NOTE

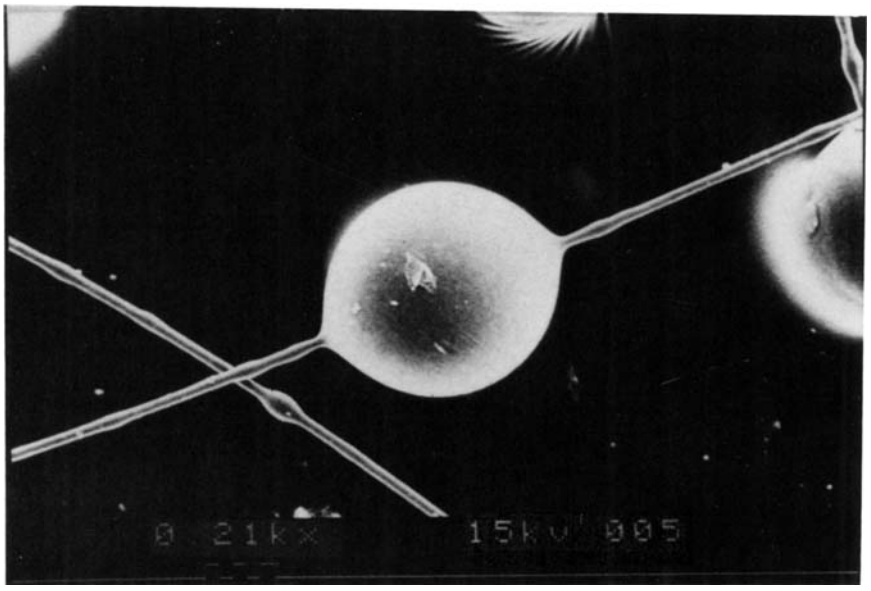
A Solution Microbond Method for Determination of the Shear Strength of a Fiber/Thermoplastic Resin Interface

The interfacial property of fiber-reinforced composites has a great influence on the performance of composite materials. Especially, the interfacial shear strength between fiber and matrix is one of the most fundamental factors in evaluating the mechanical properties of short-fiber-reinforced composites.[1] If the interfacial shear strength is too low, the mechanical properties of composites are controlled mainly by the interface of low shear strength; hence, it is hard to expect that the performance of reinforcing fiber is reflected in composites, even using the high-strength fiber.[2] On the other hand, if the interfacial shear strength is too high, there is a fear of a decrease in fracture toughness of composites because of the poor resistance against the stress crack propagation.[3] Therefore, it is necessary that the interfacial shear strength of the fiber-reinforced composite is controlled in accordance with the performance of material demanded for the final purpose. In principle, the interfacial shear strength will be able to be controlled by the suitable combination of fiber, matrix, surface modification, etc. Then, it is also very important to evaluate exactly the interfacial shear strength thus controlled.

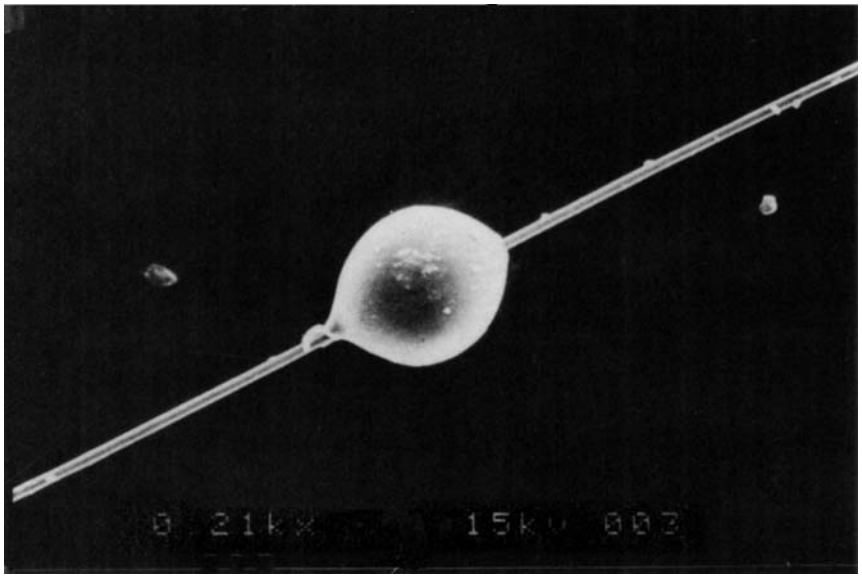
When measuring the interfacial shear strength, preparation of the test specimen is relatively easy for fibers with large-diameters, but extremely difficult for fibers with small (less than 10 μm) diameters, like glass or carbon fibers. Hence, methods such as the three-strand fiber pull-out test,[4] the microdebonding test,[5,6] the pull-out test using the special jig,[7] etc., have been proposed for this purpose. However, none of these methods has given satisfactory results for the correct estimation of the interfacial shear strength of the fiber system with a small diameter because of the problems associated with specimen preparation and meniscus effect. On the other hand, Miller et al. devised a method called the "micobonding test"[8] to remove these two difficulties. All the methods mentioned above, however, are only applicable to the thermosetting resin system, usually present in a liquid form before molding, but not to the thermoplastic resin system, solid at room temperature. There are two main approaches to the preparation of the pull-out test specimen for measuring the interfacial shear strength of the thermoplastic composite system: one, by piercing with a fiber the thin film of the matrix polymer properly heated, i.e., the bonding between fiber and matrix being formed by thermal fusion,[9] and the other, by tying a fiber with the polymer thread already formed, followed by heating it to form a polymer bead on the fiber surface.[10] Although both these methods are fairly efficient for the fiber system with large diameter, they are difficult and unreliable for the small-diameter fiber system, similarly to the case of the thermosetting composite system. Hence, we have developed the so-called "solution microbond method" for the easy application to the carbon fiber thermoplastic resin composite system. According to this method, the microdroplet of the matrix polymer is formed on the fiber surface using polymer molecules dissolved in a suitable organic solvent, and it is subjected to vacuum drying at boiling point long enough to make the solvent evaporate, then finally to reheating above the melting temperature of the matrix polymer to completely remove the residual solvent within the resin droplet by diffusion and evaporation.

To test the validity of this method, the application to the carbon fiber/high-density polyethylene (HDPE) composite system was first made, where the organic solvent used was toluene. From the IR analysis, it was confirmed that there was no solvent remaining in the completely molded resin droplet. SEM microphotographs in Figure 1(a) and (b) illustrate the shapes of microdroplets thus obtained before and after the pull-out test, respectively. In particular, from Figure 1(b), we can see that the shape of a microdroplet remains nearly intact even after the pull-out test. The pull-out test results made on the single fiber for the carbon fiber/HDPE system are illustrated in Figure 2. In this figure, the pull-out force, i.e., the tensile force required for bond breakage between fiber and matrix, is plotted against the length of the fiber embedded in the matrix resin, where the symbols ○ and ◐ represent the tensile force at debonding and the pure frictional force between fiber and matrix after debonding obtained at given fiber-embedded length, respectively. From the slopes of two lines (for ○ and ◐) determined from regressional analysis, we can estimate the mean in-

Journal of Applied Polymer Science, Vol. 44, 561–563 (1992)
 CCC 0021-8995/92/030561-03$04.00



(a) before pull-out test



(b) after pull-out test

Figure 1 Typical microdroplet shape. Carbon fiber in polyethylene resin droplet.

terfacial shear strength and the mean frictional shear strength, yielding the values of 12.94 and 4.22 MPa, respectively.

From the above consideration, we may conclude that the present solution microbond method is very effective for the evaluation (by the single-fiber pull-out test) of the interfacial shear strength for the carbon fiber/thermoplastic resin composite system, provided that a suitable solvent for the matrix polymer is available. In addition, the investigation regarding the effect of the surface modification of carbon fiber on the mechanical properties of fiber-reinforced composites based on thermoplastic resins like polyethylene, polypropylene, and nylon 6 is being carried out using the solution microbond method.

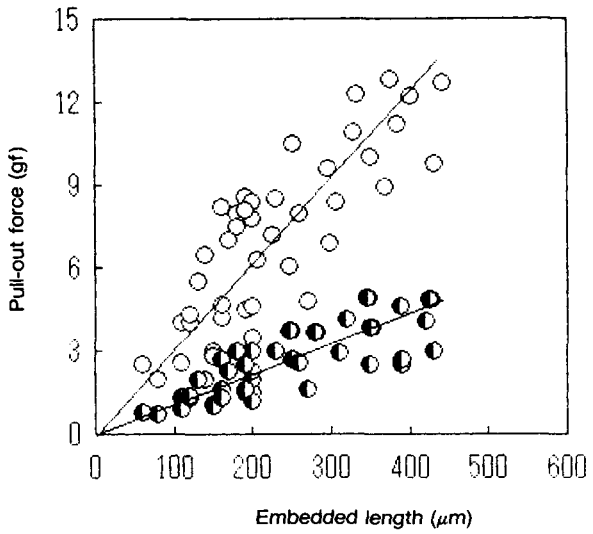


Figure 2 Pull-out force vs. fiber-embedded length. Carbon fiber in polyethylene resin droplet. (○) Tensile force at debonding; (◐) frictional force after debonding.

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Received February 11, 1991
Accepted April 8, 1991

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