Synergetic Behavior of Low T_g Polybutadiene Polymers and Polysiloxanes as Components for Glass Fiber Sizing in Composite Materials

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ABSTRACT: The effect of low T_g polybutadiene (PBD) rubbery polymer as a part of polysiloxane/PBD E-glass fibers sizing on mechanical properties of a corresponding epoxy composite material has been evaluated by the punch shear test technique. The results show that the use of hydroxyl terminated PBD led to significant increase in interface shear strength, energy absorption, as well as dynamic modulus and T_g in a corresponding composite material. The sizing composition and fiber morphology were characterized by FTIR spectroscopy and AFM microscopy, respectively. Possible rational for such sizing composition/material property relationship is discussed. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 118: 841–848, 2010

Key words: polybutadiene rubbers; siloxane sizing; composite materials; sizing; interfacial shear strength; energy absorption

INTRODUCTION

The interface layer between fibers and polymer matrix plays a pivotal role in defining mechanical properties of a composite material.[1,2] To achieve optimum stress transfer from the matrix through the interface into the reinforcing fiber, extensive research has been done to tailor the interface adhesion by controlling the degree of chemical bonding between fiber and resin.[3–11] For example, it has been demonstrated that by using compatible silane coupling agent the interfacial shear strength (IFSS) could be increased as much as 40% through controlled chemical bonding.[12] However, avoiding the tradeoff between decline of fracture toughness versus the increase of the shear strength in a composite material still remains a challenge.[13,14]

Several ways were sought to improve both strength and energy absorption of a composite material at the same time. Thus, recent studies have unambiguously shown that the mechanical interlocking created between fiber and resin can significantly increase the impact performance of composites while maintaining its structural integrity. Novel hybrid sizing materials designed by Jensen and McKnight have shown great capability to improve both strength and energy absorption simultaneously, especially during high impact loading using a drop tower test.[15–17] The hybrid sizing used was comprised of a blend of siloxanes compatible (3-glycidopropyltrimethoxy silane, GPS) and incompatible (tetraethoxysilane, TES) with epoxy resin, as well as silica nanoparticles. Such combination leads to considerable increase of interface strength through mechanical interlocking between fiber and resin through higher surface roughness, and energy absorption through frictional sliding. However, despite the growing interest into nanoparticles as fiber sizing component, research into other sizing additives, such as organic polymers, has been largely overlooked. For example, it has been shown that the addition of liquid rubber such as carboxyl-terminated butadiene acrylonitrile polymers to epoxy resins promotes the increase of both adhesive and energy absorption properties of the final composite material part.[18] Thus, the polymer alloy resin obtained exhibited a broad glass transition temperature range, which resulted in the high loss factor ($\eta > 0.1$). The resin also demonstrated higher adhesive strength on aluminum substrates, which yielded high strength and strain energy to failure of the bulk resin. Earlier data also confirm that the addition of epoxidized polybutadiene to epoxy resin can greatly improve the fracture toughness of a corresponding cured material.[19,20] In another example, carbon fibers

grafted with hydroxyl-terminated PBD through the isocyanate linkage showed the threefold increase of impact resistance from 51 to 148 J/m for unsized and polybutadiene /isocyanate sized carbon fibers, respectively.[21] The use of styrene/isoprene copolymer layer as a sizing for glass fibers induced the growth of the flexural strength of a corresponding glass/epoxy composite.[22,23] Finally, our recent studies showed the considerable enhancement for the energy absorption values for E-glass/epoxy composite system with hybrid siloxane sizing containing a combination of hard silica and soft latex nanoparticles, when compared to hybrid siloxane only sizing.[24]

To explore the effect of rubbery polymers as polysiloxane sizing additives on the overall strength and energy absorption of a composite, commercially available hydroxyl-terminated PBD (Chart 1) has been selected as a model macromolecule for this study. As a model composite system, an E-glass fiber/epoxy resin composite has been chosen.

PBD polymer additive has low T_g (< -90 °C) and belongs to the class of liquid rubbers. Moreover, PBD has a C=C double bond, which makes it easily traceable with a variety of analytical spectroscopic methods, e.g., FTIR or NMR.

Herein, we present our study on evaluating the effect of low T_g polybutadiene-based polymers as components of polysiloxane sizing for energy absorption and strength in E-glass fibers/epoxy composites. Particularly, we compare the strength and energy absorption of neat GPS/TES/ polysiloxane sizing with silica nanoparticles, which showed the highest combination of strength and energy absorption amongst the range of polysiloxane-only sizings, against TES/GPS polysiloxane sizing with a combination of hard silica and soft polymer macromolecules, and investigate qualitative siloxane sizing composition as well as the morphology of the fiber surface to establish sizing composition/mechanical properties relationship in E-glass/epoxy composite.

EXPERIMENTAL

Materials and preparations

Solvents [tetrahydrofuran (THF), deionized water] and reagents [tetraethoxysilane (TES), 3-glycidopropyltrimethoxy silane (GPS), hydroxyl functionalized polybutadiene ($M_n = 1200$), Ludox® silica nanoparticles (34 wt % aqueous solution, 20 nm average size)] were purchased from Aldrich and were used as supplied. The Amicure PACM curing agent was purchased from Air Products and the D.E.R. 353 Epoxy Resin was purchased from Dow Chemical Company: both were used as received. The E-glass fibers were purchased from Owens-Corning Fiberglas Corporation.
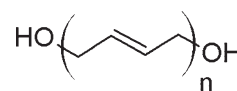
PBD

Chart 1 Chemical structure for hydroxyl-terminated PBD.

Infrared spectra were recorded on a Nicolet Magna-860 Fourier Transform spectrometer (4 cm^{-1} resolution) using KBr discs. All spectra were recorded at ambient temperatures. Surface morphology of sized fibers was studied by AFM microscopy on Veeco Dimension 3100 instrument in a tapping mode. AFM tips (TESPA model) were also purchased from Veeco Instruments, Inc.

E-glass fibers were desized using Sonics Vibra Cell ultrasonic tip. Punch shear tests were performed on Instron 4434 apparatus using Bluehill 2.0 software, and DMA tests were performed on Mettler-Toledo DMA861 instrument.

Sizing of E-glass fabric. General procedure

For fiber sizing solution, 10 mL each of TES and GPS were mixed with 400 mL of deionized water in a 1-L Erlenmeyer flask. Then, 30 mL of 30% colloidal silica was added to this solution, and the solution was stirred on a magnetic stirrer for 1 h. For the TES/GPS/SiO$_2$/PBD sizing, 3 g of polymer (hydroxyl-terminated polybutadiene) was weighed in a separate flask and dissolved in 400 mL of THF, whereupon it was combined with aqueous siloxane sizing solution, and the resulting mixture was stirred for 30 min. This step was omitted for the TES/GPS/SiO$_2$ sizing.

The final sizing mixture was transferred into a bucket and diluted with 2000 mL of 1 : 1 mixture of THF and deionized water. Four of 2′ × 2′ E-glass fiber sheets were completely immersed in this solution and kept soaked for 30 min. Then, the fabric was taken out of the solution, dried first at ambient temperature for 12 h, and then oven dried at 210°F for 12 more hours.

Composite manufacture by vacuum-assisted resin transfer molding

Epoxy SC-15 epoxy resin and curing agent were mixed in 100 : 37.5 ratio (by weight) and degassed under vacuum, whereupon the mixture was infused into four plies of sized E-glass fiber sheets using vacuum-assisted resin transfer molding (VARTM) technique. After infusion, the panels were cured inside at 250°F for 12 h. The panels were then tested to determine the mechanical properties of the E-glass/epoxy composite system and correlate the mechanical properties and morphology with sizing composition.

TABLE I
Selected Physical Properties for Composite Panels with TES/GPS/SiO$_2$ and TES/GPS/SiO$_2$/PBD Sizings

Properties	TES/GPS/SiO$_2$	TES/GPS/SiO$_2$/PBD
Thickness, mm	2.33 ± 0.12	2.42 ± 0.23
Density, g/cm^3	1.91 ± 0.2	1.87 ± 0.2
Fiber volume fraction, %	74.18 ± 0.85	72.36 ± 0.85
Void content, %	1.25 ± 0.11	1.17 ± 0.12

Physical properties measurements of composite panels

Selected physical properties of composite panels manufactured by the VARTM process are listed in the Table I. The thickness of a panel was measured with a digital caliper, and the average of five measurements was recorded. To determine the density of a composite panel, the sample was cut into 2.54 cm × 2.54 cm thickness cubes, the weight in air and water were measured; the composite density was then calculated using ASTM D792-98 dry/wet weight method. Void content was measured according to ASTM D2734-70 standard. After the actual density of a material was determined, the sample and crucible were weighed together. The sample was then placed into a furnace and burned at 550°C for 1 h in air, whereupon the crucible was cooled down and weighed again. The resin content can be calculated as a weight percent from available data. By comparing the actual and theoretical densities, void content was calculated as follows [eqs. (1) and (2)].

$$V = (T_d - M_d)/T_d \quad (1)$$

$$T_d = 100/(R/D + r/d) \quad (2)$$

where V is the void content; T_d and M_d theoretical and measured composite densities, respectively; R, resin weight percent in composite; D, resin density, r, E-glass fiber percentage in composite; and d, E-glass fiber density (2.54 g/cm^3).

The fiber volume fraction is measured according to ASTM D3171-76 standard according to eq. (3).

$$V_f\% = (W \times M_d/d \times w) \times 100 \quad (3)$$

where $V_f\%$ is the fiber volume fraction; W, weight of fiber in a composite; w, weight of a corresponding composite; and d, E-glass fiber density (2.54 g/cm^3).

Selected physical properties of composite panels obtained are listed in Table I.

Quasi-static punch shear test

Punch shear test data reduction was performed according to the previously described method.[25] Punch diameter of 2.54 cm was used with span-to-punch ratio of 2.

The thickness of composite panels was 2.4 mm and 1.8 mm for TES/GPS/SiO$_2$ and TES/GPS/SiO$_2$/PBD sizings, respectively. IFSS was calculated using eq. (4).

$$IPSS = P_{max}/A, \quad (4)$$

where $A = \pi \cdot d_p \cdot l_e$, P_{max} is the maximum load of the load-displacement curve as defined in Figure 1, d_p is the punch diameter and l_e is the thickness of a composite panel.

Energy absorption was divided into three regions: initial deformation (ID), compression shear (CS), and tension shear (TS), according to the different failure modes (Fig. 1).

Total (E_t) and elastic (E_e) energies for a specific region were calculated by eqs. (5) and (6), respectively. Energy absorption (E_a) was calculated as a difference between E_t and E_e according to eq. (7).[25]

$$E_t(\delta) = \int_{\delta 1}^{\delta 2} F(\delta)d\delta \quad (5)$$

$$E_e(\delta) = F(\delta)^2/2 \times K(\delta), \quad (6)$$

$$\text{where } K(\delta) = K_e(\delta)(\delta \leq \delta_e);$$

$$K(\delta) = K_e - \alpha \times (K_e - K_{nl}) \times (\delta - \delta_e)/\delta \ (\delta > \delta_e)$$

$$E_a(\delta) = E_t(\delta) - E_e(\delta) \quad (7)$$

The specific energy absorption (E_{abs}^{sp}) was defined by eq. (8).

$$E_{abs}^{sp} = E_a/l_e \times \rho \quad (8)$$

where E_a is the total energy absorption for the ID, CS, and TS regions, ρ is the density of a composite material and l_e is the thickness of a composite panel.
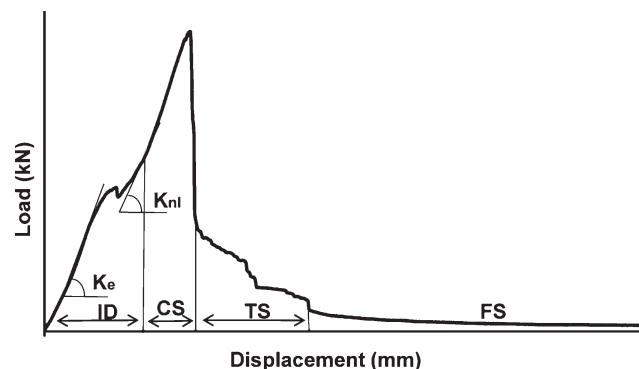

Figure 1 Representative load-displacement curve for the punch shear test showing the distinct deformation regions: Initial Deformation (ID), Compression Shear (CS), Tension shear (TS), and Frictional Sliding (FS).

Dynamic mechanical analysis

The composite panels were subjected to three-point bending test by DMA-861 in the range of 30–200°C. A force of 0.1 N at the working frequency of 1 Hz (10% strain) was applied on composite panels with a heating rate of 2°C/min.

E-glass fiber desizing

Sized E-glass fiber was put into acetone (200 mL) and treated with 5s ultrasound pulse for 7 h at ambient temperature. The solution was decanted off the fiber, stripped to dryness, and analyzed by FTIR spectroscopy.

RESULTS AND DISCUSSION

E-glass fiber sizing and interface chemical analysis

The general sizing mechanism of E-glass fibers with TES/GPS/SiO$_2$ has been discussed previously,[16] where the first stage involves acid catalyzed hydrolysis of labile RO-Si (R = Me, Et) group of a siloxane precursor to HO-Si moiety. Such intermediates further condense with the E-glass fiber surface hydroxyl groups to form Si$_{surface}$–O–Si$_{sizing}$ chemically bonded interface layer. We postulate similar behavior for the TES/GPS/SiO$_2$/PBD sizing. Because of the presence of terminal hydroxyl groups, PBD polymer could also link to the fiber surface/siloxane sizing by the mechanism similar to siloxane condensation to form Si–O–C$_{polymer}$ bridges. Alternatively, our recent studies also suggest that the polymer macromolecules could be incorporated into the sizing even without formation of covalent bonds by virtue of physical absorption.[24]

To confirm the sizing composition on firm basis, the desizing of E-glass fibers was undertaken. Thus, sized fibers were treated with ultrasound in the presence of acetone as extracting solvent. Solutions were stripped to dryness, and the residue was analyzed by FTIR (Fig. 2) spectroscopy techniques.

FTIR spectroscopy confirms the presence of siloxane Si–CH$_2$– stretch as a medium intensity double absorption at 2300–2400 cm^{-1} and the free silanol groups as a broad band between 3200–3500 cm^{-1}; also the absorptions of Si–O–Si and Si–O–C moieties are observed at the range 1030–1090 cm^{-1} as medium/strong bands [Fig. 2(a,b)].[26] Epoxy group in glycidoxypropyl siloxane in GPS/TES/SiO$_2$ sizing is represented by very weak absorption at 909 cm^{-1} almost at a threshold level; however, it grows into strong absorption stretch for TES/GPS/SiO$_2$/PBD sizing (916 cm^{-1}) [C–O stretch, Fig. 2(a,b)]. The IR spectrum of TES/GPS/SiO$_2$/PBD sizing also exhibits the strong C=C double bond vibration at 1642 cm^{-1} [Fig. 2(b)]. The increased intensity of IR absorptions
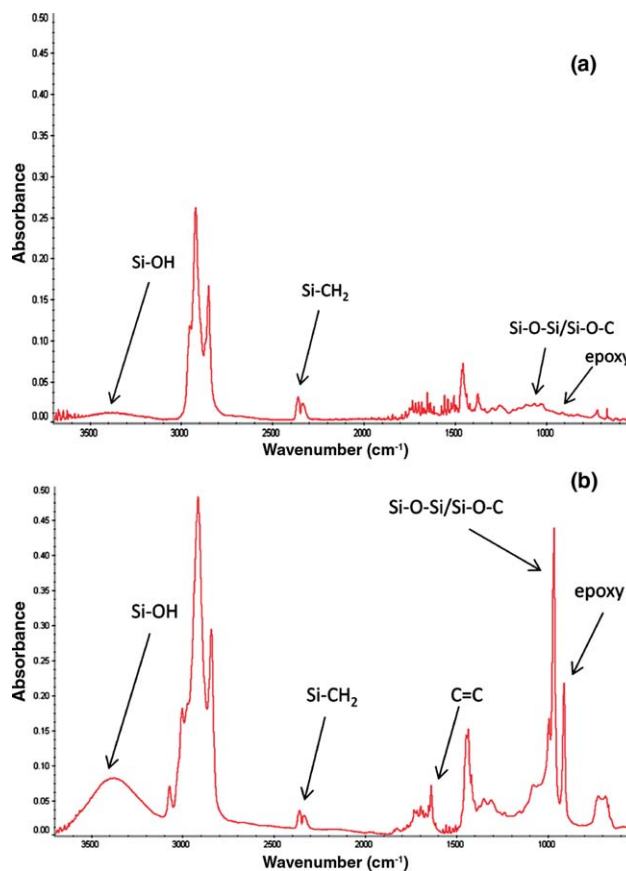

Figure 2 FTIR absorption spectra for (a) TES/GPS/SiO$_2$ and (b) TES/GPS/SiO$_2$/PBD sizing. [Color figure can be viewed in the online issue, which is available at www. interscience.wiley.com.]

for the TES/GPS/SiO$_2$/PBD in comparison with IR spectrum for TES/GPS/SiO$_2$ sizing [Fig. 2(a,b)] could be attributed to higher miscibility of glycidoxypropyl moiety with PBD, and higher solubility of PBD in acetone compared to the polysiloxane-only sizing.

Surface morphology studies of E-glass fibers TES/GPS/SiO$_2$ and TES/GPS/SiO$_2$/PBD sizings

Surface morphology of the sized fabric plays a pivotal role in controlling the interface properties of a corresponding composite material. Hence, in our study, the sized E-glass fabric was examined next by AFM microscopy techniques. The results of the study are shown in Figure 3.

Contrary to the homogeneous surface of the siloxane-only sized fibers [Fig. 3(a,b)], the PBD-containing sizing shows the distinct phase separation behavior on the fiber surface [Fig. 3(c,d)]. Thus, on the phase diagram [Fig. 3(d)], areas attributable to the rigid siloxane and soft PBD domains can be seen. Cross-section surface analysis of the two sizings also confirms the high degree of nonhomogeneity and surface roughness for
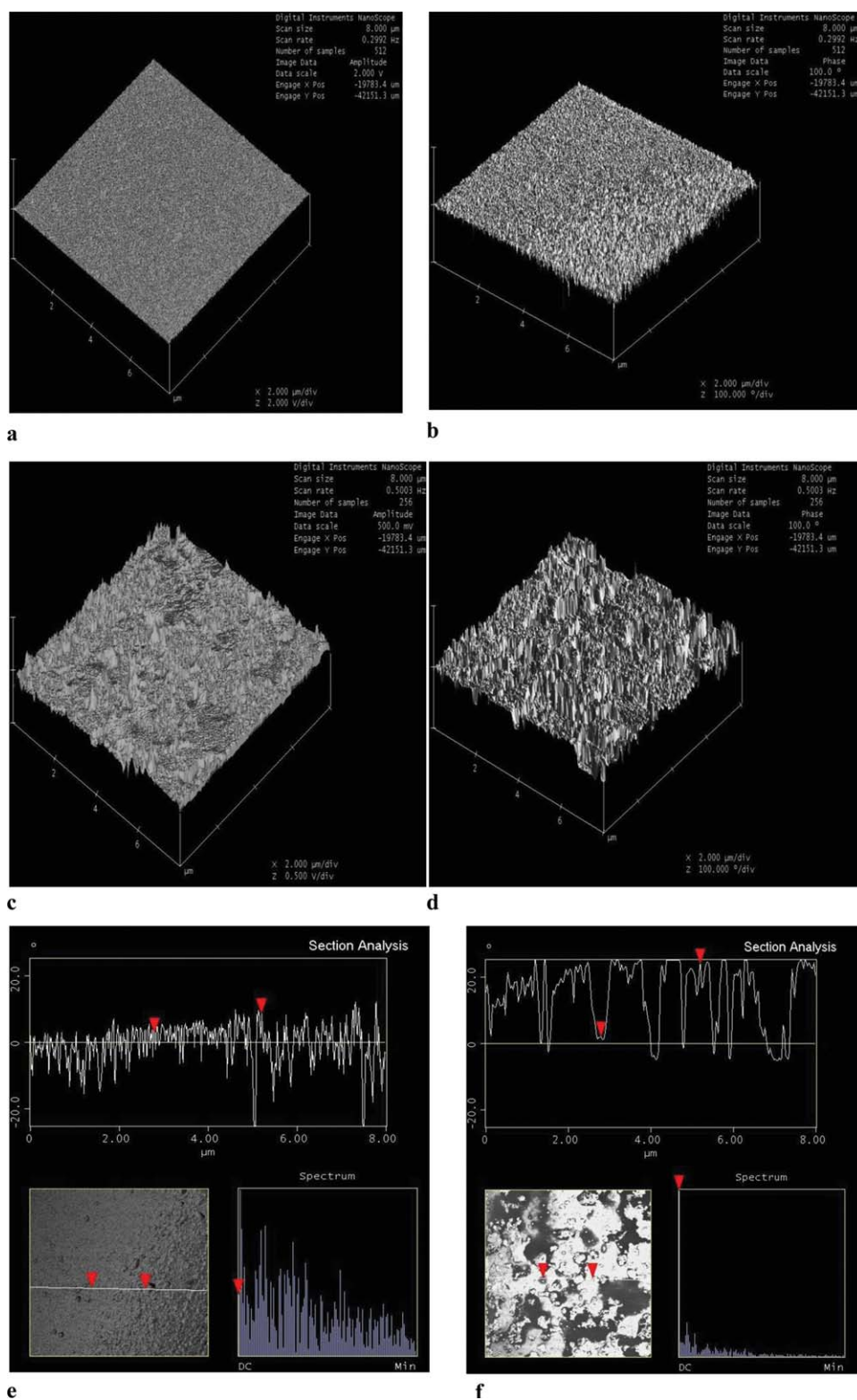
Figure 3 AFM images for (a) TES/GPS/SiO$_2$ sizing amplitude; (b) TES/GPS/SiO$_2$ sizing phase; (c) TES/GPS/SiO$_2$/PBD sizing amplitude; (d) TES/GPS/SiO$_2$/PBD sizing phase; (e) TES/GPS/SiO$_2$ cross-section analysis; and (f) TES/GPS/SiO$_2$/PBD cross-section analysis. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

the sizing [Fig. 3(e,f)]. Based on prior studies[27] and the comparison with the polysiloxane/silica-only sizing [Fig. 3(a,b)], we tentatively assign higher areas to the soft polymer-rich domains, whereas hard crystalline si-

loxane domains tend to locate in the valleys between elevated surface areas. Because of such phase separation behavior the overall surface roughness for the PBD-containing sizing is higher than for the
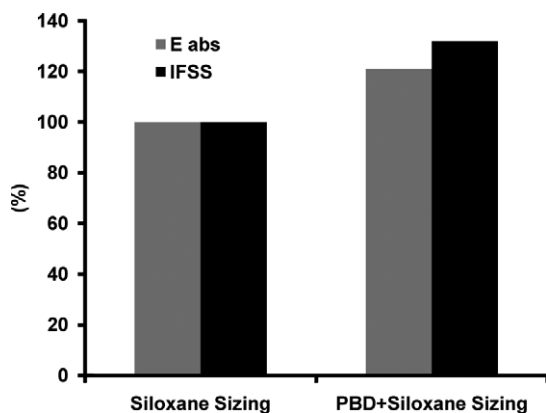
Figure 4 IFSS and total energy absorption for E-glass/epoxy composites with TES/GPS/SiO$_2$ and TES/GPS/SiO$_2$/PBD sizing mixtures.

corresponding homogeneous polysiloxane only sizing with mean RMS for the 3rd order plane fit of 24.8 nm and 18.7 nm, respectively. Such increase in surface roughness and formation of polymer-rich surface domains miscible with epoxy resin for the sizing compared to siloxane-only sizing have the profound effect on the mechanical properties of a corresponding composite material, which will be discussed below.

IFSS, energy absorption, and dynamic modulus evaluation in E-glass/epoxy composites with TES/GPS/SiO$_2$ and TES/GPS/SiO$_2$/PBD fiber sizings

Punch shear test technique has proved to be a reliable macromechanical way to estimate interfacial properties in composite materials such as IFSS and energy absorption.[25] Therefore, this test method was selected as a primary choice for IFSS and energy absorption evaluation in our studies. The results for TES/GPS/SiO$_2$ and TES/GPS/SiO$_2$/PBD sizings are summarized on Figures 4 and 5 [energy absorption values correspond to the total energy absorption at initial deformation (ID), compression shear (CS) and tension shear (TS) regions normalized by the shear volume] and Table II.

The data in Figure 3 and Table I show that the addition of rubbery polymer into the fiber sizing leads to increase in both IFSS and energy absorption by 32% and 19%, respectively. As has been shown on Figure 1, the typical load-displacement curve for the punch shear tests is comprised of three major
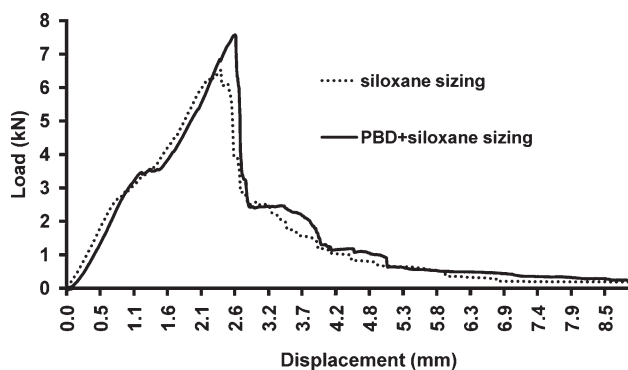

Figure 5 Punch shear test load-displacement curves for E-glass/epoxy composites with TES/GPS/SiO$_2$ and TES/GPS/SiO$_2$/PBD sizing mixtures.

regions: initial deformation (ID), compression shear (CS), and tension shear (TS) regions. Analysis of the load-displacement curves (Fig. 4) shows similar level of energy absorption for both samples at the ID region with matrix cracking being the primary energy absorption mode. The CS region, next to ID, involves debonding between cross-over tows, initiation and propagation of interlaminar delamination, shear cutting of fibers around the punch head as well as formation of a shear plug, apart from continuing matrix cracking. Finally, at the TS region, the shear plug is pushed out through the laminate with tensile fiber fracture taking place at the back side of the laminate. Hence, the energy absorption there is fully governed by tension forces.[25] Further examination of the load-displacement curves (Fig. 4) demonstrates that the energy absorption increase for the TES/GPS/SiO$_2$/PBD sizing mixture comes from both CS and TS regions. We speculate that such effect of the polymer could be attributed to the significant deformation of rubber domains under the compressive shear load, resulting in energy absorption increase. Whereas the energy absorption increase at the CS region suggests that the rubbery domains undergo significant degree of deformation compared to rigid silica nanoparticles and siloxane sizing; the increase of energy absorption on TS region can be attributed to the enhanced interface surface roughness leading to higher frictional forces. Increased IFSS can be rationalized by both higher mechanical fiber-resin interlocking due to the higher interface roughness, and by increased miscibility of polymeric domains in epoxy resin, which also

TABLE II
IFSS and Energy Absorption Data for E-glass/Epoxy Composites with TES/GPS/SiO$_2$ and TES/GPS/SiO$_2$/PBD Sizing Mixtures

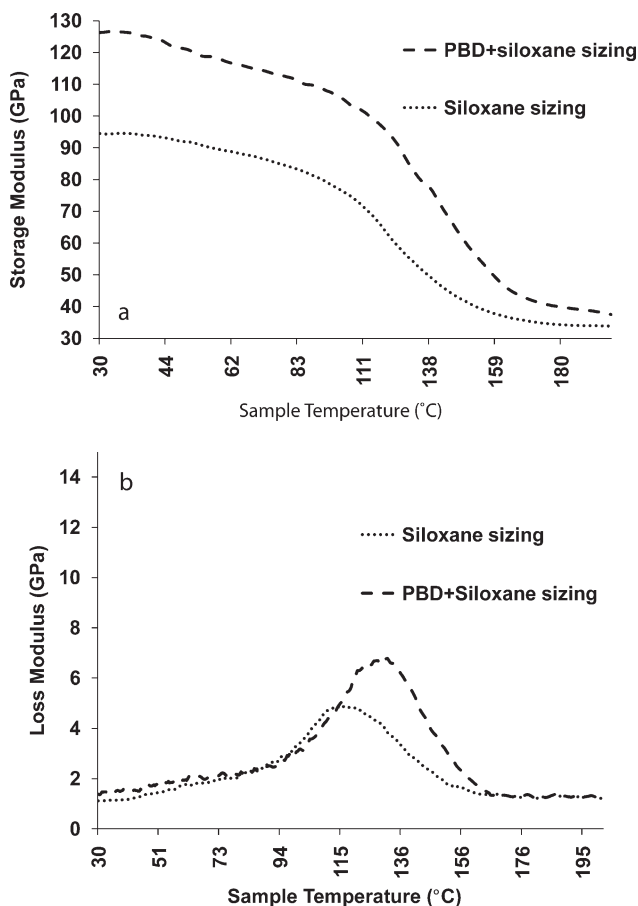| Sample	IFSS (MPa)	IFSS, %	E_{abs}^{sp}, kJ $\times$ m^2/kg	E_{abs}^{sp}, %
TES/GPS/SiO$_2$	76.79 $\pm$ 3.89	100	31.41 $\pm$ 2.94	100
TES/GPS/SiO$_2$/PBD sizing	101.27 $\pm$ 2.74	132	39.18 $\pm$ 3.02	121

Figure 6 (a) Storage and (b) loss modulus–sample temperature DMA curves for E-glass/epoxy composites with TES/GPS/SiO$_2$ and TES/GPS/SiO$_2$/PBD sizing mixtures.

provides additional interface strength. The superior behavior of the PBD-containing sizing versus siloxane-only analog in E-glass/epoxy composite was confirmed by a Dynamic Mechanical Analysis (DMA) test (Fig. 6).

Thus, in a three-point bending test, the composite with PBD-containing sizing exhibited 34% higher storage modulus (E') in a glass region, as well as 23% higher modulus in rubbery region [Fig. 6(a)]. Such behavior is indicative of the fact that higher, though by a smaller margin, IFSS retains by silica/polymer sizing even at elevated temperatures. The analysis of the loss modulus (E'') behavior provides further insights into molecular motion of interface layer [Fig. 6(b)]. First, the T_g for TES/GPS/SiO$_2$/PBD sizing is 14°C higher than for the composite with siloxane only sizing (128°C and 114°C, respectively). As the loss modulus is a measure of dissipated energy or lost as heat per cycle of sinusoidal deformation, it's very sensitive to the change in molecular motions.[28,29] Shift of T_g value to a higher temperature for TES/GPS/SiO$_2$/PBD sizing is indicative of lower segmental motion at the fiber-resin interface due to the increased interface surface

roughness and fiber-resin adhesion. Once again, the DMA studies demonstrate the synergetic behavior of hard silica and rubbery macromolecules as a part of polysiloxane sizing.[*]

These experiments simulate the effect played by low T_g polymers (such as PBD) as components of polysiloxane sizing on strength and energy absorption of a composite material. Further investigations are pursued to optimize the polysiloxane/polymer sizing mixture composition to achieve the best possible combination of strength and energy absorption. The studies are also on the way to compare the effect of a homopolymer as a sizing component on interface properties with related block and random copolymers.

CONCLUSIONS

The data reported here expand our understanding of the role of low T_g polymers on composite material IFSS and energy absorption as fiber sizing components. Thus, rubbery polymer such as PBD can be used as components for polysiloxane-based sizing for E-glass fibers/epoxy resin composites. The use of PBD as polysiloxane sizing component considerably improves both the IFSS and energy absorption of a composite material compared to the hybrid TES/GPS/SiO$_2$ sizing. Moreover, the introduction of a rubbery polymer into the rigid polysiloxane sizing leads to 34% higher dynamic modulus in a glass region, as well as 23% higher modulus in rubbery region. As has been shown, the energy absorption rise comes from both compression shear and tension shear regions due to higher fiber-resin miscibility of polymer-containing surface domains and the resin, resulting in superior interface adhesion. Furthermore, the induced nonhomogeneity of the fiber surface in the latter case leads to increased surface roughness, which can also yield higher energy absorption via elevated frictional forces at the tension shear region. Both factors: increased interfacial adhesion and mechanical fiber-resin interlocking resulting from the rougher surface roughness, also contribute to a significant growth of IFSS. Overall, this study demonstrates that incorporation of rubber-type polymers miscible with polymer matrix into the fiber/matrix interface can serve as a promising route to tailor the mechanical properties of a composite material.

*The corresponding composite material with PBD only sizing was also attempted. However, due to the inherent problems with resin infusibility was not pursued further. Thus, the PBD only sizing impedes resin infusion due to increased interfiber adhesion.

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