Mechanical Properties of Poly(styrene-*co*-acrylonitrile)-Modified Epoxy Resin/Glass Fiber Composites

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ABSTRACT: Poly(styrene-*co*-acylonitrile) was used to modify diglycedyl ether of bisphenol-A type epoxy resin cured with diamino diphenyl sulfone and the modified epoxy resin was used as the matrix for fiber-reinforced composites (FRPs) to get improved mechanical properties. E-glass fiber was used as fiber reinforcement. The tensile, flexural, and impact properties of the blends and composites were investigated. The blends exhibited considerable improvement in mechanical properties. The scanning electron micrographs of the fractured surfaces of the blends and tensile fractured surfaces of the composites were also analyzed. The micrographs showed the influence of morphology on the properties of blends. Results showed that the mechanical properties of glass FRPs increased gradually upon fiber loading. Predictive models were applied using various equations to compare the mechanical data obtained theoretically and experimentally. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 110: 3431–3438, 2008

Key words: epoxy resin; SAN; glass fiber; fracture toughness; mechanical properties

INTRODUCTION

In the past few decades, thermosetting resins have found extensive use in many industrial applications; consequently, extensive development has been made to understand the effect of modifications and resulting properties of such systems.[1–4] Epoxy resins are one of the most important classes of thermosetting polymers, which are widely used as matrices for fiber-reinforced composites (FRPs), coatings, and structural adhesives.[5–7] Epoxy resins are characterized by excellent mechanical and thermal properties, high chemical and corrosion resistance, low shrinkage on curing, and the ability to be processed under a variety of conditions.[8,9] However, because of their highly crosslinked structure, these materials have some undesirable properties such as low toughness and poor crack resistance, which constraint many of its applications. The most successful methods of improving the toughness of epoxy resin are incorporating secondary rubbery phase particles in to the crosslinked polymer[10–12] and the modification with engineering thermoplastics.[13,14] These studies indicate that in most cases, thermoplastic modification resulted in improved toughness of the resin. The toughness of the resulting system depends mainly on its morphology.

The modified epoxy has large number of applications such as matrices for FRPs and structural materials.[15–17] The use of epoxy resin as the matrix for FRPs in structural applications has been increased significantly in recent years. Most of such FRPs offers a combination of strength that is either comparable or better than many of conventional metallic materials. The mechanical performance of such composites mainly depends upon the properties of the matrix and reinforcement and the interaction between the matrix and reinforcement. Nowadays, fiber-reinforced plastic composites are used in thousands of structural applications such as sports and recreation equipment, boats and office products, construction, machinery, housing, etc. Moreover, these composite materials now dominate the aerospace, leisure, automotive, communication, and chemical processing.[18,19]

Usually a composite is expected to sustain both static and dynamic loads without the danger of sudden catastrophic failure. The local response of the fiber–matrix interface within the composite during fracture is particularly important. Glass fiber represents excellent performance reinforcement for FRPs. High strength, light weight, dimensional stability, resistance to corrosion, low cost (compared to aramid and carbon), etc., are major advantages of the glass-reinforced composites. Glass fiber effectively reinforces thermoplastics as well as thermosets. Using glass fiber as reinforcement to make glass fiber-reinforced polymer composites had aroused great interest of

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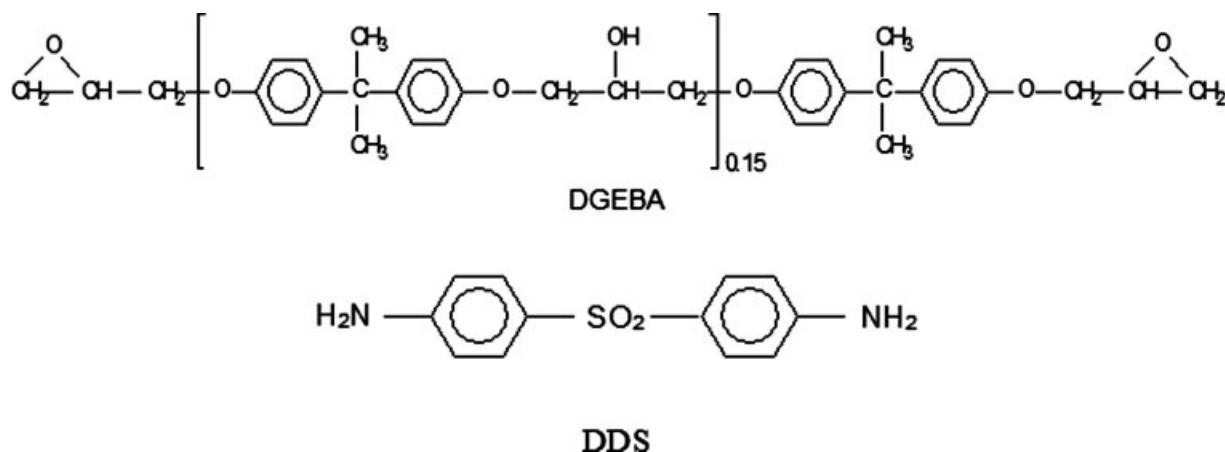
Figure 1   The chemical structures of epoxy resin (DGEBA) and DDS.

materials scientists and engineers all over the world. Many studies have been done in recent years to find an efficient way to improve the interfacial bonding between the fiber and polymeric matrices. Mechanical strength of the composite is dependent upon the amount, type, and arrangement of the fiber within the composite. On the application basis, glass is used as a continuous strand mat, chopped strand mat, milled fibers, and as glass flakes.[20]

The epoxy/poly(styrene-co-acylonitrile) (SAN) composites were also studied by other authors.[21] The morphological and dynamic mechanical analyses of the present system have been discussed in detail in our previous publication.[22] It has been proved that 10-phr epoxy/SAN blend showed good thermal and viscoelastic properties compared to other blends. This investigation concentrates primarily on the mechanical behavior of the blends of bisphenol-A type epoxy resin and poly(styrene-co-acrylonitrile) (SAN). SAN used is commercially available thermoplastic polymer with 25 wt % acrylonitrile (AN) content. In the case of thermoplastic modification of epoxy resins, the toughening effect should be relative to phase structure, interfacial adhesion, and mechanical properties of the thermoplastic used. The main objective of the study is to explore the effect of thermoplastic modification of epoxy resin on the mechanical properties of the blends and the glass FRP developed using the SAN-modified epoxy resin as the matrix. The other objective is to investigate the effect of fiber loading on the mechanical properties of glass FRPs.

EXPERIMETAL

Materials used

Commercially available diglycedyl ether of bisphenol-A (DGEBA) epoxy resin (L-12, Atul Ltd., India) with an epoxide equivalent of 180–200 was used.

The curing agent used was 4,4′-diaminodiphenyl sulfone (DDS) (Atul Ltd.). The poly(styrene-co-acryloni-triole) used was (SAN-A, Bayer, Germany) with 25% of AN content. The chopped E-glass fiber mat was supplied by Ceat Ltd., India. The chemical structures of epoxy resin and DDS are given in Figure 1.

Blend Preparation

Melt mixing technique was employed for preparing all the blends. SAN was first dissolved in epoxy resin at 180°C with constant stirring. After getting a homogeneous solution, stoichiometric amount of DDS was added and dissolved completely without changing the temperature. The resulting solution was poured in to an open mold. The blend was cured at 180°C for 3 h and then post cured at 200°C for 2 h in an oven. After curing, blends were allowed to cool slowly to room temperature. Blends with 0, 5, 10, 15, 20 phr SAN were prepared.

Composite preparation

Compression molding technique was used to fabricate the composites. The matrix for composites was 10-phr epoxy/SAN blend. Glass fiber mat was cut to size and heated in an air oven at 150°C to make it moisture-free before processing. The SAN modified epoxy resin and hardener mixture, before curing, was applied to the preweighed glass fiber sheets. To get 3 mm thickness for the composites, eight layers of fiber mats were added successively. The laminates were compressed in a mold, cured at 180°C for 3 h and then postcured at 200°C for 2 h. The laminates were allowed to cool slowly to room temperature. Six different compositions were prepared, in which the fiber volume varies from 10 to 60 vol %.

Blends were designated as ES5, ES10, ES15, and ES20, where E and S represent Epoxy resin and SAN, respectively; the numbers 5, 10, 15, and 20

TABLE I
Tensile Properties of Epoxy/SAN Blends and Epoxy/SAN/Glass Fiber Composites

Sample	Tensile strength (MPa)	Young's modulus (GPa)	Strain at break (%)
Neat epoxy	51 ± 4	2.6 ± 0.23	3.1 ± 0.11
ES5	63 ± 4	2.5 ± 0.32	4.6 ± 0.12
ES10	70 ± 3	2.6 ± 0.35	4.7 ± 0.09
ES15	53 ± 5	2.6 ± 0.18	3.7 ± 0.06
ES20	48 ± 4	2.5 ± 0.20	2.8 ± 0.02
ESG10	126 ± 8	6.3 ± 0.35	6.9 ± 0.11
ESG20	174 ± 8	7.5 ± 0.32	7.1 ± 0.09
ESG30	203 ± 6	9.8 ± 0.36	7.8 ± 0.11
ESG40	264 ± 9	12.1 ± 0.40	7.3 ± 0.06
ESG50	307 ± 7	16.9 ± 0.35	8.0 ± 0.05
ESG60	231 ± 8	16.2 ± 0.29	7.9 ± 0.10

represent the wt % of SAN (in phr). The composites were designated as ESG10, ESG20, ESG30 and ESG40, ESG50, and ESG60, where G represents the Glass fiber and the numbers 10 to 60 represent the fiber volume percentage.

CHARACTERIZATION

Scanning electron microscopy

The morphology of the blends and composites was examined using scanning electron microscopy (SEM). The SEM of failed specimens was analyzed with a Zeiss FESEM Supra 25 scanning electron microscope.

Tensile tests

Tensile properties were determined according to ASTM D 638. The measurements were taken with a universal testing machine (Schimadzu AGS-1000G) at a cross head speed of 10 mm/min. Rectangular specimens of dimension having $120 \times 13 \times 3$ mm^3 were used for determining tensile strength. The tests were performed on 5–7 different specimens of the same sample and the average was taken as the final value.

Flexural tests

Flexural tests were performed according to ASTM D 790 with a universal testing machine (TNE-500). The sample dimension was $140 \times 13 \times 2.5$ mm^3. The cross head speed was 10 mm/min. The flexural modulus was calculated from the slope of initial portion of the flexural stress–strain curve.

Impact tests

Impact tests were performed on an Instron charpy impact testing machine model PW5. The test method adopted was consistent to ASTM D 256-78 method

B. All the test specimens were un-notched. Impact loading was done with a 15 J-hammer. The tests were performed on 5–7 different specimens of each composition. The sample dimension was $80 \times 10 \times 2.5$ mm^3.

All the mechanical characterization was carried out on 5–7 identical test specimens. The values were deviated very slightly. The error was calculated from different values obtained from the results of 5–7 specimens of the same sample. All the as prepared samples were tested at room temperature. Samples were dried in vacuum at 100°C for 3 h before taking the measurements. The given values of tensile and flexural properties are the maximum at the breaking point of the sample.

RESULTS AND DISCUSSION

Tensile properties

Tensile properties of DDS cured epoxy/SAN blends and epoxy/SAN/glass fiber composites are given in Table I. In the case of blends, the data revealed a remarkable increment in tensile strength for 5 ES5 and ES10 blends. The tensile strength is maximum for ES10 blend. The Young's modulus is increased slightly in the case of ES10 and ES15 blends. The increase in tensile strength in the case of blends may be due to the decrease in the crosslink density of the system The tensile stress/strain curves of the blend systems, given in Figure 2, show that tensile strain is also higher for blends than cured neat epoxy resin. It is known that epoxy is highly brittle. So the samples break before the ductile region showing almost a straight curve for the tensile stress–strain graph. The increase in the tensile properties can be explained using the SEM of tensile fractured surfaces
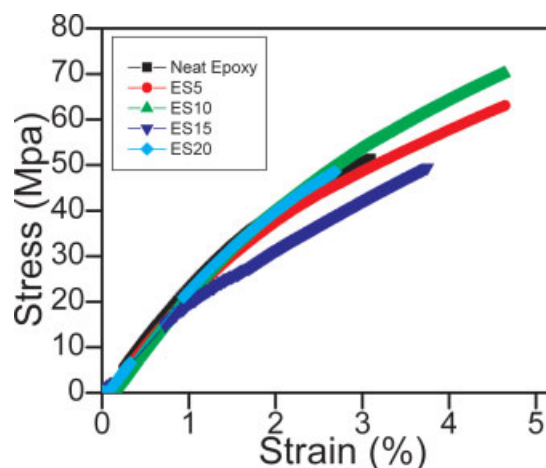
Figure 2 Tensile stress–strain curves of epoxy/SAN blends. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
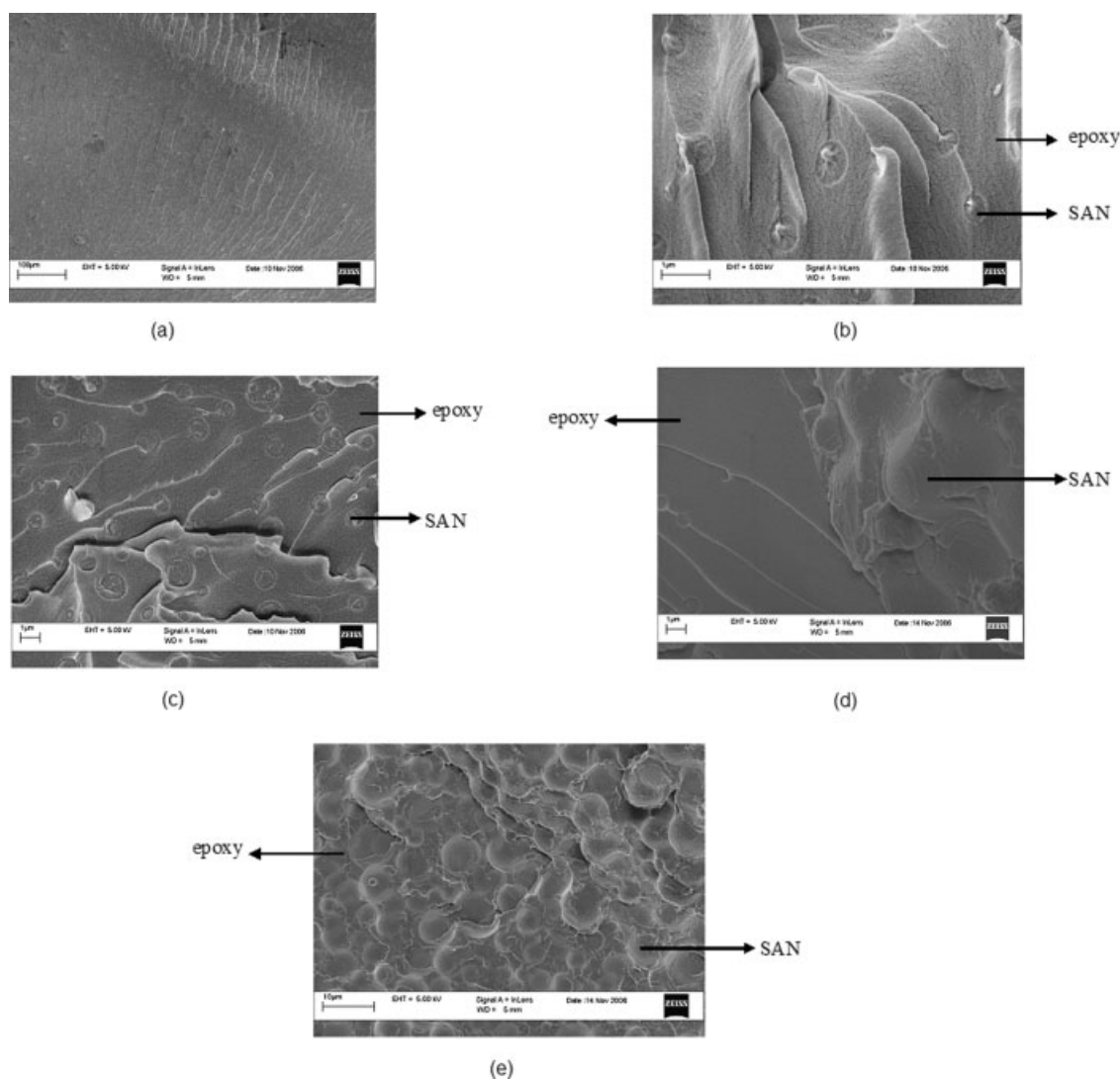
Figure 3 Scanning electron micrographs of failed surfaces of epoxy/SAN/blends. (a) Neat epoxy, (b) ES5, (c) ES10, (d) ES15, (e) ES20.

of blends given in Figure 3. The SEM micrographs of failed surface revealed the two-phase morphology of the blends. The surfaces of blends were rough and ridgy and river marks were observed on the surface. But the surface of unmodified epoxy resin was smooth with free and regular crack propagation, indicating the characteristics of a brittle material. Blends with 5 and 10 phr SAN content showed better properties. This may be due to the highly uniform dispersion of SAN particles in the epoxy matrix as shown in Figure 3. The SAN particles act as stress concentrator on applying external load and that will lead to an improved load bearing capacity of the matrix. The rough surface shows that the systems become more ductile. In the case of ES15 blend, the SEM micrograph shows some plastic deformation on the interface of this phase. The plastic deformation might cause the energy absorption and consequently properties improve.

In FRPs, the applied load is transmitted from the matrix to fiber at the interface. The tensile stress–strain behavior of epoxy/SAN/glass fiber composites with different glass fiber content is shown in Figure 4. The tensile properties are presented in Table I. It can be seen that the tensile strength and modulus increased with increase in the glass fiber content, reaches a maximum in the case of ESG50. When the glass fiber content is 50 vol %, ultimate tensile strength is 307.19 MPa, which is 500% higher than neat epoxy. Tensile modulus was also increased by 550% compared to the cured neat epoxy. The effect of fiber volume on the tensile behavior is also clear from the table. The tensile strength increased by 145% when the volume percentage of the fiber increased from 10 to 50%. A similar increase in the tensile modulus was also observed. A higher increase in the modulus value with the fiber loading can be attributed in part to the high stiffness of glass
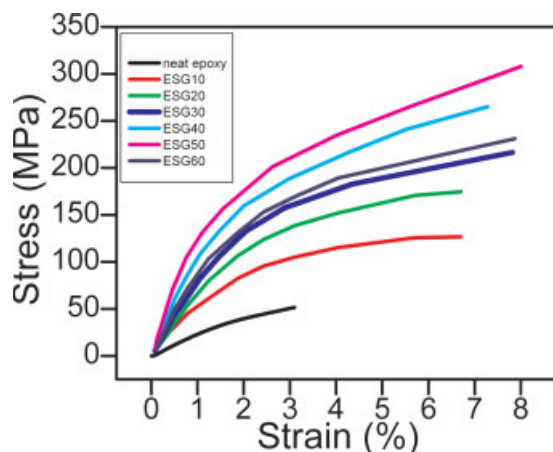
Figure 4 Tensile stress–strain curves of epoxy/SAN/glass fiber composites. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

fiber and better distribution of fibers inside the matrix. The increase observed in the modulus indicated that the fibers imparted some stiffness to the matrix.

It is known that fiber–matrix adhesion and the stress transfer efficiency of the interface play an important role in determining the strength of a composite. The increase in the tensile strength and modulus can also be explained through fiber straightening because of applied stress. As each specimen is subjected to an applied stress, the fibers align themselves in the direction of the load. The alignment of the fibers makes the material more efficient in carrying the load, thus increasing the strength and enabling the material to be more competent. The decrease in the tensile strength beyond 50 vol % is mostly due to poor matrix/fiber adhesion which may promote microcrack formation as well as nonuniform stress transfer due to fiber agglomeration within the matrix.[23] The cured epoxy resin and glass fiber composites exhibit brittle rupture after peak stress. Failure is marked by a complete loss of load carrying capacity and abrupt stress

drop to near zero. However, elastic modulus of the glass fiber-reinforced matrix was higher than the unreinforced resin matrix. That is, the elongation-to-failure of the composites was much higher as failure was delayed to larger strains and higher stresses.

The morphology of the tensile fracture surfaces of composites show phase information and fracture characteristics reflecting the reasons why the mechanical properties have been changed, and in turn decide the mechanical properties of polymeric composites.[24] Morphology studies revealed the aspects of fiber bonding and adhesion between fiber and matrix. Figure 5 shows the SEM micrographs of tensile fractured surface of glass fiber-reinforced epoxy resin composite specimens with and without SAN modification of epoxy resin. The interaction between matrix and glass fibers is clear from the figure. The composite shown in Figure 5(a) was developed using neat epoxy as the matrix, which produces poor interface adhesion. In contrast, when modified epoxy was used as the matrix [Fig. 5(b)], the adhesion becomes strong, indicating good wetting and strong interface which is clear from the matrix traces found stick to the surface of fibers.

The SEM pictures of composites with different fiber volume percentage are shown in Figure 6. These micrographs show that fiber pull-out and delamination are the major features of failure in such composites. In addition, most fibers on the fracture surface were orientated in the flowing direction of molding. This indicates that composites have higher degree of fiber orientation under the stress, which results in higher fiber efficiency factor and hence higher mechanical strength. There is very little matrix degradation witnessed by the 50 vol % fiber-reinforced specimens. In the figure, the glass fibers protruding from the composite indicate the degree of fiber pull-out and crack deflection. The fiber surfaces that protrude are not clean, with some adhering matrix material. The fibers are dispersed well and situated within the cell walls of the matrix. From
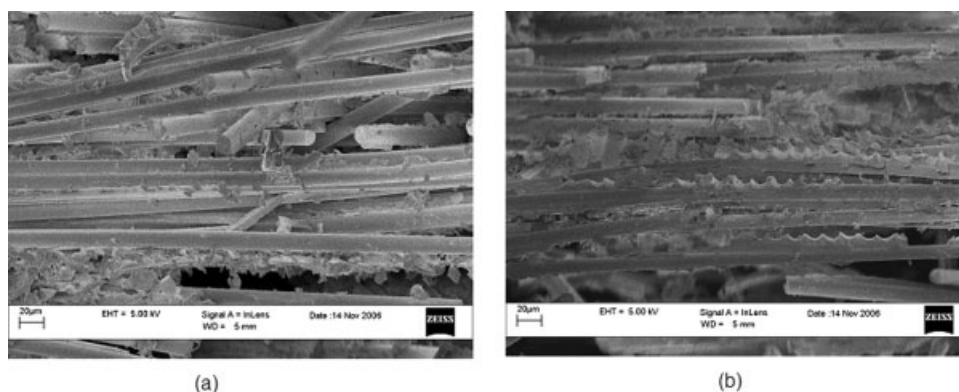
Figure 5 Scanning electron micrographs of failed surfaces of epoxy resin glass fiber composites. (a) Neat epoxy resin as matrix (50 vol % glass fiber). (b) Epoxy resin modified with SAN as matrix (ESG50).
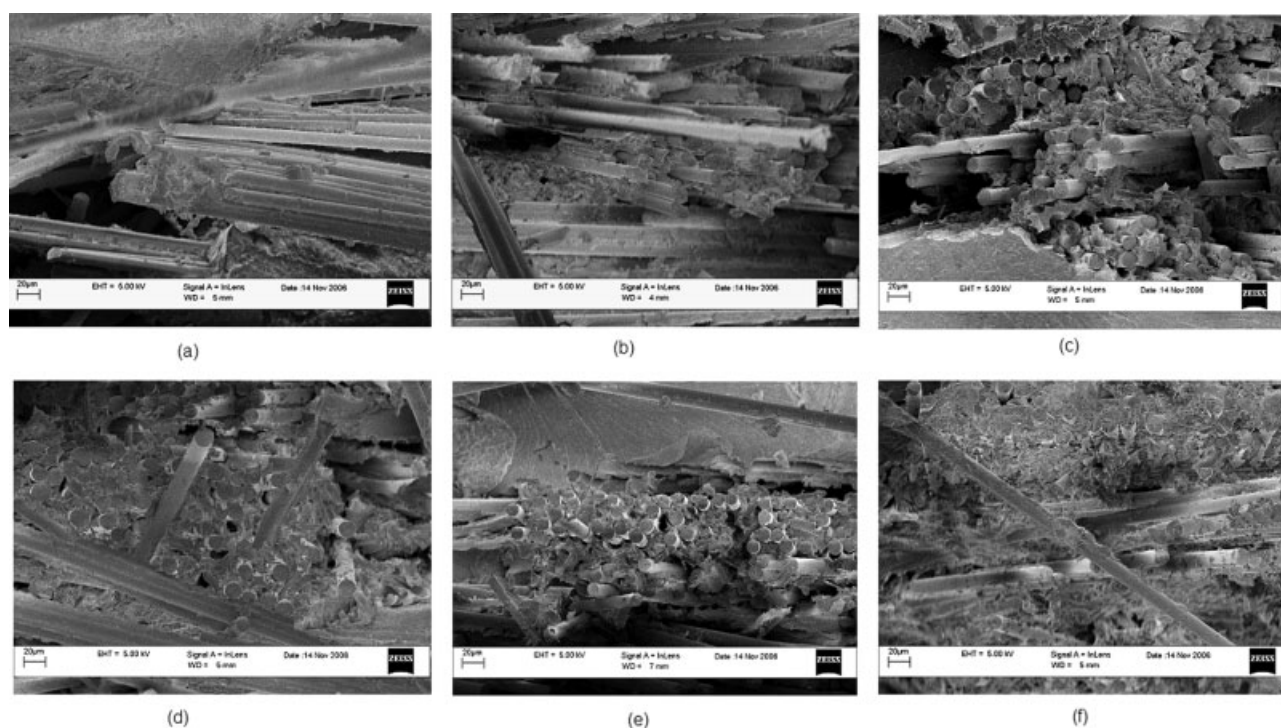
Figure 6 Scanning electron micrographs of failed surfaces of epoxy/SAN/glass fiber composites. (a) ESG10, (b) ESG20, (c) ESG30, (d) ESG40, (e) ESG50, (f) ESG60.

this we can conclude that fibers were adhered well to the matrix.

Flexural properties

The flexural properties of epoxy/SAN blends and epoxy/SAN/glass fiber composites are given in Table II. From the table it is seen that in the case of blends, the flexural strength almost remained the same except a marginal increase showed by ES5. But flexural modulus was increased considerably in the case of ES10 and ES15 blends. The flexural stress–strain curves of epoxy/SAN blends are shown in Figure 7. There is an increment in flexural modulus

by 21% and 20% in the case of ES10 and ES15 blends, respectively. The flexural strain also showed decreasing effect upon blending. The flexural stress–strain behavior of epoxy/SAN/glass fiber composites with different glass fiber content is shown in Figure 8. The curves exhibit a nonlinear nature, after the initial rise, indicating the flexible nature of the composites. An increment of 430% in flexural strength and 700% in flexural modulus was observed in the case of composites (ESG50) when compared with cured neat resin. It was also found

TABLE II
Flexural Properties of Epoxy/SAN Blends and Epoxy/SAN/Glass Fiber Composites

Sample	Flexural strength (MPa)	Flexural modulus (GPa)	Flexural strain at break (%)
Neat epoxy	69 ± 2	2.2 ± 0.11	5.0 ± 0.09
ES5	77 ± 3	2.3 ± 0.09	2.8 ± 0.08
ES10	65 ± 2	2.7 ± 0.02	2.0 ± 0.12
ES15	55 ± 3	2.6 ± 0.03	2.2 ± 0.11
ES20	55 ± 2	2.5 ± 0.07	2.4 ± 0.10
ESG10	269 ± 5	6.9 ± 0.23	3.1 ± 0.15
ESG20	295 ± 4	9.8 ± 0.43	3.2 ± 0.12
ESG30	327 ± 6	11.4 ± 0.53	3.2 ± 0.16
ESG40	332 ± 4	14.3 ± 0.21	3.6 ± 0.23
ESG50	367 ± 6	17.0 ± 0.60	3.7 ± 0.18
ESG60	355 ± 5	18.3 ± 0.35	3.9 ± 0.31
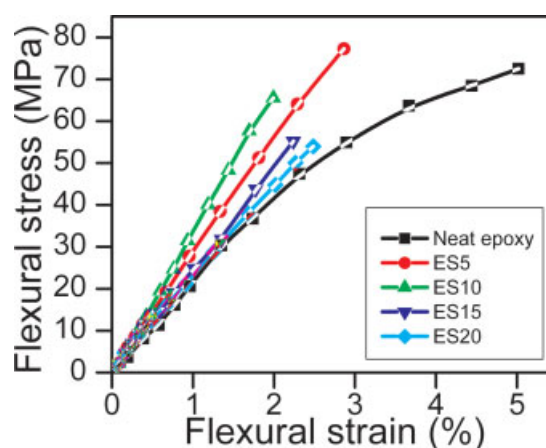
Figure 7 Flexural stress–strain curves of epoxy/SAN blends. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
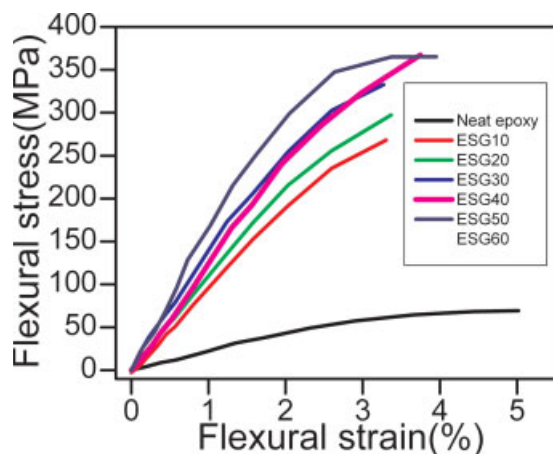
Figure 8 Flexural stress–strain curves of epoxy/SAN/glass fiber composites. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

that the flexural strength and modulus of glass fiber-reinforced composites increased gradually with increasing glass fiber content. There is a reduction in flexural strength beyond 50 vol % fiber content. A marginal reduction in flexural strain can be observed in the case of composites. In three-point bending test, when the load is applied on the specimen through the cross head, the load is transferred to the matrix and then to the fibers.

Impact properties

The charpy impact energies of DDS cured epoxy/SAN blends are given in Table III. It can be observed that the impact properties of blends were increased upon blending. ES10 showed the maximum increase. The increase in the impact strength can be attributed to the increased toughness of the matrix upon blending. The impact strength of a material describes the energy required to break the specimen. The magnitude of impact strength reflects the ability of the material to resist impact. The addition of the thermoplastic makes no significant changes in the impact property of the material.

Theoretical modeling of mechanical properties

Since the mechanical properties of blends and composites have great potential applications industrially and academically, their predictive models will be advantageous for the polymer technologist. Several equations have been discussed in the literature for predicting the behavior of polymer blends and composites.

Theoretical modeling of Young's modulus of epoxy/SAN blends

Predictive models of Young's modulus were used to realize the level of interaction between the compo-

nent polymers in epoxy/SAN blends. Different models can be employed for this purpose such as parallel, series, Coran, and Takayanagi models. The upper-bound parallel model is given by the rule of mixtures[25] as follows:

$$E_u = \phi_1 E_1 + \phi_2 E_2 \qquad (1)$$

where, E_u is the modulus of the blend in the upper-bound parallel model and E_1 and E_2 are the Young's moduli of components 1 and 2, respectively; ϕ_1 and ϕ_2 are their corresponding volume fractions. This model is applicable to materials in which the components are connected parallel to one another. The modulus prediction in the lower-bound series model is given by the inverse rule of mixtures as

$$\frac{1}{E_L} = \frac{\phi_1}{E_1} + \frac{\phi_2}{E_2} \qquad (2)$$

where E_L the modulus of the blend in the series model. This model can be applied to materials in which the components are arranged in series (Reuss prediction).

Another relation for predicting the modulus of blends is given as Coran's equation[26]:

$$M = f(M_U - M_L) + M_L \qquad (3)$$

where f can vary between zero and unity. The value of f is given by

$$f = V^n_H(nV_S + 1) \qquad (4)$$

where n contains the aspects of phase morphology, and V_H and V_S are the volume fractions of the hard phase and soft phase, respectively.

A phenomenological series-parallel model proposed by Takayanagi[27,28] consisting of mixing rule between two simple models involving connection in series (Reuss prediction) or in parallel (Voigt prediction) of the components. The equation is given as follows:
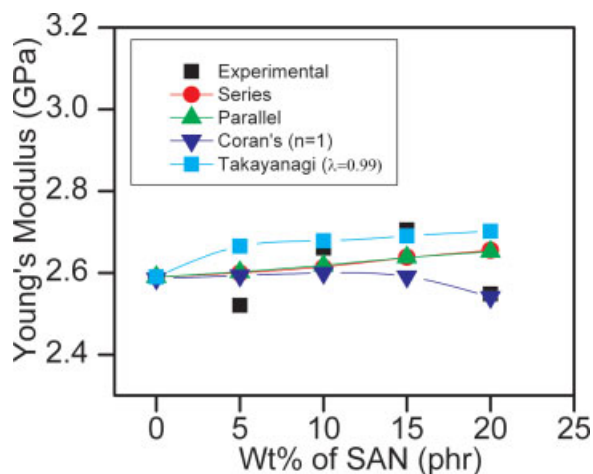
$$E = (1 - \lambda)E_1 + \lambda[(1 - \phi)/E_1 + (\phi/E_2)]^{-1} \qquad (5)$$

where, E_1 and E_2 are the moduli of the matrix phase and dispersed phase, respectively, and ϕ is the vol-

TABLE III
Impact Energies of Epoxy/SAN Blends

Sample	Impact energy (kJ/m^2)
Neat epoxy	11.4 ± 2.2
ES5	12.9 ± 1.9
ES10	13.9 ± 2.3
ES15	12.2 ± 3.0
ES20	11.8 ± 3.0

Figure 9 Plots of experimental and theoretical Young's moduli of epoxy/SAN blends as a function of wt % of SAN. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

ume fraction of the dispersed phase and is related to the degree of series-parallel coupling. The degree of parallel coupling of the model can be expressed by

$$\% \text{ parallel} = [\phi(1 - \lambda)/(1 - \phi\lambda)] \times 100 \qquad (6)$$

The graphical comparison of experimental and theoretical data is given in Figure 9. From the figure it is clear that none of the models is in good agreement with the theoretical values. However, Takayanagi model shows some degree of agreement with 10 and 15 phr blends.

CONCLUSIONS

The morphological and mechanical properties of epoxy/SAN blends were analyzed to determine the effect of thermoplastic modification on the toughness and mechanical properties of blends. Moreover, the analysis of epoxy/SAN/glass FRPs was performed to determine the effect of modification as well as fiber loading on the overall performance of the composites. The blends were homogeneous before curing and become heterogeneous after curing because of reaction induced phase separation (RIPS). The SEM micrographs clearly revealed the two-phase morphology of the blends in which different morphologies such as dispersed, cocontinuous, and phase-inverted morphologies were obtained. The tensile strength of the blends increased on curing, which is because of better interfacial adhesion. The flexural and impact properties were not varied significantly. Glass fiber was found to be an effective reinforcement for epoxy resin for high-performance application. Dramatic increase in the mechanical properties

was observed in the case of composites. The tensile and flexural properties enhanced with increase in fiber loading up to 50 vol % fiber content. The micrographs show good adhesion between the fiber and matrix because of the modification of the matrix. These results indicate that glass fiber-reinforced composites possess excellent potential for various applications as structural materials.

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