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Mechanical Properties of Carbon/Polypropylene Fiber Reinforced Rubber-Modified Epoxy System

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Abstract

Epoxies as a thermoset polymer have gained a considerable attention in structural, electrical, and marine applications. To widen their usage and overcome their brittleness, many polymers were blended with it. The addition of Polysulfide rubber helps in increasing the impact resistance of Epoxy, while in the other hand; it reduces strength , modulus, hardness and creep resistance. That leads to the need for another material to compensate the reduction in these properties.

Carbon, polypropylene as an inorganic and polymeric short fibers with their exceptional and different mechanical properties will compensate many drawbacks of polysulfide addition in enhancing mechanical properties. The composite material with Epoxy-Polysulfide matrix properties were evaluated by conducting the mechanical tests which include compression test, hardness test, impact resistance test. The fracture surface of composite impact specimens was observed by Scanning Electron Microscopy SEM on all designed experimental samples.

Elongation and impact strength of epoxy- polysulfide blend were found to increase with increasing rubber content while compressive strength and modulus decrease. The influence of fiber adding on the mechanical properties of the composite was studied , the mechanical properties of the blend matrix were found to be better than those of the pure epoxy. The morphology of the fractured surfaces showed significant signs of plastic deformation such as shear bands, high deformation, and cavitations due to rubber addition, and this correlates well with mechanical properties which resulted in an increase in toughness of the composites when rubber content was increased. The results of this investigation clearly show the possibility of balancing strength and toughness of the material when adding rubber, fiber to epoxy.

Results of compression test showed that the compressive strength decreases as Polysulfide percentage (PS) increase. While the carbon fiber addition helped in balance or overcomes the original compressive strength reduction up to 30 vol.%. of fiber. The results confirmed that the best modification is the addition of 6% polysulfide considering compressive strength, Impact and hardness. While 20% carbon fiber and 30% Polypropylene fiber gives the optimum mechanical properties in compression and Impact, respectively, for the composite samples.

Keywords: Epoxy resin, Liquid rubber, Mechanical properties.

1. Introduction

Epoxy resins are widely employed as structural adhesives, matrices in fiber-reinforced composites, and coatings for metals because of their excellent bulk properties such as high modulus, low creep, and good performance at elevated temperature. However, like other thermoset resins, the crosslinking character of cured epoxies produces a highly undesirable property: they are relatively brittle, having poor resistance to crack initiation and growth[1-2]. This lack of toughness severely impacts the performance of these thermoset in almost all applications. To address this defect, resin formulators have developed technology that permits some thermosets to be toughened by the addition of a second elastomeric phase[3,4], by introducing an acceptable sacrifice of desirable properties. By increasing the concentration of elastomer phase, an increase in fracture energy of the unmodified epoxy has been achieved [5,6].

Improving fracture toughness will lead to significantly enhanced performance when used as is, and will also enhance the damage initiation threshold and long term reliability for fiber reinforced composites. There are various reactive liquid elastomers which are used to modify or toughen epoxy resins [3]. Liquid polysulfide elastomer is one of the most important reactive modifiers for epoxy resin. Polysulfide modified epoxy adhesive systems are widely used in the construction, electrical and transportation industries due to the following benefits [7-11]. Thus improving fracture toughness will lead to significantly enhanced performance when used as is, and will also enhance the long term reliability for fiber reinforced composites[12].

One of the prime benefits is improved adhesion, particularly to substrates which are oily, wet and rusted. The excellent adhesion of polysulfide epoxy systems leads to the use of these materials in surface-tolerant adhesives and coatings. In addition to providing improved initial adhesion, polysulfide elastomers provide long-term adhesion and durability to epoxy resins. Since polysulfide is a very flexible elastomer, addition of liquid polysulfide elastomer to epoxy resin gives good flexibility and impact strength to epoxy resin[9].

However, there are some limitations to the use of polysulfide modified epoxy systems. Polysulfide elastomer disrupts the most desirable properties of neat epoxy resin, they reduce the glass transition temperature (Tg), tensile strength and tensile modulus of neat epoxy resin due to the dilution effect of the polysulfide elastomer in the epoxy matrix. This limits the application areas in which they can be used [11].

Improving the fracture toughness of thermosetting resins has been the subject of considerable research. Typically, these approaches have involved the addition of micron-sized soft (elastomeric or thermoplastic) or rigid (glass or ceramic) particles into the polymer matrix. These filler are then expected to provide extrinsic toughening mechanisms.

In the first approach, micron-sized rubber particles are dispersed in the polymer matrix to enhance the overall fracture toughness by triggering localized shear yielding of the polymer [13-16]. However the addition of rubber introduces deleterious effects, such as a reduction in the elastic modulus and tensile strength, decreased creep resistance, poor hot/wet performance, and a lowering of the glass transition temperature. Alternatively, ductile thermoplastic particles may be dispersed in the thermosetting polymer to provide extrinsic mechanisms for increasing the overall toughness [17,18]. However, only moderate increases in the fracture toughness have been observed, especially for low volume fractions of added thermoplastic particles [19].

While these studies have focused on fracture toughness as the primary issue, there are clear indications that the reinforcement could lead to significant improvements in fracture toughness and resistance to crack growth. Nevertheless, despite these observations there is limited fundamental information regarding the effects of reinforcement type and volume fracture on fracture processes, toughening mechanisms and the resulting overall mechanical properties.

This paper discusses the effects of combining the benefits of adding polysulfide elastomer and two deferent types of fibers to the epoxy so as to develop improved matrix material with the aim of attaining good adhesive strength without compromising the other desired mechanical properties of the epoxy resin. The objective of this study is to examine the influence of polysulfide elastomer and fibers concentration on the morphology and mechanical properties of fiber reinforced rubber-modified epoxy system.

2. Experimental

2.1 Materials

A diglycidyl ether of bisphenole A (DGEBA) Quickmast 105 (DCP) epoxy was selected for its low viscosity to overcome voids formation during specimens preparation, and for its wide range usage and applications. Table 1. lists the specification of epoxy resin used.

Polyseal PS PG (pouring grade) polysulfide rubber which supplied in the shape of white liquid with density 1.30 gm/cm3 was used. This type was chosen for its low molecular weight which means high compatibility with epoxy and enables the formulation of pouring highly reactive compounds. All chemical materials supplied by Ayla chemicals company, Jordan. Properties of polysulfide polymers are listed in Table 2.

Table 1. The specification of epoxy resin

PROPERTIES	Density	Compressive strength	Tensile strength	Pot life	Boiling point
VALUES	1.1 ±0.05 [g/cm^3]	>70MPa	>25MPa	50-70 min. @ 25°C	>200 °C

Table 2. Properties of polysulfide polymers

PROPERTIES	Density	Shore A' Hardness	Elongation,	Tack free time	Service temp.
VALUES	1.3±0.05 [g/cm^3]	15-30	>300 [%]	5 hrs.	-20 to 80

A 10, 20, 30 vol.% short fibers were added to the modified epoxy, and also added to the plane epoxy for purpose of comparison. The carbon fiber chosen for its distinct compression strength, while polypropylene fiber for its ductility and high toughness. Each of this fiber are available in chopped form and have low cost so they can be used industrially.

2.2 *Preparing epoxy- polysulfide Blends and fiber reinforced composite*

Preparation of polymer blends was achieved from mixing epoxy resin with different percentages of Polysulfide rubber (0,3,6, 9, 15, and 25 wt%). First, mechanical mixing for 15 minutes of epoxy resin with varying concentration of liquid polysulfide using a mechanical mixer. The blend then heated to 70^{o}C over a 2 hours period using magnetic stirrer with stirring speed of 500 rev/minute to allow better reaction and interlocking,. The blend was subsequently put into a vacuum desiccator and degassed at 1.7 bar for at least 20 minutes until the mixture was free of bubbles. A stoichiometric amount of the anhydride curing agent was then added and stirred thoroughly for 15 minute and at 40^{o}C,worth noting that curing of the blended resins is achieved using conventional epoxy hardener only. the blend was poured into the molds and placed in a vacuum desiccator again under 1.7 bar for 24 hours. Test samples were obtained after curing for 7 days and machined by torn. The mechanical properties were measured at room temperature (25-30) ^{o}C.

Same procedure was followed in fabrication of fiber reinforced samples with adding fiber to the blend at the stage of adding hardener.

2.3 *Characterization of prepared materials and composites*

2.3.1 Compression Test

For the brittle polymers, Compression test usually used instead of the tensile test to obtain the stress and the high strain behavior, this is because of the brittleness of the samples. The test were performed in accordance with ASTM D695 at room temperature using Gunt 20 KN, Universal Material Tester. The loading of the specimen is carried out (with a cross head velocity of 5mm/min). The compression strength (σ), compression strain (ε), and Young modulus (E) were calculated.

2.3.2 Impact Test

Charpy impact tests of the composites were performed on a Gunt WP 400 , Pendulum impact tester, New York Company, the measurements were made according to ASTM D256 method, by using rectangular unnotched specimens which were 55 mm long and 10×5 mm in cross section. The unnotched specimens were chosen because the volume fraction of the fibers in the composite is known to influence the mechanical properties, the notch gives rise to stresses in different fiber positions in that point which could affect the impact results. Unnotched impact strength (acU) is calculated by the following equation [1]:

$$acU = (W / hb)\times 10^{3} \qquad (1)$$

Where ; W: energy (J) , b: width of the sample (m), h: thickness (m).

2.3.3 Scanning electron microscopy

A low voltage JEOL scanning electron microscope (JSM-5800,Japan) was used to examine the fracture surfaces of the tough epoxy samples, the test was performed in Materials and Engineering Research Institute (MERI) in Sheffield, UK. A thin section of fracture surface was cut and cleaned with cleaning solution, followed by sonication, nitrogen blow, then mounted on an aluminum stub using conducting (Carbon) paint and was sputter coater with 2 nm thick gold to reduce any charge build up on the surface. The images were analyzed at an acceleration voltage of 10KV.

3. Results and Discussion

3.1 *Influence of rubber and fiber addition on compression behavior*

3.1.1 Effect of PS rubber addition on Epoxy

A representative compression curves are plotted in (Figure 1), (Figure 2, a,b).The addition of liquid polysulfide reduces the modulus and compressive strength, but it also allows more elongation compared to the pure Epoxy, indicating greater ductility of the material.

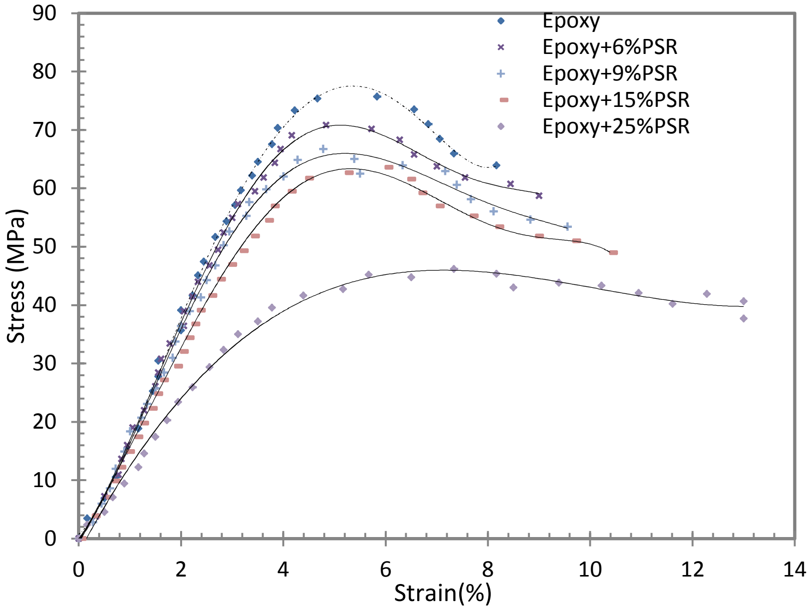
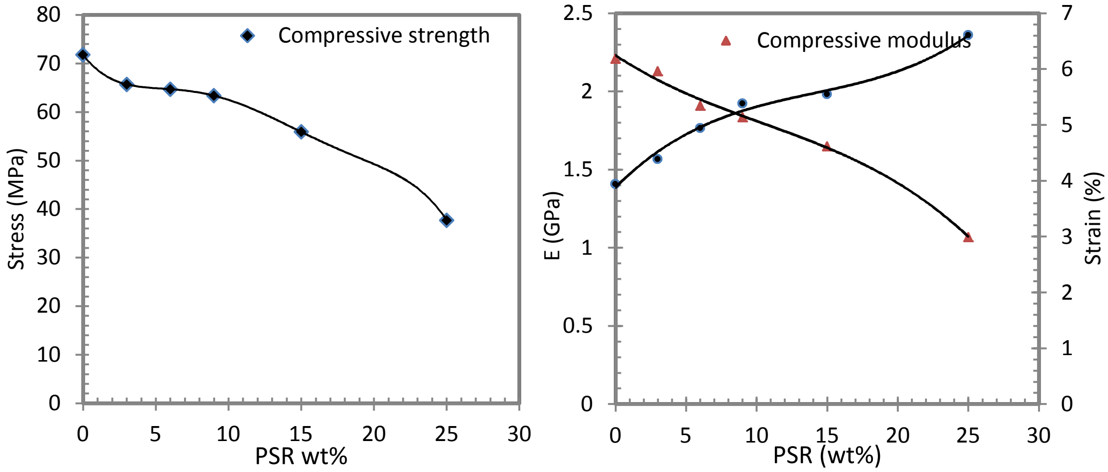


Figure 1. Compressive stress-strain curves for Epoxy-PSR blends

From (Figure 2,a), the data indicate that the polysulfide-Epoxy blend has a lower modulus and strength than the Epoxy. This is clearly demonstrated for blend with 30 wt% of Polysulfide Rubber (PSR) when modulus and compressive strength drop dramatically to about 0.8 GPa and 45 MPa.

The elongation to break of Epoxy is improved by adding PSR (Figure 2, b), compared to the pure Epoxy where the elongation to break is 4% , the elongation to break of 6 wt% PS modified Epoxy is 5%, and become 6.6% after adding 25 wt% of PSR. The rubber particles are known to break the Epoxy via interaction of the stress field ahead of the crack tip with the rubber particles.



(a) (b)

Figure 2. (a) Compressive strength (b) compressive modulus, strain at failure of Epoxy as a function of PSR content.

3.1.2 Influence of fiber addition on compression behavior of both Epoxy and blend (Epoxy- 6 wt% PSR)

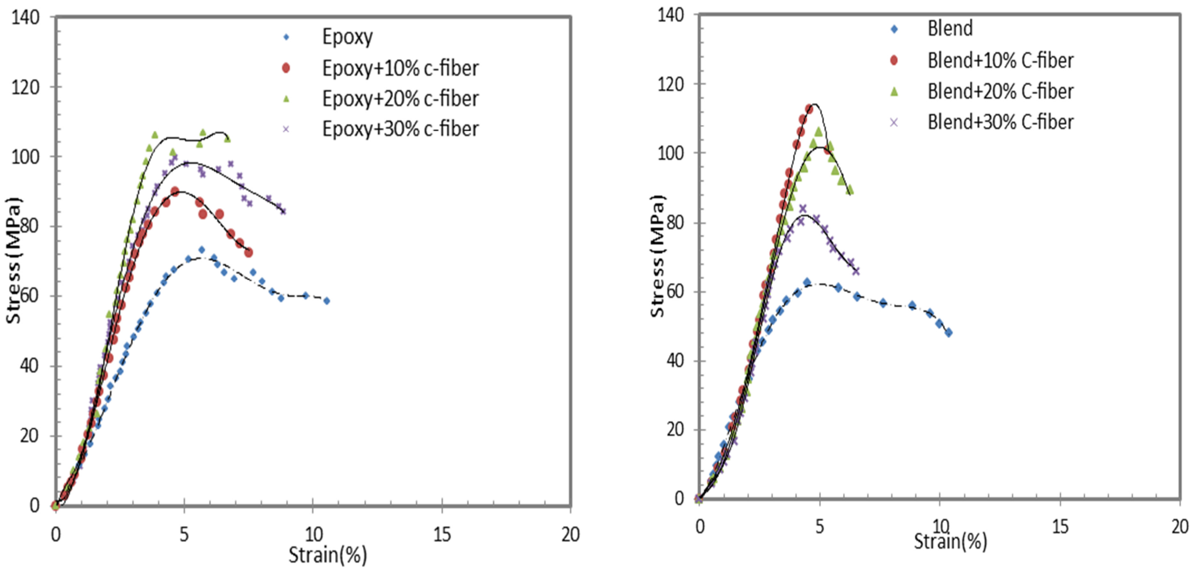
To overcome the drawback of PSR addition, like the reduction of modulus or loss in strength, this can be eliminated when using fiber together with the PSR. The compressive strength of the composites increases with the incorporation of the reinforcing elements within the matrix of both Epoxy and blend. The difference in slop in their stress strain curves reflects the difference in their respective fiber modulus.

- Carbon fiber reinforced composite

A compressive stress strain curves for the fiber reinforced composite materials are shown in (Figure 3, a&b). There is a significant change in the compressive strength of the carbon fiber reinforced Epoxy and blend until concentration of 20 vol.% after which the strength and modulus begin to drop. The increase in compressive strength and modulus is about 45%, 50% over that of Epoxy and 70%, 68% over the blend (Figure 4, a&b) . The enhancement is not surprising given that fiber modulus is much higher than that of Epoxy [20]. However, this increasing is up to 20% of C-fiber in the Epoxy or blend, after which it start to drop, this may attributed to the agglomeration of fiber and voids formation as the fiber increases in content, this leads to fiber-fiber contact, as stated by Manikandan [21], with voids, initiating cracks leading to fracture.

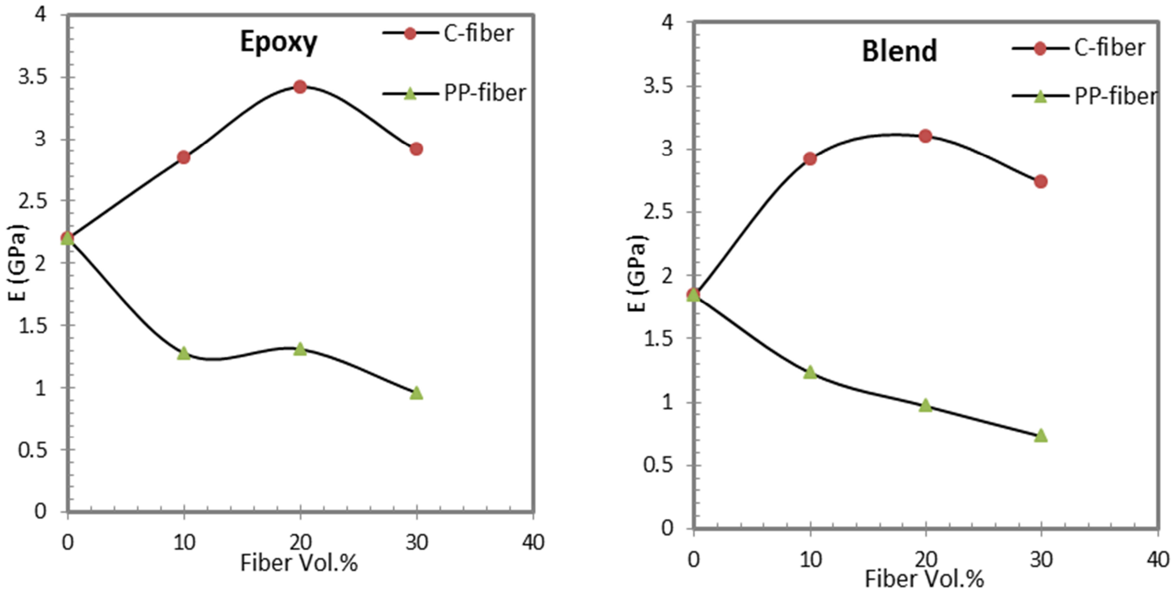
- Polypropylene fiber reinforced composite

Addition of up to 30 vol.% of polypropylene fiber reduces both strength and stiffness. This can be seen in the form of (i) reduction in the slope of the linear portion of the stress-strain curves which represents the stiffness or modulus of the material, and (ii) reduction in compressive stress, (Figure 5, a&b). However, the elongation at break increases with increasing fiber content. This is expected since Polypropylene, which has a low value of modulus, tends to force the material to yield at lower value of stress as it acts as a stress concentrator. A reduction of 44% in compressive strength, 56% in modulus is observed after adding 30 vol.% PP fiber to the epoxy, while 40%, 60% respectively after addition to the blend,(Figure 4, a&b).



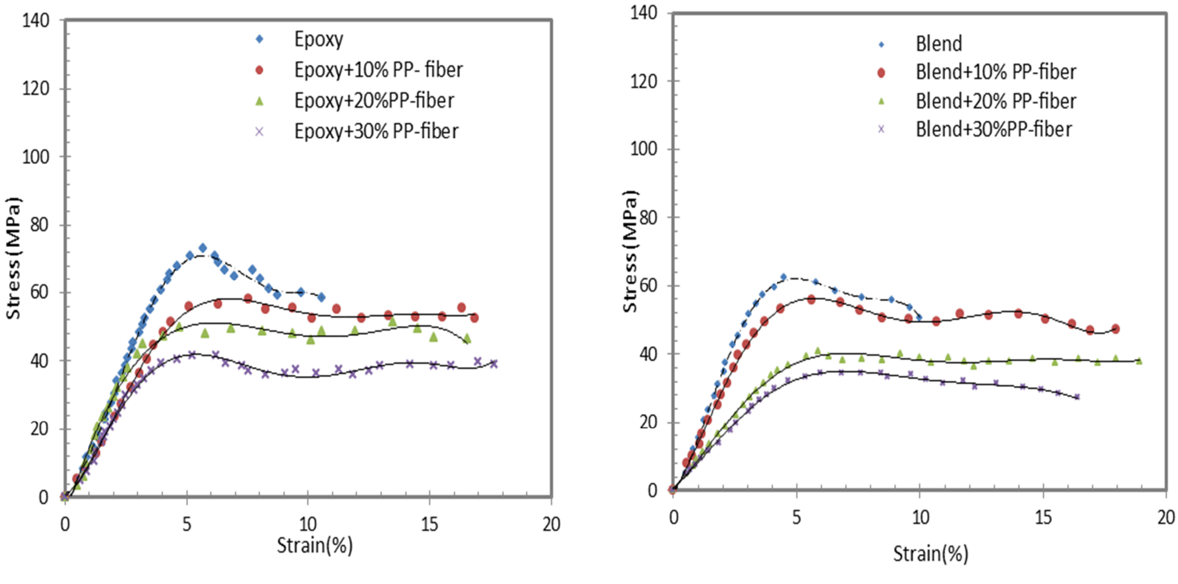
(a) (b)

Figure 3. Stress-strain curves of (a) Epoxy, (b)blend (PS-modified Epoxy), reinforced with different loading of Carbon fiber



(a) (b)

Figure 4. Compressive modulus of (a) Epoxy, (b) blend reinforced with different fiber as function of fiber load



(a) (b)

Figure 5. Stress-strain curves of Epoxy and blend (PS-modified Epoxy) reinforced with different loading of polypropylene fiber

3.2 Impact resistance

3.2.1 Influence of rubber addition on the Impact strength of the Epoxy

The pure Epoxy has a comparatively low value of energy absorption, a reflection of the brittle characteristic of this cross linked system. With the addition of the PS to Epoxy, there is a an improvement of the energy absorption of the composite system. These small amount of rubber which get incorporate into the Epoxy matrix, thereby flexibilizes the thermoset network and cause reduction in compressive strength and increase in impact strength. Previous works on rubber-modified epoxies support this observation [22,23].

The results of impact strength of the Epoxy and blend samples with different amounts of the rubber are depicted in (Figure 6). All modified networks show higher impact resistance than pure Epoxy.

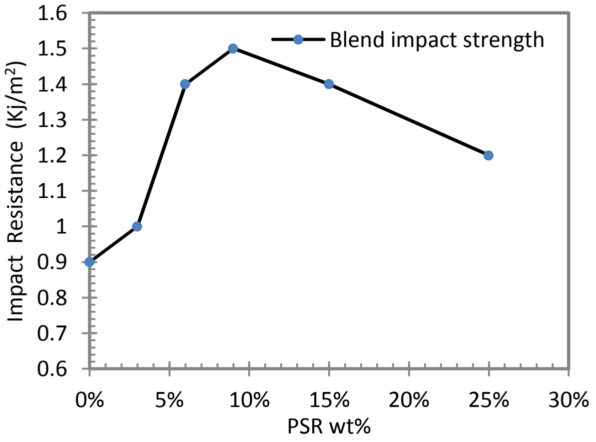


Figure 6. Impact strength as a function of rubber content

The blend network containing a 9 % rubber has a maximum impact strength of 1.5 J/m^2 which is about 66% higher than that of pure Epoxy and for a 25% rubber the value is 17% lower than the maximum.

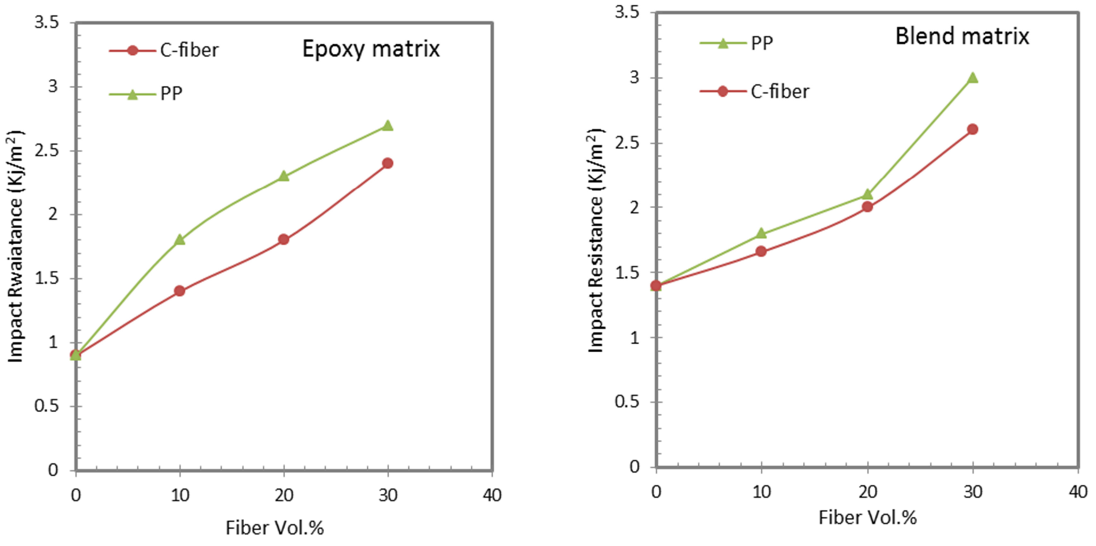
The decreasing tendency of impact strength after an optimum level of 9 % rubber inclusion may attributed to the aggregated size of rubber particles as concentration of rubber increases. Similar behavior has been reported in other rubber-modified Epoxy systems [24,25].

Although addition of 9% PSR gives the highest impact improvement to the Epoxy, but, on the other hand, the compression strength is reduced much higher compared with addition of only 6% PSR.

3.2.2 Influence of fiber addition on Impact Strength of both Epoxy and blend (Epoxy- 6 wt% PS)

- Carbon fiber modified composite

The lower impact strength related to the carbon-matrix composites compared to the glass–matrix composite is supposed to be originated from the interface between the fiber and matrix. The interactions at this interface and the brittle energy absorbtion in carbon fiber composite inhibit the elastic and plastic deformations of the composite. (Figure 7) shows the effect of fiber load on impact strength of the composite. A linear increase is observed with increasing fiber content. The addition of carbon fiber up to 30 vol.% loading gives 2.43 Kj/m^2 of impact strength which is 166% higher than that of pure Epoxy, while for blend, an increase of 86% of impact strength of blend to give 2.6 Kj/m^2.



(a) (b)

Figure 7. Impact strength of (a) Epoxy, (b) blend reinforced with different fiber as function of fiber load

- Polypropylene fiber modified composite

Polypropylene (PP) fiber – Epoxy, blend composites displayed higher impact strength than the carbon fiber composite. Since PP fiber has no active sites at the surface [26], the better impact performance of this composite may be associated with the ductile characteristic of the PP fiber together with debonding phenomena. The addition of PP fiber gave a significant improvement in the toughness. For both Epoxy and blend, the value of impact strength increased lineary with the increasing load of fiber, see (Figure 7, a&b). Maximum value of impact strength 3 Kj/m^2 was measured for the 30vol.% PP fiber reinforced blend, which is 114 % higher than the corresponding value for the blend without fiber, and 2.66 Kj/m^2 for the fiber reinforced epoxy, which is 194% higher than that of pure Epoxy. This type of behavior has been also reported for plain polyethylene (PE) fiber based composite [26,27].

3.3 Effect of rubber addition on Hardness of Epoxy

(Figure 8) shows the hardness of the and modified Epoxy resins. As can be observed, the hardness of the samples decreased with an increase in PSR content. This suggests that some of the rubber is dissolved in the Epoxy phase and plasticizes the system. Moreover, incorporation of PSR, which has little resistance to penetration, creates domains of rubber particles in between the cross-links which reduce the cross-linking density [28,29].

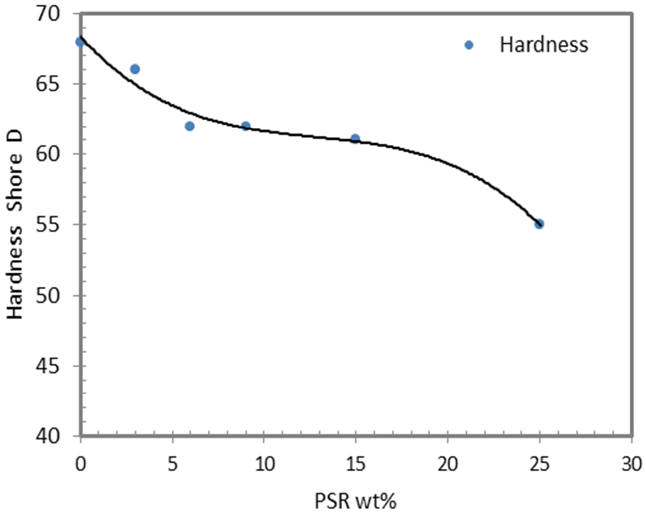


Figure 8. PSR effect on hardness of Epoxy

3.4 Morphology of fracture surfaces.

3.4.1 Epoxy and blend

The pure Epoxy matrix, (Figure 9, a,b), shows smooth, glassy, and rivery fractured surfaces. The relative smoothness of the fractured surface, irrespective of the presence of some shear deformation lines, indicates that no significant plastic deformation had occurred. Similar behavior has been also reported by others [30, 31].

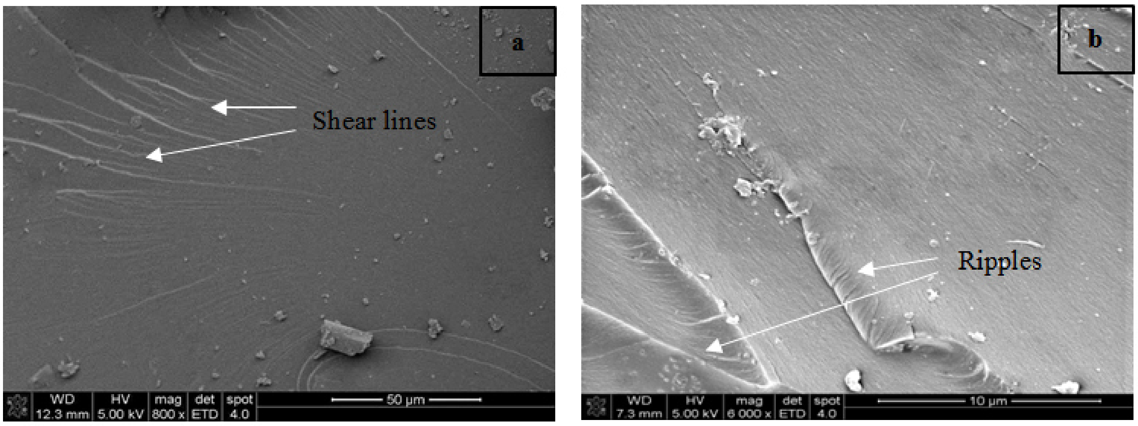


Figure 9. (a), (b), Fracture surface of the Epoxy.

In epoxies modified with 6 wt% of rubber, as shown in (Figure 10) the PS particles are uniformly distributed throughout the matrix with a narrow particle size distribution. This distribution of smaller particles is responsible for lower crack growth in these specimens which is indicated by the presence of relatively large number of deformation lines. Also, the fracture surfaces, unlike pure Epoxy, are not very smooth, indicative of a ductile manner of fracture.

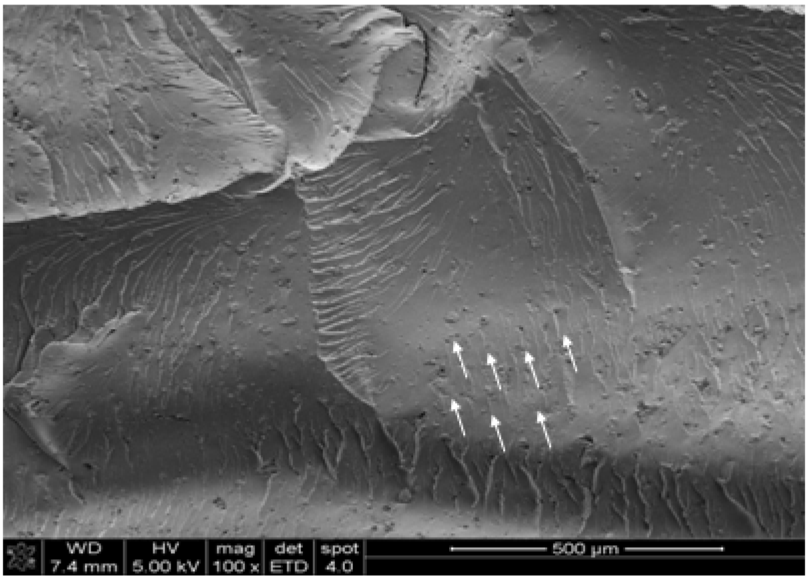


Figure 10. Fracture surface of 6%PS- Epoxy blend. White arrows point to some rubber particles

3.4.2 Fiber reinforced Epoxy and blend

Failure in impact of a fiber-reinforced polymer can occur by one or more of three possible mechanisms(1) fiber fracture, (2) interface shear fracture or debonding, and (3) matrix shear fracture. A detailed analysis of the of the failure mechanism in short fiber-reinforced polymers is complex because of the need account for such variables as the distribution of the fiber lengths and fiber orientations, stress concentrations due to interaction of fiber ends, residual matrix interface.

An examination of the fracture surfaces of the carbon fiber reinforced Epoxy and blend by SEM, (Figure 11), reveals that these materials do not fail by fiber fracture, but rather by either matrix failure or interfacial debonding.

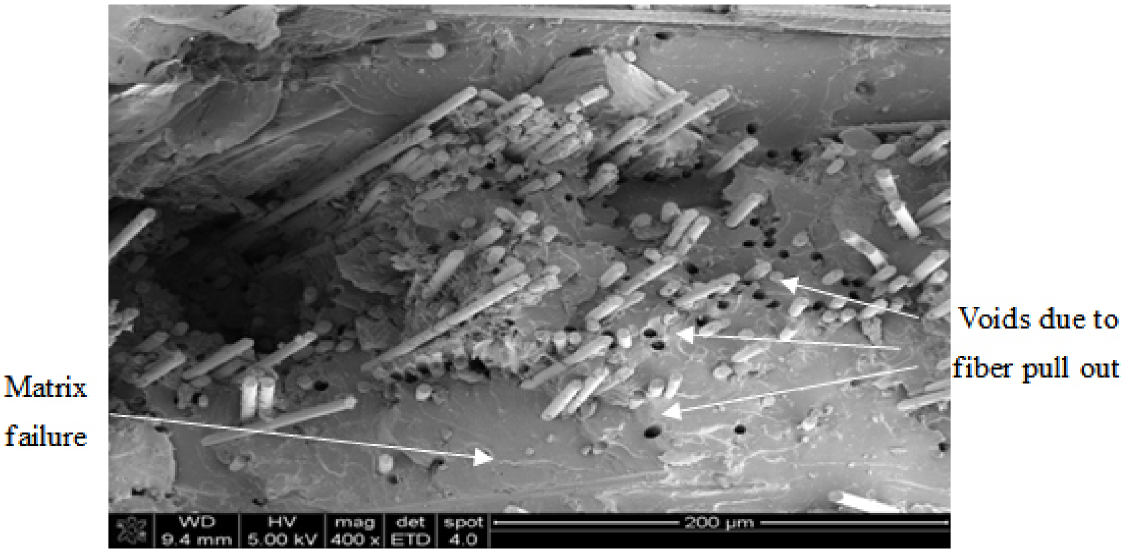


Figure 11. SEM image of impact fracture showing fiber/matrix debonding of carbon fiber

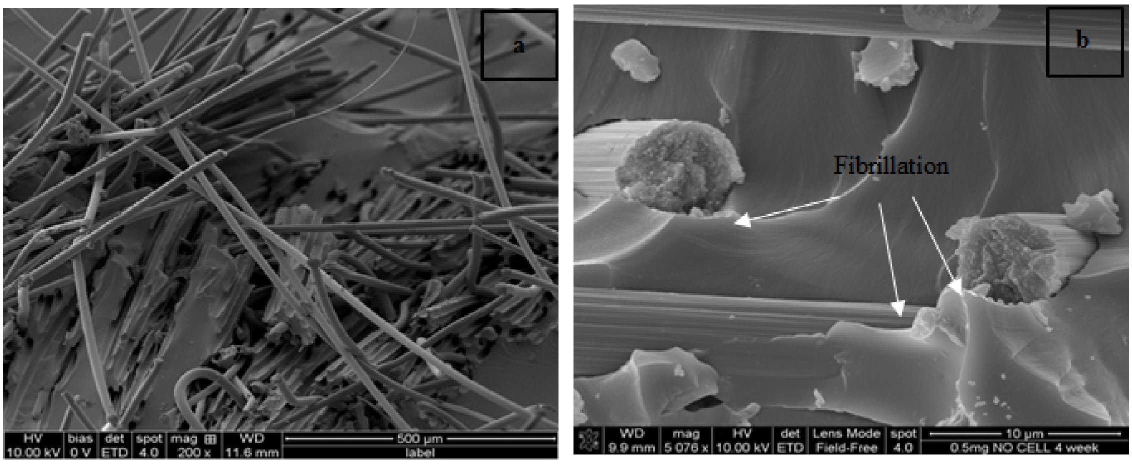


Figure 12. SEM image of impact fracture showing (a) 30% PP fiber reinforced Epoxy with no matrix adherent to them, (b) fibrillation near carbon fiber ends

A low magnification SEM photograph of the PP fiber reinforced composite in (Figure 12, a) shows that the PP fiber surface did not have much matrix material adhering to them, indicating relatively weak fiber/matrix interface.

A considerable fibrillation of the matrix is observed in some pictures, Figure (12, b), which suggests that failure was initiated by local yielding of the polymer. similar behavior has been also reported in other fiber reinforced polymer systems [26, 32,33].

4. Conclusions

Based on the results obtained and discussed previously, the following conclusions can be drawn.

1. The results shows that the liquid polysulfide rubber is a good potential toughening agent for epoxy resin.
2. The mechanical performance of the epoxy was changed with the incorporation of varying weight percent of PS rubber, which was explained by the evolution of morphological changes. The elastomeric nature of the rubber caused reduction in compressive strength andhardness, but impact strength values increased and attained a maximum for 9 wt% of PS., this can be attributed to the small rubber particle size, uniform distribution, and better matrix adhesion.
3. The combination of rubber toughening and fibers offers the possibility to formulate tough and stiff

epoxy resin system.

4. Losses in strength caused by PS rubber can be compensated and even improved by addition carbon fibers, while reduced even more in incorporation of Polypropylene fiber.
5. Reinforcing Epoxy and blend with carbon improves the ultimate strength, hardness and impact strength. The best mechanical properties obtained when 30vol.% of glass fiber added to the blend. it have balancing effect to compensate the PS reduction in mechanical properties and gives the highest toughness.
6. Most of the composite material of reinforced blend possess better compression modulus, compression strength, and impact resistance than epoxy composites.
7. Incorporation of the ductile polypropylene fiber reduces the compressive strength to a high extent, But, on the other hand improves the impact resistance. Considering the good properties as low density and low cost, the interesting features may be useful for development of highly deformed composite with a good toughness.
8. The SEM analysis indicates rubber particles cavitations and localized plastic shear yielding induced by the presences of the dispersed rubber particles.
9. The microscopy showed evidence of debonding for the Polypropylene fiber composite, while both matrix failure and shear failure of fiber/polymer interface are involved in glass and carbon fiber reinforced composites.

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