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Multiple Fracture in a Steel Reinforced Epoxy Resin Composite

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Specimens of an epoxy resin reinforced by steel wires have been made to fail by a process which involves extensive cracking of the matrix before the UTS of the composite is reached. Such behaviour can result in energy being absorbed by the composite under constant or rising load conditions even when the composite is composed of two brittle phases. The influence of fibre size and volume fraction on the cracking process have been examined, and it has been shown that for low fibre volume fractions and large fibre sizes, the cracking process is governed by a simple relationship. When the fibre size becomes small, or the volume fraction becomes large, the cracking process is hindered, and this relationship breaks down. Under extreme conditions, cracking of the matrix can be completely suppressed and the matrix can be forced to exhibit properties markedly better than it would have shown when tested by itself. The design of materials which behave in this way may provide an important means for producing ceramic-matrix compositions with very good mechanical properties.

1. Introduction

Many of the new composite materials which have been developed for use in engineering rely for their reinforcement upon fibres which, although strong and stiff, are seldom ductile. This lack of ductility is often carried over into the composite and leads to problems of low fracture toughness and difficulty in the design of structures. Some progress has been made in developing composites which are notch-resistant even though they are not ductile [1, 2] but the difficulty of designing with a non-ductile material remains. The problem lies in making structures which are inherently stable, even under conditions of accidental overload, i.e. of designing so that a "shakedown state" is possible, and that progressive deformation occurs under rising-load conditions. This is most easily achieved if the material is not brittle, but itself has a capacity for deforming and absorbing energy under rising-load conditions (like a work-hardening metal). Elsewhere [3] we have considered the various types of fracture which a composite material can show, and conclude that it is possible to obtain deformation with the absorption of energy under constant or rising load conditions in a composite in which the matrix has a lower failure strain than that of the fibres. The energy is absorbed in causing multiple fracture of the matrix, and this may occur even if neither fibre nor matrix is itself ductile.

In this paper, we report the results of experiments on composites which show multiple fracture of the matrix before the UTS. We have observed the absorption of energy by the composite under rising-load conditions, due to the multiple fracture process, and we have investigated the variation in matrix crack spacing with fibre size and volume fraction.

2. Experimental

Experiments were conducted upon composites made of steel wires or rods embedded in an epoxy resin. Our initial studies were made with a resin supplied by Messrs CIBA (ARL) Ltd, and composed of 60 parts by weight of resin MY750, 40 parts plasticiser CY208 and 10 parts hardener HY951.

Specimens were made by two methods depending upon whether the wire diameter was greater

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or less than 0.3 mm. In all cases the steel was thoroughly cleaned with detergent and hot water, and then with alcohol, before fabrication. Specimens with large wires or rods were made by casting in an open silicone rubber mould, the fibres being placed by hand in a uniformly spaced single row.

Specimens reinforced by the thinner wires were made on a coil winding machine provided with a brass drum of 175 mm diameter. The drum was coated with a film of PTFE release agent, and then a layer of resin was spread on the drum and cured. A single layer of steel "piano wire" was then laid down and a further coating of resin applied to enclose the wires. By varying the thickness of the two resin layers and the spacing of the wires, a series of composites was made with fibre volume fractions in the range 10 to 70%. Attention was paid to maintaining geometrical similarity in the composites when changing the fibre volume fraction by making proportionate increases or decreases in both the fibre spacing and resin thickness simultaneously. By making an axial cut through the composite cylinder it could be peeled from the drum as a single sheet of overall dimensions 60 × 550 mm. From these sheets were cut specimens of dimensions 10 × 200 mm for tensile testing.

The testing was carried out in an "Instron" machine fitted with a "Polanyi cage" so that the specimen could be immersed in liquid nitrogen during the test. The elongation to failure of the resin at room temperature was of the order of 50%, because of the presence of the plasticiser, and since this was considerably greater than the failure strain of the wire, single fracture was observed at room temperature. At liquid nitrogen temperatures, however, although the wire was embrittled, the resin proved to have an even lower failure strain, and so multiple fracture of the resin was obtained, before failure of the wires. A typical stress-strain curve for a low-temperature test is shown in fig. 1, in which the characteristic drops in load caused by the progressive fracture of the matrix can be seen. On unloading the specimen from any stress between the "yield point", at which the first fracture of the matrix was observed, and the UTS, a permanent elongation of the specimen was recorded.

In the present case, the permanent set derived partly from a slight ductility in the wires which persists even at 77 K, and partly from a hysteresis effect which is inherent in the multiple

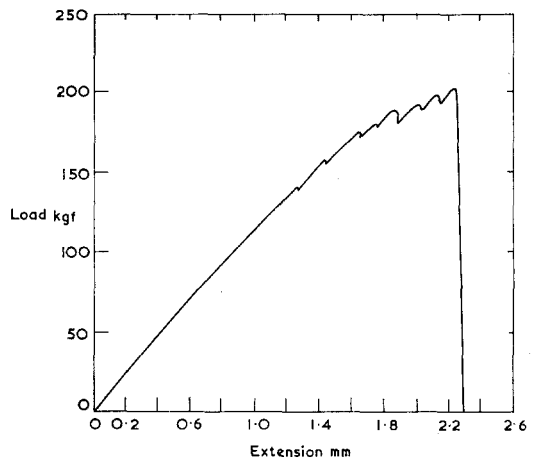

Figure 1 Load-extension curve for specimen of steel-reinforced epoxy resin, test at 77 K.

fracture process. (This arises because of the frictional stresses which exist between the fibres and the blocks of matrix through which they pass; on unloading, the wires contract, but this contraction is opposed by the frictional stresses. The result is that at zero load, the blocks are in compression, the fibres are in tension, and there is a net permanent extension of the specimen. The magnitude of this effect is discussed in greater detail in another article [4].)

After testing, the specimen was removed and the spacing between the matrix cracks was measured A micrograph of a cracked specimen is reproduced in fig. 2 and a histogram of the crack spacings measured on the same specimen is shown in fig. 3. The data have been plotted both as the crude number of spacings observed in the given interval (stippled), and also as the number in the interval multiplied by the mean value of that interval (hatched). The latter treatment removes the skewness associated with any population which has been produced by a process of progressive breakdown (since each single large piece which breaks produces two of the smaller size, and the population is thus always biased towards the smaller values). It will be noted that the great majority of values lies in the range 0.3 to 0.6 mm. This is in good agreement with the theory, which requires the distribution of cracks to be such that the maximum spacing is no greater than twice the minimum (see discussion, below). Exceptionally high or low values were nearly always associated with crack branching, several examples of which may be seen in the micrograph.

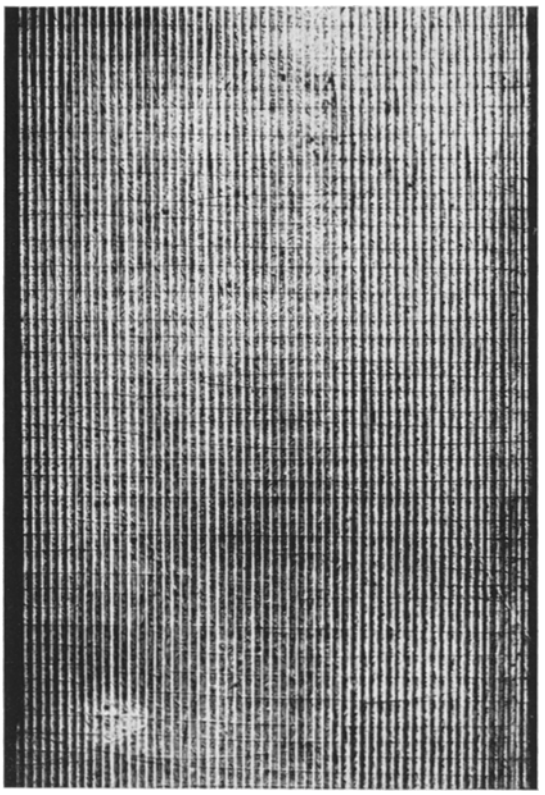

Figure 2 Specimen after test, showing multiple matrix cracking (fibre radius 0.06 mm, $V_f = 0.33$) (× 6).

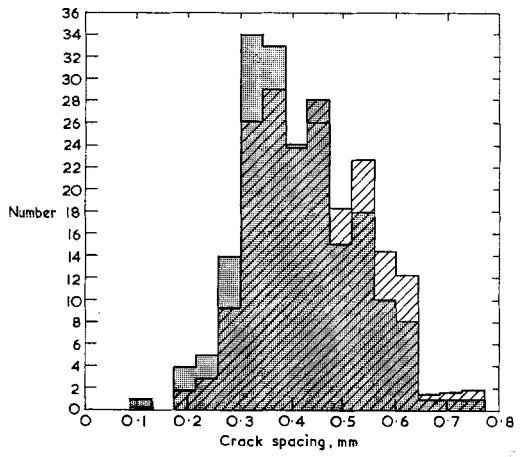

Figure 3 Histogram of crack spacings measured on the specimen shown in fig. 2.

After several specimens had been made and tested in this manner, it was discovered that the resin could be modified in such a way as to reduce the failure strain even further, by reducing the quantity of hardener to 5 parts of HY951 in the formula given above. (At normal temperatures, such a departure from the normal composition of the resin produces a plasticising effect, since the number of cross-links is reduced. At 77 K, however, all compositions are completely brittle, and the effect of reducing the quantity of hardener is to reduce the failure strain by reducing the strength.) When composites made from this resin were cooled slowly to liquid nitrogen temperature, multiple fracture of the matrix could be obtained as a consequence of the differential thermal contraction between wire and resin. The contraction was greater for the resin and amounted to approximately 1.3% linear strain at 77 K.

Following this discovery, all our subsequent experiments on the variation of the crack spacing with fibre volume fraction or size were performed using this method.

The experimental technique consisted of holding the specimens on a metal frame in the vapour of boiling liquid nitrogen, the whole being contained in an insulated vessel. The progress of the cracking was followed by observation through a binocular microscope, or with the naked eye in the case of the specimens with large fibres, where the cracking was on a much coarser scale.

A series of tests was made to measure the variation of crack spacing with fibre volume fraction at constant fibre radius. This procedure was then repeated for different fibre radii, so that in all, data were obtained from six different systems, having fibre radii of 1.18, 0.59, 0.39, 0.14, 0.06 and 0.05 mm.

Some mechanical properties of the resin were measured at room temperature and at 77 K. The tests at room temperature were carried out in tension in the normal manner on specimens 100 mm long × 12 mm square, at a strain rate of 10% per min. Tests at the low temperature were made both in tension and bending.

The tensile tests were carried out on waisted specimens with a central portion 5 mm in diameter and 40 mm in length. The test pieces were gripped at room temperature and slowly cooled (to prevent cracking by thermal shock) by enclosing them in a polythene cover and introducing the vapour of boiling liquid nitrogen.

Specimens 40 mm long × 6 mm square were used for the tests performed in the 3-point bending mode. These specimens were also cooled in the vapour of boiling liquid nitrogen, on a frame

similar to that used for making observations on the composites. They were then quickly transferred to the test rig, which was also cooled with liquid nitrogen, and the test carried out. Results obtained from these and the tensile tests are shown in table I.

TABLE I

Temp. °C	σ ult.m MN/m²	E MN/m²	ϵ ult.	Test method
20	0.176	0.264	0.65	Tensile
− 196	10.25	790	0.013	Tensile
− 196	17.5	2370	0.007	Flexure (3-point bend)

An attempt was made to measure the work of fracture of the resin at 77 K by means of a Hounsfield Plastics Impact Machine, using specimens prepared as described above for the 3-point bend tests. However, the fracture energy was extremely low and was small compared with losses inherent in the test, e.g. energy lost in vibration and kinetic energy given to the broken test pieces. The *apparent* fracture energy determined in these tests was 120 J/m².

Another estimate was made using a method described by Tattersall and Tappin [5]. This method is similar to a 3-point bend test but here the material is severely notched, to ensure that the failure crack begins to propagate before the bulk of the specimen is significantly stressed. In this way, the stored elastic energy in the specimen is kept at a low level, and conditions are favourable for slow crack growth. Under such conditions, the fracture energy can be found directly from the total area under the load-extension curve divided by the area of crack surface produced.

For these tests a 500 kg maximum load cell (hardest available) was used, on its lowest range, fitted to a bench model Instron. The specimens used were 140 mm long × 12.5 mm square. The two outer supports in the 3-point bend test rig were 89 mm apart and the load was applied centrally between them. It was found that a specimen with a transverse notch of uniform depth had a lower failure load than when a triangular shaped remainder was used (as in the original experiments of Tattersall and Tappin). In later work by Davidge and Tappin [6], however, a parallel notch was also preferred. Using this method we obtained a value of 1.75 J/m² for the fracture surface energy and consider it a better estimate than the value of 120 J/m² found using the impact machine.

3. Results and Discussion

3.1. Normal Behaviour

Multiple fracture in a brittle-matrix composite leads to a progressive fracturing of the matrix on planes normal to the fibre direction. The limiting spacing of these cracks can be deduced by considering a block of matrix, of unit cross-sectional area, and height $2x$, traversed by a number of parallel fibres. As load is applied to the fibres and they elongate, stress is transferred to the block by shear stresses τ at the fibre-matrix interface. If we assume that τ has a constant limiting value, then the stress on the mid-plane of the block will reach the fracture stress if:

$$2\pi rN\tau x = (1 - \pi r^2 N)\sigma_m \qquad (1)$$

where N is the number of fibres in unit cross-section, r is their radius and σ_m is the strength of the matrix. As the matrix is broken down, a block of height just over $2x$ will be cracked into two pieces of height just greater than x, whereas one of just under $2x$ will remain unbroken. Thus, when the fracturing of the matrix is complete, it will be broken into blocks of height between x and $2x$, where x is given by (rearranging equation 1):

$$x = \left(\frac{1 - V_f}{V_f}\right)\frac{\sigma_m r}{2\tau} \qquad (2)$$

In order to check the validity of this equation, we plotted our observed crack spacings against the factor $([1 - V_f]/V_f)$, since the crack spacing is proportional to x. The result obtained for most of the fibre radii was a straight line, passing near, if not through, the origin. An example is shown in fig. 4 for wires of radius 0.59 mm. The relationship obtained with the two smallest sizes (radii 0.05 and 0.06 mm) was not so simple, however (figs. 5 and 6). At low volume-fractions of fibre, i.e. at high values of $([1 - V_f]/V_f)$, a straight-line relationship was obtained, although the extrapolated line did not pass through the origin. At higher volume fractions, the observed crack spacings were larger than predicted and at the highest loadings of fibre the matrix was completely uncracked. We have considered whether this suppression of cracking was due to abnormal conditions of curing or some other chance effect in these specimens but we have since prepared sheets of material by the coil winding technique described above using a final resin coat with local variations in thickness. On cutting out a specimen with both thick and thin regions in it, and cooling in liquid nitrogen in the normal way, we have seen cracks form in the thicker regions

(lower volume-fractions) which run towards the thinner regions, but do not enter them. We have also been able to take specimens with a high volume fraction ($\sim 60\%$) of fine wire and plunge them directly into liquid nitrogen without them cracking; the normal effect is to produce extensive cracking, if not disintegration, of the specimen.

We have also examined the validity of equation 2 as a function of fibre radius. It was not possible to make specimens of a predetermined fibre volume fraction with our techniques, so we have no direct comparison of crack spacing with fibre size at constant fibre content.

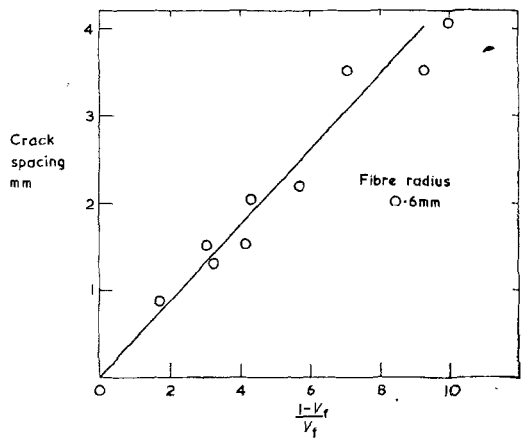

Figure 4 Variation in crack spacing with $([1 - V_f]/V_f)$; fibre radius 0.59 mm.

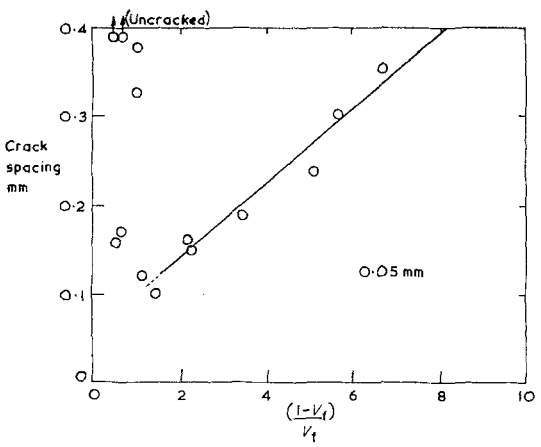

Figure 5 Variation in crack spacing with $([1 - V_f]/V_f)$; fibre radius 0.05 mm.

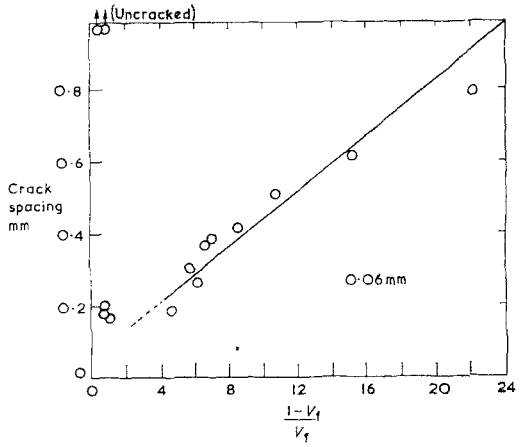

Figure 6 Variation in crack spacing with $([1 - V_f]/V_f)$; fibre radius 0.06 mm.

By differentiating equation 2, however, we obtain:

$$\frac{\partial x}{\partial\left(\frac{1-V_f}{V_f}\right)} = \frac{\sigma_m r}{2\tau} \tag{3}$$

and so we have plotted the gradients of the family of curves exemplified by figs. 4, 5 and 6 against fibre radius, and this is shown on fig. 7. A straight line fit was obtained, passing through the origin, with gradient 0.68. (Although it should be borne in mind that in figs. 5 and 6 the gradients were measured in the "normal" region, and so the abnormal behaviour with small fibres at high volume fractions is disguised.)

It was noted above that the crack spacing should be between x and $2x$, so we have assumed that our measured average crack spacing, C, is close to the arithmetic mean, i.e. $C = 1.5\ x$. Thus, from equation 3,

$$\frac{\partial C}{\partial\left(\frac{1-V_f}{V_f}\right)} = \frac{3\sigma_m r}{4\tau} \tag{4}$$

and so the gradient of the line in fig. 7 should be $\frac{3}{4}\ \sigma_m/\tau$. From our measured gradient we obtain the ratio $\sigma_m/\tau = 0.91$.

Although we have alternative values for the strength of the resin at low temperature we consider that obtained in flexure, 17.5 MN/m² (2537 psi) to be more applicable considering the susceptibility of the material to surface defects and the possibility of stress concentrations from internal defects such as small bubbles. Sims [7] measured the shear strength of the steel-resin

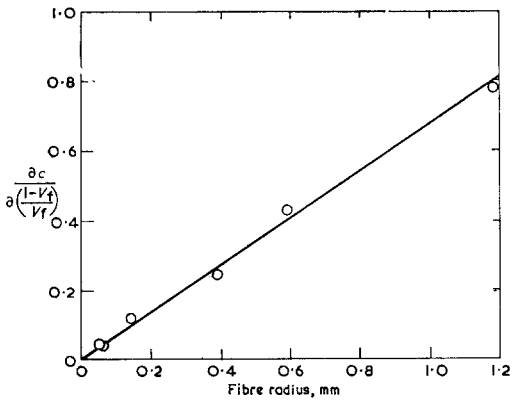

Figure 7 Variation of gradients from the family of plots exemplified by figs. 4, 5 and 6 against fibre radius.

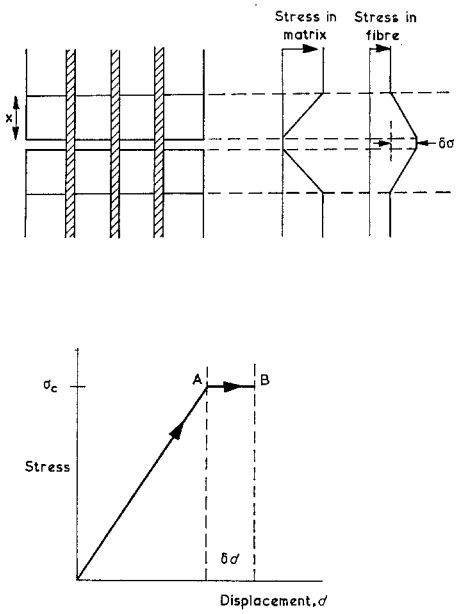

Figure 8 Stress distribution and the change in the stress-displacement curve after the formation of a single matrix crack.

interface at 77 K by means of a pull-out test and obtained a value of 8.0 MN/m² (1120 psi). Using these direct measurements we obtain a value of 2.19 for σ_m/τ.

Thus, in the conditions of normal behaviour considered above, i.e. low volume fractions, and large fibre sizes, we conclude that equation 2 gives an aedquate description of the fracture of the brittle matrix, both for variations in fibre size and volume-fraction.

3.2. Abnormal Behaviour

In this section, we examine the reasons for the suppression of matrix-cracking under conditions of small fibre size and high concentration. This theory has been developed in conjunction with J. Aveston and A. Kelly and is published in detail elsewhere [4]. It is reproduced here for convenience. We consider the energy balance which governs the formation of a single crack in the matrix, at strain ϵ_m, and under conidtions of fixed load. In fig. 8 is sketched the simplified distribution of stress in fibres and matrix near such a crack, and the effect on the load-extension curve of the composite when the crack forms. We assume that after the formation of the crack, the stress in the matrix is relaxed on either surface of the crack, and builds up to its former value (σ_m) at a distance x on either side of the crack, the value of x being given by equation 2.

Over this same region, the fibres carry an additional stress which has a maximum value:

$$\delta\sigma = \sigma_m \frac{V_m}{V_f} \qquad (5)$$

This increment of stress causes an additional extension of the composite under conditions of fixed load:

$$\delta d = \frac{2x}{2E_f} \cdot \delta\sigma \qquad (6)$$

Terms in the energy balance which govern the formation of the matrix crack include:

1. The work done by the applied stress in extending the composite. If the stress on the composite when the crack forms is σ'_c, this energy is (per unit area of fracture surface)

$$\Delta W = \sigma_c' \, \delta d. = E_f V_f \epsilon_m^2 x \alpha(1 + \alpha) \qquad (7)$$

where

$$\alpha = \frac{E_m V_m}{E_f V_f}$$

2. The matrix is relaxed in the volume extending a distance x on either side of the crack. This leads to a release of strain energy:

$$\Delta U_m = E_f V_f \epsilon_m^2 x \frac{2\alpha}{3} \qquad (8)^*$$

3. The fibres are extended in the same region. The fibres therefore absorb energy:

$$\Delta U_f = E_f V_f \epsilon_m^2 x \alpha \left(1 + \frac{\alpha}{3}\right) \qquad (9)$$

*The detailed derivations of this equation will be published by A. Kelly in a forthcoming issue of Composites.

4. Because the matrix contracts and the fibres extend, there is a relative displacement between them. If we assume a constant sliding frictional stress τ at the interface and a negligible debonding energy, the energy loss can be shown to be:

$$U_s = E_f V_f \epsilon_m^2 x \frac{\alpha}{3}(1 + \alpha) \qquad (10)$$

5. Work must be done to fracture the matrix as the crack forms. If the fracture surface energy is γ_m, this amounts to $2\gamma_m V_m$ per unit area of composite.

The energy balance is thus:

$$\Delta W + \Delta U_m \geqslant \Delta U_f + U_s + 2\gamma_m V_m \qquad (11)$$

for the cracking to be a spontaneous process.

Collecting the first four terms in equation 11, it becomes:

$$E_f V_f \epsilon_m^2 x \frac{\alpha}{3}(1 + \alpha) \geqslant 2\gamma_m V_m \qquad (12)$$

Thus, noting that the term $E_f V_f \epsilon_m^2 x$ is common to equations 7 to 10, we see that one third of the work done by the applied stress (ΔW) is available to produce the matrix crack. (One third goes in overcoming frictional stresses, and the other third, together with the energy derived from the relaxation of the matrix, is stored as elastic strain energy in the fibres.)

Taking equation 2, and putting it in the form

$$x = \frac{E_f \epsilon_m \alpha r}{2\tau}$$

and substituting it in equation 12 we have:

$$2\gamma_m V_m \leqslant \frac{E_c E_f \epsilon_m \alpha^2 r}{6\tau} \qquad (13)$$

Thus, if other variables are held constant, it is clear that by reducing the fibre radius or by increasing the fibre volume fraction (since $\alpha = (E_m V_m)/E_f V_f$), it should be possible to reach a condition where it is energetically unfavourable to form a crack in the matrix at its normal breaking strain.

In the case of cracking caused by thermal stresses, the method used to produce multiple fracture in many of our experiments, a similar argument can be applied. Multiple fracture of the matrix can be expected on cooling if the thermal expansion coefficient of the matrix, a_m, is greater than that of the fibres, a_f. If the fibres and matrix were not bonded together, the differential strain between them would be:

$$\epsilon_t = (a_m - a_f)\Delta T$$

If they are bonded, there is no differential strain, but the fibres are in compression and the matrix is in tension. At the moment that the matrix cracks, the tensile strain in the matrix equals ϵ_m, and a simple force balance then requires that the tensile load in the matrix, $E_m \epsilon_m V_m$ equals the compressive load in the fibres, $E_f V_f(\epsilon_t - \epsilon_m)$. Thus:

$$\epsilon_m = \frac{E_f V_f \epsilon_t}{E_c} \qquad (14)$$

and the degree of cooling necessary to cause fracture of the matrix can be found from the expression

$$\Delta T = \frac{\epsilon_t}{(a_m - a_f)} = \frac{E_c \epsilon_m}{E_f V_f (a_m - a_f)} \qquad (15)$$

An energy balance similar to that of equation 11 can then be constructed, taking note of the following factors:

1. Since the composite is not stressed externally, ΔW is zero.
2. The energy released by the relaxation of the matrix ΔU_m is the same as that given in equation 8.
3. The fibres are initially in compression, at a uniform strain of $\epsilon_t - \epsilon_m = \alpha \epsilon_m$ (compressive). When the matrix fractures, the fibres relax to zero stress at the plane of the break. Making the usual assumption of uniform shear stress at the interface, the amount of strain energy lost by the fibres can be shown to be:

$$\Delta U_f = E_f V_f \epsilon_m x \frac{2\alpha^2}{3} \qquad (16)$$

4. The relative displacement between fibres and matrix after fracture is the same as under conditions of fracture in tension, so U_s is as given by equation 10.
5. The fracture surface energy term is assumed to be the same as before, and is taken as equal to $2\gamma_m V_m$.

The energy balance thus becomes:

$$\Delta U_f + \Delta U_m \geqslant U_s + 2\gamma_m V_m \qquad (17)$$

and collection of the first three terms gives:

$$E_f V_f \epsilon_m^2 x \frac{\alpha}{3}(1 + \alpha) \geqslant 2\gamma_m V_m \qquad (18)$$

which is identical to equation 12.

In order to compare equations 12 or 18 with our experimental findings, we have substituted for x and α, and obtain:

$$r < \frac{12\gamma_m \tau E_f V_f^2}{E_c E_m^2 \epsilon_m^3 V_m} \qquad (19)$$

as the condition for the suppression of matrix cracking at ϵ_m.

In our particular system $E_f \gg E_m$ and therefore E_c is approximately equal to $E_f V_f$ and equation 19 becomes:

$$r < \frac{12 \gamma_m \tau V_f}{E_m^2 \epsilon_m^3 V_m}$$

Taking our experimental values of $\tau = 8 MN/m^2$, $\gamma_m = 1.75 J/m^2$, $E_m = 2370 MN/m^2$ and $\epsilon_m = 0.007$ we obtain a value of $r < 0.09$ mm (at $V_f = 0.5$) from the above equation which, it should be noted, is not a general expression. Again we have chosen the flexure values as an estimate of matrix properties to be more significant than our tensile values. The strain, and hence the modulus, in the tensile test is believed to be inaccurate because the measured value is the same as the applied differential strain and observation shows that the cracking process starts before the specimens have reached their lowest temperature.

Assuming a lower volume-fraction of fibres gives a correspondingly smaller value for the critical radius. Substitution of the value $V_f = 0.2$ for example decreases r by a factor of four. Experimentally, we observed crack suppression at fibre radii of 0.05 and 0.06 mm, and for fibre concentrations greater than about 50% in each case (see figs. 6 and 7). In view of the uncertainties in the determination of γ_m and τ, we conclude that the agreement with the theoretical predictions is quite satisfactory.

4. Conclusions

In this paper, we have described experiments to investigate the behaviour of a composite in which the matrix is more brittle than the fibre. Final failure in tension is preceded by multiple fracture of the matrix. The theory suggests that this progressive failure, which takes place when the strain is increased beyond ϵ_m, should occur at constant load. This is not borne out in the shape of the load-extension curve, as in fig. 1, which shows the matrix failure occurring for rising load on the composite. The divergence between the expected and actual results may be explained by considering the value for the strength of the matrix which, as is common with most brittle materials, is not well defined. This spread of values for σ_m could lead to the type of curve shown in fig. 1. We have investigated the factors governing the multiple fracture process, and have shown that for composites reinforced by large fibres in low concentrations, the fracture is governed by a simple equation. When fine fibres are present in high concentrations, however, the cracking process is hindered, and under extreme conditions, it can be completely suppressed.

We have attempted to account for this behaviour by means of a simple theory, which predicts that at a particular concentration of fibres, there will be a fibre size below which the matrix will not fail at its normal fracture strain.

It should be noted, however, that this condition does not mean that the matrix will never crack. As the applied stress on the composite is increased, the energy stored in it increases, and so the terms ΔW, ΔU_m, ΔU_f and U_s in the energy balance all change with a net effect of making the cracking more easily achieved. Rearrangement of equation 19 enables the expected failure strain of the matrix to be calculated for any given fibre size or concentration

$$\epsilon_m = \left(\frac{12 \gamma_m \tau E_f V_f^2}{E_c E_m^2 V_m r} \right)^{1/3} \qquad (20)$$

For suitably chosen values of r and V_f, this expression can give values of ϵ_m which are either smaller or greater than the normal breaking strain of the matrix. If the strain predicted by equation 20 is less than the normal failure strain of the matrix, "normal" multiple fracture will be observed at the normal failure strain of the matrix; if equation 20 predicts a greater strain, then multiple fracture will not occur until this strain is reached.

There is a possibility then, of designing a brittle matrix composite with a fixed yield strain – i.e. the normal fracture strain of the matrix – and some degree of fracture toughness; or, by adjustment of V_f and r, ϵ_m can be increased until ϵ_f is reached, at which stage the composite will fail in a single fracture mode. It is probable that the condition of equation 20 may have been exceeded in composites such as carbon fibre glass and experimentalists have unwittingly obtained improved matrix properties.

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