

increase in long period due to the anneal measured for each.

It is apparent from Fig. 1 that the irradiation has no detectable effect on the increase in long period due to annealing. In quantitative terms the ratios of the means of the irradiated and unirradiated long periods at each annealing temperature average out at 1.002.

An interesting feature of the curve is the slight "knee" apparent at a long period of approximately 200 Å. The error bars shown represent 90% confidence limits calculated from values of the standard deviation of the mean for each temperature. It is not possible to draw a curve of continuously increasing slope (i.e. without the "knee") within the bounds of these confidence limits.

The "knee" could be interpreted in terms of a doubling of the thickness of the crystallites associ-

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ated with the operation of the chain unlooping mechanism.

References

1. A. H. WINDLE, *J. Mater. Sci.* **10** (1975) 1959.
2. A. CHARLESBY, "Atomic Radiation and Polymers" (Pergamon, 1960).
3. D. H. RENEKER, *J. Polymer Sci.* **59** (1962) 39.
4. P. DREYFUSS and A. KELLER, *Polymer Letters* **8** (1970) 253.

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Poisson contraction effects in aligned fibre composites

The phenomenon of multiple cracking that is observed under certain conditions in fibre-reinforced brittle matrix composites has generated a great deal of interest in studies of fibre pull-out. The frictional force developed between the fibre and matrix resisting pull-out after debonding results in increased post-cracking strength and multiple fracture. The effect of Poisson contraction of the fibre and matrix before and after matrix cracking has been the subject of a recent letter by Kelly and Zweben [1]. A theoretical treatment considering composite behaviour in three dimensions has been performed in our laboratory and leads to conclusions very different from those of Kelly and Zweben. In this note the results of our investigation will be presented and the differences in the two treatments considered. The results will also be applied to a number of practical examples treated in the earlier paper.

Consider an ideal aligned continuous fibre composite consisting of fibres of radius r_f and Poisson's ratio ν_f arranged in a hexagonal or square array (Fig. 1). Each fibre is surrounded by a cylinder of matrix of radius r_m and Poisson's ratio ν_m . As pointed out by Kelly and Zweben and by Hill [2] in the absence of any matrix shrinkage in speci-

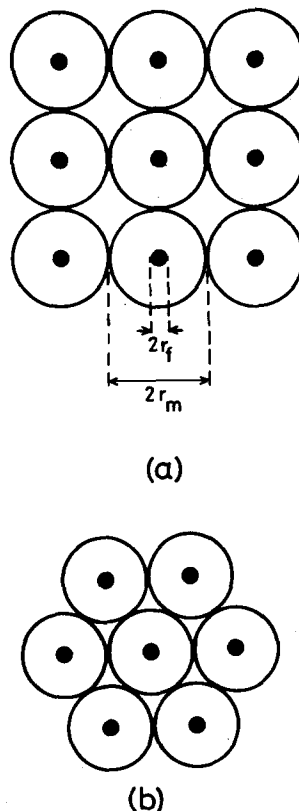


Figure 1 Idealized fibre arrays in composite: (a) square array, (b) hexagonal array. Larger circles (radius r_m) represent imaginary cylinders of matrix associated with each fibre.

men preparation, the interfacial traction when the composite is stretched by an axial strain e is a normal stress p given by

$$p = \frac{2e(\nu_m - \nu_f)V_m}{\left[\frac{V_m}{k_f} + \frac{V_f}{k_m} + \frac{1}{G_m}\right]} \quad (1)$$

where V is the volume fraction, k is the plane strain bulk modulus and G is the shear modulus. The subscripts f and m refer to fibre and matrix.

When the strain in the ideal composite described above exceeds the matrix failure strain, fracture of the matrix will occur. For simplicity we consider a single crack in the matrix across the specimen. The stress in the matrix at this crack surface will be zero and the fibres bridging the crack will carry the load formerly carried by the matrix. We wish to consider the conditions under which the embedded fibre and matrix will remain in contact by considering the displacement of the fibre and matrix separately.

Before the crack is formed and far from the crack the fibre and matrix have undergone the same strain e and the fibre will have a Poisson contraction of $\nu_f e$. At the crack the fibre has undergone an additional strain $(E_m V_m / E_f V_f) e$ and an additional Poisson contraction $\nu_f (E_m V_m / E_f V_f) e$.

The displacement of the inner cylindrical surface of the matrix near the crack surface is more difficult to treat. Before cracking, when the composite is strained, each matrix element shown in Fig. 1 can contract freely with no constraint from the surrounding matrix. The only constraint that may exist is that of the central fibre if an interfacial pressure develops (i.e. if $\nu_m > \nu_f$). If the cylinder of matrix material in Fig. 2 is subjected to an axial strain e the displacement at the outer surface is given by

$$U_{r_0} = -\nu_m r_0 e. \quad (2)$$

After matrix cracking, if the matrix could freely relax, it would return to its original unstrained configuration and the hole for the fibre would return to its original size. This would always result in the loss of fibre/matrix contact. In a composite, however, after the matrix has cracked the fibres bridging the crack resist the free expansion of the matrix at the crack surface. In the middle of an array of fibres as in the bulk of the

composite, the matrix element is restrained by the neighbouring elements which are also attempting to expand laterally. In the idealized aligned composite of Fig. 1 the restraint of the surrounding matrix causes a pressure p_0 at the outer surface of the matrix cylinder and the points on the outer surface of the matrix cylinder are fixed by this restraint. To determine the resultant displacement of the inner surface of the matrix element consider the element shown in Fig. 2. If it is (1) subjected to an axial strain e with no constraint at the inner or outer surface and then (2) the axial stress is removed while the outer surface is held fixed, the displacement of the inner surface can be determined from the results in Love [3] for a tube under pressure with the following boundary conditions:

- (i) $\sigma_z = 0$, the net axial stress is zero, i.e. at the crack surface;
- (ii) $p_1 = 0$, the pressure at the inner surface at the matrix cylinder is zero if the fibre and matrix lose contact;
- (iii) $U_{r_0} = -\nu_m e r_0$, the displacement at the outer surface of the matrix cylinder.

Using these conditions we find

$$U_{r_1} = \frac{-2 \nu_m r_1 e}{1 - \nu_m + (1 + \nu_m)(r_1^2 / r_0^2)} \quad (3)$$

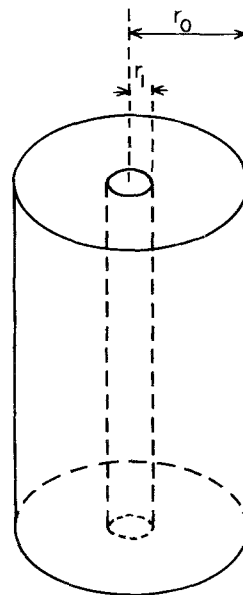


Figure 2 Cylinder of matrix material (no embedded fibre).

TABLE I

System fibre/matrix	V_f	E_f (GN m ⁻²)	ν_f	E_m (GN m ⁻²)	ν_m	M_s	F_s	Equation 5 satisfied
Steel/cement	0.05	200	0.28	20	0.23	0.54	0.81	No
Steel/epoxy	0.2	200	0.28	3.5	0.3	0.58	0.30	Yes
Graphite/cement	0.02	200	0.35	20	0.23	0.57	2.07	No
Graphite/cement	0.05	200	0.35	20	0.23	0.54	1.02	No
Glass/epoxy	0.6	72	0.25	3.5	0.3	0.35	0.26	Yes
Graphite/glass	0.5	200	0.35	72	0.25	0.32	0.48	No
Glass/cement	0.05	72	0.25	20	0.23	0.54	1.57	No
Polypropylene/ cement	0.1	10	0.3	20	0.23	0.50	5.7	No

where e is the original strain on the matrix (positive for tension). The fibre and matrix will remain in contact in the absence of matrix shrinkage in curing or preparation if the reduction of the radius of the hole in the matrix is greater than the reduction of the radius of the fibre. That is if

$$\frac{2\nu_m}{1 - \nu_m + (1 + \nu_m)(r_1^2/r_0^2)} > \nu_f \left[1 + \frac{E_m V_m}{E_f V_f} \right] \quad (4)$$

If we assume a square array as in Fig. 1a ($r_1 = r_f$, $r_0 = r_m$)

$$\frac{\pi r_1^2}{4r_0^2} = V_f$$

we obtain the condition

$$\frac{2\nu_m}{1 - \nu_m + (1 + \nu_m)(4V_f/\pi)} > \nu_f \left[1 + \frac{E_m V_m}{E_f V_f} \right] \quad (5)$$

Data for a number of systems collected by Kelly and Zweben are given in Table I. The left hand side of Equation 5 is called M_s and the right hand side F_s . It can be seen that only two systems, in which the matrix is epoxy, obey the criterion for fibre/matrix contact after cracking with subsequent multiple fracture. Similar results with slightly different constants are obtained for a hexagonal array but are not presented here. All the systems presented are known to undergo multiple fracture with the exception of polypropylene in cement. This implies that the consideration of Poisson contraction alone is insufficient to determine whether multiple fracture will occur in a given system. There are several reasons for this. If we treat the first example of Table I assuming a

reasonable matrix cracking strain of 5.0×10^{-4} and a fibre radius of 0.15 mm the Poisson contraction of the fibre at this strain after cracking is 6×10^{-5} mm and of the matrix is 4×10^{-5} mm. The fibre/matrix separation is hence of the order of 0.02 μ m. This will certainly be less than the asperity height on all but the smoothest fibre so some frictional load transfer will occur even in perfectly aligned fibrous composites. Matrix shrinkage in specimen preparation resulting in a residual stress at the interface is also important in many of the examples shown in Table I. This shrinkage may be due to temperature effects, curing shrinkage, or drying shrinkage, particularly with cement matrices. Observation of steel fibres being pulled from cement matrices in our laboratory shows that failure often occurs several microns from the actual steel/cement interface in the bulk of the cement. The crack path is quite tortuous and thus even if the fibre is smooth, contact will be maintained between the fibre and matrix [4].

The difference between the present treatment and that of Kelly and Zweben is due to two factors. Kelly and Zweben in their paper assume that the matrix can freely expand with no restraint from the surrounding matrix on cracking. The treatment also considers that a point midway between the two fibres under consideration is fixed and allows the matrix to expand around this point. This approach will only maintain fibre matrix contact at one point on the fibre circumference. The actual composite behaviour does not allow reduction to one dimension and the restraint of the surrounding matrix must be considered. Their model also assumes fibre/matrix contact with no interfacial pressure when the composite is under strain just before cracking. This will not be the case unless ν_m and ν_f have the same value.

The effect of Poisson contraction, while not having as direct an influence on multiple fracture as the Kelly-Zweben paper would indicate, will certainly result in a decreased fibre/matrix normal force when the matrix cracks. Only in the case of composites prepared with an epoxy matrix when $\nu_m > \nu_f$ was the Poisson contraction alone found to be sufficient to maintain fibre/matrix contact. However, the final result indicates that the Poisson contraction will lead to a decreased pressure at the interface on matrix cracking and hence a reduced frictional stress transfer at the interface. This reduction in normal stress at the interface will be the subject of a future paper from our laboratory.

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References

1. A. KELLY and C. ZWEBEN, *J. Mater. Sci.* **11** (1976) 582.
2. R. HILL, *J. Mech. Phys. Solids* **12** (1964) 199.
3. A. E. H. LOVE, "A Treatise on the Mathematical Theory of Elasticity" p. 144.
4. D. J. PINCHIN and D. TABOR, Mechanical Properties of the Steel/Cement Interface: Some Experimental Results, in "Fibre Reinforced Cement and Concrete", Vol. 2 (Construction Press, Hornby, Lancs, 1976) in press.

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A new method of film casting for electron microscopy

Many studies have been reported of microphase separation in block co-polymers [1] which can lead to ordered arrays of regular domains [2-4]. One widely used method of studying these arrays in thermoplastic elastomers such as SBS and SIS block co-polymers is electron microscopy. This requires the preparation of very thin films of the material either by cutting using an ultramicrotome or by solvent casting. The present article deals with the application of the latter technique to rubbery styrene-butadiene block co-polymers, and describes an improved solvent casting method.

A variety of casting techniques have been described in the literature [5-7]. With block co-polymers, the Kato [5] method gave a very poor success rate. Hall [6] has suggested a modification of Kato's technique involving casting the film on a clean water surface. However, Lewis and Price [7] have reported that Hall's method is unsatisfactory for ABA poly (styrene-*b*-butadiene) co-polymers as the films tended to break when dried. They suggested a method [7] involving casting on a freshly distilled and cleaned mercury surface. The grids are held by tweezers and dragged over the surface to collect the film. This

method has also been found to be rather unsuccessful with highly rubbery materials which appear to collect at the edge of the grid. However, when this technique worked the film was very satisfactory suggesting that the surface obtained is good for ABA poly (styrene-*b*-butadiene) co-polymers and that the problem simply lies in the transfer of the film onto the grids.

Fig. 1 shows a cross-sectional view of the apparatus developed for solvent casting on to electron microscope grids. Freshly cleaned and distilled mercury (A) is placed in a circular steel vessel (B) fitted with a push-fit Teflon ring (C). The ring just touches the mercury surface to prevent solution running into the trough round the edge of the vessel. A circular disc composed of two layers (D and E) is then placed in the mercury pool and allowed to float. The top half of the disc is formed

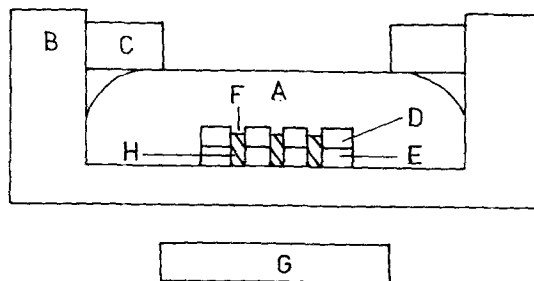


Figure 1 Cross-sectional view of casting apparatus.