

# The effect of matrix stresses on fibre pull-out forces

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A pull-out test was developed to measure the bond strengths and frictional forces between steel wires, and polycarbonate and epoxy matrices when the matrix was under tensile stress. Some debonding occurred due to the matrix stress. Despite this, the nominal bond strength, in the polycarbonate case, increased with increasing matrix applied stress. When the pull-out force had caused complete debonding, sliding under approximately constant friction coefficient,  $\mu$ , occurred. The value of  $\mu$  for steel sliding in polycarbonate was 0.6, and for epoxy it was 0.19. The values were reduced to 0.12 and 0.10 respectively when the steel was coated with a fluorocarbon release agent. The normal stresses at the interface, in the absence of any applied stresses, were found to be about  $7 \text{ MN m}^{-2}$  in the polycarbonate, and  $3.0 \text{ MN m}^{-2}$  in the epoxy case. It was observed that the frictional forces due to these residual stresses could be less than one third of those generated by the applied stresses on the matrix. Thus residual stresses are not as important for fibre reinforcement as are matrix Poisson's shrinkage stresses.

## 1. Introduction

Numerous measurements have been made on the interfacial strength and pull-out forces for fibres and wires in a number of different matrices. The results obtained up to 1970 have been reviewed by Broutman [1]. These measurements have been very useful in promoting a better understanding of the processes that take place when composites fail. However, two factors are frequently overlooked.

(1) In the fracture process the fibres can bridge cracks, and hence, at the crack faces, are under very high stresses, and yet embedded in a matrix which is bearing almost no stress at all. Hence Poisson's shrinkage results in the fibres contracting radially towards their centre-lines, and away from the matrix. This can have very significant consequences [2], and Takaku and Arridge [3] have made pull-out measurements and calculations that take this into account.

(2) The converse of this occurs at the fibre end in a highly stressed short fibre composite. Here the fibre stress is small or zero, while the matrix stress can be quite large. In this case Poisson's shrinkages in the matrix can result in high compressive forces

at the fibre surface. The effect of this on fibre stress distribution has been discussed by Amirbayat and Hearle [4], and Piggott [5] has discussed its effect on the stress-strain curve of the composite. The initial bond between fibres and matrix is generally not important for reinforcement with polymer matrices, since it is broken at quite low applied stresses by stress concentrations at the fibre ends. Stress transfer takes place near the fibre ends mainly by frictional forces generated by the differential contraction between fibres and matrix.

The effect on the frictional forces of putting the matrix under uniform compressive stress has been investigated by Bowden [6]. This paper reports measurements of frictional forces, and the shear forces required to break the bond between fibres and matrix, under conditions which simulate those near the fibre ends in a reinforced polymer. These are obtained by applying a tensile stress to the matrix while pulling the fibre out.

## 2. Experimental method

Fig. 1 indicates the type of specimen used for the test. The polymer matrix was kept under constant

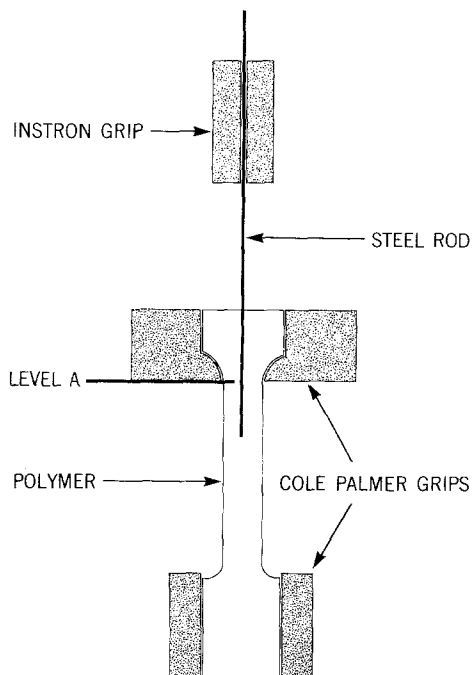


Figure 1 Specimen used for tests.

stress using a modified Cole-Palmer fibre tester. Load was applied to the upper end of the polymer by specially shaped grips that supported, rather than compressed, the polymer. The Cole-Palmer tester was mounted on an Instron tensile tester (Fig. 2) and the Instron cross-head was used to pull the wire out at a rate of  $1 \text{ mm min}^{-1}$ .

In order to minimize end effects, the wire was coated with fluorocarbon release agent (Miller Stephenson Chemical Co, MS 122) between the upper surface of the polymer specimen, and the top end of the reduced section (level A, Fig. 1).

This was done by forcing carefully measured short lengths of polyethylene tube, 1.09 mm i.d., over the wire ends. The wire diameter was 1.14 mm. The wire was then dipped in the release agent, and the agent was dried by gentle heat (65 to

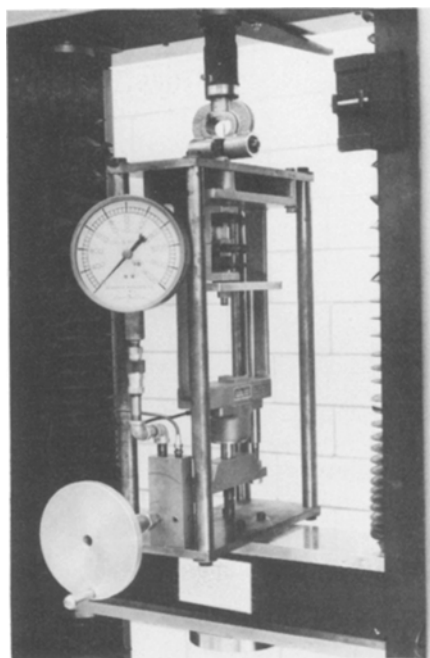


Figure 2 Testing set-up showing the specimen mounted in the Cole-Palmer Tester; it is near the dial at the top. The Cole-Palmer Tester is fixed to the Instron cross-head.

$70^\circ \text{C}$ ) for 20 min before removal of the polyethylene, and embedding in the polymer matrix.

The wire was in the form of straight, high carbon steel rods. These were embedded in Lexan polycarbonate (General Electric Co), and in Shell epoxy resin (type 815) with 50 vol% V-40 curing agent. The mechanical properties of these materials are given in Table I.

Before being used, the rods were cleaned as described previously [7]. Hot pressing at  $240$  to  $250^\circ \text{C}$  and  $10 \text{ MN m}^{-2}$  pressure were used to embed them in the polycarbonate. The pressure was maintained during cooling, and the specimens were kept for at least 30 days before use, to allow any temporary internal residual stresses in the polycarbonate to relax.

TABLE I Mechanical properties of materials used

	Poisson's ratio	Youngs modulus ( $\text{GN m}^{-1}$ )	Tensile strength ( $\text{MN M}^{-2}$ )	Shear strength ( $\text{MN m}^{-2}$ )	Shear yield stress ( $\text{MN m}^{-2}$ )
Steel	0.29*	210*	$880 \pm 90$	—	—
Polycarbonate	0.37*	2.4*	68*	$63 \pm 2$	$42 \pm 3$
Epoxy	0.34*	2.2*	$50 \pm 3$	$35 \pm 4$	$18 \pm 2$

\*Manufacturers data. Other results come from specimens made for this work.

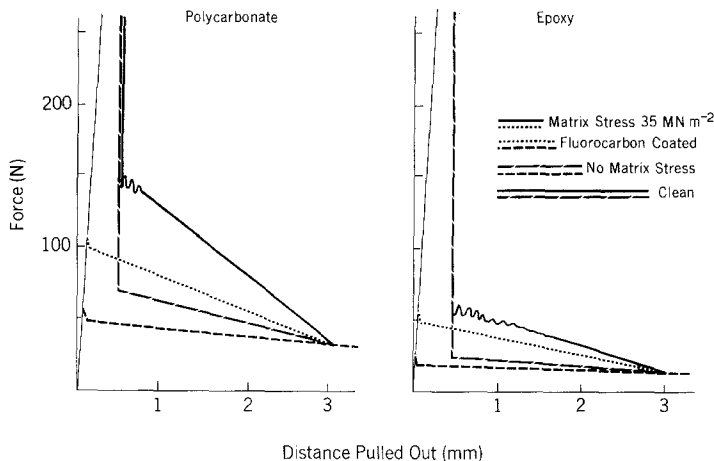


Figure 3 Typical pull-out curves obtained with steel rods in epoxy and polycarbonate matrices.

In the case of the epoxy specimens, the rods were embedded and cured at room temperature. These specimens were also kept for at least 30 days before use.

The shear properties of polycarbonate and epoxy matrices were determined by the ASTM D 732 method.

### 3. Experimental results

Fig. 3 shows typical pull-out curves obtained with rods having clean end regions, and rods coated with fluorocarbon near the ends (as well as the fluorocarbon used above level A). It can be seen that the fluorocarbon has a large effect on the force required to overcome adhesion, and start the sliding process. Matrix stresses increase the frictional forces, but have little effect on the force required to overcome adhesion. The sliding process is not continuous when the matrix is highly stressed.

Visual observation of the specimens indicated that the adhesion near the fibre end was failing when the matrix was stressed, before any force had been applied to the rod by the Instron.

With the epoxy specimens, this only affected the force required to overcome adhesion when the effective embedded length was less than 3 mm. With the polycarbonate, on the other hand, the adhesive strength appeared to increase at the highest applied stress (Fig. 4). Apart from this, the adhesive forces were proportional to surface area embedded, and gave bond strengths of  $42 \text{ MN m}^{-2}$  for polycarbonate and  $25 \text{ MN m}^{-2}$  for epoxy.

Separate tests showed that end effects could be neglected. These tests were carried out by embedding rods which were entirely clean (i.e. no release

agent was used). The matrix applied stress used was zero. The results are plotted as solid circles in Fig. 4, and can be seen to agree reasonably well with the results of the corresponding, partly coated, specimens.

When the rod end regions were coated with fluorocarbon, even the 5 mm embedded length lost its adhesion before pull-out forces were applied in

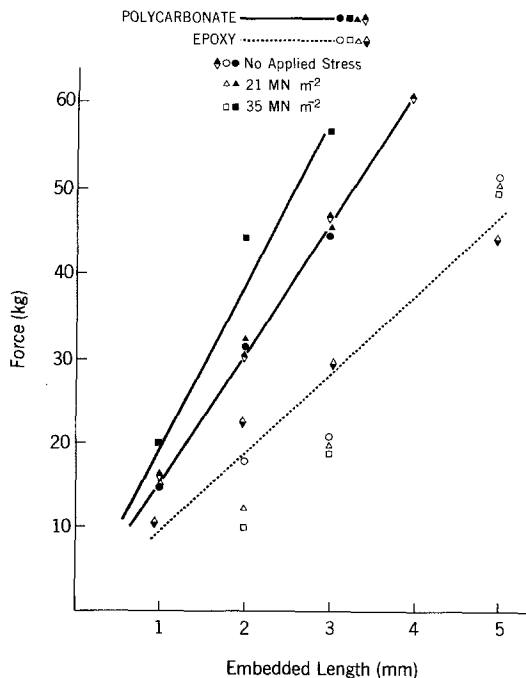


Figure 4 Force required to overcome adhesion and initiate sliding with rods having clean end regions. The points marked  $\diamond$  were taken from experiments in which the whole rod was clean (i.e. no release agent was used at all). The lower sets of points all came from epoxy specimens.

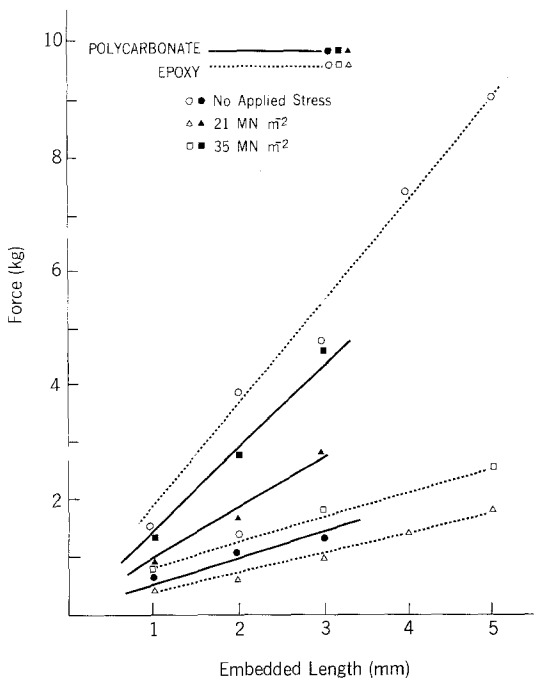


Figure 5 Force required to overcome adhesion and initiate sliding with rods coated with fluorocarbon.

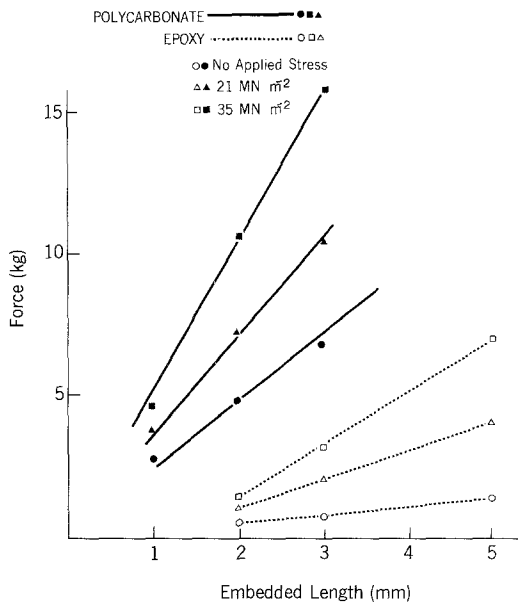


Figure 6 Force required to maintain sliding immediately after the adhesion had failed, for rods with clean end regions.

the epoxy case (Fig. 5). In contrast, the applied stresses increased the force required to overcome adhesion in the polycarbonate case.

The friction forces, measured just after sliding started, increased linearly with embedded length in the case of the polycarbonate matrix and the

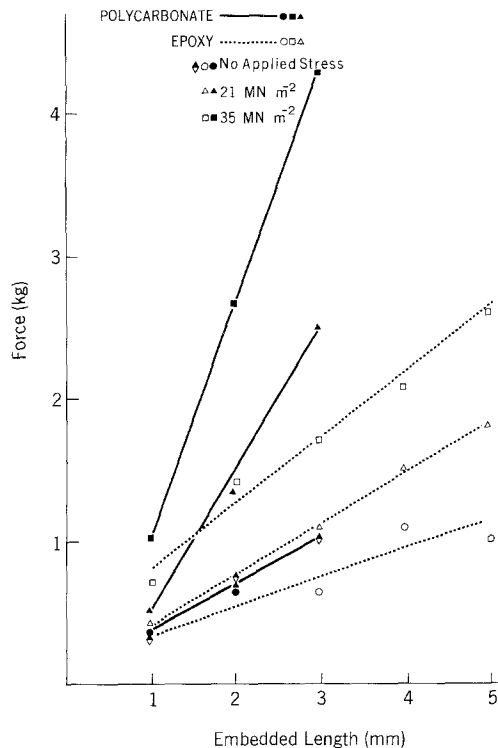


Figure 7 Force required to maintain sliding immediately after the adhesion has failed, for rods coated with fluorocarbon.

results extrapolated to the origin (Fig. 6). This was not true in the epoxy case, however; the coefficient of friction in this case appears to increase with embedded length. Once pull-out has started, the pull-out force decreases approximately linearly with embedded length and falls to zero at the instant of separation. (The wire could not be embedded in the polycarbonate to a depth any greater than 3 mm, since at 4 mm and greater depths complete interface failure occurred when the specimens were cooled to room temperature during manufacture.) When the rod end regions were coated with fluorocarbon, the friction forces were much smaller, and the increase with embedded length was not linear from the origin with either matrix (Fig. 7) except for the unstressed polycarbonate. In all cases, however, the pull-out force decreased approximately linearly with pull-out distance as the fibre was being extracted. Each result plotted in Figs. 4 to 7 is the average from at least four specimens.

When the average frictional forces for the different embedded lengths are plotted as a function of matrix applied stress, the results for coated and clean rods are approximately linear (Fig. 8) and

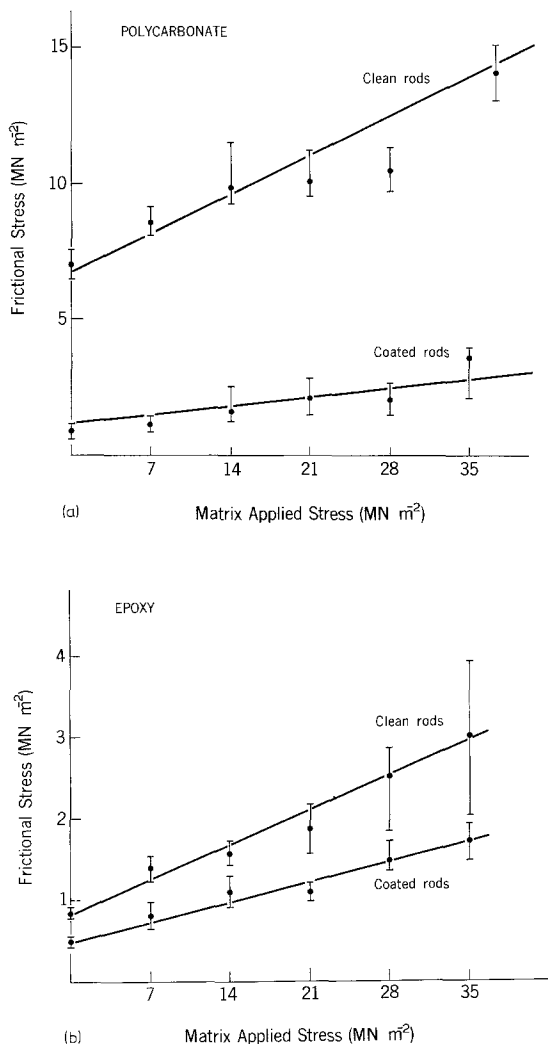


Figure 8 Frictional force as a function of matrix applied stress (a) with polycarbonate matrix, (b) with epoxy resin matrix.

extrapolate to about the same point on the stress axis. The intersection point for the polycarbonate matrix is  $-28 \text{ MN m}^{-2}$ , and for the epoxy it is  $-12 \text{ MN m}^{-2}$ . The coefficients of friction calculated from the slopes of these lines come to 0.6 for polycarbonate and 0.19 for epoxy. When fluorocarbon is used, these figures are reduced to 0.12 and 0.10 respectively.

#### 4. Discussion

The results obtained yield information about the adhesive forces, the frictional forces, and the residual (shrinkage) stresses in the matrix.

#### 4.1. Adhesive strength

With polycarbonate, the apparent adhesive strength was about the same as the shear yield stress of the polycarbonate. That the results were substantially independent of matrix stress, and if anything increase at high matrix stresses is surprising, since it was observed that adhesion failure occurred near the fibre end at high matrix stresses. This reduced the bonded area, and should thus have reduced the total adhesive force. Thus, when the matrix was under stress, the interfacial strength approached, and in one case exceeded, the ultimate shear strength of the polycarbonate. Some polycarbonate was found to be still adhering to the steel after it was pulled out, attesting to the strength of the adhesion.

The results all fell quite close to lines which extrapolated to the origin, indicating that stress concentration effects were not influencing the results very seriously. The epoxy results were not so well behaved however, especially for short embedded lengths. The adhesive strength was lower than for polycarbonate, but still exceeded the shear yield stress of the material. The matrix stresses reduced the apparent adhesion at short embedded lengths but had no significant effects at 5 mm embedded length. This reduction in apparent adhesion was probably largely due to partial interface failure, occurring when the matrix was stressed, before any pull-out forces were applied to the fibre. Since the bonded area was less when the matrix was stressed, the 5 mm results indicate that the matrix Poisson's contractions do increase the adhesive strength over the regions still adhering.

The effect of the fluorocarbon on the adhesive strength was very large, reducing it by a factor of about five for the epoxy, and about forty for the polycarbonate. The matrix applied stress decreased the epoxy "adhesive" strength, but increased that for the polycarbonate. The epoxy, when stressed to  $35 \text{ MN m}^{-2}$ , showed anomalous behaviour, since the results did not extrapolate to the origin, and fell between those for zero applied stress and  $21 \text{ MN m}^{-2}$  applied stress. This may indicate that some small force is being transmitted across the tip of the rod in this case. Such a force, having a value of about 0.5 to 0.7 kg would be sufficient to bring the results into agreement with those at  $21 \text{ MN m}^{-2}$ , and extrapolate to the origin. Apart from this, the effect of stress suggests that the rheological properties of the fluorocarbon are modified by the

matrix due to solvent action or other physical effects.

## 4.2. Friction forces

With clean rods, the initial frictional forces are approximately proportional to embedded length in the stressed and unstressed polycarbonate. This is not the case, however, with the stressed epoxy matrix.

This anomalous behaviour of the epoxy matrix cannot be due to Poisson's shrinkage of the fibres, since this would cause an apparent decrease in friction coefficient as embedded length was increased. Nor could the effect be accounted for by the variation of matrix stress due to stress transfer from the fibres. For example, at the lower end of the specimen, the matrix stress is only 8% higher than at the upper end, due to this process, for the 5 mm embedded length in epoxy stressed to  $35 \text{ MN m}^{-2}$ . Yet the apparent friction coefficient has almost doubled as compared with the 2 mm embedded length.

This anomaly has also been observed by Amirbayat and Hearle [8], and is probably due to the bond failure being progressive, rather than occurring suddenly and completely when the fibre is relatively long. Thus when a long fibre is being pulled out the force initially increases linearly with time as the interfacial shear stresses build up. These stresses are not, of course, constant along the fibre length but have their maximum value at the matrix surface [9]. The failure of the bond occurs first at the matrix surface, and then progresses along the fibre until sufficient of the fibre has been freed. This will be when its elastic extension increases the compliance of the specimen, so that the applied force can fall to a low value. Further displacement of the tensile machine than results in frictional sliding together with further bond failure. The progressive bond failure will contribute an apparent friction force which will increase with embedded length. The magnitude of this effect will depend on the compliances of both testing machine, and specimen. Friction forces can thus only be reliably measured when the embedded length is small.

When the fluorocarbon release agent was used, all the results were anomalous, in that they did not extrapolate to the origin, except for the case of the unstressed polycarbonate. The shear flow behaviour of the release agent is evidently complex, and, like its yielding behaviour, is influenced by

the matrix, giving a higher average coefficient of friction with the polycarbonate (0.12) than with the epoxy resin (0.10).

## 4.3. Residual stresses

In all cases, the frictional forces increase with applied stress, and from the straight lines shown in Fig. 8 the residual stresses may be calculated. Let us assume that the fibre Poisson's contractions can be neglected. The radial stress at the fibre surface is then the sum of the matrix residual stress,  $\sigma_0$ , and the stress resulting from the Poisson's shrinkage of the matrix,  $\nu_m \sigma_m / (1 + \nu_m)$  approximately. (Here  $\nu_m$  = Poisson's ratio for the matrix and  $\sigma_m$  is the stress applied to the matrix.) Thus the fibre pull-out stress,  $\sigma_f$ , is given by

$$\sigma_f = \frac{4l\mu}{d} \left( \frac{\nu_m \sigma_m}{1 + \nu_m} - \sigma_0 \right)$$

where  $l$  is the embedded length and  $d$  the diameter of the steel rod.

Thus, for each matrix, the two lines in Fig. 8 should intersect the matrix stress axis when  $\nu_m \sigma_m / (1 + \nu_m) = \sigma_0$ , if the residual stress is unaffected by the use of the release agent. From the value of the intersection,  $\sigma_m = -28 \text{ MN m}^{-2}$ , for the polycarbonate matrix; we therefore conclude that the residual stress is  $-7 \text{ MN m}^{-2}$  in this case. For the epoxy matrix the corresponding intersection is  $-12 \text{ MN m}^{-2}$  giving a residual stress of  $-3 \text{ MN m}^{-2}$ . The sign indicates that the stresses are compressive. These are equivalent to shrinkages of about 0.3% and 0.15% respectively, which are small compared with the differential thermal contraction between steel and polycarbonate from the melting temperature (1.4%), and the cure shrinkage of the epoxy (2%). A large degree of stress relaxation must have taken place, the final stress being 30% or less of the probable initial value. Even larger stress relaxations have been observed with silica fibre-reinforced aluminium [10]. The polycarbonate shrinkage of 0.3% is also slightly less than the thermoforming shrinkage of 0.5 to 0.7% quoted by the manufacturer.

The average fibre Poisson's shrinkage strains, once adhesion had failed, were always less than 10% of matrix radial strains, so that we are justified in neglecting them.

## 5. Fibre reinforcement

The results may be useful in clarifying the processes that take place in fibre-reinforced materials.

In earlier discussions of frictional stress transfer, for example by Outwater [11], and in the text by Kelly [12] the matrix Poisson's shrinkage has been neglected. That this assumption is not justified is clearly shown by Fig. 8, where frictional forces can increase by a factor of 3 due to the matrix applied stress, as compared with the value at zero matrix applied stress. The fibre Poisson's shrinkage will mitigate this effect, but not where frictional stress transfer is most important, since the fibre stress, becomes very small at the ends. A theory which takes these effects into account has been developed by Piggott [5]. With a knowledge of the coefficient of friction and the matrix shrinkage stress, some estimate can be made of the fibre length required to obtain efficient reinforcement, and avoid the "yield drop" and early failure that occur with short fibre reinforced polymers.

## 6. Conclusions

Matrix Poisson shrinkage has a large effect on the interfacial forces between fibres and matrix, and can cause a three-fold increase in the frictional forces. It also appears to have some effect on the strength of the adhesion between fibres and matrix. However, the measurement of adhesive strength is complicated by partial bond failure caused by the interfacial shears generated due to the stress transfer process. In the absence of matrix stresses, the adhesive strength can equal and sometimes exceed the matrix shear yield strength.

The average coefficient of friction is quite high for steel-polycarbonate (0.6) but not for steel-epoxy resin (0.19). It is much reduced by fluorocarbon release agent. Reliable values for coefficient

of friction are only likely to be obtained with short embedded lengths, owing to the possibility of progressive bond failure during the pull-out process when long fibres are used.

Residual stresses in the matrices are much smaller than expected, and are not likely to contribute very significantly to the fibre-reinforcement process except at very low matrix applied stresses.

## Acknowledgement

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