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howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

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Measurement of interfacial shear strength of carbon fibre/epoxy composites using a single fibre pull-out test

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Abstract—Single fiber pull-out tests were conducted for G34-700/Araldite-F, T700S/Araldite-F and T800H/Araldite-F model composites using an improved experimental set-up to evaluate the interfacial shear strength (IFSS). The modified method made it possible to reduce the formation of resin meniscus and to accurately define the embedded fibre length, and it also provided a practical approach to conveniently estimate the IFSS for comparison of the fibre–matrix adhesion between different composite systems or different fibre surface conditions. A strong interfacial adhesion and unstable debonding process without detectable friction stages were found for all types of model composites with fibres being chemically treated/sized or sizing being removed. The electrochemical oxidation treatment and sizing on the carbon fibres noticeably increase the IFSS of the composites.

Keywords: Single fibre pull-out test; fibre–matrix interface; fibre–matrix adhesion; carbon fibre/epoxy composites.

1. INTRODUCTION

There are three main micromechanical tests which have been developed to evaluate interfacial shear strength (or interfacial fracture toughness) of continuous fibre-reinforced composites, namely, the fragmentation test, the indentation test and the single fibre pull-out test [1–3]. The last may also be subdivided into the pull-out test and the microbond test [4]. Among these methods the single fibre pull-out test is the most extensively used because of its simplicity and versatility as well as the fact that it is a direct method for measuring fibre–matrix adhesion. However, there are some inherent drawbacks associated with the single fibre pull-out method [5]. A main problem is that the resin meniscus formed on the fibre using current procedures of specimen preparation makes the determination of the embedded fibre length difficult. Also, the presence of the meniscus has a certain effect on the interfacial bond strength due to possible premature failure caused by stress

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concentrations in the meniscus region before interfacial debonding. Moreover, for a single fibre pull-out test designed to determine the interfacial properties it is essential that the interfacial debonding occurs before the fibre breaks. Since in pull-out tests the interfacial debonding force is directly related to the embedded fibre length, there is a maximum embedded fibre length which permits the fibre to be pulled out without breakage. The maximum embedded fibre length is usually very short, especially for composite systems with very fine fibres and/or strong interfacial bonding. For example, the maximum embedded fibre length is generally less than 200 μm for carbon fibre/epoxy matrix systems, which causes certain difficulties in specifying the embedded fibre length in the preparation of specimens for pull-out tests.

A modified pull-out test procedure was adopted in this work to evaluate the interfacial adhesion between fibres and matrices for three carbon fibre/epoxy systems. The basic test geometry is similar to that proposed by Gu *et al.* [6], but a special procedure was applied in this work, which made it possible to reduce the formation of resin meniscus and to accurately define the embedded fibre length.

2. EXPERIMENTAL PROCEDURE

The carbon fibres used in this study were G34-700 (Grafil Inc., USA), T700S and T800H (Torayca Inc., Japan). The G34-700 fibres were supplied with two different surface conditions, i.e. one without any surface treatment and the other treated using electrochemical oxidation and then coated with a thin layer of epoxy sizing (0.4% by mass). The 'as received' T700S and T800H fibres were treated and sized by the manufacturer. Some of the T700S and T800H fibres were washed in a methylethylketone (MEK) solution for 1 h and then dried for 2 h in an oven at 200°C to remove the sizing. An epoxy resin (Araldite-F, Ciba-Geigy, Australia) was chosen as the matrix material. Piperidine was added as the hardener to the epoxy with a weight ratio of 5 : 100 after degassing the epoxy at 100°C in a vacuum chamber.

The procedure for preparing a fibre pull-out specimen is schematically illustrated in Fig. 1. Two small pieces of glass slides (one 0.17 mm, the other 0.34 mm in thickness) were placed at a distance of about 1.5 mm apart on a base glass slide. A small drop of the uncured resin was placed on the edge of the 0.17 mm thick glass slide. A liquid silicone release agent was applied to fill in the gap between the two glass slides and on the top surface of the 0.34 mm thick glass slide, as shown in Fig. 1. A single carbon fibre was laid with one end in the middle of the resin drop. Then, the whole system was moved onto the stage of an optical microscope, with the base glass slide being fixed. Under view of the microscope, the fibre was gradually drawn out of the resin until a desired embedded fibre length was achieved. Finally, the whole sample was carefully put on a steel plate, to be cured in an oven at 120°C for 16 h. After curing, the glass slide with the cured resin drop and the fibre was mounted on a paper frame similar to that described in the ASTM test standard (ASTM D3379-75) with a gauge length of

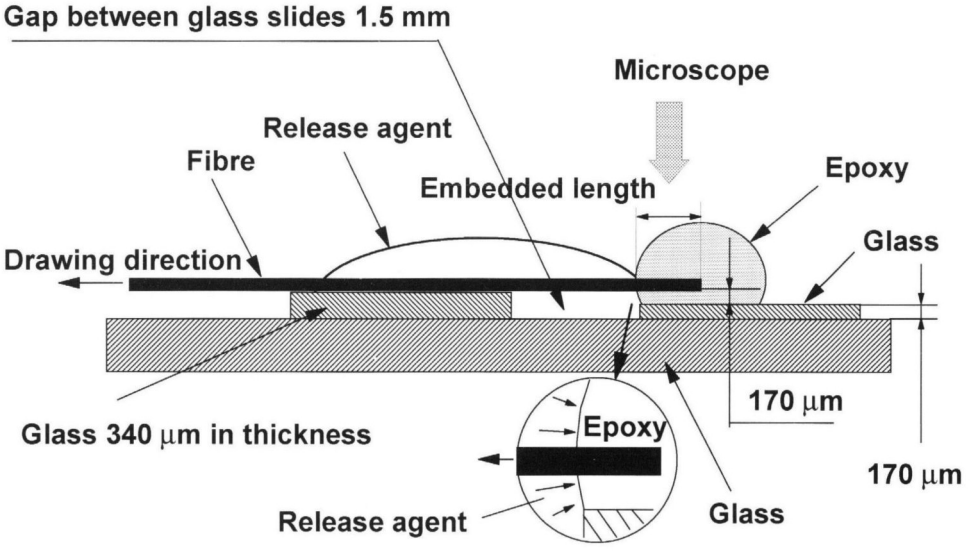


Figure 1. Schematic of a fibre pull-out specimen.

20 mm. The embedded fibre length was measured using a MD-30 Plus image analysis system in a computer which was connected to the optical microscope. There are several advantages for the use of the silicone release agent in the preparation of the fibre pull-out specimens. The release agent pushes back the uncured resin from around the carbon fibre when the fibre is drawn, and it also prevents the resin from migrating along the fibre during curing by decreasing the wettability of the resin on the fibre, so that the resin meniscus formed around the fibre can be reduced.

The single fibre pull-out tests were carried out at ambient temperature using an Instron 5567 universal test machine with a very sensitive load cell (2.5 N). A crosshead speed of 0.1 mm/min was applied. After testing, the specimen was checked under the optical microscope to determine whether the fibre had been pulled out or fibre breakage had occurred. The pull-out length (or embedded fibre length) was confirmed by measuring the length of the matrix socket. A high resolution SEM was used to determine the fibre diameter and define the interfacial failure mode of the pull-out specimens.

The apparent average interfacial shear strength, τ_a, at failure for the model composite systems was calculated from the pull-out test using the peak load, F_d, the embedded fibre length, L, and the fibre diameter, d [1]

$$\tau_a = \frac{F_d}{\pi d L}.$$

3. RESULTS AND DISCUSSIONS

Optical micrographs of a typical fibre pull-out specimen before and after testing for the T700S/Araldite-F model composite are shown in Fig. 2. It can be seen that only a tiny meniscus formed on the fibre, and there is a matrix socket after the fibre was pulled out.

Some typical load–displacement curves are shown in Fig. 3 for the single fibre pull-out tests of the treated/sized and untreated/unsized G34-700 fibres with different embedded fibre lengths. The characteristics of load–displacement curves for the untreated/unsized G34-700 fibres are similar to those of the treated/sized

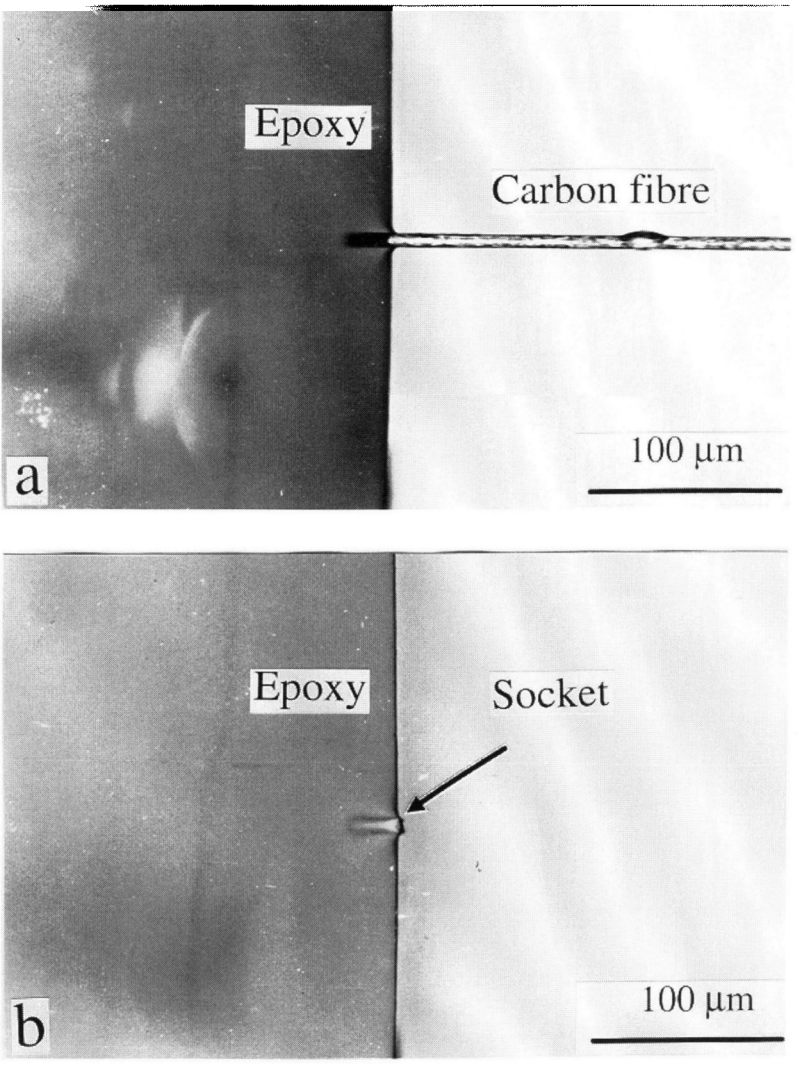
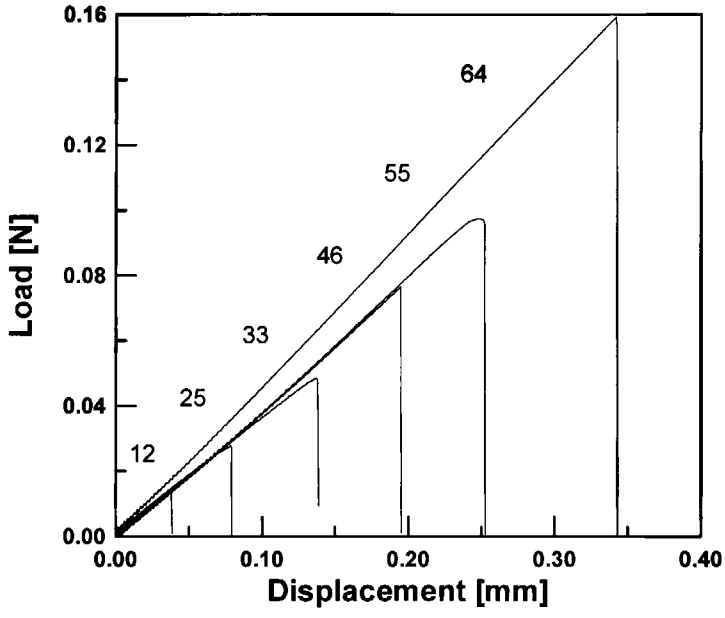
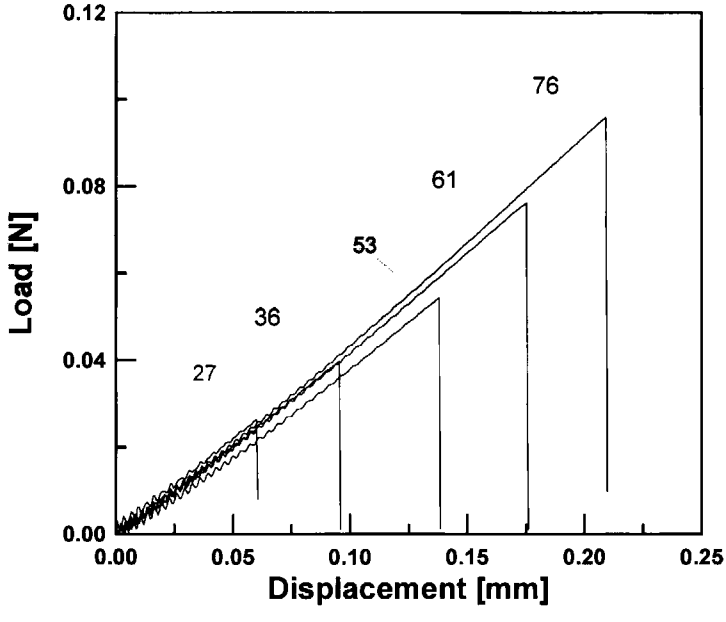


Figure 2. Optical micrographs of a typical fibre pull-out specimen for treated/sized G34-700/Araldite-F with an embedded fibre length of 28 μm: (a) before pull-out test; (b) after pull-out test.

G34-700 fibres, but with lower peak loads for the same embedded fibre lengths, shown in Fig. 3. For T700S and T800H fibres with and without sizing, similar load–displacement curves were obtained. Generally speaking, interfacial adhesion in these carbon fibre/epoxy composite systems is fairly strong. The applied load in-



(a)



(b)

Figure 3. Typical load–displacement curves for single fibre pull-out tests of G34-700 fibres: (a) treated/sized; (b) untreated/unsized (numbers in the figure indicate embedded fibre lengths in μm).

creases almost linearly with the displacement until the peak load is reached, followed by a sudden drop to zero, indicating the instantaneous debonding of the fibre–matrix interface. The embedded fibre lengths, allowing a successful pull-out of the fibre, for these model composite systems are very short, generally less than 100 μm as shown in Fig. 3.

In the single fibre pull-out tests conducted in this study, the interfacial debonding process proceeded in an unstable manner and no detectable interfacial friction stage can be found on the load–displacement curves. This is due to the short embedded fibre length and the strong fibre–matrix adhesion. Since the tests were carried out at a constant strain rate with a fixed gauge length which is much longer than the embedded fibre length, elastic deformation will occur at the fibre free end when the load is applied. When debonding occurs, a sudden release of the elastic strain energy stored in the system under tension leads the instantaneous fibre pull-out from the resin and, as a result, the fibre motion in the resin is undetectable because of its dynamic behaviour. Moreover, the elastic contraction of the fibre in the radial direction at a high debonding load during the pull-out process also assists in its quick extraction from the resin.

The debonding load was found to be correlated approximately linearly with the embedded fibre length for G34-700/Araldite-F, T700S/Araldite-F and T800H/Araldite-F model composites (Figs 4, 6 and 8). The fibre surface treatment and the sizing on G34-700 and T700S carbon fibres obviously improved the interfacial shear strength of the model composite systems (Figs 5 and 7). However, no obvious effect of the

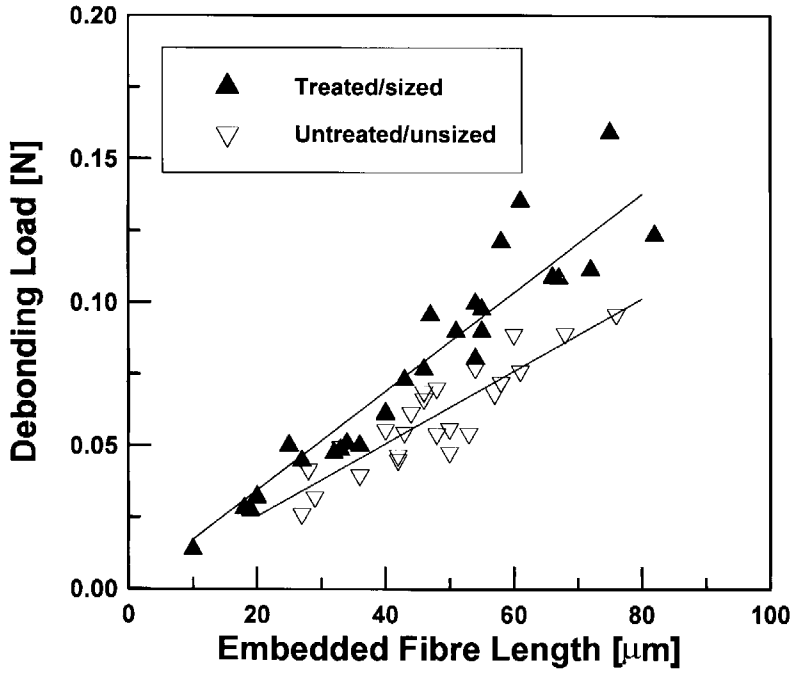


Figure 4. Debonding load *versus* embedded fibre length for G34-700 fibres.

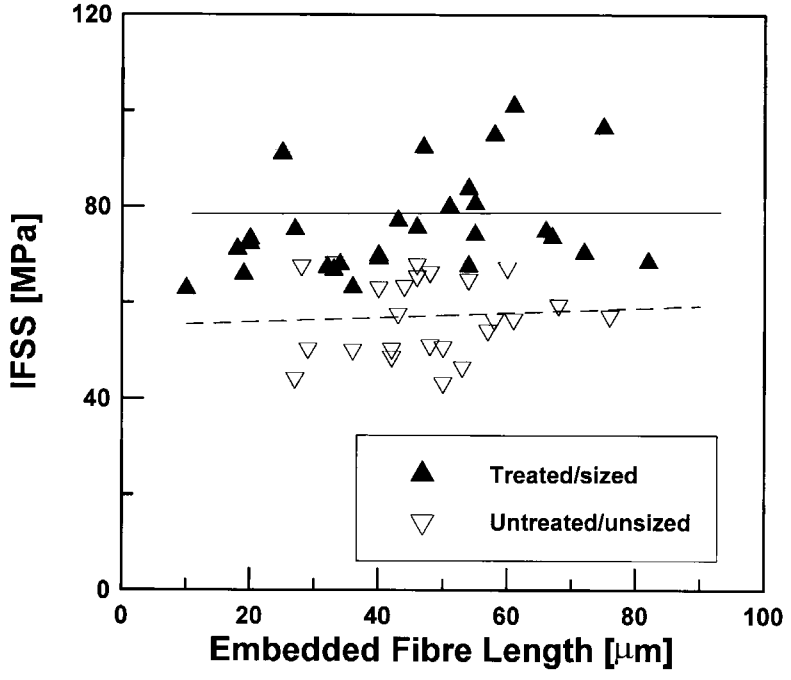


Figure 5. Interfacial shear strength *versus* embedded fibre length for G34-700 fibres.

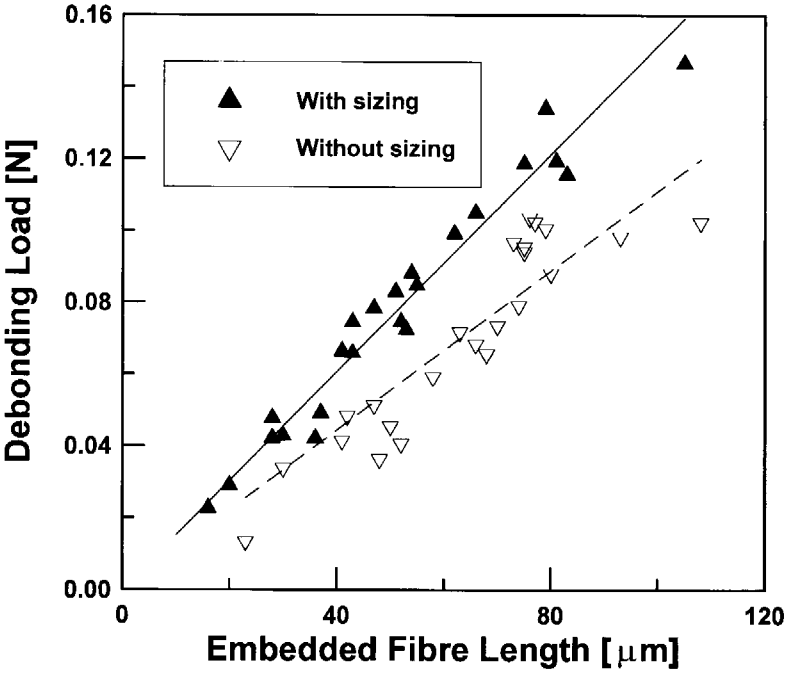


Figure 6. Debonding load *versus* embedded fibre length for T700S fibres.

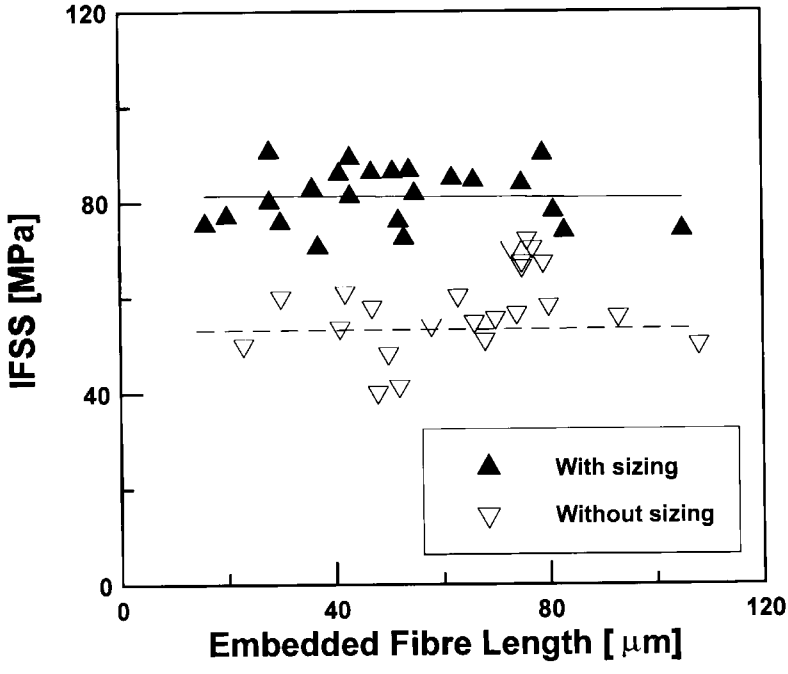


Figure 7. Interfacial shear strength *versus* embedded fibre length for T700S fibres.

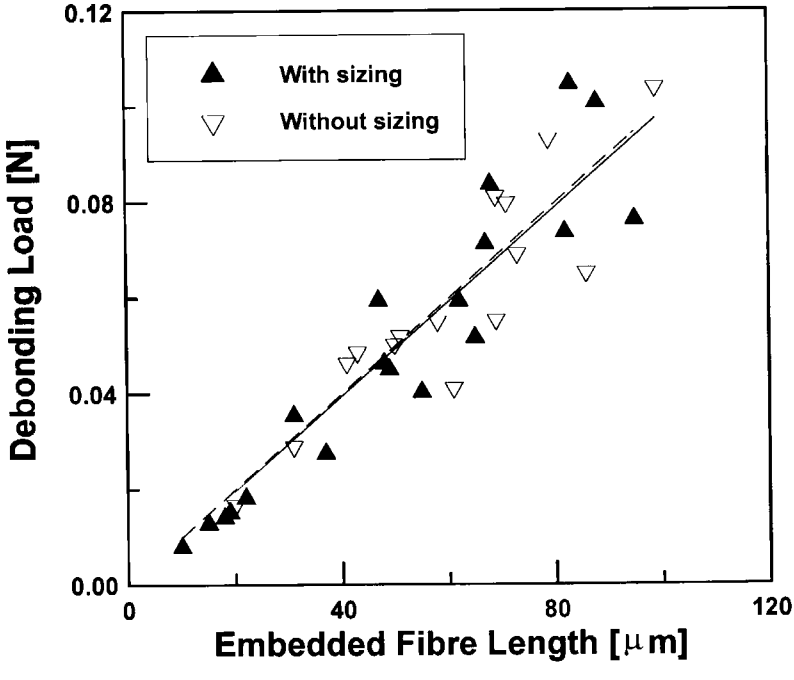


Figure 8. Debonding load *versus* embedded length for T800H fibres.

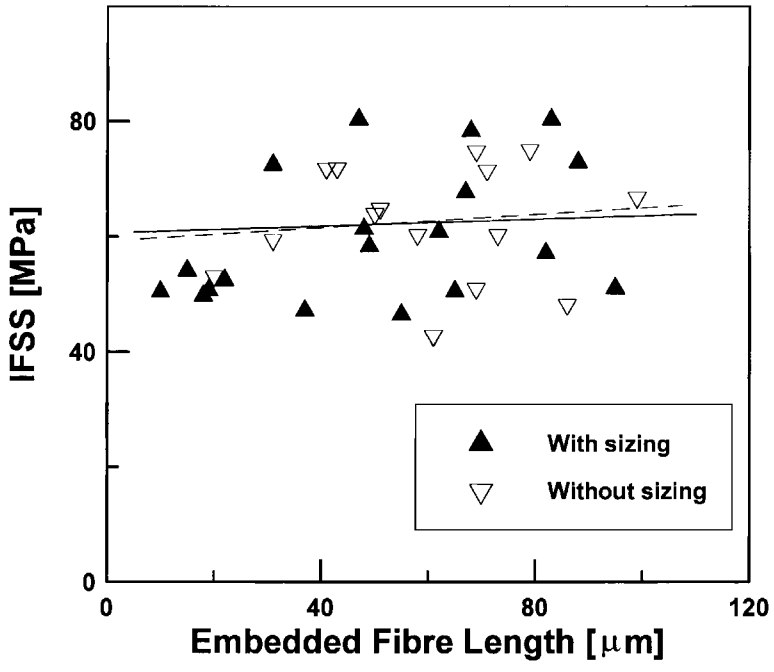


Figure 9. Interfacial shear strength *versus* embedded length for T800H fibres.

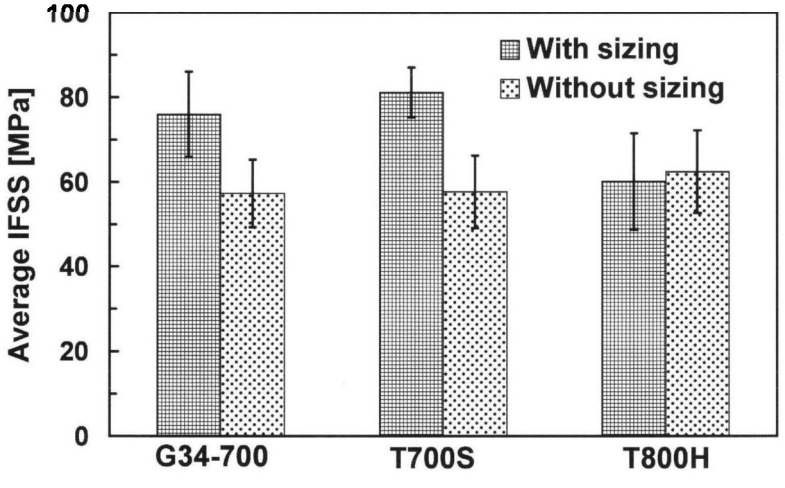


Figure 10. Comparison of interfacial shear strengths for G34-700, T700S and T800H carbon fibres in Araldite-F epoxy.

sizing on the IFSS values was found for the T800H/Araldite-F model composite system (Fig. 9). In recent years various methods in relation to fibre surface treatments and sizing have received extensive attention [7–10]. For carbon fibre–epoxy systems, a fundamental relationship has been shown to exist between fibre surface chemicals and interfacial adhesion. It is believed that some surface treatment methods

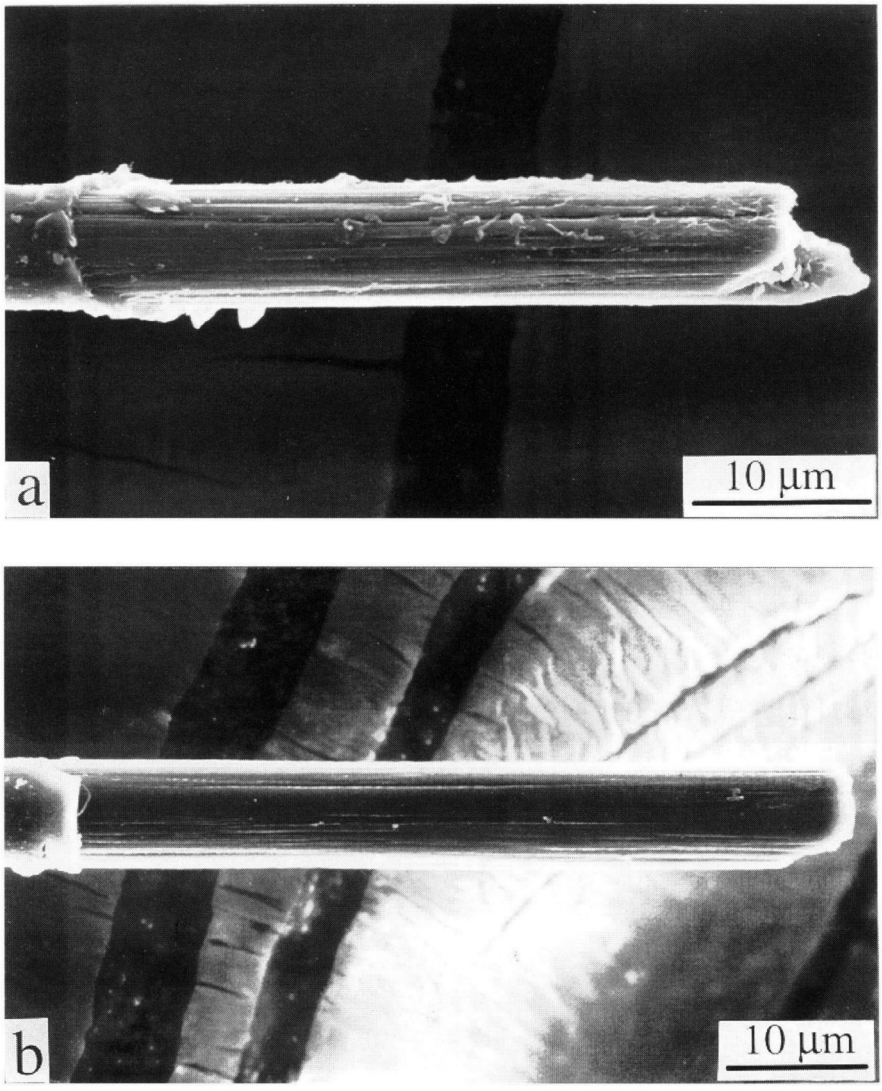


Figure 11. SEM micrographs of typical G34-700 carbon fibres after pulled-out from the resin (carbon fibre diameter is 7 μm): (a) treated/sized; (b) untreated/unsized.

such as oxidation treatment increase the content of oxygen-containing groups on the fibre surface, so that the chemical reaction between fibres and resins is enhanced. The sizing applied to the G34-700, T700S and T800H fibres with a tiny thickness is generally an epoxy compatible material. Although the exact composition is not known, it is assumed to be an epoxy formulation deficient in a curing agent. Such a formulation is proved to increase the fibre–matrix interfacial shear strength [8, 9]. In the present study, the G34-700 fibres were supplied with two different surface conditions, untreated/unsized and treated/sized. Therefore, the increase of the interfacial shear strength for G34-700/Araldite-F system is attributed to both surface treatment

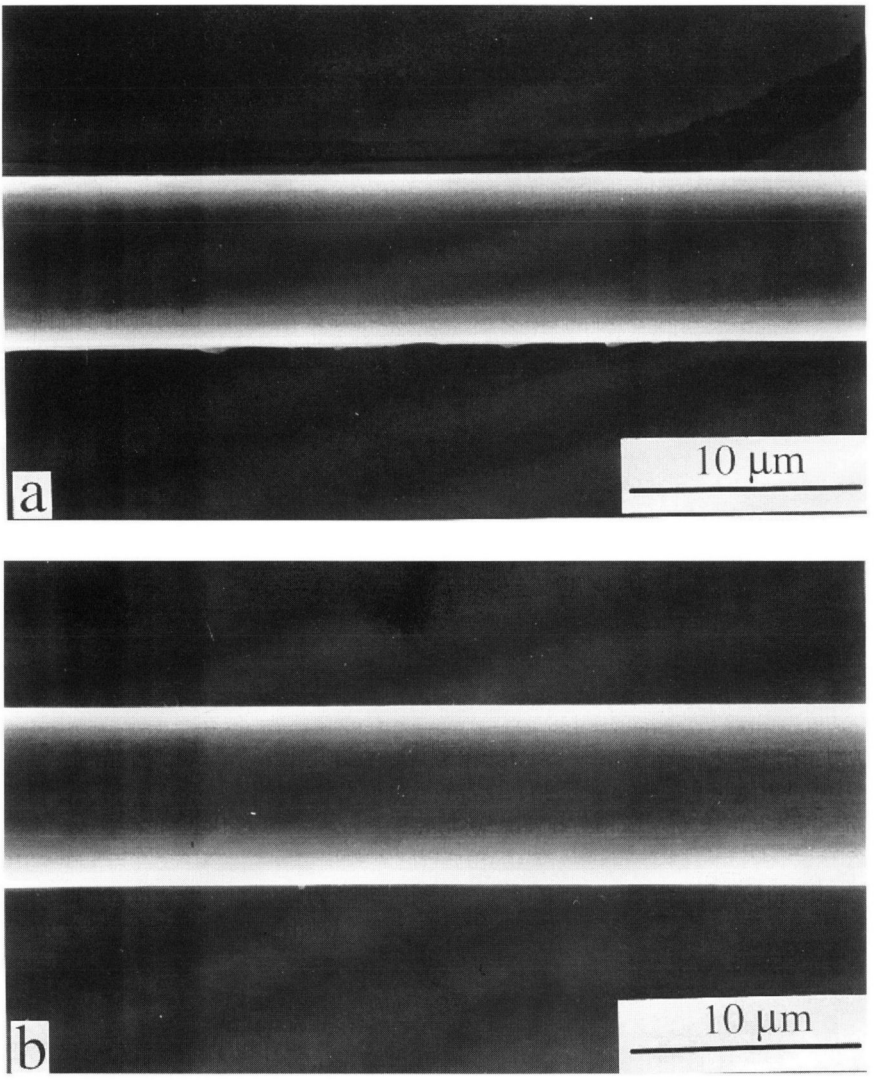


Figure 12. SEM micrographs of T700S carbon fibres: (a) with sizing; (b) without sizing.

and sizing (Figs 4 and 5). For T700S and T800H fibres, since the surface treatment and sizing have been made by the manufacturer, the effect of the surface treatment may still exist after the sizing on the fibre surface was removed using MEK solution. Consequently, Figs 7 and 9 show only the influence of sizing on the interfacial shear strength of the composite systems.

A comparison of the interfacial shear strength between different composite systems was made in Fig. 10. As expected, the untreated/unsized G34-700 and desized T700S fibres show low IFSS values, and the surface treatment and sizing improve the fibre–matrix adhesion substantially. Figure 11 illustrates typical SEM micrographs of the carbon fibres after they were pulled out from the resin. The surface along the

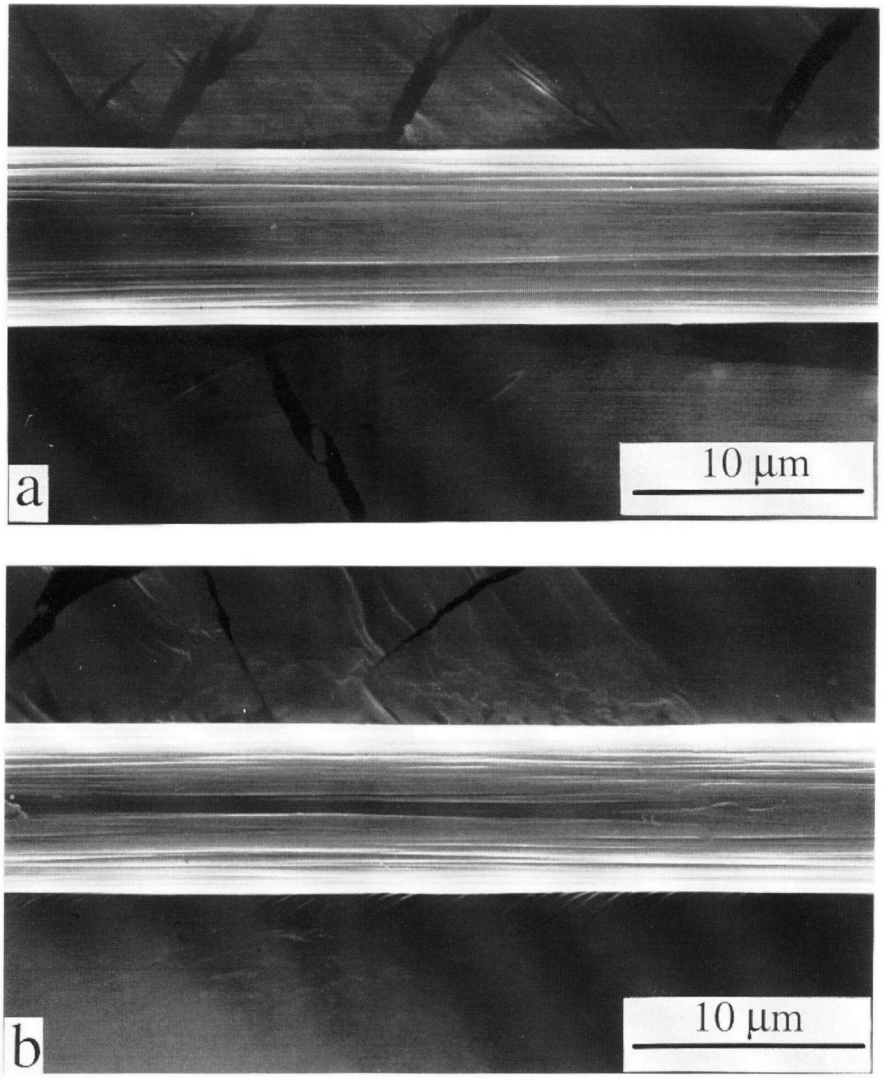


Figure 13. SEM micrographs of G34-700 carbon fibres: (a) untreated/unsized; and (b) treated/sized.

pull-out length of the untreated/unsized G34-700 fibre is almost free of matrix material, indicating a poor interfacial adhesion, but for the treated/sized G34-700 fibre there is some matrix material stuck on the surface along the pull-out length, showing a strong interfacial adhesion.

The sizing on the T800H carbon fibres does not show a clear effect on the IFSS values, which may be attributed to the differences in fibre surface topography and fibre surface chemical compositions. The amount and type of the sizing on G34-700, T700S and T800H fibres are different from each other according to the specifications from the manufacturers. The sizing on the surface of T700S carbon fibres can be clearly seen by SEM (Fig. 12), but the sizing on G34-700 and T800H carbon fibres

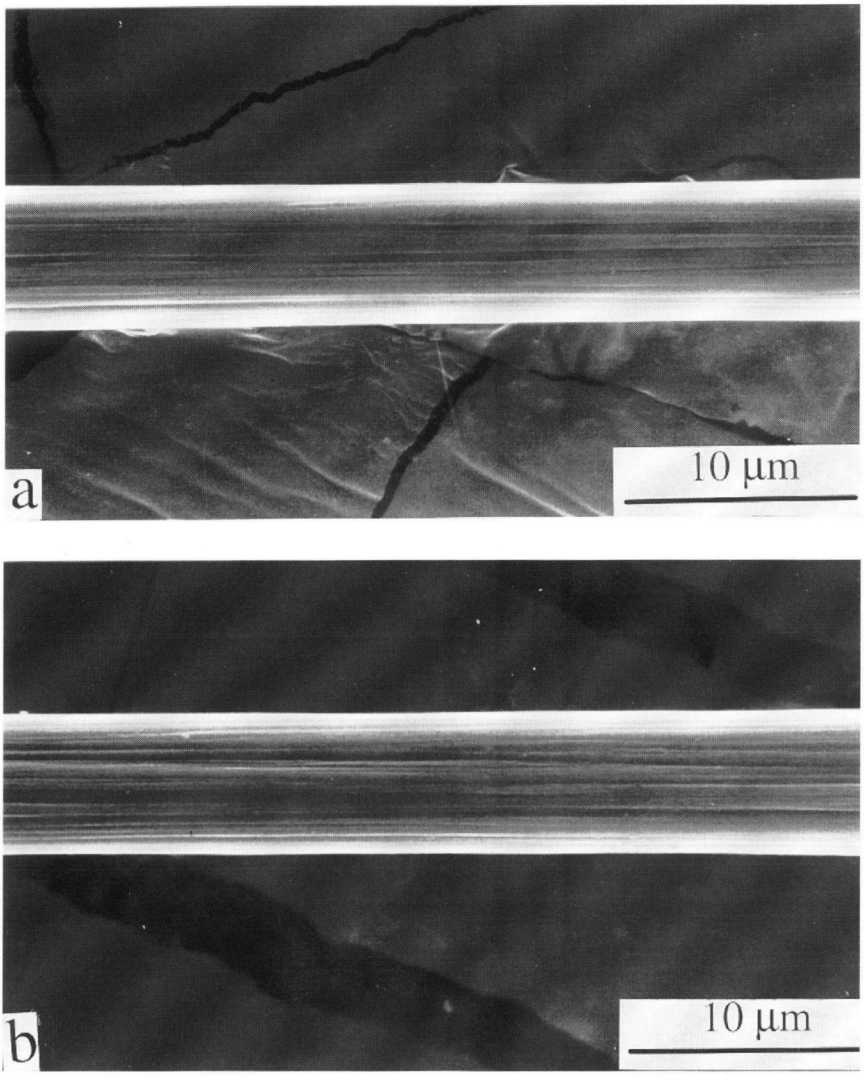


Figure 14. SEM micrographs of T800H carbon fibres: (a) with sizing; (b) without sizing.

cannot be identified (Figs 13 and 14). The fibre surface topography was also found to be significantly different between the three types of carbon fibres. The surface of T700S fibres is very smooth, but the surface of G34-700 and T800H fibres is rather rough, and tiny surface grooves parallel to the fibre axis can easily be seen (Figs 13 and 14). The fibre surface roughness may have an over-riding influence on the IFSS values compared with the sizing for the T800H/Araldite-F model composite. However, for the G34-700S/Araldite-F system, the double effects of the fibre surface treatment and sizing on the IFSS exist, which could improve the fibre–matrix adhesion greatly though the fibre surfaces are rough, so that a noticeable increase of the IFSS for the treated/sized G34-700 fibre model composite can be expected.

4. CONCLUSIONS

The single fibre pull-out test procedure applied in this study makes it possible to reduce the formation of resin meniscus and to accurately define the embedded fibre lengths, and it also provides a practical approach to conveniently estimate the IFSS for comparison of the fibre–matrix adhesion between various composite systems or different fibre surface conditions. A strong interfacial adhesion and an unstable debonding process without detectable friction stages were found for all three types of model composites. The electrochemical oxidation treatment and/or sizing on carbon fibres noticeably increased the IFSS of the composites.

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REFERENCES

1. Kelly, A. and Tyson, W. R. Tensile properties of fibre-reinforced metals: copper/tungsten and copper/molybdenum. *J. Mech. Phys. Solids* **13**, 329–350 (1965).
2. Drzal, L. T. and Herrera-Franco, P. J. Composite fibre–matrix bond tests. In: *Engineered Materials Handbook: Adhesives and Sealants*, Vol. 3. ASM International, Metals Park, OH (1990), pp. 391–405.
3. Mandell, J. F., Chen, J. H. and McGarry, F. J. A microdebonding test for *in situ* assessment of fibre/matrix bond strength in composite materials. *Int. J. Adhes. Adhesives* **1**, 40–44 (1980).
4. Miller, B., Muri, P. and Rebenfeld, L. A microbond method for determination of the shear strength of a fibre/resin interface. *Compos. Sci. Technol.* **28**, 17–32 (1987).
5. Herrera-Franco, P. J. and Drzal, L. T. Comparison of methods for the measurement of fibre/matrix adhesion in composites. *Composites* **23**, 2–26 (1992).
6. Gu, X. H., Young, R. J. and Day, R. J. Deformation micromechanics in model carbon fibre-reinforced composite: Part I. The single-fibre pull-out test. *J. Mater. Sci.* **30**, 1409–1419 (1995).
7. Drzal, L. T., Rich, M., Koenig, M. and Lloyed, P. Adhesion of graphite fibres to epoxy matrices. II. The effect of fibre finish. *J. Adhesion* **16**, 1–30 (1983).
8. Drzal, L. T., Madhukar, M. and Waterbury, M. C. Surface chemical and surface energetic alteration of the IM6 carbon fibre surface. In: *Proc. of the American Society for Composites*. Albany, NY (1991), pp. 732–741.
9. Drzal, L. T. and Madhukar, M. Fibre–matrix adhesion and its relationship to composite mechanical properties. *J. Mater. Sci.* **28**, 569–610 (1993).
10. Baillie, C. A. and Bader, M. G. Some aspects of interface adhesion of electrolytically oxidised carbon fibres in an epoxy-resin matrix. *J. Mater. Sci.* **29**, 3822–3836 (1994).