in an epoxy resin matrix.

TABLE II Shear strength of various Nylon 6 composite systems

Fibre type	Composite polymerization temperature (K)	Shear strength (MN m^{-2})
Glass fibre	453	63
	473	64
Carbon fibres:		
Le Carbone AC69	453	40
(Type II treated)	473	44
Courtaulds HM–S	453	55
(Type I treated)	473	53
Modmor I untreated	473	39
Modmore I treated	473	44
Modmore II treated	473	63

3.3. Deformation modes and fracture behaviour

The deformation and fracture behaviour of the specimens tested in three-point bending is complex due to the interaction of three different modes of failure; compressive buckling, shear and flexure failure. These three fracture modes observed in carbon fibre–nylon composites are shown in Fig. 4. Because of this complex deformation the

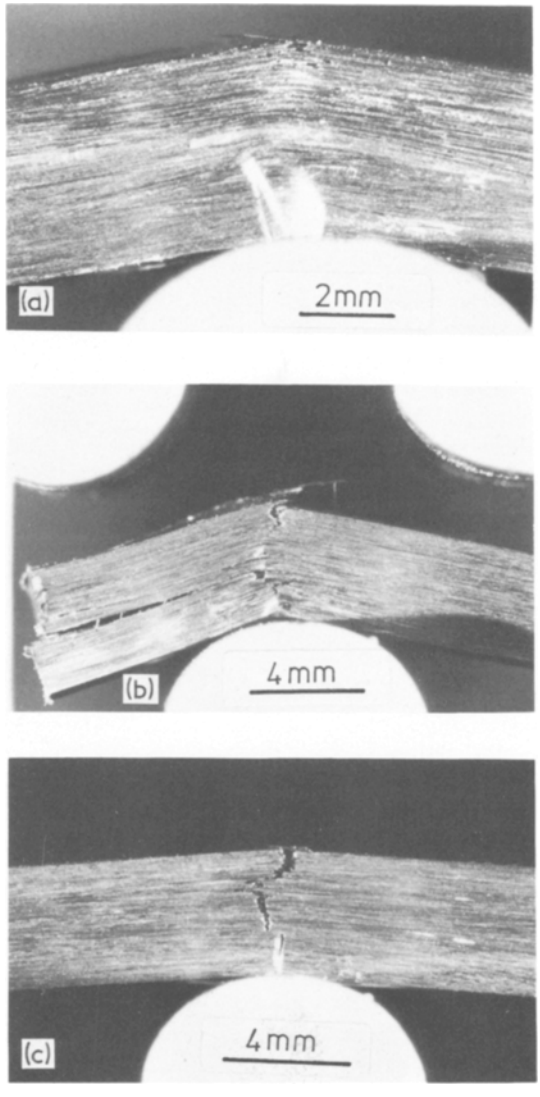

Figure 4 (a) Compressive, (b) shear and (c) flexure failure modes in carbon fibre–nylon composites as a result of bend testing.

numerical values of shear and flexure strength of the composites may be somewhat misleading since the basis of calculating these results depends on inserting the maximum values of load (P), obtained from the load–deflection curve, into the relevant equation [7].

$$\text{flexure stress, } \sigma = \frac{3pl}{2bd^2}, \quad (1)$$

$$\text{shear stress, } \tau = \frac{3p}{4bd}, \quad (2)$$

where l, b and d are the length, breadth and thickness of the specimen respectively. Such an analysis depends on only one deformation mechanism, either shear of flexure, being operative at the point of maximum load. However, in the carbon fibre composites this was not the case. In Fig. 5, which shows the load–deflection curve of a specimen which was filmed during deformation, the yield point is seen to coincide with the onset of compressive buckling, evident as a white streak above the loading nose. This behaviour was observed in all samples which did not fail in a catastrophic brittle manner. Although either shear or flexure failure were responsible for the final fracture of the specimens the onset of compressive failure was always visable at or just after the yield point had been reached. Thus the initiation and propagation of compressive buckling will tend to decrease the value of load P and give a reduced value for the flexure and shear strengths calculated using Equations 1 and 2.

Compressive failure has been observed as a result of impact and bend testing in carbon fibre–epoxy resin composites [10, 11] and in glass–polyester resin composites [7]. However, the compressive zone in these thermoset matrix materials is smaller than that in nylon and does not appear at the yield point of the load–deflection curve; this can be attributed to the higher compressive strength of the resin matrices. It can be concluded that in composites in which the matrix has a low compressive yield strength, compressive buckling will considerably influence the interpretation of the results of the three-point bend test.

However, it should be noted that this compressive failure is associated specifically with three-point bend testing due to the non-uniform stress distribution at the central loading point. In four-point bending, where the load is distributed between two points, the compressive failure is seldom encountered.

Because of the importance of compressive deformation on the fracture of nylon composites subject to bending stresses the compressive failure zone was examined using optical and scanning electron microscopy. The first stage of the deformation process is the yielding in compression of the nylon matrix, which subsequently causes fibre buckling and eventually fracture. However,

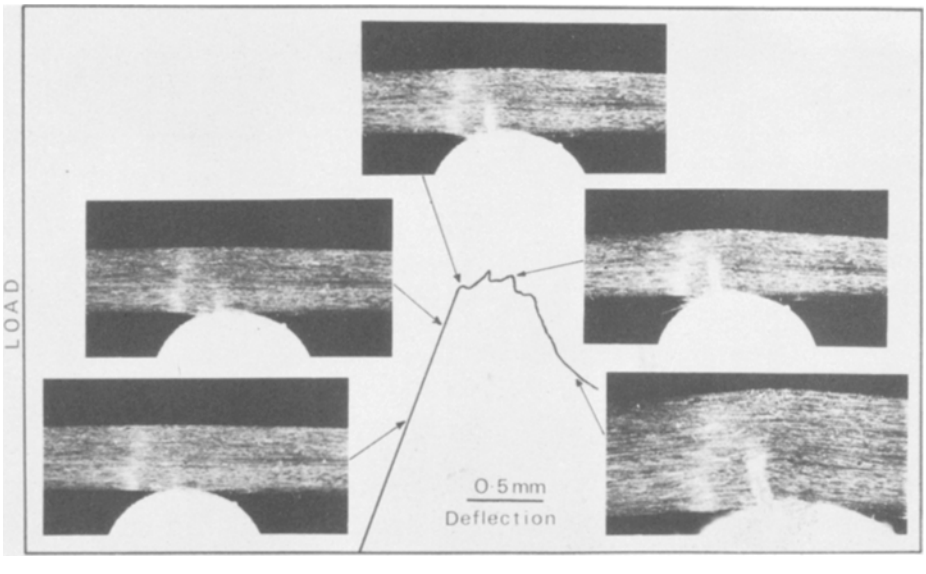

Figure 5 A load–deflection curve of a carbon fibre composite tested in bending showing the compressive failure associated with yield.

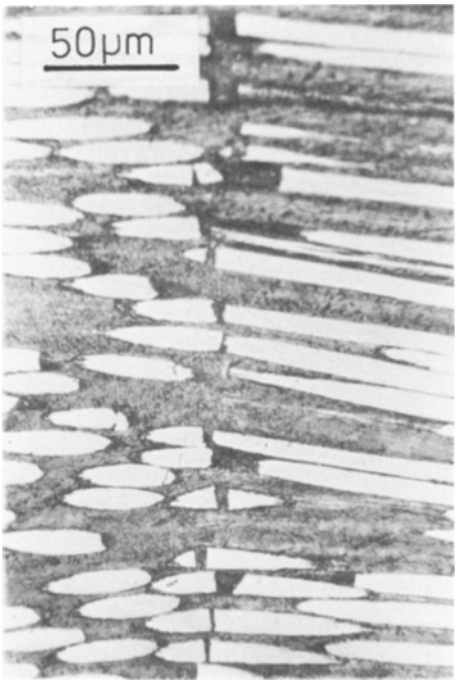

Figure 6 Optical micrograph of a polished section through the compressive failure regions in a carbon fibre composite, showing fibre fracture but no matrix fracture.

in most cases, and especially those with a low matrix crystallinity the fibre failure does not propagate through the nylon matrix until considerable deformation has occurred. Fig. 6 shows fibre fracture but no matrix failure in a carbon fibre composite; although the composite must be considered to have fractured total separation into two or more parts has not occurred and the nylon matrix is still capable of supporting some load.

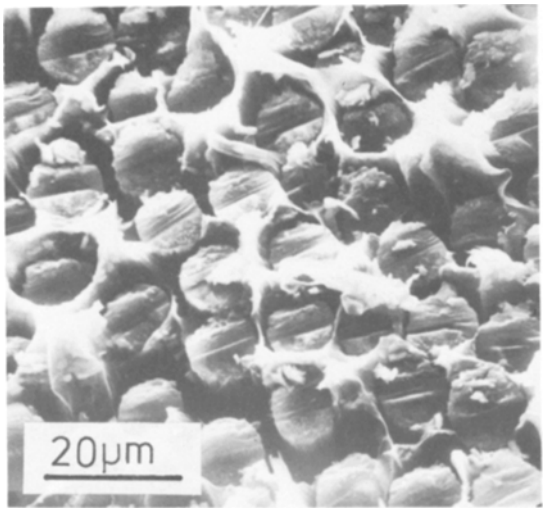

Figure 7 Scanning electron micrograph showing matrix drawing associated with compressive failure of a type II carbon fibre composite.

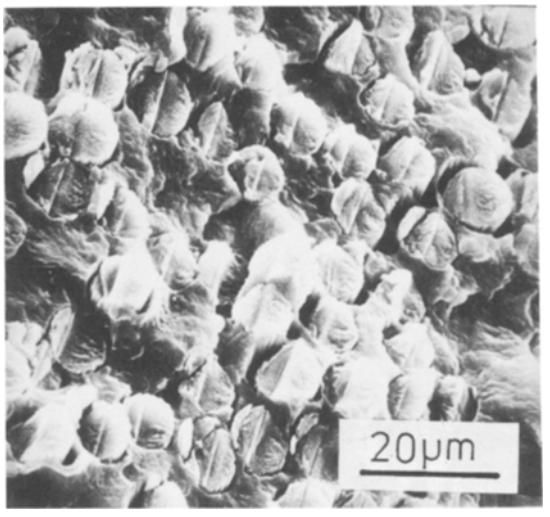

Figure 8 Scanning electron micrograph showing the lack of matrix deformation in the compressive failure zone of a surface-treated type I carbon fibre composite.

This is also evident from the stereoscan micrograph presented in Fig. 7 which shows the severe deformation and drawing of the nylon matrix in a type II carbon fibre composite. This is in contrast to the compressive fracture shown in Fig. 8 in which a high matrix crystallinity and surface-treated type I carbon fibres combine to give a very brittle fracture with no apparent matrix drawing after fibre buckling. Fig. 8 also reveals the failure zone in the fibre.

The effect of matrix toughness on the fracture in bending of nylon composites is shown in Fig. 9 in which typical load–deflection curves for various composites are presented. It can be seen that glass-filled composites polymerized at 453 K (curve a) were not as tough as those polymerized at 473 K (curve b) in which the matrix crystallinity was about 15% lower. Very severe brittleness was exhibited by all the composites containing surface-treated type I carbon fibres in which the failure strain was small and fracture occurred in a catastrophic manner (curve c). Nylon reinforced with untreated type I carbon fibres, however, showed large failure strains (curve d) during which considerable fibre pull-out had occurred with the material still supporting a load. Stereoscan microscopy of type I carbon fibre composites revealed that the treated fibres gave little fibre pull-out on the tensile half of the fracture surface, whereas the untreated fibres

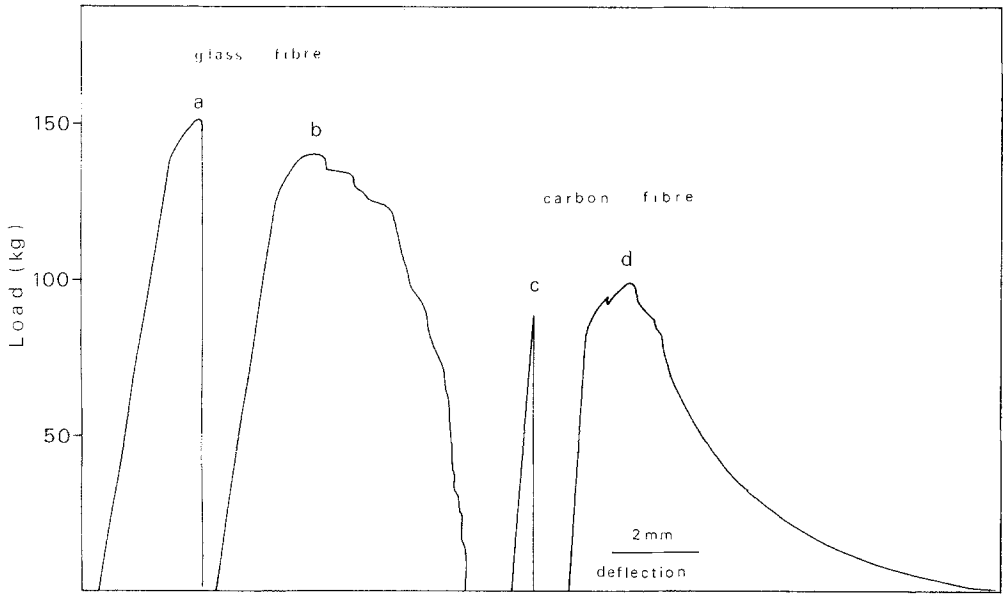

Figure 9 Load–displacement curves from three-point bend tests for (a) glass fibre composite polymerized at 453 K; (b) glass fibre composite polymerized at 473 K; (c) surface-treated type I carbon fibre composite; (d) untreated type I carbon fibre composite.

showed substantial pull-out in this region (Fig. 10). Thus the apparently weaker fibre–matrix bond between the untreated type I carbon fibres and the nylon gives greater pull-out and a tougher mode of failure in bending.

4. Conclusions

It has been shown that in general the tensile strengths of uniaxially aligned continuous fibre-reinforced Nylon 6 lie within the limits predicted by the law of mixtures, although the composites containing surface treated type I carbon fibres have tensile strengths approaching the lower limit due to the extreme brittleness of the composites. The fracture surfaces of these composites are characterized by very small fibre pull-out lengths.

In three-point bending similar findings are obtained and the treated type I fibres are seen to give brittle failures. An important mode of fracture in bending is shown to be compressive failure which occurs at the same time as a yield drop is

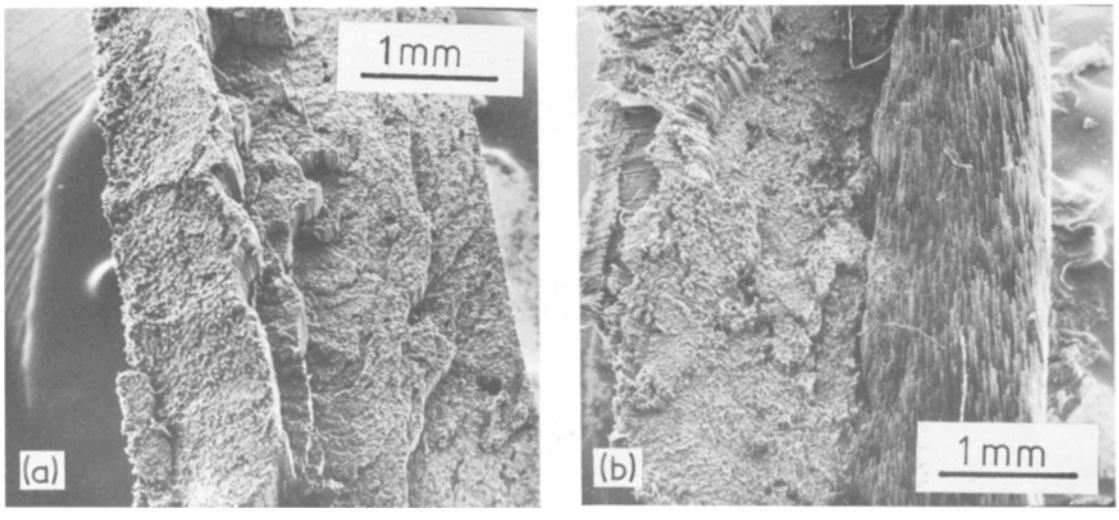

Figure 10 Stereoscan micrographs showing composites fractured in bending, the compressive zone is on the left; (a) treated type I carbon fibres, and (b) untreated type I carbon fibres.

observed on the load–deflection curve. However, provided the matrix has a sufficiently low crystallinity or fibres other than surface-treated type I are used the fibre failure within the compressive zone need not lead to catastrophic failure of the composite since, after fibre fracture, the nylon matrix can undergo substantial deformation prior to ductile failure.

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