EFFECT OF TYPE AND FILLER LOADING ON THE STATIC MECHANICAL PROPERTIES OF GLASS-BASALT HYBRID FABRIC REINFORCED EPOXY COMPOSITES

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Abstract—Fiber-reinforced polymer composites (FRPCs) are rapidly gaining market share in structural applications, but further growth is limited by their lack of toughness. Fiber hybridization is a promising strategy to toughen composite materials. By combining two or more fiber types, these hybrid composites offer a better balance in mechanical properties than single fiber reinforced composites. This work concerns the production of glass-basalt hybrid fiber reinforced composite with and without different micro fillers like graphite and Polytetrafluoroethylene(PTFE). All the composites were fabricated by hand layup technique followed by compression molding. The mechanical properties such as tensile strength, tensile modulus, flexural strength, flexural modulus and inter laminar shear strength have been investigated in accordance with ASTM standards. From the experimental investigations, it has been found that loading of graphite filler to glass-basalt hybrid fiber reinforced composites shows superior mechanical performance compared to unfilled and PTFE filled composites. Scanning electron microscopy (SEM) photomicrographs of the fractured samples revealed various aspects of the fractured surfaces. The failure modes of the tensile and flexure fractured surfaces have also been reported.

Keywords—Fiber-reinforced polymer composites, Graphite, Polytetrafluoroethylene, Scanning electron microscopy.

I. Introduction

Fiber reinforced polymer composites (FRPCs) are used in almost all type of advanced engineering structures like aircraft, helicopters, boats, ships and off-shore platforms, automobiles, sporting goods, chemical processing equipment etc.[1, 2]. A key factor driving the increased applications of composites over the recent years is the development of new advanced forms of FRPCs. These include developments in high performance resin systems and wide variety of fabric reinforcement.

Epoxy resins are widely used as matrix in many FRPCs. They are a class of thermoset materials of particular interest to structural engineers owing to the fact that they provide a unique balance of chemical and mechanical properties combined with wide processing versatility. For epoxy-based fiber laminates, the typical reinforcements are glass, carbon, aramid and basalt fibers (or a combination of them, through a process known as hybridization, in order to take advantage of both fibers) have been used to improve the overall properties of the composite laminate.

Due to environmental issue natural fibers are widely used as reinforcement for polymer composites [3-5]. Despite the advantages of natural fibers over traditional ones (low cost, low density, acceptable specific strength properties, reduced tool wear and biodegradability), they suffer from several drawbacks, such as their hydrophilic nature (which affects the compatibility with hydrophobic polymeric matrix), the scattering in mechanical properties and the low processing temperature required. A possible solution that takes into account the environmental issues is represented by the use of mineral fibers like basalt. Since deep studies on this material are only recent, in the last 10 years, number of researchers has been investigating properties and behavior of various composites made of continuous or short basalt fibers [6–12]. These emerging mineral fibers are natural, safe and easy to process also at the recycling stage. Basalt fibers are continuously extruded from high temperature melt of selected basalt stones, which are volcanic, over-ground, effusive rocks saturated with 45–52% SiO_2. These fibers show high modulus, excellent heat resistance, heat and sound insulating properties, good resistance to chemical attack and low water absorption. These advantages make basalt fibers a promising alternative to glass fibers as reinforcing material in composites, when considering that the price of basalt fibers lies between that of E and S-glass and that it is continuously dwindling as new market opportunities arise [13]. As a consequence, over the last year's basalt fibers have been studied extensively as reinforcement in thermosetting matrices [14–16]. Zhang et al. [17] found that the ultimate tensile strength of unidirectional glass/flax composites increased by 15% with better dispersion and bonding. Dong et al. [18] obtained experimental flexural strength for carbon/glass interlayer hybrids, which are 40% and 9% higher than the pure carbon/ glass composites. De Rosa et al. [19] confirmed that using the stronger basalt fiber at the top and bottom of the glass laminate improved the post-flexural strength of the hybrids. However, it is difficult to transfer the conclusions from one hybrid to the other, as there is currently no theoretical framework available to assess the importance of the various material parameters.

Apart from FRPCs, the composites made from both fiber/filler reinforcement performed well in many practical situations. Koh et al. [20] stated that silicon carbide (SiC) filler particles enhance the mechanical properties of fiber reinforced epoxy composites. Yamamoto et al. [21] found that change in particle size, shape, and percentage content of filler particles on the mechanical properties of FRPCs is an area of keen interest. High cost of polymers is sometimes a limiting factor in their use for commercial applications. Use of low cost easily available fillers may be useful to bring down the cost of polymer based components. Study of effect of such filler addition is necessary to ensure that the mechanical properties are not affected adversely by such inclusions. Available references suggest that investigations on a large number of materials to be used as fillers in polymers as reported by Katz and Milewski [22], but only a few of them deal with the material systems containing fibers and fillers simultaneously [23]. The purpose of fillers can be divided into two basic categories, first, to improve the properties of the material and second, to reduce the cost of component. The present study focuses on the evaluation of the mechanical properties like tensile, flexure and inter laminar shear strength of glass–basalt hybrid fiber reinforced epoxy composite with and without fillers like graphite and PTFE (2, 4 and 6 wt. % each).

II. EXPERIMENTAL

A. *Materials*

Two types of reinforcement used in the present study are basalt fabrics, 360 g/m^2, plain-weave (warp 5F/ 10 mm, weft 5F/10 mm), tex 330, from Nickunj Pvt Limited, and E-Glass fabric, 360 g/m^2 , plain-weave (warp 5F/10 mm, weft 5F/10 mm), tex 330 (Suntech fibers Ltd.). Table 1 summarizes the basic mechanical properties of the adopted basalt fibers as well as E-glass fibers. The matrix used is an epoxy (LY556) is a bifunctional one that is diglycidyl ether of bisphenol-A (DGEBA) and the high temperature hardener HT972 is a solid, aromatic amine, viz., 4,4' – diamino diphenyl methane (DDM). The resin and hardeners were kindly supplied by M/s. Huntsman Advanced Materials, Mumbai. The wt. % of glass and basalt fibers in the final formulations was varied and is listed in Table 2.

B. *Preparation of Composite Laminates*

The epoxy resin is mixed with the hardener in the ratio 100:28 by weight. Dry hand lay-up technique is employed to produce the composites. The stacking procedure consists of placing glass and basalt fabric one above the other with the resin mix well spread between the fabrics to obtain hybrid fiber reinforced composites. A porous Teflon film is placed on the completed stack. To ensure uniform thickness of the sample a spacer of size 3 mm is used. The mold plates have a release agent smeared on them. The whole assembly is pressed in a hydraulic press at pressing temperature and pressure of 100 ^{o}C and 0.5 MPa. The laminate so prepared has a size 500mm X 500mm X 3 mm was kept it in a hot air oven at a temperature of 120 ^{o}C for 2 h. To prepare the particulate filled fiber reinforced composites graphite and PFTE powder (average particle size of about 10 µm) is mixed with a known weighed quantity of epoxy resin. The details of the final composites fabricated are listed in Table 2.

TABLE 1 Basic mechanical property of the used basalt and glass fibers

Properties	Basalt	E-glass
Filament diameter (µm)	17	7
Density (g/cc)	2.8	2.54
Tensile strength (MPa)	4800	3200
Elastic modulus (GPa)	90	70
Elongation at break (%)	3.15	4.0
Maximum service temp. (^{o}C)	650	460^{o} C

TABLE 2 Weight percentage of matrix, fiber and filler of prepared composites

Sample code	Matrix wt. %	Fiber wt. %	Filler wt. %
G-E	45%	55% (Glass)	-
B-E	45%	55% (Basalt)	-
GB-E	45%	55% (50% Glass-50%Basalt)	---
GB-E+2Gr	43%	55% (50% Glass-50% Basalt)	2% Graphite
GB-E+4Gr	41%	55% (50% Glass-50% Basalt)	4% Graphite
GB-E+6Gr	39%	55% (50% Glass-50% Basalt)	6% Graphite
GB-E+2PTFE	43%	55% (50% Glass-50% Basalt)	2% PTFE
GB-E+4PTFE	41%	55% (50% Glass-50% Basalt)	4% PTFE
GB-E+6PTFE	39%	55% (50% Glass-50% Basalt)	6% PTFE

C. *Mechanical characterization*

Tensile testing was performed according to ASTM D 638 [24] using a universal testing machine (Kalpak Instruments). The composite plates were cut into dog-bone shape test specimens (L = 165 mm) by water jet cutting. At least five specimens were tested for each sample and the average of which is reported here. The test was conducted at a crosshead speed of 5 mm/min at room temperature. Flexural (three-point bending) test was carried out according to ASTM D790 [25]. The average of at least five measurements of each laminate sample is reported here. Rectangular shape (L = 90 mm, W= 12 mm) specimens were used. The crosshead speed was maintained at 2.5 mm/min.

The flexural strength was calculated using:

$$\sigma_f=3PL/2bd^2 \quad (1)$$

Where σ_f is the flexural strength (MPa), P the maximum load (N), L is the support span (mm), b measured specimen width (mm) and d is the measured specimen thickness (mm).

The interlaminar shear strength was evaluated in accordance to ASTM D 2344 [26]. Five specimens were tested, having the following dimensions: 60 mm X 12 mm X 3 mm. A span to- depth ratio of 4:1 and a cross-head speed of 2.5 mm/min were used. Eq. (2) was used to evaluate the interlaminar shear strength:

$$ILSS=0.75P_m/bh \quad (2)$$

Where ILSS is the interlaminar shear strength (MPa), Pm the maximum load (N), b is the measured specimen width (mm) and h is the measured specimen thickness (mm).

D. *Scanning Electron Microscopy*

Fractography examination for the failure specimens were examined using Scanning Electron Microscope (SEM), JEOL model 6390. The surface of the fractured specimens under tensile, flexural and impact tests was examined with different magnifications

III. RESULTS AND DISCUSSION

The tensile strength of the fiber reinforced epoxy composites depends upon the strength and modulus of the fibers, strength and chemical stability of the matrix, fiber matrix interaction and fiber length. Fig. 1 shows typical tensile load versus displacement curves of glass fiber, basalt fiber and unfilled and filled hybrid (glass-basalt) fiber reinforced epoxy composites. Here the load linearly increased with increasing displacement until reaching failure of all the samples.

From the Fig. 2 it is observed that basalt fabric type shows an increase of 5% of the tensile strength when compared with glass fabric reinforced composites. As listed in Table 3 hybridization of glass and basalt fiber (GB-E) showed the tensile strength of 321.8 MPa which is about 13.5% and 8.5% higher than the plain glass and plain basalt fiber reinforced composites. This is due to positive hybridization between glass and basalt and also the better interfacial bonding between the fiber and matrix. Similar result was observed from different authors that Wang et al. [27] proposed an enhancement method for basalt fiber reinforced polymer (BFRP) based on hybridization, where its design was optimized for the application in a long-span cable based bridge. The results obtained showed that, due to the effect of hybridization of the fiber, the composite showed an increase in the overall modulus, potential strength and also fatigue behavior. Similarly, Zhang et al. [28] have fabricated basalt fiber (BF) reinforced with polybutylene succinate (PBS) composite with different wt. % of fibers. They employed an injection molding method and assessed the tensile strength, flexural, and impact properties which increases with increasing wt.% of fibers in PBS matrix. They observed an improvement in the tensile and flexural properties of the PBS matrix due to the synergistic strength offered with an increasing number of basalt fibers in the composite. Dorigato and Pegoretti [29] compared the quasi static tensile and fatigue properties of epoxy based laminates reinforced with woven fabrics of basalt, E-glass and carbon fibers with the same areal density (200 g/m2). All the laminates were prepared by means of vacuum bagging technique, thus obtaining volume fiber content equal to 63.5%, 56.3% and 61.3 % for carbon, glass and basalt reinforced epoxy, respectively. The experimental result showed that the basalt fibers laminates present elastic module and strength values higher than those of the corresponding laminates reinforced with glass fibers, with tensile strength values near to that of carbon fibers based laminates.

Further incorporation of graphite particles into GB-E composite increases the tensile strength and modulus. This may be due to good particle dispersion and strong polymer/filler interface adhesion for effective stress transfer. But increase in addition of graphite and PTFE content up to 6% weight to the composites the tensile strengths and modulus is found to be less this is due to more filler material in the composites damages matrix continuity, less volume of fiber and more void formation in the composites. Subagia et al. [30] fabricated basalt/epoxy composites with tourmaline micro/nanoparticles (0.5–2 wt. %) using the vacuum assisted resin transfer molding technique. Laminates of TM/basalt/epoxy showed that the tensile and flexural strength increased by 16 %, whereas an increase of 27 % and 153 % was observed for the tensile and flexural modulus, respectively. On the other hand the shape of the curves remains essentially unchanged as PTFE filler is added to the matrix, but both the maximum strength and strain decrease.

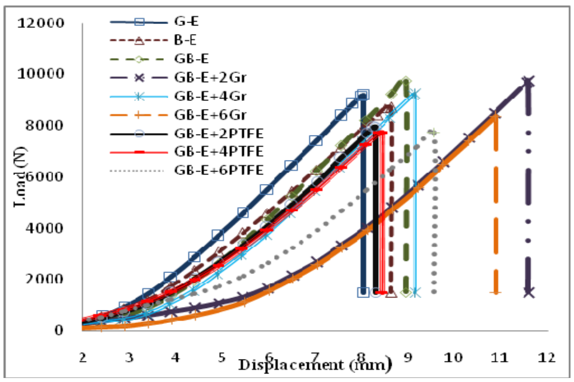


Figure 1. Typical load as a function of displacement of G-E, B-E and unfilled and filled GB-E composites.

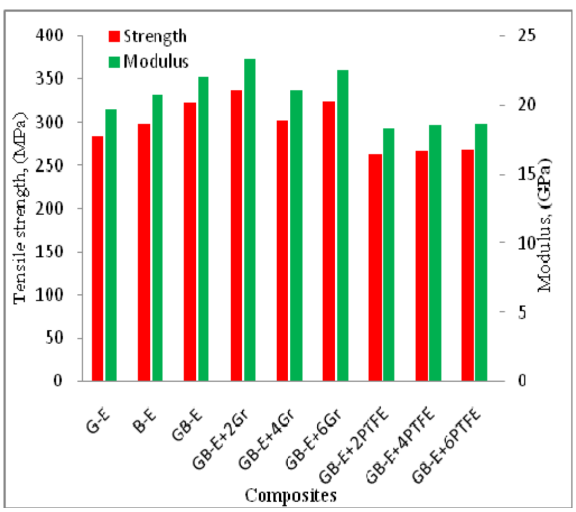


Figure 2. Tensile strength and modulus of G-E, B-E and unfilled and filled GB-E composites.

TABLE 3 Mechanical properties of prepared composites

Composites	**Tensile strength MPa**	**Tensile modulus GPa**	**Flexural strength MPa**	**Flexural modulus GPa**	**Interlaminar shear strength MPa**
G-E	283.33	19.674	334.07	20.967	13.36
B-E	297.15	20.635	244.4	14.7	11.95
GB-E	321.8	21.964	268.41	15.737	13.25
GB-E+2Gr	335.7	23.312	359.91	23.302	14.2
GB-E+4Gr	302.01	20.979	364.21	18.819	13.6
GB-E+6Gr	323.58	22.471	347.8	19.606	13.9
GB-E+2PTFE	262.6	18.236	304.1	16.724	12.08
GB-E+4PTFE	266.4	18.5	331.8	22.487	11.79
GB-E+6PTFE	267.34	18.565	290.3	17.587	8.8

Flexural (3-point) bending tests were performed to check the effect of two different filler particles on the stiffness and strength of composites. Fig. 3 shows the load-deflection curves of all the prepared samples under flexural loading. From the Fig. 4 it is observed that GB-E composites showing higher flexural strength compared to pure basalt reinforced composites and lower as compared to pure glass fiber composite. This can be explained by the fact that failure in basalt laminates takes place in the compressed half section, whilst in glass laminates the whole section is involved in the fracture process upto the tensile face. A similar behavior was also observed by Wittek and Tanimoto [31]. The GB-E with graphite particle composites showed a rapid load rise, the highest maximum load, and catastrophic failure. This means that the composite failed in a brittle manner. On the other hand, the GB-E showed a slow load rise, large yield displacement, and the lowest maximum load. This behavior suggests that the composite failed in a ductile manner because of the high elongation property of basalt fiber. The average value of flexural strength and modulus of the composites are listed in Table 3.

From Fig. 4, it is also observed that addition of both graphite and PTFE filler particles enhances the flexural strength of GB-E composites. Flexural strength of GB-E laminates increased by 34%, 36% and 30% for incorporation of 2%, 4% and 6% graphite particles and also it is increased by 13%, 24% and 8% for increasing wt. % of PTFE in GB-E composites.

Graphite particles filled GB-E composite showed the highest improvement in flexural strength from all the samples. The increased flexural properties signify that graphite particles were homogeneously dispersed in the epoxy matrix.

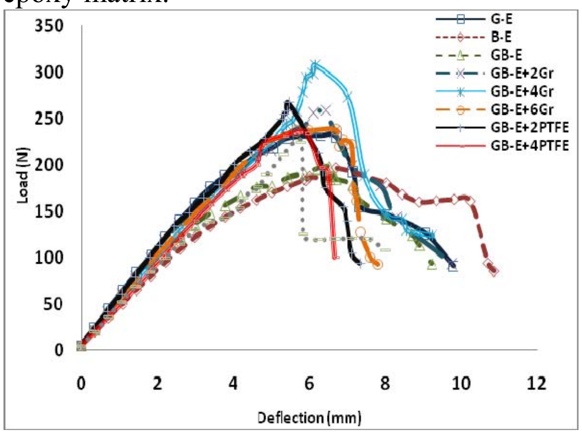


Figure 3. Typical load as a function of deflection of G-E, B-E and unfilled and filled GB-E composites.

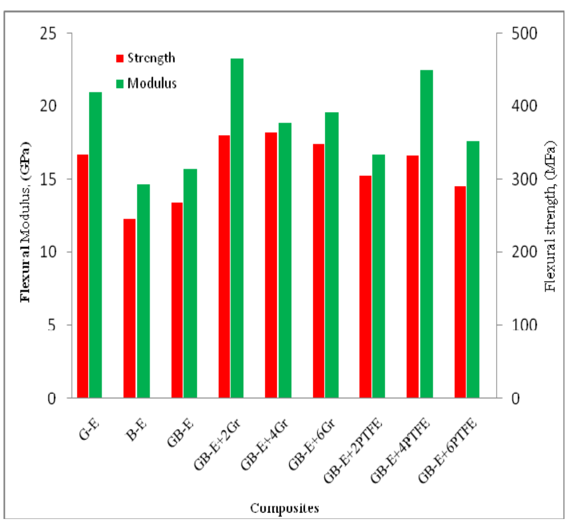


Figure 4. Flexural strength and modulus of G-E, B-E and unfilled and filled GB-E composites.

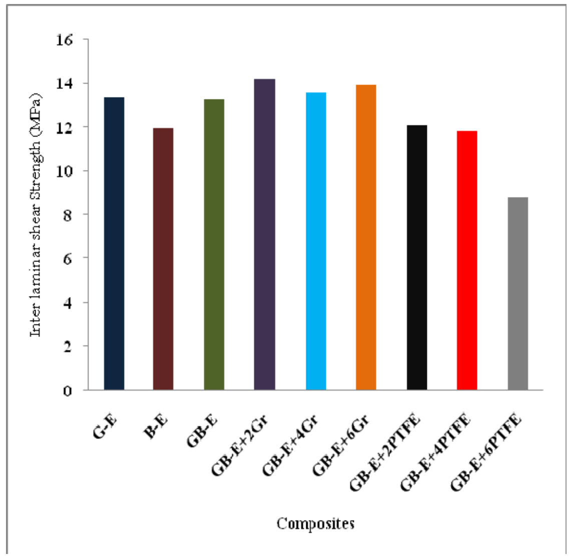


Figure 5. ILSS strength of G-E, B-E and unfilled and filled GB-E composites.

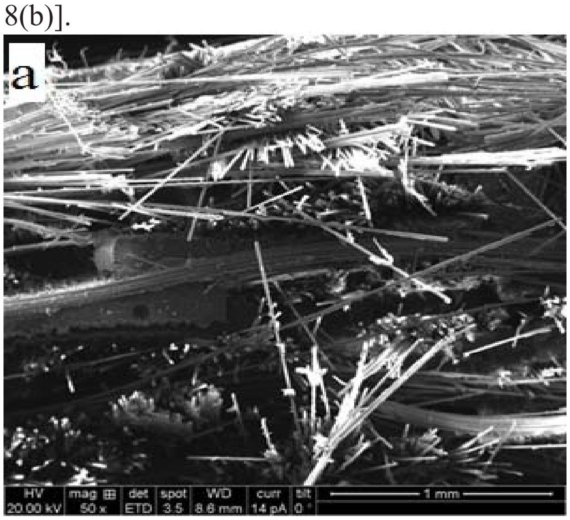
Interlaminar shear strength (ILSS) is usually a limiting design property for composites. When the transverse shear load experienced by a laminated composite exceeds the interlaminar shear strength, a delamination failure will occur between the layers of reinforcing fibers. To measure the ILSS of a composite, a state of pure shear stress should be generated between laminate to induce an interlaminar shear failure. A number of different tests have been developed for the purpose of assessing the ILSS. In this regard, the short beam shear (SBS) method is the simplest and therefore the most used in practice. This test is designed to create interlaminar shear through three-point bending with a small span to- depth ratio (between 4 and 5). The bond strength between the fiber and the matrix resin in the laminated composites was determined by an inter-laminar shear strength test. Generally in the laminated composites, when the transverse shear load exceeds the inter-laminar shear strength, a delamination failure will occur between the layers of reinforcement.

Fig. 5 shows that ILSS of GB-E composite is higher than that of pure glass and basalt fiber reinforced composites. This confirms that a good bond exists between the two different fibers and epoxy resin. Further with the incorporation of graphite particles in GB-E composite increases the ILSS, but in contrast a decrease in ILSS has been observed with increase in PTFE particles. It shows the better graphite particles dispersion in the epoxy matrix. From among the samples GB-E+2Gr showed the best result in the improvement of ILSS.

Addition of graphite and PTFE particles in GB-E composites improves an ILSS property. The presence of micro sized filler particles resulted in formation of roughness of the fiber without damaging the fiber surfaces, and strong interfacial bonding at the fiber matrix interfaces was caused by thermal residual stresses on the fiber surface. These roughness and strong interfacial adhesion act as mechanical interlocking and improved frictional coefficient which contribute the higher flexural and interlaminar shear strength [32].

IV. Fractography

The SEM micrographs in Figure 7(a, b) and Figure 8(a,b) showed the tensile fractured surface of GB-E and GB-E+2Gr composite systems, respectively. The fracture is due to delamination between the layers of the composite samples and fiber-pull out [Fig. 7(a)]. The SEM micrograph shown in Figure 7(b) indicates brittle fracture failure mechanism because as evident from the clean fibers on fractured surfaces. Other important failure mechanisms of composites such as fiber fracture and fiber-matrix debonding are also observed in SEM micrograph [Fig. 7(b)]. SEM characterization of the GB-E+2Gr fractured surface [Fig. 8(a,b)] confirmed the presence of graphite particles on the surface of basalt and in the epoxy matrix showing the elemental components of graphite. This is a qualitative indication of a greater interfacial strength between the fiber filler and the matrix [Fig. 8(b)].



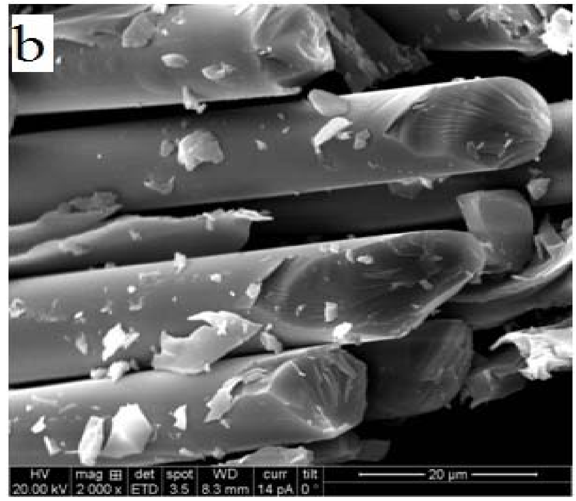


Figure 7. SEM images of tensile fractured surface of GB-E samples: (a) At 50X magnification; and (b) At 2000X magnification
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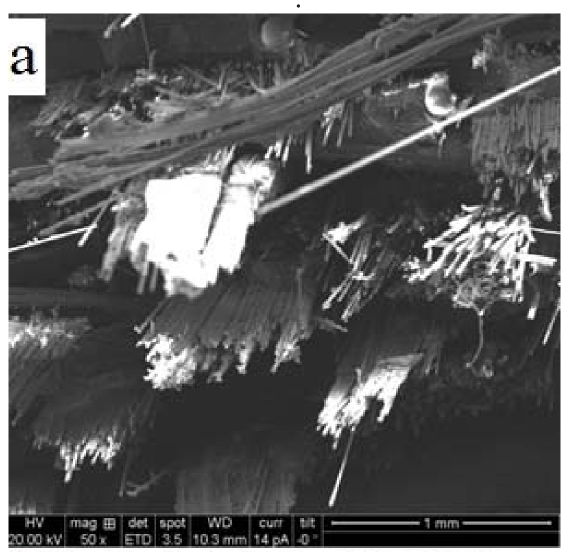

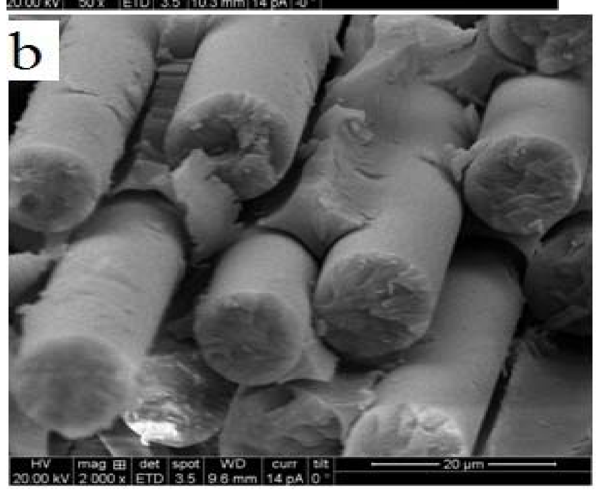

Figure 8. SEM images of tensile fractured surface of GB-E+2Gr samples: (a) At 50X magnification; and (b) At 2000X magnification.

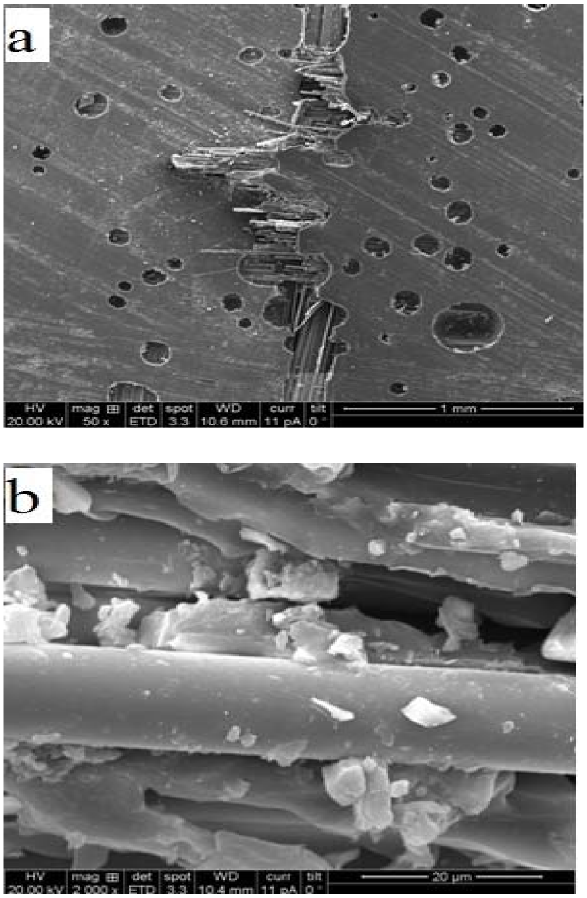

Figure 9. SEM images of flexure fractured surface of GB-E samples: (a) At 50X magnification; and (b) At 2000X magnification.

Fig. 9(a,b) and 10(a,b) shows the fracture surfaces GB-E and GB-E+2Gr of the present samples after flexural test. GB-E (Fig. 9a) showed some fiber pull-outs and voids and also shows crack initiation (Figs. 9 a) at the fiber layer and in the matrix and fabric buckling and delamination (Figs. 9b) were also observed. In Fig. 9b, some delamination and fiber–matrix debonding were seen to occur in the composite and also Delamination was also observed in the region between the glass and plain basalt fiber. Some broken fibers are observed in Fig. 10a, which relates to strong interfacial bonding with Gr/epoxy matrix, i.e., less fiber pull-outs. A good epoxy-fiber bonding and well-impregnated Gr particles were observed in Fig. 10b. In Fig. 10b good fiber–matrix bonding was observed and the dispersed basalt fibers hindered the crack damage in the matrix and glass fibers from propagating leading to high flexural modulus.

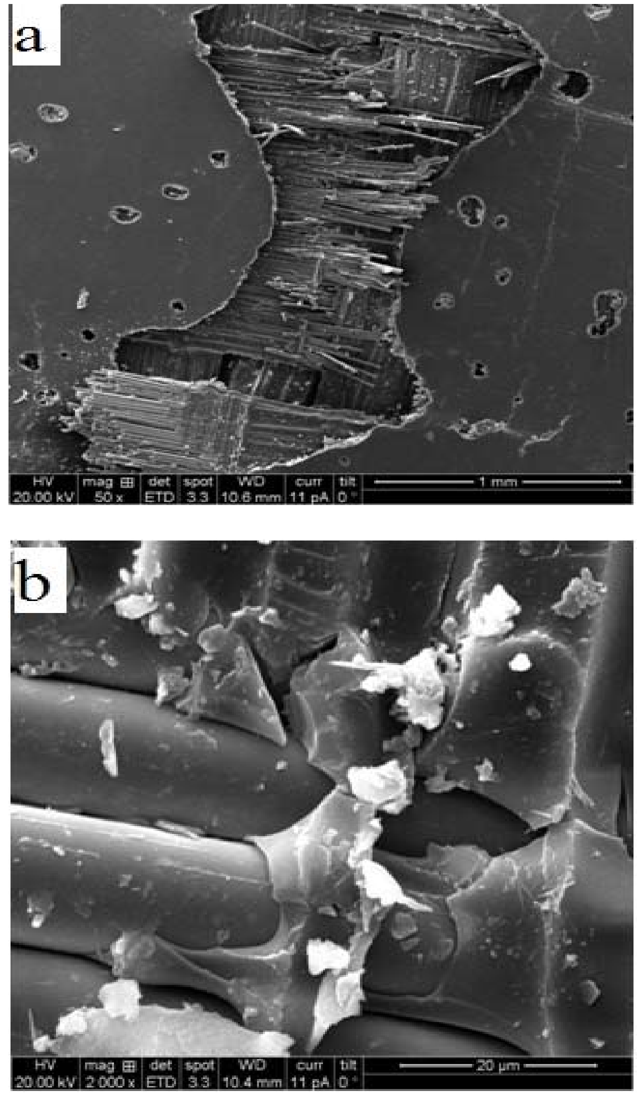

Figure 10. SEM images of flexure fractured surface of GB-E+2Gr samples: (a) At 50X magnification; and (b) At 2000X magnification.

V. Conclusions

Some important conclusions of this investigation are:

- The incorporation of micron sized fillers improves the mechanical properties such as tensile strength/modulus and flexural strength/modulus of GB-E composite. The improvement is more pronounced with the combined addition of graphite microfiller in to the GB-E system.
- The improved results are obtained with 2 wt % and 6 wt. % of graphite filler loading in respect of tensile and flexural properties of GB-E composites. The tensile and flexural strengths show an

increase of 5%, and 35% , respectively as compared to unfilled GB-E composite. The enhancements in mechanical properties are attributed to the good dispersion of particulates in the epoxy matrix which lead to high surface area for strong interfacial bonding, and better load bearing from hybrid fibers.

- Typical failure of unfilled, filled GB-E composites under static tension and flexure as shown that the fracture is accompanied by an extensive amount of delamination, matrix cracking, partitioning of fiber bundles, and breakage of fibers. However, in partilcuate filled GB-E composites, the fracture is associated with less matrix cracks, less breakage of fiber and the fibers remains intact due to the good interaction between fillers in the composites.
- The expanding applications of GB-E in industries would benefit from this study and could provide additional information on the exploitation of micro particles to further enhance the properties of epoxy-based hybrid fiber reinforced composite materials

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