Effect of Solubility and Miscibility on the Adhesion Behavior of Polymer-Coated Carbon Fibers with Vinyl Ester Resins

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ABSTRACT: Interfacial shear strength (IFSS) of carbon fibers with vinyl ester resin was investigated as a function of the structure of the polymer coating on carbon fibers via microdroplet tests. For coating carbon fibers, high-performance polymers such as poly-(arylene ether phosphine oxide) (PEPO), Udel® P-1700, and Ultem® 1000, water-soluble poly(hydroxy ether ethanol amine) (PHEA), water-dispersed carboxy-modified poly(hydroxy ether) (C-PHE), and water-insoluble poly(hydroxy ether) (PHE) were utilized. Adhesion of polymers to carbon fibers was also evaluated and the failure surface of the fibers was analyzed by SEM to understand the adhesion mechanism. Diffusion between polymers and vinyl ester resins was investigated and the solubility parameters of the polymers were calculated, with the results being correlated to the IFSS. A highly enhanced IFSS was obtained with PEPO coating, while marginally improved IFSS resulted from PHE, Udel®, and C-PHE coatings. However, PHEA and Ultem® coatings showed no improvement. Such results were attributed to the extent of solubility and/or miscibility of polymer coatings in vinyl ester resin, with better solubility and miscibility leading to a higher IFSS. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 79: 1042–1053, 2001

Key words: interfacial shear strength; vinyl ester resin; carbon fiber; polymer coating; microdroplet test; solubility; miscibility

INTRODUCTION

Vinyl ester resins are widely utilized thermoset resins in composites and coating applications due to their good electrical and mechanical properties, as well as excellent corrosion resistance.[1–3] They are prepared by the esterification of epoxy resins with methacrylic or acrylic acid to provide the vinyl end groups, followed by the addition of styrene monomers to reduce the viscosity of the resin. Thus, they have excellent processability and can be cured by free-radical reaction. This is one of the attractive features of vinyl ester resins.

Recently, much interest has focused on vinyl ester resins for infrastructural composite applications such as bridges. Unfortunately, their inadequate adhesion to reinforcing fibers proved to be a major obstacle in the way of wider usage. Because the performance of composite materials are strongly dependent on interfacial adhesion between fibers and the matrix resin,[4–5] the surface of reinforcing fibers has to be properly tailored to achieve maximum adhesion. To this end, silane coupling agents have been commonly utilized for

Poly(arylene ether phosphine oxide)[PEPO]-NH₂

Udel® P-1700

Ultem® 1000

Poly(hydroxy ether)

Carboxy modified poly(hydroxy ether)
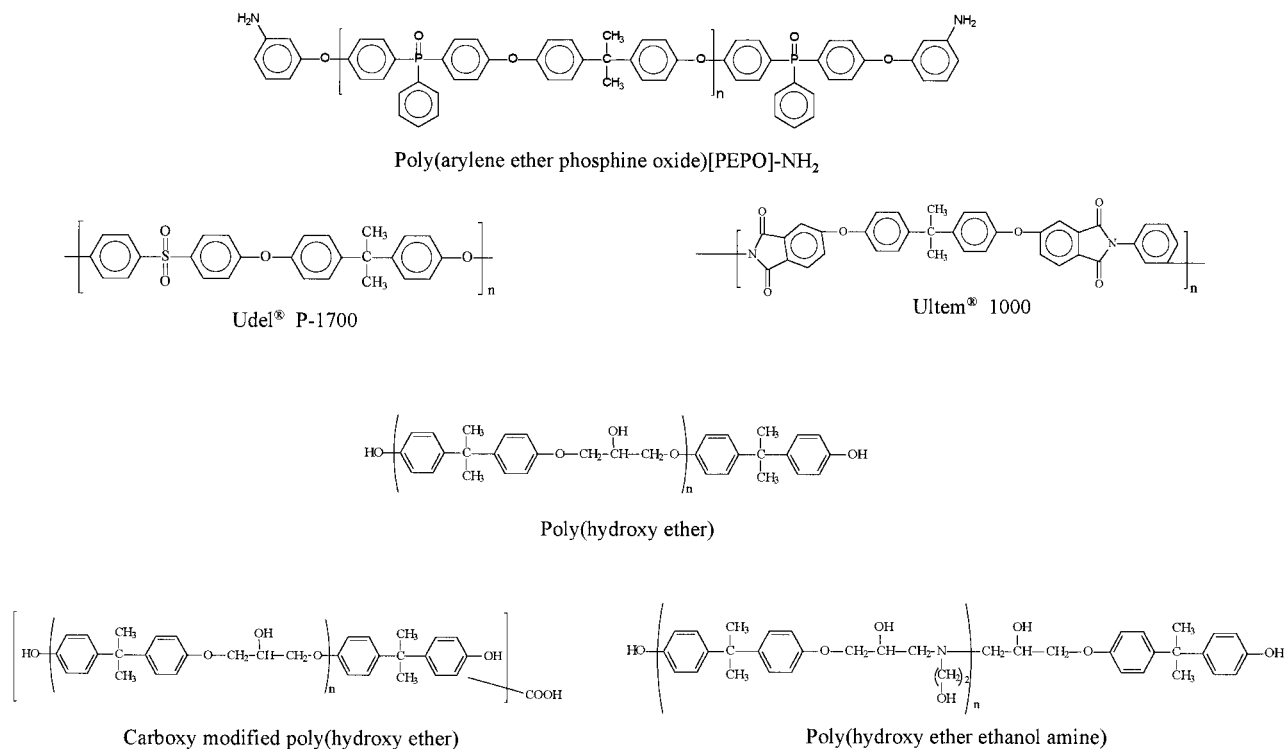
Poly(hydroxy ether ethanol amine)

Figure 1 Chemical structure of polymers: PEPO, Udel®, Ultem®, and poly(hydroxy ethers).

glass fibers,[6–7] whereas methods such as surface oxidation,[8–9] electrochemical deposition,[10] plasma etching,[11–12] plasma polymerization,[13–14] cryogenic treatments,[15] and polymer coatings[16–20] have been employed to modify carbon fibers.

Among them, polymer coatings have received a great deal of attention, having the advantage of enhanced interfacial adhesion as well as improved toughness at the interface. Recently, poly(vinylpyrrolidone),[18–19] poly(arylene ether phosphine oxide),[20–21] and others[22] were successfully utilized to enhance the interfacial adhesion of carbon fibers to vinyl ester resins, or glass fibers to polyurethanes. Improved adhesion, and thus fatigue resistance and mechanical properties of composites, with polymer coatings are attributed to good miscibility or compatibility of polymer coatings with resins.

However, environmental concerns regarding solvent-involved processes have generated an increasing demand for water-soluble polymer coatings for carbon fibers. Therefore, in this study, interfacial adhesion of carbon fibers coated with high-performance thermoplastic polymers such as poly(arylene ether phosphine oxide) (PEPO), Udel® P-1700, and Ultem® 1000 were evaluated.

The results were compared to interfacial shear strength (IFSS) obtained with coating of water-soluble poly(hydroxy ether ethanol amine) (PHEA), water-dispersive carboxy-modified poly(hydroxy ether) (C-PHE), and water-insoluble poly(hydroxy ether) (PHE). Diffusion behavior between polymer coatings and the vinyl ester resin was also investigated via SEM analysis and correlated to the interfacial adhesion and polymer structure.

EXPERIMENTAL

Materials

Vinyl ester resin with 33 wt % styrene (DERA-KANE® 441–400) was donated by Dow Chemical (Midland, MI), whereas styrene monomers (Junsei, Japan) and benzoyl peroxide (Aldrich, Milwaukee, WI) were purchased. PEPO with a molecular weight of 20,000 g/mol was synthesized in our laboratory, as reported elsewhere.[23–24] Commercial polymers such as Udel® P-1700 and Ultem® 1000 were provided by Amoco (Alpharetta, GA) and GE (Pittsfield, MA), respectively (Fig. 1).
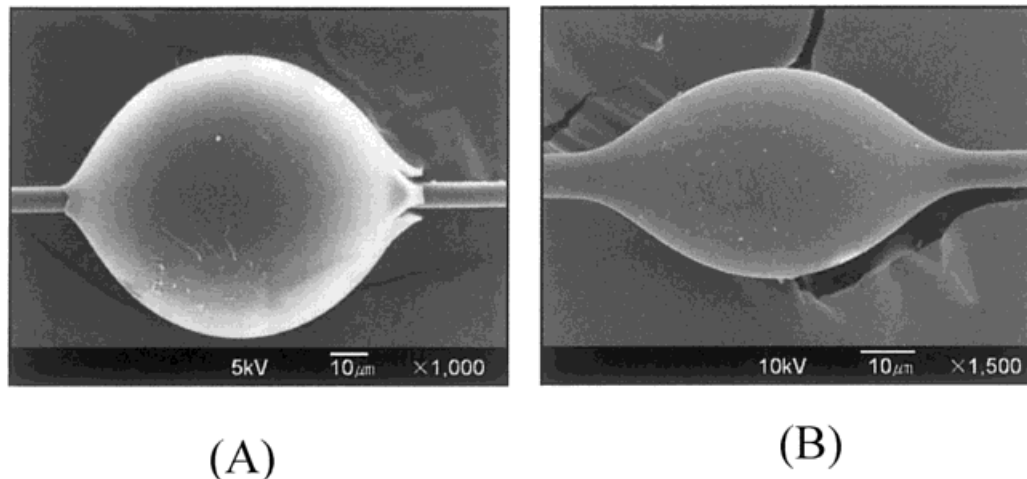
Figure 2 SEM micrographs of vinyl ester and PEPO droplets. (A) Vinyl ester, (B) PEPO.

Water-dispersed C-PHE from Phenoxy Associates (Rock Hill, SC) as well as water-soluble polymers such as PHEA from Dow and water-insoluble PHE from Phenoxy Associates were also utilized. Polymer coatings were applied to unsized AS-4 carbon fibers with an average diameter of 8 μm, provided by Hercules (Wilmington, DE).

Interfacial Adhesion Study

Carbon fiber coating with PEPO, Ultem®, or Udel® was carried out by dipping a single fiber into a 2 wt % chloroform solution for 1 min, followed by drying at 100°C for 12 h. Because C-PHE is water-dispersed, 2 wt % aqueous solution was utilized to coat the carbon fiber. With PHE and PHEA, 2 wt % in THF or aqueous acetic acid (3.75% acetic acid) solution was used, respectively, followed by drying at 100°C for 12 h.

Microdroplet specimens were prepared by a liquid method for vinyl ester resin droplets or film method for polymer droplets, as described previously.[20,25] Vinyl ester resin was mixed with 1.1 wt % benzoyl peroxide and stirred at room temperature until a clear mixture was obtained. Subsequently, the resin mixture was degassed by the freeze/thaw technique to remove oxygen, which is known to be a radical scavenger. A series of droplets was formed by dipping a short single carbon fiber into the resin, then pulling. One of the resin droplets was transferred to a single carbon fiber on an aluminum fixture by contact. The samples were cured under N_2 atmosphere via an optimized cure cycle of 130°C for 20 min as reported.[20]

Polymer microdroplets were prepared by melting a piece of polymer film, which was hung on a carbon fiber. The films were compression molded to afford a thickness of approximately 100–150 μm and cut into 10 × 1.5 mm size. The microdroplets were formed by heating the film on the fiber in an oven at 350–400°C for 5–8 min. The size of the microdroplets was measured by SEM and used in the calculation of IFSS.

The IFSS of microdroplets was measured with Instron 5567 at a speed of 0.3 mm/min. Because the load from the tests was very small, a microbalance (BB 2400, Mettler) connected to a personal computer was utilized to measure the debonding load. IFSS was calculated by
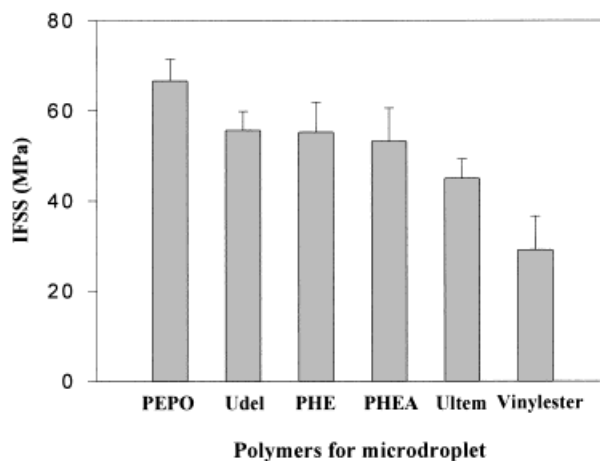

Figure 3 Interfacial shear strength of microdroplet with polymers and as-received carbon fibers.
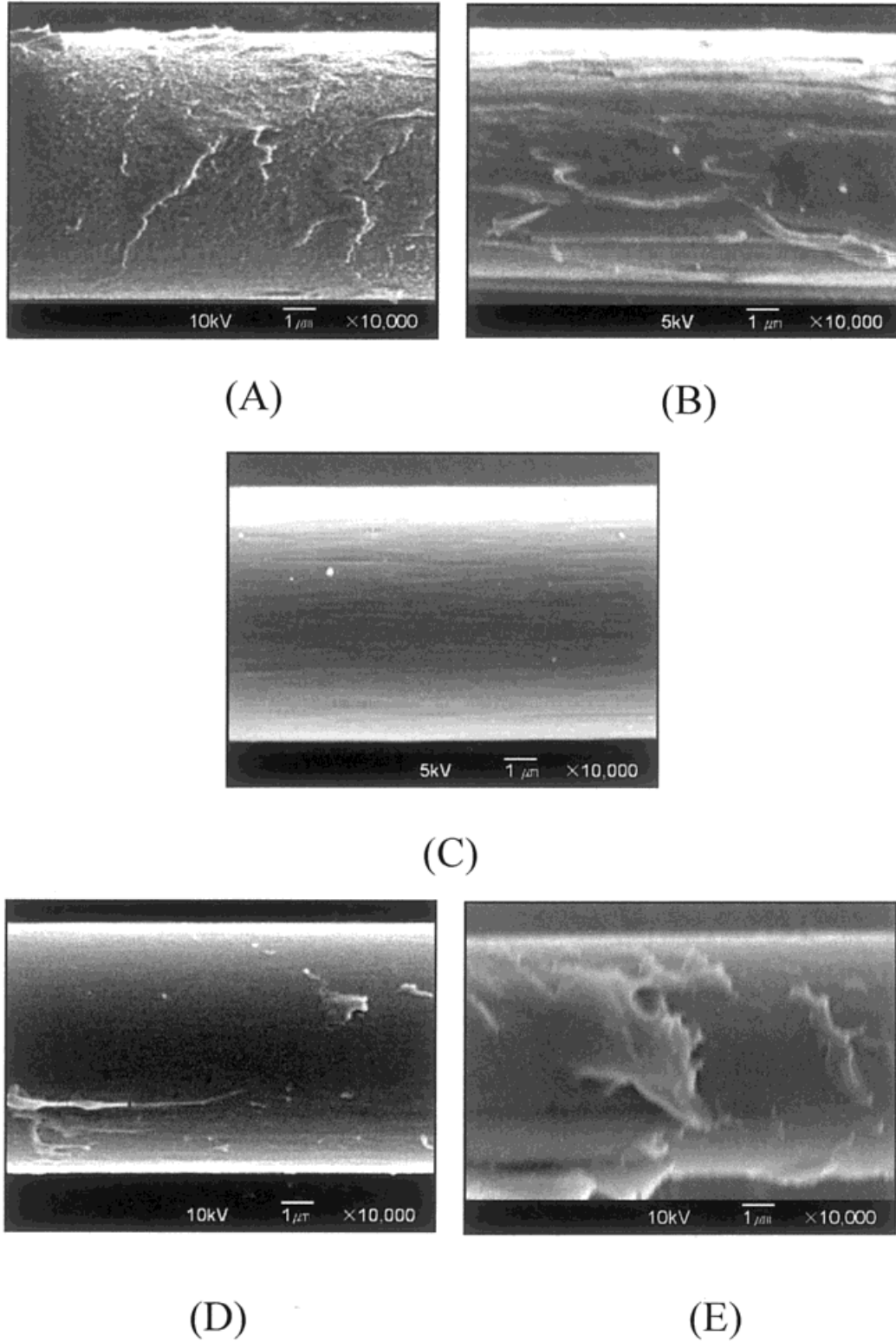
Figure 4 SEM micrographs of carbon fiber after testing. Polymer droplets of (A) PEPO, (B) Udel®, (C) Ultem®, (D) PHE, (E) PHEA.

$$\tau = F_d/\pi d_f L$$

where F_d is the maximum debonding load, d_f is the fiber diameter, and L is the embedded fiber length in a droplet. At least 30 specimens were tested and the results were averaged.

Diffusion Study

The samples for the diffusion study were prepared from a piece of film and vinyl ester resin. Polymer films with a thickness of 100–150 μm were prepared by compression molding and cut into 3 × 6 mm size. Excess styrene monomers were added to DERAKANE® 441–400, which already contained 33 wt % of styrene monomer to afford 40 and 50 wt % styrene, to study the role of styrene in diffusion and thus interfacial adhesion. Styrene monomers were purified by passing them through an aluminum oxide column, followed by vacuum distillation. The specimens (3 × 6 × 15 mm) for diffusion study were prepared by inserting the polymer film into the vinyl ester resin in a silicon rubber mold and by curing at 130°C for 20 min. The samples were cut in half to observe the diffused region and to investigate the diffusion behavior by SEM (JEOL-JSM 5800). SEM samples were coated with Au prior to the analysis. The diffusion behavior of polymers was correlated to their solubility parameters, which were calculated according to group contribution theory.

RESULTS AND DISCUSSION

Interfacial Adhesion of Polymer to Carbon Fibers

Interfacial adhesion of polymers to carbon fibers was evaluated to understand the adhesion mechanism between the polymer coating and fibers. The microdroplets prepared from polymer films were slightly ellipsoidal and 40–80 μm in length (Fig. 2). The IFSS with C-PHE could not be measured, as it tended to degrade rather than melt at 400°C. IFSS was the highest with PEPO at 66.7 ± 4.8 MPa, followed by Udel® (55.6 ± 4.3 MPa), PHE (55.2 ± 6.7 MPa), PHEA (53.2 ± 7.4 MPa), and Ultem® (45.0 ± 4.3 MPa) (Fig. 3).

SEM analysis revealed that the failure surfaces of carbon fibers from PEPO and Udel® seemed to be fully covered with polymer (Fig. 4), attesting to their excellent adhesion to carbon fibers. However, as expected from the low IFSS, the Ultem® samples exhibited clean and smooth failure surfaces, indicating adhesive failure. The
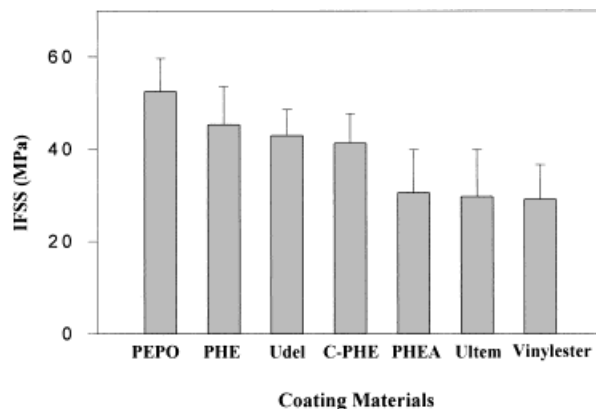

Figure 5 Interfacial shear strength of microdroplet with polymer-coated fibers and vinyl ester resin.

samples from PHE and PHEA droplets showed some polymer residue after fracture (Fig. 4). Therefore, it can be said that failure occurred in the polymer layer of PHE or PHEA (cohesive failure) providing high-interfacial adhesion.

Interfacial Adhesion with Polymer-Coated Carbon Fibers

The microdroplets prepared from vinyl ester resin and polymer-coated carbon fibers were also slightly ellipsoidal and ranged from 40 to 80 μm in length. As expected from polymer droplet studies, PEPO coating exhibited higher interfacial shear strength, at 52.5 ± 7.2 MPa, than PHE (45.3 ± 8.3 MPa), Udel® (42.9 ± 5.7 MPa), and C-PHE (41.3 ± 6.4 MPa) coatings. However, PHEA (30.5 ± 9.4 MPa) and Ultem® (29.7 ± 10.2 MPa) coated carbon fibers exhibited IFSS as low as those of as-received carbon fibers (29.1 ± 7.5 MPa) (Fig. 5).

It is noted that higher IFSS was obtained from PEPO droplets than from vinyl ester droplets with PEPO-coated carbon fiber. The same could be said for all polymers, indicating better adhesion of polymer to carbon fiber than to vinyl ester resin. PHE coating provided very good interfacial adhesion to vinyl ester resin, possibly due to the similar chemical structure of PHE and vinyl ester resin. Water-soluble PHEA coating, however, showed very low IFSS, although PHEA droplets exhibited relatively high IFSS. Therefore, it can be said that adhesion between carbon fiber and PHEA coating is much stronger than that between PHEA coating and vinyl ester resin.

The failure surface of PEPO-coated carbon fibers was almost fully covered by a polymer layer with a thickness of a tenth of a micron [Fig. 6(B)].
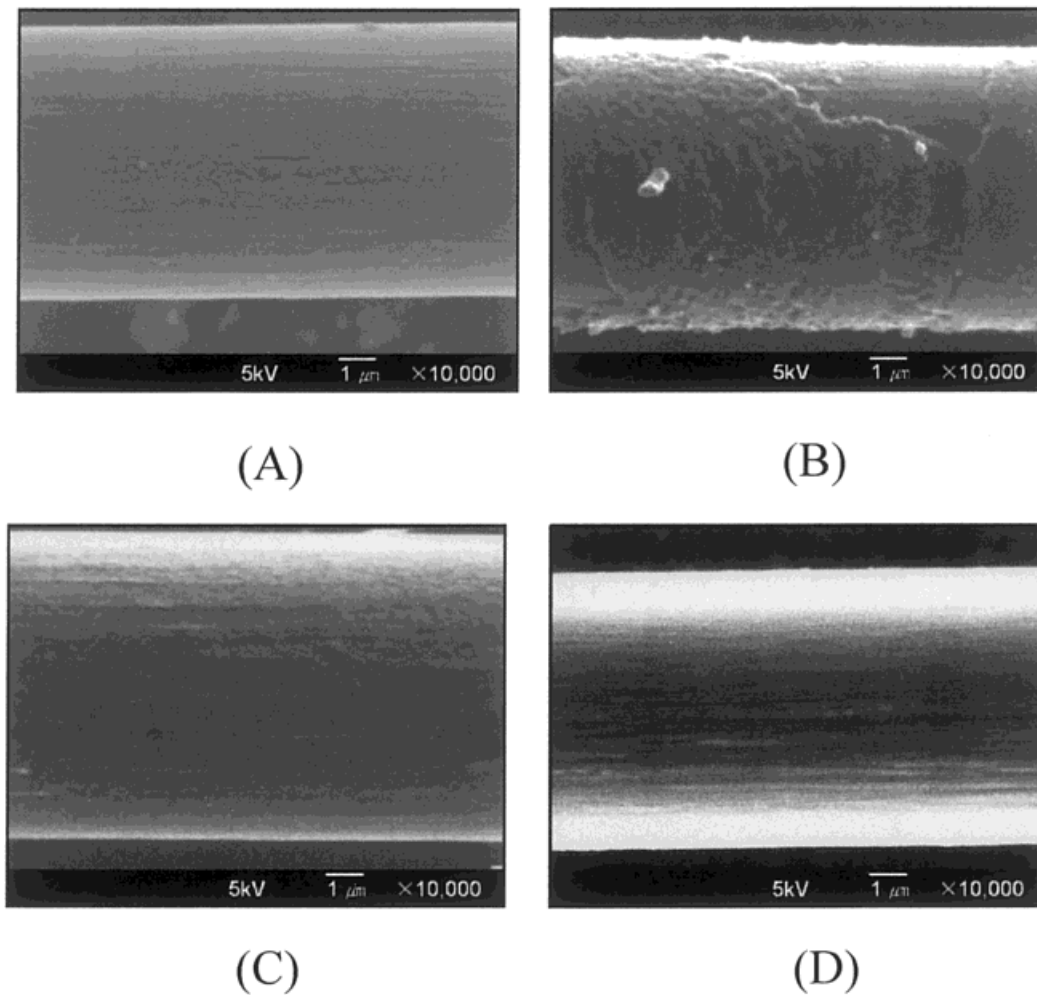
Figure 6 SEM micrographs of polymer-coated carbon fiber after testing, samples prepared from (A) as-received carbon fiber, (B) PEPO-coated carbon fiber, (C) Udel®-coated carbon fiber, (D) Ultem®-coated carbon fiber.

It is believed that the failure occurred in the layer of vinyl ester resin, due to strong adhesion of PEPO coating to carbon fibers and also to vinyl ester resin, resulting in high IFSS. The failure surface of Udel®-coated carbon fibers was slightly rough, but similar to that of Udel®-coated carbon fiber, which points to failure occurring near the interface of Udel® coating and the vinyl ester resin layer [Fig. 6(C)]. The failure surface of Ultem®-coated fibers were relatively smooth [Fig. 6(D)] and somewhat similar to that of as-received fibers [Fig. 6(A)]. Thus, it is believed that the failure occurred at the interface of vinyl ester and Ultem® coating, resulting in low IFSS.

The failure surface of PHE-coated fibers was almost fully covered by the polymer layer, as seen from PEPO-coated fibers, indicating that the failure occurred in the vinyl ester resin layer (Fig. 7),

which explains the high IFSS with PHE coating. However, the failure surfaces of PHEA-coated carbon fibers was smooth and similar to that of Ultem®-coated carbon fibers, owing to interfacial failure, while C-PHE-coated fibers exhibited similar surface characteristics as Udel®-coated fibers, with the exception of white spots.

Diffusion Study

SEM micrographs from PEPO/vinyl ester resin system revealed morphology different from other samples. As shown in Figure 8, it was difficult to differentiate PEPO film from the vinyl ester region, the only clue being that of surface roughness. Interestingly, the rough surface was not observed at 50 wt % styrene, suggesting that PEPO film was completely dissolved in vinyl ester
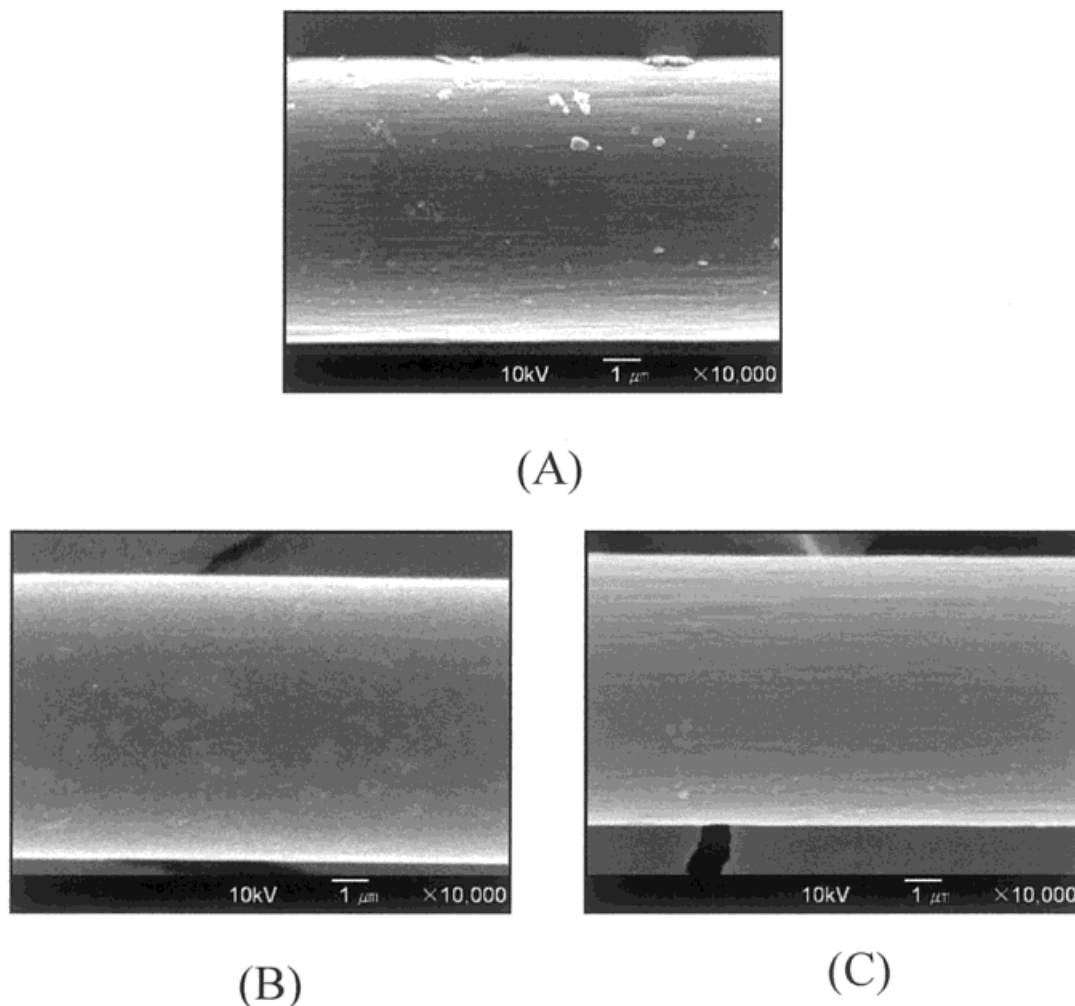
(A)

(B)

(C)

Figure 7 SEM micrographs of polymer-coated carbon fiber after testing; samples prepared from (A) PHE-coated carbon fiber, (B) C-PHE-coated carbon fiber, (C) PHEA-coated carbon fiber.

resin (before cure) and is completely miscible with vinyl ester resin (after cure), possibly due to the strong interaction of the phosphine oxide moiety, as previously reported.[20,26] Therefore, the highest IFSS with PEPO coating is attributed to the excellent solubility and miscibility of PEPO in vinyl ester resin, leading to complete interdiffusion at the interface, which in turn resulted in very good adhesion to carbon fibers, as demonstrated by PEPO droplet tests.

However, the samples with Udel® film exhibited a distinct film region and somewhat clear interface between the film and the vinyl ester region even at 50% styrene, as shown in Figure 9. At 33 wt % of styrene [Fig. 9(A)], the film region can be divided into three parts: outer, inner, and central. The outer part has high roughness and shows small spheres which are believed to be

formed by diffused vinyl ester resins. On the other hand, the inner part was fairly smooth, although the spheres were still observed, whereas the central part seems to be free from diffusion. As the styrene content increased to 40 and 50%, the entire film was diffused with vinyl ester resin, while interdiffusion at the interface and film distortion were observed at 50% [Fig. 9(C)].

It is believed that Udel® film is easily diffused by vinyl ester resin, but is not miscible with it after cure. In other words, vinyl ester resin diffused into Udel® film (before cure) and formed spheres due to immiscibility with Udel® (during the cure). Relatively high IFSS with Udel® coating can be attributed to good diffusion of vinyl ester resin into Udel® film and marginal interdiffusion at the Udel®/vinyl ester interface. Slightly rough failure surface of Udel®-coated fibers in
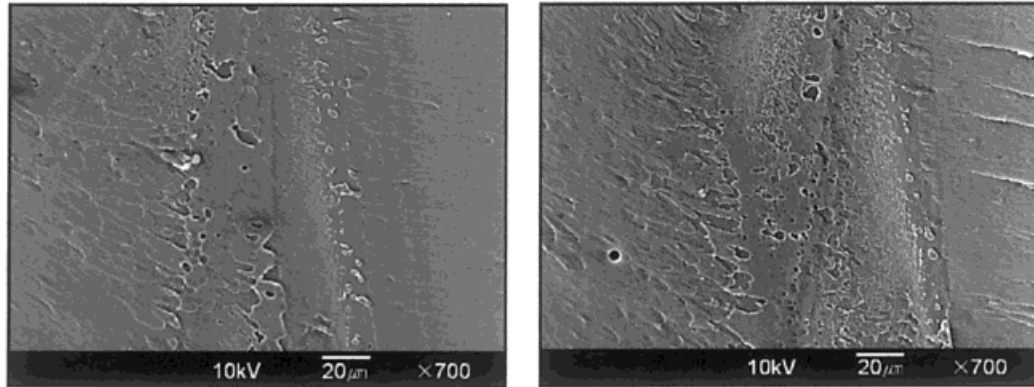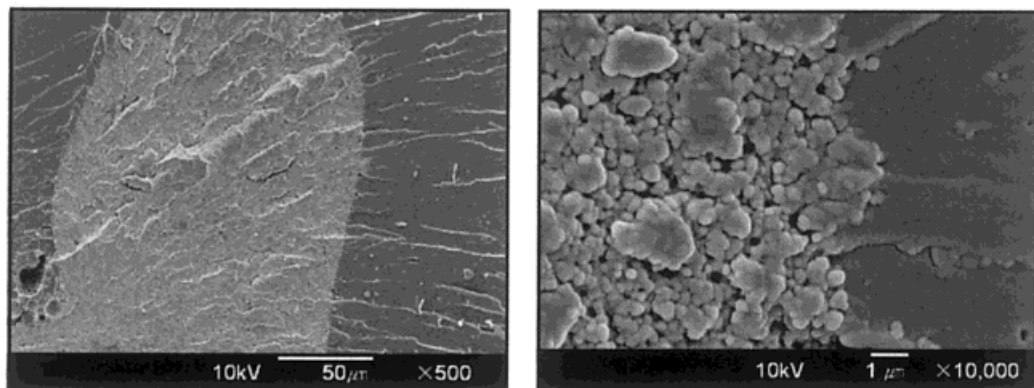
Figure 8   SEM micrographs of diffusion layer of PEPO/vinyl ester resin. (A) 33 wt % styrene, (B) 40 wt % styrene.
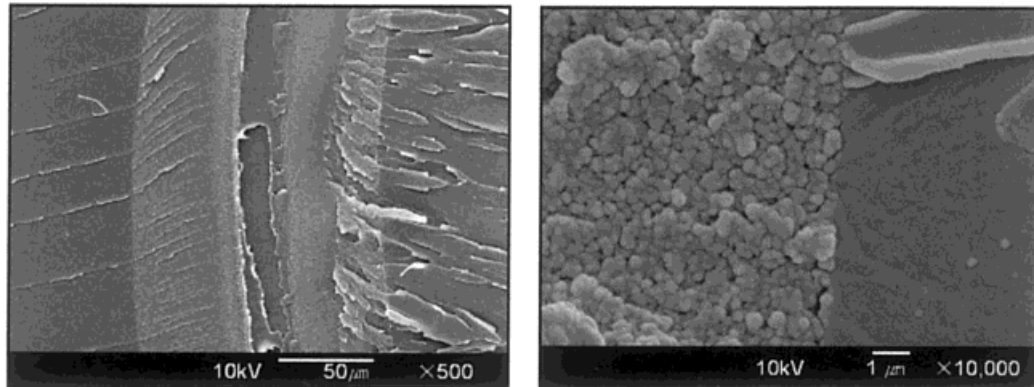
Figure 9   SEM micrographs of diffusion layer of Udel®/vinyl ester resin. (A) 33 wt % styrene (×500), (B) 33 wt % styrene (×10,000), (C) 50 wt % styrene (×500), (D) 50 wt % styrene (×10,000).
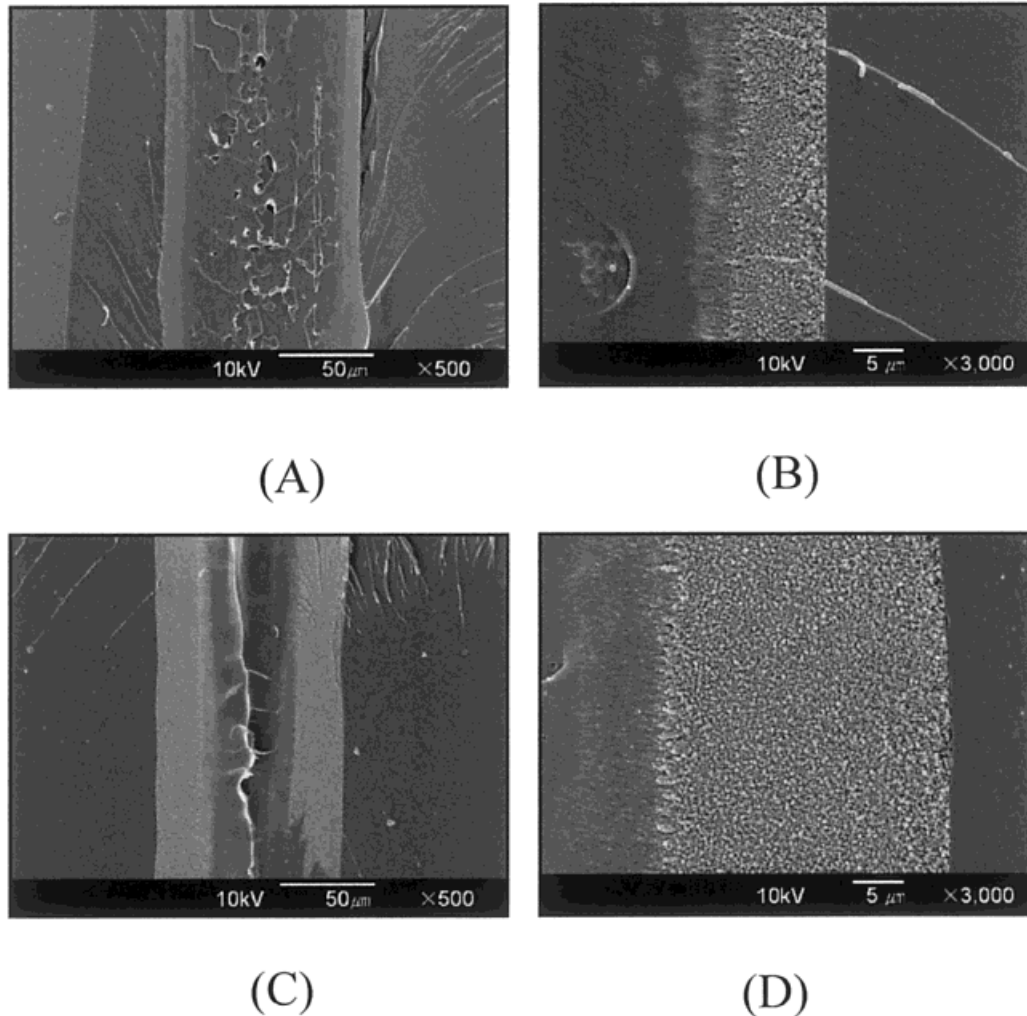
(A)

(B)

(C)

(D)

Figure 10 SEM micrographs of diffusion layer of Ultem®/vinyl ester resin. (A) 33 wt % styrene (×500), (B) 33 wt % styrene (×3000) (C) 50 wt % styrene (×500), (D) 50 wt % styrene (×3000).

Figure 6(C) can be attributed to the spheres formed in the Udel®-coating layer by diffused vinyl ester resin, which is immiscible in Udel®.

As shown in Figure 10, an Ultem® film region with sharp interface was observed. The diffused area of the film region was very small, one tenth of the total film width [Fig. 10(A)], and even at 50% of styrene only half of the film was diffused. At high magnification, small spheres were observed [Fig. 10(D)], as seen in the diffused part of Udel® samples [Fig. 9(B)]. Thus, it can be said that the solubility of Ultem® in vinyl ester resin as well as that of vinyl ester resin in Ultem® is very limited. Moreover, Ultem® is not miscible with vinyl ester resin after curing, as observed from Figure 10(D). Therefore, very low IFSS obtained from Ultem® coating most likely resulted from very limited diffusion of vinyl ester resin in Ultem® and a sharp interface, leading to interfacial failure, which is responsible for the smooth failure surface in Figure 6(D).

PHE samples exhibited a clear film region, but interdiffused interface (or interphase) rather than a sharp interface between the film and the vinyl ester region [Fig. 11(A)]. Spheres were also seen at the edges of the film region, exhibiting poor miscibility of diffused vinyl ester resin in the PHE film. At 40% styrene, most of the film was dissolved, disappearing completely at 50% styrene, which suggests good solubility of PHE in vinyl ester resin [Fig. 11(B)]. Therefore, it can be said that the solubility of PHE in vinyl ester resin was very good,
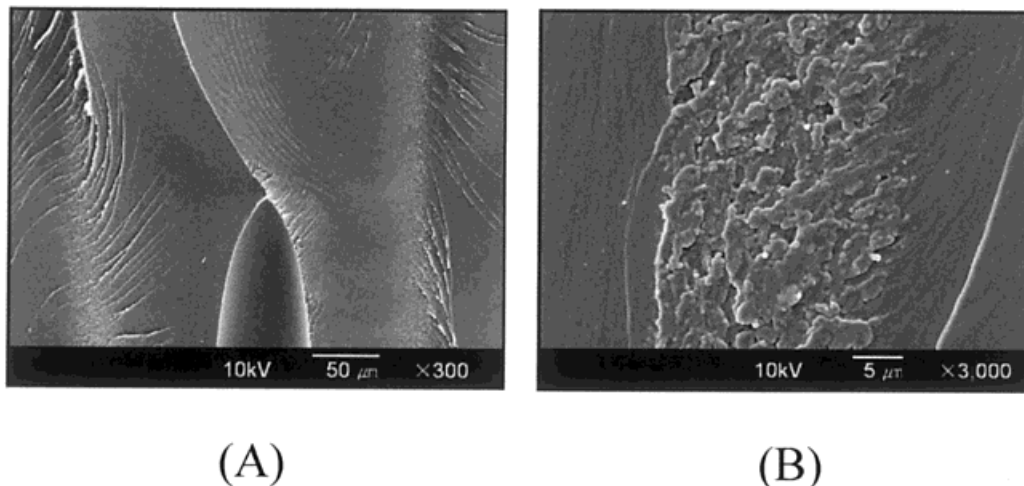
Figure 11   SEM micrographs of diffusion layer of PHE/vinyl ester resin. (A) 33 wt % styrene (×300), (B) 50 wt % styrene (×3000).

whereas the miscibility of vinyl ester resin in PHE was limited, resulting in high, but lower IFSS than that with PEPO coating.

In C-PHE diffusion samples, interdiffused interface (interphase) spheres in the film region and a clear film region were observed even at 50% styrene. At 33% styrene, only the edge of the film was diffused by vinyl ester resin, whereas the entire film was diffused at 50% (Fig. 12). What differed greatly from other samples was the mutual solubility (before cure) and immiscibility (after cure) between vinyl ester resin and C-PHE, as evidenced by the spheres in the interdiffused region. It is most probable that spheres were formed not only by the vinyl

ester resin (dissolved into C-PHE) but also by C-PHE (dissolved into vinyl ester resin). Relatively high IFSS obtained with C-PHE coating can be attributed to good interdiffusion, despite the immiscibility of C-PHE in vinyl ester. The white spots in Figure 7(B) can be attributed to the spheres formed by diffused vinyl ester resin in C-PHE film.

PHEA samples showed a clear interface and very narrow diffused region with spheres (Fig. 13), as observed from Ultem® samples. As styrene content increased, the diffused layer increased slightly, but still remained very small. Limited diffusion of vinyl ester into PHEA film and poor miscibility of vinyl ester resin in PHEA resulted
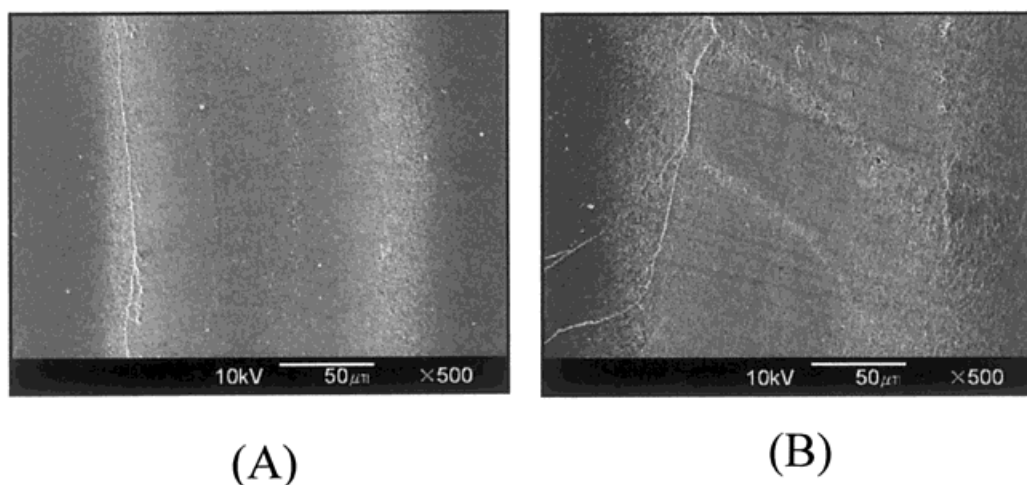

Figure 12   SEM micrographs of diffusion layer of C-PHE/vinyl ester resin. (A) 33 wt % styrene, (B) 50 wt % styrene.
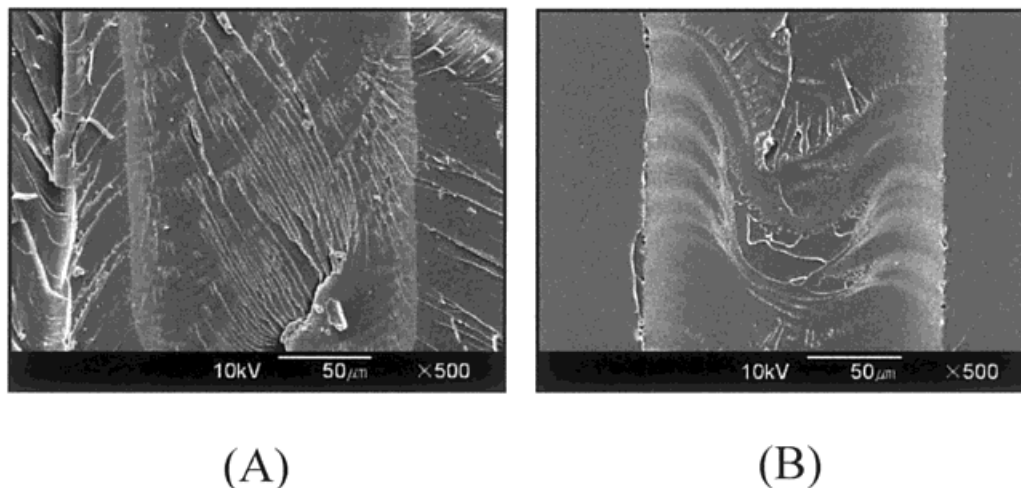
(A)   (B)

Figure 13   SEM micrographs of diffusion layer of PHEA/vinyl ester resin. (A) 33 wt % styrene, (B) 50 wt % styrene.

in very low IFSS and a smooth failure surface in Figure 7(C). PHEA, having a similar chemical structure as PHE, exhibited very different diffusion behavior and IFSS, possibly due to ethanolamine moiety in PHEA which is also responsible for the high IFSS in PHEA droplet tests.

Diffusion behavior of polymer in vinyl ester resin was evaluated in terms of solubility parameters which were calculated via group contribution theory with the table given by Fedor.[27] The solubility parameter of PEPO and Udel were calculated with PO_3 and SO_3 instead of PO and SO_2, respectively, due to lack of data. The solubility of vinyl ester resin (33% styrene) was calculated to be 9.72, which was close to 9.6 reported by Li et al.,[19] but those of Udel and Ultem were 11.58 and 15.24, respectively, which were slightly different from 10.61 and 10.51 found in CRC Handbook.[28] The solubility differences between vinyl ester and polymers corresponded well to the IFSS of polymer-coated carbon fibers, as shown in Figure 14. As expected, PEPO showed the lowest solubility parameter (the smallest difference), whereas Ultem® provided the highest value (the largest difference).

It should be noted that the diffusion behavior of microdroplet samples may be different from large diffusion samples, since the former only deals with a thin layer of polymer coating and rather small droplet size, which affect cure rate and thus diffusion behavior. Diffusion study, however, provided excellent guidelines for a better understanding of the adhesion mechanism in polymer-coated microdroplet samples.

CONCLUSION

The IFSS of carbon fiber/vinyl ester was evaluated via microdroplet tests as a function of the structure of polymers used to coat carbon fibers. The adhesion mechanism was also investigated by diffusion studies. Major findings are summarized below.

1. The IFSS of polymer-coated carbon fibers was greatly increased by PEPO coating, and marginally by PHE, Udel®, and C-PHE coating, but not by PHEA and Ultem® coatings. Increased IFSS with PEPO coating can be attributed to phosphine oxide groups which provided strong interaction
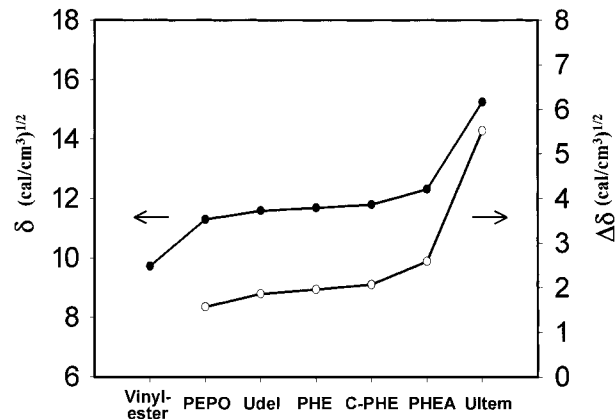

Figure 14   Solubility parameter differences between vinyl ester and polymers for coating.

not only to carbon fibers but also to vinyl ester resin.

2. The highest IFSS was also obtained with PEPO droplet testing, followed by Udel®, PHE, and PHEA, with no improvement observed from Ultem®. However, the polymer droplet samples exhibited higher IFSS than polymer-coated samples, indicating that the adhesion between polymer coating and carbon fiber is stronger than that between polymer coating and vinyl ester resin.

3. PEPO films exhibited excellent solubility and complete miscibility with vinyl ester resin, showing high IFSS. However, Udel®, PHE, and C-PHE have good diffusion of vinyl ester resin but limited miscibility, resulting in marginally improved IFSS, whereas Ultem® and PHEA exhibited very limited diffusion by vinyl ester resin and poor miscibility, showing the same IFSS as the as-received carbon fibers.

4. PEPO provided the smallest difference in solubility parameters with vinyl ester resin, showing the highest IFSS, whereas the largest difference was obtained from Ultem®, which resulted in the lowest IFSS.

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