The Effect of Hydrostatic Pressure on the Fibre-Matrix Bond in a Steel-Resin Model Composite

P. B. BOWDEN

Metallurgy Department, The University, Cambridge, UK

The shear strength of the adhesive bond between a steel wire and an epoxy resin has been measured and it has been found that when the composite is under pressure the strength corresponds closely to the shear yield stress of the resin determined in a plane strain compression test. Discrepancies at low pressure may be due to the nucleation of stress-concentrating cracks. Measurements of the friction stress between the wire and the resin as a function of pressure indicated that the coefficient of friction was 0.5 and that the compressive stress at the interface due to resin shrinkage was 7 Nmm⁻² (1000 psi).

1. Introduction

The properties of fibre-reinforced composite materials are critically dependent on the breaking stress of the bond between fibre and matrix, and also on the friction stress at the fibre-matrix interface during pull-out after the bond is broken. In this investigation both these quantities have been measured in a model system as a function of applied hydrostatic pressure. This extra variable makes it possible to obtain more information about the processes operating. Also the effect of hydrostatic pressure is important in real composites since the stress at the interface can have a hydrostatic component which may affect the shear strength of the interface and hence the properties of the composite. The particular system investigated was a steel wire embedded in epoxy resin.

2. Experimental

2.1. Experimental Technique

The type of specimen used is illustrated in fig. 1a. A length of 0.56 mm diameter wire (24 swg) was embedded centrally in a block of epoxy resin 1.6 mm thick and approximately 10 mm square. The resin block was bonded on both faces to $\frac{1}{8}$ in. thick mild steel plates. The resin used was 60 parts by weight CIBA MY 750, 40 parts by weight plasticiser CY 208 and 10 parts by weight hardener HY 951, cured at 100° C for 30 min. During a test a predetermined pressure was applied to the steel-resin sandwich by a hydraulic ram and anvil as indicated in fig. 1b. The load-extension curve was then determined as the wire (C) 1970 Chapman and Hall Ltd.

was pulled through the resin block. All tests were at a constant cross-head speed of 2 mm min^{-1} .

When the sandwich is compressed the bond between the resin block and the steel plates prevents any lateral expansion of the resin and an internal stress is built up in a direction perpendicular to the compression direction equal to a fraction $\nu/(1 - \nu)$ of the compressive stress applied. For rigid polymers Poisson's ratio, $\nu_{\rm r}$ has a value of about 0.4 for small strains, increasing to 0.5 as the strain is increased [1]. The transverse stress will then be 0.7 or more of the stress in the compression direction. Thus the state of stress inside the resin block is approximately hydrostatic and the stress normal to the interface between the wire and the resin will be everywhere approximately equal to the compressive stress applied.

In addition there will be circumferential shear stresses at the interface between the wire and the resin because of the difference in elastic moduli, but these will be relatively small because the shear modulus of the resin is only 20% of the bulk modulus. There will be no longitudinal shear stresses at the interface under compression alone because the resin undergoes no change in length in this direction.

2.2. Results

Preliminary experiments were carried out using phosphor-bronze wires (24 swg) but it was found that these wires either broke in the grips or pulled out of the resin without breaking at a load of 25 kg, the breaking strength of the wire. Since



Figure 1 Specimen configuration. (a) Perspective view with one plate removed. (b) Testing arrangement.

the object of the experiment was to measure the strength of the bond, it was necessary to use piano wire (24 swg) with a breaking strength of 60 kg.

An example of the type of stress-strain



Figure 2 Variation of the shear stress at the wire-resin interface as the wire is pulled through the resin and the pressure is varied.

behaviour obtained is shown in fig. 2. The load initially increased approximately linearly until at some critical value it dropped suddenly as the bond between wire and resin broke. As straining was continued the load settled down to a value corresponding to the friction stress between the wire and the resin. This friction stress sometimes remained constant, and sometimes oscillated indicating stick-slip motion. The friction stress could be changed by changing the applied pressure and it was found that the initial load necessary to break the bond was also a function of applied pressure.

The results have been analysed in terms of $\tau_{\rm B}$, the shear stress at the wire-resin interface at the instant the bond broke, and $\tau_{\rm S}$, the shear stress at the interface during sliding after breaking the bond. Both quantities were obtained by dividing the measured load by the surface area of the embedded length of the wire. The pressure in the resin was obtained by dividing the load applied by the hydraulic ram by the area of the resin block.

The results obtained from a number of specimens are summarised in fig. 3. It is seen that $\tau_{\rm B}$ increases markedly with pressure over the limited pressure range investigated. The limit was set by the breaking strength of the wire. For measurements at higher pressures it would be necessary to use wire of larger diameter, or to reduce the embedded length of wire which would increase end effects. The friction stress $\tau_{\rm S}$ also increases with an initial gradient of 0.5. The coefficient of friction appears to fall slightly at higher pressures but this is unlikely to be a real



Figure 3 Plot showing the variation with pressure of the friction stress, $\tau_{\rm S}$, the bond breaking stress, $\tau_{\rm B}$, and the resin shear yield stress, $\tau_{\rm Y}$.

effect and is probably due to gross elastic distortion of the resin as the stress approaches its yield stress.



Figure 4 Stress-strain curve for plane strain compression of the epoxy resin at a strain rate of 0.14 min^{-1} .

The shear yield stress of the resin can be determined independently from a plane strain compression test in which a sheet of resin is compressed between parallel dies [2, 3]. Fig. 4 is the stress-strain curve determined in this manner at a strain rate of 0.14 min⁻¹ using sheets of resin 1.4 mm thick and dies 6.1 mm broad. End effects due to the material near the ends of the dies not being constrained to plane strain have been corrected for by carrying out tests using resin sheets of two different widths. It is seen that there is a pronounced yield point at a stress of 88 Nmm⁻² followed by a load drop and subsequent rise as strain hardening occurs. Yield in glassy polymeric materials takes place in shear on planes

that are inclined at approximately 45° to the compression direction at the instant of yield [3, 4] so that a compressive yield stress of 88 Nmm⁻² implies that yield in shear will occur at a stress of 44 Nmm⁻². This value is for shear under a hydrostatic pressure of 44 Nmm⁻² and it is plotted in fig. 3. There will be a significant variation of resin yield stress with hydrostatic pressure [3]. From experiments on a similar epoxy resin it was deduced that the slope of a plot of shear stress against pressure had a gradient of 0.21 [4], and this gradient is indicated by the dashed line.

3. Discussion

3.1. The Shrinkage Stress in the Resin

It can be seen from fig. 3 that the friction stress is finite at zero applied pressure, and will extrapolate to zero only at a negative applied pressure of -7 Nmm⁻². It seems reasonable to suppose that this effect is due to internal stresses in the resin which give rise to a compressive stress of this magnitude at the wire-resin interface. These internal stresses could have arisen both because of the shrinkage that takes place during the curing of the resin and because of differences in thermal contraction between wire and resin during cooling from the cure temperature of 100° C.

3.2. The Strength of the Wire-Resin Bond

It is not certain that the measured average shear stress at the interface at failure, $\tau_{\rm B}$, represents the actual failure shear strength of the bond. Certainly the strength of the bond cannot be less than $\tau_{\rm B}$, but there is a possibility that a shear crack opening up at the point where the wire emerges from the resin concentrates the stress and leads to failure at a mean stress below the actual breaking stress. The free surface of the resin will bulge outwards when the wire is pulled and tensile forces will be set up tending to initiate a crack. Once this crack is present it will concentrate the shear stress at its tip and spread along the wire breaking the bond progressively. The observed rapid increase of $\tau_{\rm B}$ with applied pressure could be because pressure will progressively suppress this mechanism of failure.

Whatever the true value of the shear strength of the bond it cannot be greater than the shear strength of the resin, τ_Y , since this is the maximum load the resin can ever support in shear. Looking at the measured value for τ_Y plotted in fig. 3, and taking into account its expected variation with pressure, it can be seen that τ_B is very close to τ_Y , particularly at high pressures. This agreement is remarkable considering the widely differing nature of the tests, and suggests that the breaking strength of the bond is equal to the shear yield stress of the resin. However, the agreement is bound to be to some extent fortuitous since the yield stress is sensitive to strain rate and the shear strain rate at the interface is not known.

If the thickness of the deforming layer at the interface were known it would be possible to work out a shear strain rate based on the crosshead speed of 2 mm min⁻¹. If the layer were 0.1 mm thick the strain rate would be 20 min⁻¹. But it is doubtful if it is realistic to compute a strain rate assuming a speed of 2 mm min⁻¹ since initially some of the cross-head movement will be taken up elastically elsewhere in the system. A better measure of effective strain rate is the time to failure. The bond between wire and resin broke 15 sec after the start of the test, while in the measurement of yield stress yield occurred 60 sec after the start of the test. If it is assumed that loading was approximately linear in both cases then the difference in strain rate between the two tests was only a factor of four. For this resin the yield stress varies linearly with the logarithm of the strain rate and increasing the rate by a factor of four would increase the yield stress by about 4 Nmm⁻², an increase of 10%.

The bond breaking strength, $\tau_{\rm B}$, is certainly close to the shear yield stress of the resin particularly at high pressures. The discrepancy at low pressures might be explained if the shearcrack mechanism described above operates in this region.

3.3. Fibre-Reinforced Composite Materials

For material reinforced with discontinuous fibres where the matrix is brittle, such as fibreglass, the bond between matrix and fibre is broken at quite low stresses by stress concentration effects and the important parameter determining the properties of the composite is the friction stress, $\tau_{\rm S}$ [5]. The value for the compressive stress in the resin deduced above is only 7 Nmm⁻² (1000 psi) and it would be comparatively simple to double the value of the friction stress by applying a compressive stress of this magnitude to the composite, so producing a stronger and tougher material. This could be done either by winding pre-tensioned fibres around a rod of the composite, or possibly by 520

using some helically-wound fibres incorporated in the composite to generate compressive stresses when the composite is under load (the principle of the rope). It might be possible to increase the resin shrinkage stress by changing the curing treatment, but it is unlikely that this could produce a very large increase.

4. Conclusions

Pull-out measurements under applied hydrostatic pressure can give useful information about the properties of the fibre-matrix interface. The shear strength of the bond between metal wires and epoxy resin is close to the shear yield strength of the resin as deduced from a plane strain compression test if the effect of stressconcentrating cracks is suppressed. The friction stress between wire and resin after the bond is broken is due to a compressive stress at the interface of approximately 7 Nmm⁻², and consequently quite small changes in hydrostatic pressure, due either to external applied stresses or to internal stresses in the composite, can lead to large changes in the friction stress.

Acknowledgement

This work was carried out during a visit to the Division of Inorganic and Metallic Structures at the National Physical Laboratory, Teddington. I am very grateful to Dr A. Kelly both for making the visit possible and for many stimulating discussions; also to other members of the Division, in particular Dr G. C. Cooper, for discussion, help and advice.

References

- 1. I. KRAUSE, A. J. SEGRETO, H. PRZIREMBEL, and R. L. MACH, *Mat. Sci. Eng.* 1 (1966) 239.
- 2. J. G. WILLIAMS and H. FORD, J. Mech. Eng. Sci. 6 (1964) 405.
- 3. P. B. BOWDEN and J. A. JUKES, J. Mater. Sci. 3 (1968) 183.
- 4. *Idem*, paper presented at the Conference on the Yield, Deformation and Fracture of Polymers at Cambridge, March 1970.
- 5. V. R. RILEY and J. L. REDDAWAY, J. Mater. Sci. 3 (1968) 41.

Received 15 February and accepted 18 February 1970.