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A buckled plate test to measure interfacial toughness in composites

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A new, buckled plate (BP) test has been used to measure transverse toughness as the parameter characterizing interfacial adhesion in unidirectional, continuous-fibre composites. The test is simple, with advantages over other interfacial methods. The theory and experimental details are presented. The results of BP tests are discussed for polycarbonate/carbon fibre composites. Evaluations have been made with regard to specimen dimensions, testing speed, crack length, modulus, fibre volume fraction, and processing conditions. Transverse toughness is a sensitive measure of the interfacial adhesion, giving results similar to transverse tensile strength. The test has also been used to measure longitudinal toughness. This test should be widely applicable to many composite systems.

1. Introduction

Interfacial adhesion plays a crucial role in the mechanical properties of composites, therefore mechanical, as well as morphological, characterization of the interface is necessary to understand composite behaviour. Much has been done in developing methods for mechanical characterization of the interface, particularly in fibre-reinforced composites, as recently reviewed [1]. There are, however, difficulties with each of these tests. A new method for measuring interfacial adhesion is introduced here that has advantages over the prior tests. The new method measures transverse toughness by use of a buckled plate (BP) specimen, recently developed by Chang and Donovan [2] for slow crack-growth studies.

In a BP test, a rectangular plate is buckled in compression until a pre-introduced crack propagates across the sample. The major advantages that the BP specimen has over other fracture mechanics specimens are that it is a simple plate, yet the crack driving force is independent of crack size. If a unidirectional composite plate is buckled transverse to the fibre direction, the crack propagates along the fibre direction, and the BP test measures transverse toughness. Although longitudinal toughness is generally higher with poor fibre–matrix adhesion because fibre pull-out can occur, good transverse toughness requires good fibre–matrix adhesion. Thus transverse toughness is a sensitive measure of fibre–matrix adhesion.

Common interfacial test methods for comparison with the BP method include (1) fibre pull-out, (2) single filament critical length, (3) microdebonding, (4) short beam shear, and (5) transverse tensile strength. Fibre pull-out tests require difficult sample preparation, involve non-uniform stress distributions, and often have large data scatter [1]. In addition, use of single fibres limits comparison to actual composites. The BP test does not have these problems.

Single filament critical length tests also entail difficult sample preparation, require tedious measurements of fragment lengths, again involve only single fibres, and require fitting of fragment distribution curves. Typical variance values for interfacial shear strength are $\geqslant \pm 50\%$ [1]. Variance values found here for the buckled plate test are generally $\leqslant \pm 20\%$.

Microbonding, while applicable to actual composites, requires *in situ* measuring of debonding and finite element analysis using a micromechanics model [1]. Again the BP test is much simpler and easier. The short beam shear test is a simple three-point bend test, but requires relatively thick samples (recommended span to depth ratios of 6 [3]) in order to induce shear rather than tensile failure. There can also be problems of indentation in a three-point bend test. A BP test is just as simple, yet utilizes thin samples ideal for small-scale experimental purposes, and involves no indentation problems.

Transverse tensile tests appear simple, but have a low level of reliability [1] because of the sensitivity to flaws. The BP method, on the other hand, involves failure from a precrack, but the results are not sensitive to precrack or flaw size. The BP method also involves no gripping problems as in a transverse tensile test.

Finally, in comparing tests, it should be noted that the BP test measures fracture toughness, which is fundamentally different from the interfacial shear strength measured in methods 1 to 3, the interlaminar shear strength measured in method 4, or the tensile strength measured in method 5. Toughness is a measure of the energy or work to cause separation of the composite. In linear elastic fracture mechanics the fracture toughness, a material property, is the energy required to initiate crack growth per unit crack area. The concept is based on Griffith's [4] original criterion for fracture, with the energy to create the new fracture surfaces coming from the strain energy stored in the

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specimen. The minimum energy required to fracture the composite would be the work of adhesion, if that was less than the fracture toughness of the matrix. In all systems studied to date, the toughness has been at least an order of magnitude greater than the theoretical minimum, due to the energy dissipated by deformation. Thus, although dependent on the work of adhesion, the measured toughness includes energy required to deform the matrix and the interfacial region. This means that toughness is a more fundamental measure of the resistance to failure of the composite than strength, as measured by the other tests.

This paper presents both basic theory and experimental details of the BP test. Results for a polycarbonate (PC)/carbon fibre (CF) composite system are presented and discussed with regard to test parameters and composite processing. Transverse tensile test results are included for comparison.

2. Theory

The important equations for the BP specimen are presented briefly here. Further details are given by Chang and Donovan [2]. For an elastically buckled plate, the critical load for buckling, P_c, is

$$P_c = \Pi^2 l^{-2} EI \quad (1a)$$

$$I = wh^3/12 \quad (1b)$$

where l is the specimen initial length, w the specimen width, h the specimen thickness and E the specimen modulus. Knowing specimen dimensions and measuring the critical load from the load/deflection curve as shown below, the modulus can be calculated. E can then be used in the equation for crack driving force, G, which for the BP specimen is

$$G = 0.82\, Eh^2 l^{-2}(l - x) f^*(\varepsilon) = AE \quad (2)$$

where x is the chord length of the buckled plate, ε the normalized displacement $= (l - x)/l$, $f^*(\varepsilon) = 0.158\varepsilon^2 + 0.229\varepsilon + 1$ = nearly 1 at relatively low displacements (less than 30%), and $A = 0.82h^2l^{-2}(l - x)f^*(\varepsilon)$. The $f^*(\varepsilon)$ term has been considered equal to 1 in this study. Equation 2 indicates that the crack driving force is independent of crack length, a. If the plate is buckled until fracture, and $(l - x)$ at fracture used, Equation 2 yields the fracture toughness, G_c.

3. Experimental details

Polycarbonate/continuous carbon fibre, unidirectional composites were fabricated by alternately placing previously dried 0.13 mm thick Lexan film (General Electric, $M_w = 34\,000$) on a Teflon-covered aluminium plate and wrapping unsized T500 3k PAN-based carbon fibre yarn (Amoco) around the plate in aligned fashion. The aluminium plate's edges were rounded in order to prevent fibre breakage. Typically four layers of film and three layers of fibre yarn were used, but this was also varied to control composite thickness. The layers were then consolidated at 275° C in a Carver press by holding them for 5 min with low pressure, pressing 10 min at 0.8 MPa, then either (1) cooling the composite quickly to room temperature in the press cooling cycle (less than 5 min), (2) releasing the pressure and holding the composite for a longer processing time before cooling, or (3) releasing the pressure, cooling to 245° C, and annealing the composite for 3 h before cooling. These conditions were chosen to examine adsorption and crystallization effects, which will be discussed more fully in a forthcoming paper.

Composite plates were typically 0.45 mm thick, but several other thicknesses were examined. Unless otherwise stated, composites had a fibre weight fraction of 0.364 ± 0.008 (volume fraction = 0.28) found by dissolving out the PC with methylene chloride. Samples were cut with a paper cutter, and the edges sanded with fine sandpaper. The ends of the BP specimens were rounded to permit free rotation during buckling and minimize end effects.

The BP fracture toughness test is illustrated in Fig. 1. A Model 4202 Instron testing machine with a sensitive 50 kg reversible load cell was used. One thin aluminium plate with a shallow, rounded central groove

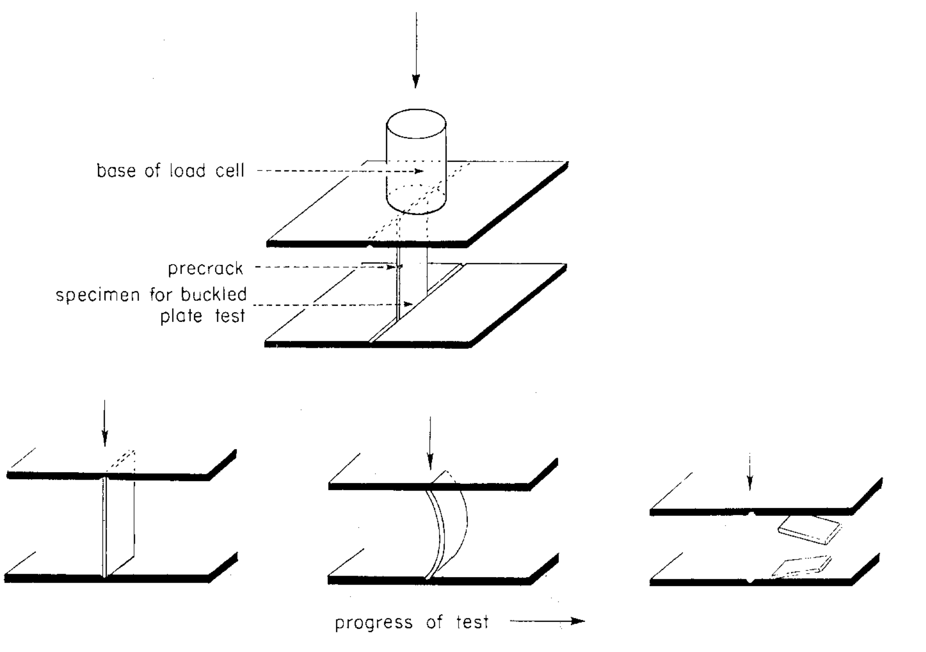

Figure 1 Buckled plate test. See text for further description.

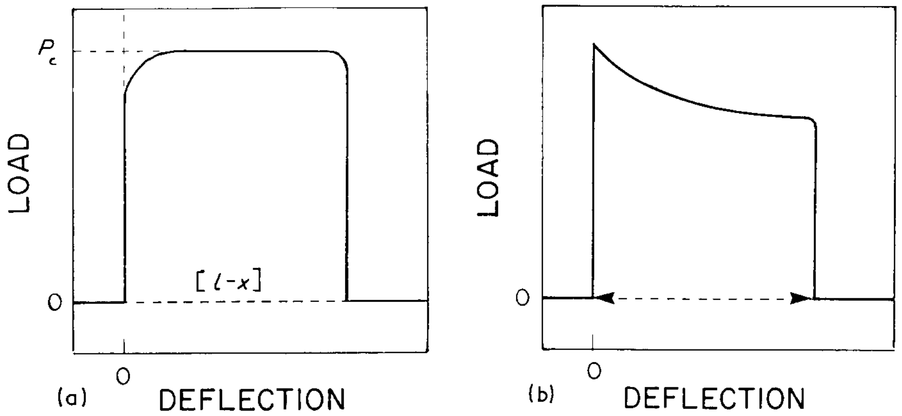

Figure 2 Example load/deflection curves for a buckled plate test: (a) typical and proper curve, (b) improper curve.

was attached directly to the load cell (on the moving cross-head), and another was attached to a stationary lower support. A small, rectangular composite specimen, typically 2.5 cm long, 0.9 cm wide and 0.045 cm thick, with fibres oriented perpendicular to the testing direction, was placed in the grooves. A central precrack parallel to the fibres and about 1 mm long was previously introduced to the BP specimen with a fresh razor blade. Samples used to determine the modulus were not precracked. The composite plates were buckled in compression at room temperature until fracture. Testing speed was 2 cm min^{-1} unless otherwise stated. A chart recorded the load/deflection curve. Averages and standard deviations were obtained by testing four to eight specimens of each kind.

Transverse tensile tests were also performed with a Model 4202 Instron testing machine, interfaced with a computer. Composites were typically strips 0.5 cm wide and 0.045 cm thick, with 2.5 cm between grips. Manilla tabs were superglued to the composite to prevent breaking in the pneumatic grips. (Epoxied tabs did not bond well to the composites.) All tests were performed at room temperature at a cross-head speed of 1 mm min^{-1}. Four to six specimens of each type were tested.

Fracture surfaces were examined in a Jeol 35CF scanning electron microscope after coating with a thin layer of gold in a Polaron E5100 SEM sputtering unit.

4. Results and discussion

4.1. Load/deflection curve

A typical load/deflection curve, in accordance with theoretical predictions [2], is shown in Fig. 2a. The critical buckling load, P_c, is taken as the intersection of the nearly vertical and horizontal portions of the load/deflection curve. For the longitudinal samples, the plateau sloped slightly upward. The deflection to fracture gives $(l - x)$ as indicated in Fig. 2a. The slight rounding of the curve before fracture is most likely due to some small amount of plastic deformation or stable crack growth.

Because of the method of fabrication, the composites are slightly different on the two sides, and therefore have a natural way to bend in compression. In the few cases where the composite buckled in the opposite direction, a curve like that in Fig. 2b resulted. This type of load deflection curve was not expected according to previous theoretical and experimental work [2], so these tests were considered invalid.

4.2. Specimen dimensions

The measured toughness of PC/CF composites processed 15 min at 275° C was independent of specimen dimensions in the range tested. The results are shown in Fig. 3 for plate dimensions varied one at a time (except that thickness changes required length changes to keep displacement to fracture in a measurable range). For all dimensions tested, the load/deflection curves were similar to that in Fig. 2a.

Thickness could affect fracture toughness if there is a transition from plane stress to plane strain in the examined thickness range [5]. The composites here are thin, therefore they would likely be in plane stress. The constraint imposed by the fibres, however, would tend to establish conditions of plane strain. The lack of thickness effect in Fig. 3 suggests plane strain, even though the specimens are thin.

Results were independent of specimen dimensions for a BP test, but several factors must be considered. (1) Samples must be thin enough to buckle at

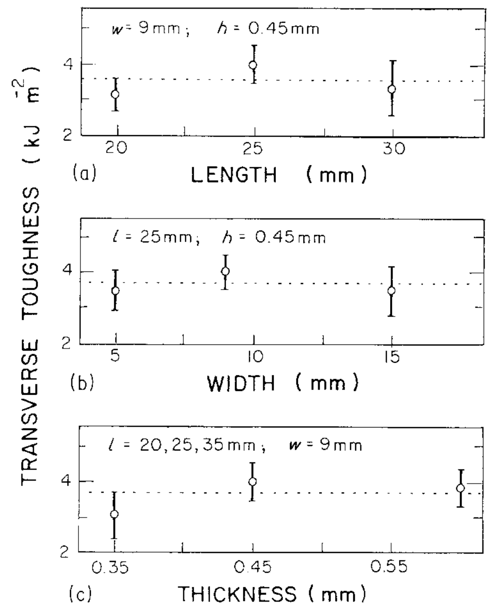

Figure 3 Transverse toughness plotted against (a) nominal length, (b) width, and (c) thickness for a PC/CF composite processed 15 min at 275° C.

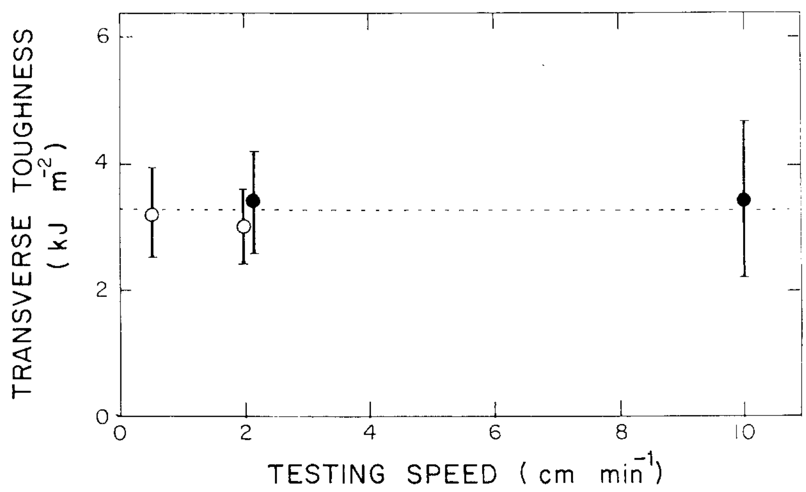

Figure 4 Transverse toughness plotted against testing speed for a PC/CF composite processed 15 min at 275° C: (○) 0.35 mm and (●) 0.45 mm thick specimens.

reasonable loads (thus very thick samples are inappropriate). Samples too thin, however, will buckle too easily and require lengths too short for study. (2) The length must be chosen so that failure occurs at a reasonable deflection. Deflections too low may be difficult to measure, while large deflections lead to $f^*(\varepsilon)$ becoming important, and to end effects. (3) Comparison of composites with different thicknesses should be done carefully because of the thickness effects that can occur in any fracture mechanics test.

4.3. Testing speed

For two different thickness PC/CF composites processed 15 min at 275° C, Fig. 4 shows that testing speed (0.5 to 10 cm min^{-1}) had no measurable effect on toughness. This is consistent with essentially elastic fracture. Composites were also buckled to near the expected breaking displacement and held overnight (static loading) without fracture, indicating little or no slow crack growth.

4.4. Crack length

Chang and Donovan [2] showed theoretically and experimentally that the crack driving force is independent of crack length in the BP specimen (Equation 2). To examine this further, crack length was varied for a PC/CF composite processed 15 min at 275° C. The results in Table I show precracked lengths of 0.5 and 1 mm gave equivalent toughness. For longer precracks, the introduction of the precrack tended to cause propagation, resulting in lower toughness. This suggests that precrack sharpness matters, and therefore consistency in precracking is considered important. It should also be noted that specimens with no precrack gave considerably higher and more scattered toughness values, most likely because of the variability in the sharpness of natural flaws. Precracking is therefore considered necessary for consistent results.

TABLE I Precrack length effect on transverse toughness for a PC/CF composite, processed 15 min at 275° C

Precrack length (mm)	Transverse toughness (kJ m^{-2})
0.5	4.1 ± 0.7
1.0	4.0 ± 0.5
2	2.5 ± 0.7*

*Precrack tends to propagate during introduction.

4.5. Modulus

It was found that the elastic modulus should be determined with specimens without precracks. When using precracked samples and assuming the effective width as the total width minus the precrack length, calculated moduli were over 10% higher than for the unprecracked composites (3.90 ± 0.12 compared to 3.49 ± 0.16 GPa). Apparently in compression, some load is transferred across the precrack, and affects the buckling load from which the modulus is calculated.

Modulus did not vary with the processing conditions studied. Table II shows moduli from BP and tensile tests for neat PC and PC/CF composites. As can be seen, the BP modulus found for pure PC is close but slightly higher than the tensile literature value of 2.4 GPa [6]. The tensile value for pure PC and the transverse tensile value for the PC/CF composites are much lower than the BP values, probably because of gripping difficulties. The transverse composite modulus of 3.49 GPa shows the small reinforcement effect of the fibres in the transverse direction.

4.6. Fibre volume fraction

Two widely different fibre volume fractions at the same composite thickness were examined for the PC/CF composites processed 15 min at 275° C. The results are shown in Table III. The modulus was lower in the low volume fraction composites, but they flexed further before breaking, as indicated by values for A (Equation 2). These two effects offset one another so that the final fracture toughness was essentially the same. The buckled plate toughness, therefore, seems to be relatively insensitive to fibre volume fraction differences. This suggests that the BP test is a fundamental measure of interfacial resistance to failure. Short beam shear and transverse tensile tests, in contrast, depend on fibre volume fraction.

TABLE II Comparison of modulus values

Sample	Method	Modulus (GPa)
PC	Buckled plate	2.72 ± 0.03
PC	Tensile	1.8
PC	[5], Tensile	2.4
PC/CF	Buckled plate, transverse	3.49 ± 0.16
PC/CF	Transverse tensile	2.71 ± 0.12

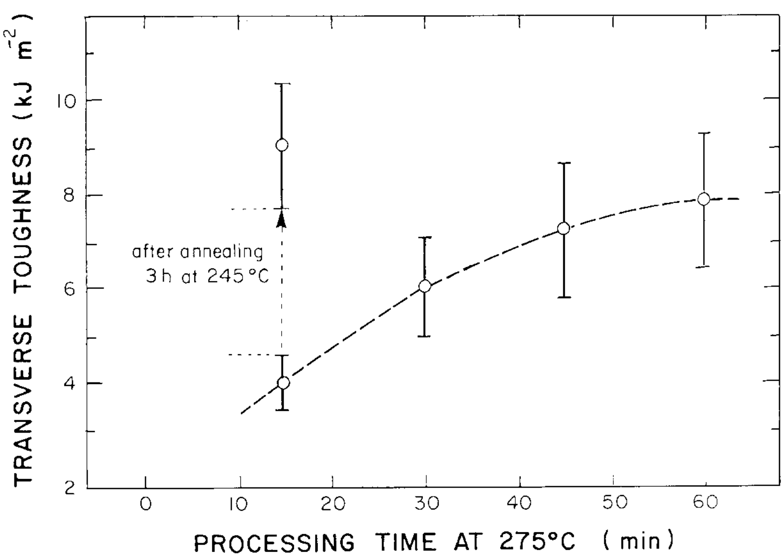

Figure 5 Transverse toughness plotted against processing time at 275° C and annealing for PC/CF composites.

4.7. Processing conditions

The processing time at 275° C, as well as annealing conditions, were changed to evaluate changes in the interfacial adsorption and crystallization of PC/CF composites. These processing conditions have been found to alter only the interface and not the bulk matrix. A more complete examination of adsorption and crystallization will be included in a forthcoming paper. Both transverse toughness and transverse tensile strength increased by about a factor of two with processing time and annealing (Figs 5 and 6). It should be noted that pure PC of the same thickness does not fracture in a BP test, indicating all composite values are less than that for pure PC.

Scanning electron micrographs of fracture surfaces confirm increased fibre–matrix adhesion with longer processing time and with annealing. In Fig. 7a the fibres can be seen to pull out cleanly from the matrix in the composite processed 15 min at 275° C, while some PC can be seen adhering to the fibres in the composite processed 60 min at 275° C (Fig. 7b).

Comparison of Figs 5 and 6 reveals that the increase in toughness is more gradual than for strength, with annealing giving a significantly higher toughness than a processing time of 60 min. The BP method sees differences where the transverse tensile strength does not. It is thus a more sensitive measure of interfacial adhesion. This is because PC begins to yield near 65 MPa, so tensile strength loses its sensitivity near this point. It should be noted that the repeatability of the transverse tensile strength values is questionable at the low end of the scale. Repeat experiments gave a value of 45.4 $\pm$ 4.3 MPa for the composite processed 15 min at 275° C, which is considerably higher than that in Fig. 6. This may be an indication of the extreme sensitivity of the transverse tensile test to flaws, especially in the more brittle composites. In contrast, repeat of the BP test always gave similar values within experimental error. Experimental error or the standard deviation in the BP test is less than $\pm$20% in all cases, and perhaps could be lower for more uniform composites and refined testing.

4.8. Longitudinal toughness

The BP test has also been used to measure longitudinal toughness (crack propagation perpendicular to the fibres) in PC/CF composites. Specimens were typically 4.0 cm long, 0.9 cm wide and 0.045 cm thick. The longer lengths were required to get adequate deformation in this stiffer direction. The toughness for composites processed 15 and 60 min at 275° C is given in Table IV. The modulus was found to be the same in both cases,

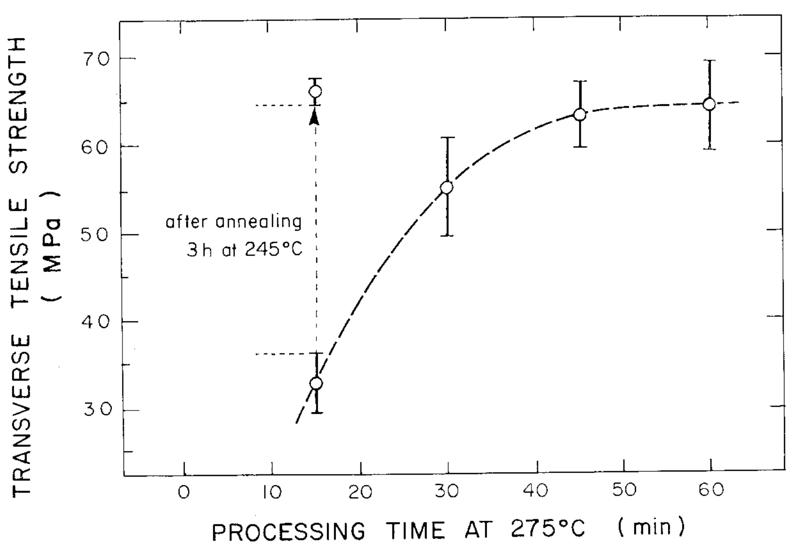

Figure 6 Transverse tensile strength plotted against processing time at 275° C and annealing for PC/CF composites.

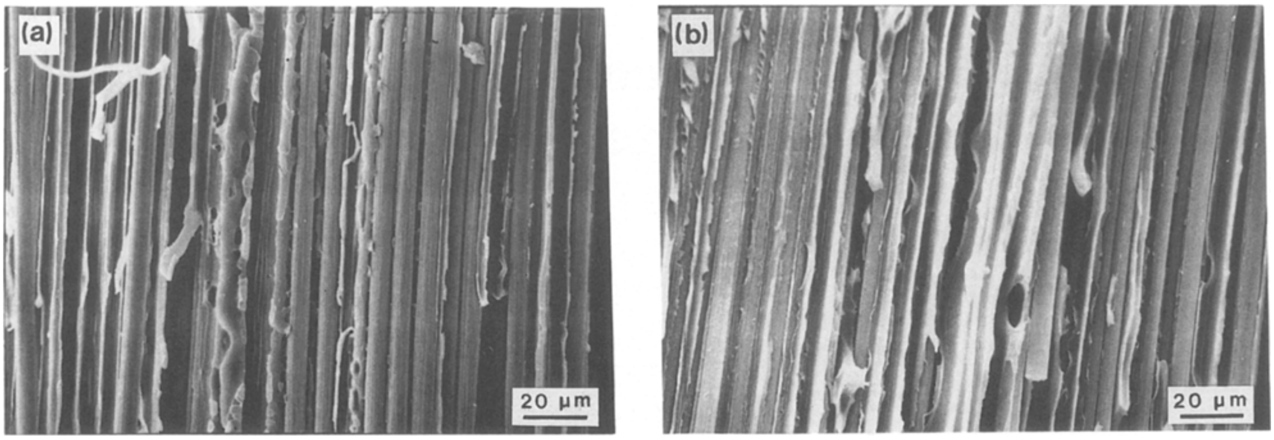

Figure 7 Scanning electron micrographs of transverse tensile fracture surfaces for PC/CF composites processed at 275° C for (a) 15 min, and (b) 60 min.

and shows the strong reinforcing effect of the fibres in the longitudinal direction. The toughness was higher in the 15 min specimens, consistent with easier fibre pull-out in composites with poorer adhesion. The longitudinal toughness thus shows an inverse relationship with adhesion, and is not nearly as sensitive as the transverse toughness to interfacial adhesion.

5. Conclusions

A new, buckled plate (BP) test has been used to measure transverse toughness as the parameter characterizing interfacial adhesion in unidirectional, continuous-fibre composites. The test is simple to perform, yet has some advances over the other interfacial evaluation techniques. Transverse toughness by the BP method was found to be independent of specimen length, width, and thickness, testing speed, crack length, and fibre volume fraction. Varying processing and annealing conditions in PC/CF composites led to transverse toughness and transverse tensile strength increases by a factor of two. Scanning electron microscopy of fracture surfaces were consistent with increased fibre–matrix adhesion. The BP method was therefore shown to be a sensitive measure of interfacial adhesion in the ideal case of unidirectional, continuous-fibre composites. The BP test was also used to measure the corresponding longitudinal toughness. The test is ideal as a measure of interfacial adhesion, and should be applicable to a range of composite systems.

TABLE III Fibre volume fraction effect for a PC/CF composite, processed 15 min at 275° C

Fibre volume fraction	Modulus (GPa)	A, Equation 2 (10^6 m)	Transverse toughness (kJ m^{-2})
0.28	3.4 ± 0.3	0.90 ± 0.15	3.0 ± 0.6
0.10	2.74 ± 0.13	1.37 ± 0.11	3.8 ± 0.4

TABLE IV Longitudinal toughness of PC/CF composites

Processing time at 275° C (min)	Modulus (GPa)	A, Equation 2 (10^6 m)	Longitudinal toughness (kJ m^{-2})
15	41.6 ± 2.1	0.96 ± 0.15	40 ± 7
60	41.6 ± 2.1	0.73 ± 0.08	30 ± 4

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