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Effect of Marble Particle on Physico-Mechanical Behaviour of Glass Fiber Reinforced Epoxy Composites

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Abstract: Materials with superior properties have been used to meet the demand for better operational performance in the world of advanced science and technology. To fulfill such needs, the importance of industrial usage of polymer composite materials filled with industrial waste instead of high cost ceramic filler has tremendously increased. In this present work, new classes of glass fiber reinforced epoxy composites with different weight content of marble powder are prepared by simple hand layup technique. Different mechanical properties test were conducted. The experimental study reveals that new hybrid composites shows better impact and hardness resistance compared with unfilled composite, while tensile and flexural strength of the composite decreases. This may be due to the presence of marble particles and a void on the upper surface induces stress concentration zone resulting crack propagation on the matrix region which increases with the addition of filler material.

Keywords: Epoxy resin, marble powder, alkali treatment, mechanical properties

1. Introduction

Polymer matrix composite material reinforced with fiber consists of two or more different elements on macro scale, and also different chemical combination which has a tremendous potential to replace the conventional material [1]. Polymer matrix modification is one the challenge by adding different filler content with different weight proportion to meet the demand. By the addition of particulate fillers, the performance of composites in structural and industrial applications improves showing better property and hence been a subject of considerable interest. But in this background, the use of such inorganic industrial waste in polymer matrices has hardly explored. Therefore, this inorganic industrial waste should be reused or recycled. Therefore, it is important to look for new options to reuse or recycle these inorganic wastes. However, an effort has given in this present work to successfully utilize the marble particulate as filler for making epoxy modified hybrid composites.

2. Literature Review

A great amount of solid waste is generated as a by-product of mineral resources to meet the most potential consumer demand in civil engineering construction industry [2]. There is an urgent need to reutilize the solid wastes for environmental as well as economic reasons. Development of new composites considering the strength, cost, and eco-

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friendliness are being major criteria for the researcher [3]. Fly ash is the principal industrial waste produced by burning the coal in thermal power plants, causes huge industrial pollution and also its storage cost is very high [4]. Previous researchers demonstrated the use of marble waste as partial replacement of natural aggregates in concrete production [5]. The effect of different ceramic filler on the mechanical properties of the composite samples have been tested earlier [6]. However, recycling has the possibility to preserve natural resources by land filling method is suitably adopted reducing the amount of industrial waste during the fabrication of composites, addition of filler to matrix improved the physical and mechanical properties of the composites as well as reduced the cost of the composites [7]. Therefore, the demand of inorganic fillers in developing new class of composites increases day by day. The previous researchers use different filler materials such as ceno-sphere, fly ash, granite etc. for preparing composites [8-13]. Many research efforts have been given for the use of ceramic filler in the composite materials in past decades [14-16]. It is also observed that, the use of different reinforcing material like glass fiber, jute fiber, oil palm fiber and bamboo fiber etc. develops the strength, stiffness and moisture resistance of the composites. Ismail et. al. seen that crushing strength of kenaf fiber composite depends on the fiber orientation and number of layer present in the composite [17].

Marble powder, which is one of the industrial wastes produced during cutting of ornamental stone in stone processing industry, can be used as good filler in polymer composites replacing the conventional ceramic fillers due to its homogeneity, uniform distribution property and chemical constancy. However, the partial replacement of marble particle with conventional cement, no effort has been made till date to critically study the mechanical properties of the marble filled glass fiber composites.

The objective of the present study is to investigate the use of marble particle as filler in epoxy-based glass fiber composite. The physical and mechanical properties of the developed composites are evaluated. The effect of addition of different weight percentage of marble particle in matrix phase improves their mechanical properties.

3. Materials and methods

3.1 Materials

In the present work, three main constituents of the composites are epoxy resin, glass fiber and marble powder correspondingly used as matrix, reinforcement and filler material. Epoxy resin (LY 556) and hardener (HY 951) are procured from Ciba Geigy Ltd., India. Bi directional glass fiber mat was supplied by Saint Govian Ltd. The epoxy resin possesses a density of 1.10gm/cc and a modulus of 3.42GPa. The glass fiber has a density of 2.59 gm/cc and a modulus of 72.5Gpa. Marble powder was collected from the local stone processing industry. The marble powder possesses a density of 2.68gm/cc [12]. The epoxy and hardener mixed thoroughly in a ratio of 10:1 before fabrication of composite. Marble powder are dried in an oven and sieved to a size of 90-120µm before fabrication.

The detail composition of marble powder is shown in Table 1[13]. Alkali treatment has been considered as an effective chemical treatment to improve the mechanical and tribological properties of the composites [14]. The tensile strength of Kenaf fiber reinforced polyester composites are increased with alkali treatment of fibers.[18]

The dried marble particles are dipped in 5% concentrated NaOH solution for 1 hour at room temperature (30ºC). The marble powder were washed in distilled water several times and neutralized with acetic acid and washed again with distilled water. The neutralized powders are then dried in oven to with a temperature of 100ºC to remove the moisture content. The process removes the impurities and the presence of O-H group increases the hyperbolic and interfacial adhesion property of the treated material [16] as presented in equation 1.

3.2 XRD analysis of marble powder

Philips X-Ray Diffractometer was used for examining the crystalline phase. The test was carried out using Cu Kα radiation.XRD analysis of the marble powder is shown in Figure 1. [13]

Table 1 - Chemical composition of waste marble powder [13]

Ceramic Oxides (%)	**Marble powder**
MgO	0.4
CaO	51.7
SiO_2	0.18
Al_2O_3	0.67
Fe_2O_3	0.44
K_2O	0.21
SO_3	0.08
LOI	46.04

LOI-Lost on Ignition

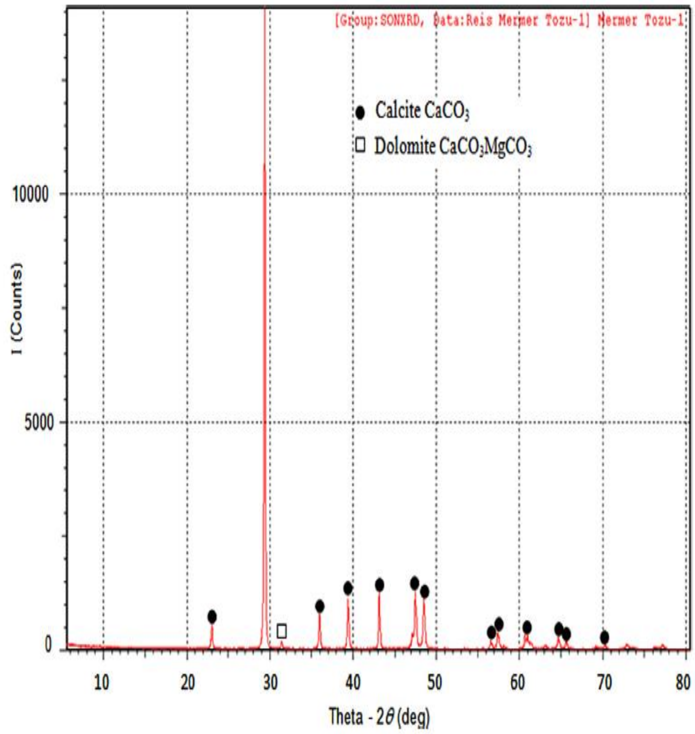

Fig. 1 - XRD analysis of waste marble powder

3.3 Composite Fabrication

The composites are fabricated by the conventional hand layup technique at room temperature afterwards light compression molding. A number of composite samples are prepared with different weight proportions of marble powder as represented in Table 2. Finally, the composites were cut into the required dimensions as per the standards for the evaluation of physical, mechanical properties.

Table 2 - Hybrid composition of the composites

Composites	Composition
EGM0	Fiber (40 wt%)+ Epoxy (60 wt %)+ Filler (0 wt%)
EGM1	Fiber (40 wt%)+ Epoxy (55 wt %)+ Filler (5 wt%)
EGM2	Fiber (40 wt%)+ Epoxy (50 wt %)+ Filler (10 wt%)
EGM3	Fiber (40 wt%)+ Epoxy (45 wt %)+Filler (15 wt%)

4. Experimental details

4.1 Test of Density, Micro hardness, Impact and Tensile Properties

The theoretical density of developed composites can be found out by using the equation stated by Agarwal and Broutman [19].

$$\rho_{ct} = \frac{1}{\left(W_f / \rho_f\right) + \left(W_m / \rho_m\right) + \left(W_p / \rho_p\right)} \qquad (1)$$

According to Archimedes principle the actual density (ρ_{ce}) of prepared samples can be found out experimentally.
The void content of respective composite samples can be calculated using following equation:

$$V_v = \frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}} \qquad (2)$$

Digital Leitz micro-hardness tester is used in the observation to measure the micro-hardness of the composites. A diamond shaped indenter, in the form of right pyramid is pressed over the sample to get the square impression on the composite surface. The Vickers hardness number is found out using the equation:

$$H_V = 0.1889 \frac{F}{L^2} \quad (3)$$

$$L = \frac{X + Y}{2} \quad (4)$$

Where:
L=Diagonal of the square impression (mm)
X= Horizontal length (mm)
Y=Vertical length (mm)

The tensile test was carried in a universal testing machine Instron 3369 as per ASTM-D-3039-76 standard using dog-bone specimen. The dimension of the specimen for the test is 150 mm × 10 mm × 4.5 mm, with the 4.5-mm thickness being maintained for all the samples. Geometry configuration for tensile test is shown in **Figure 2**. The test is carried out with a cross head speed of 10 mm per minute.

The inert laminar shear strength of the specimens is also conducted using UTM Instron 3369. The test is conducted as per ASTM-D-2344-84 standard in cross head speed of 2mm/min with a sample of dimension 60 mm × 10 mm × 4.5 mm. The geometry of the sample is shown in **Figure 3.** The *ILSS* is calculated from the following equation:

$$ILSS = \frac{3P}{4bt} \quad (5)$$

$$FS = \frac{3PL}{2bt^2} \quad (6)$$

Low velocity impact tester was used for conducting the impact test of the prepared samples (ASTM D 256). A pendulum impact tester is used to determine the impact strength of the specimen by crushing the V-notched specimens with a pendulum type hammer, recording the exhausted energy relating to the dimensions of the specimen. The composite samples with dimension 64 mm × 12.7 mm × 4.5 mm is considered for the test as shown in **Figure 4**.

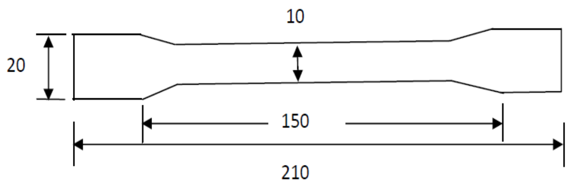

Fig. 2 - Composite sample for tensile test

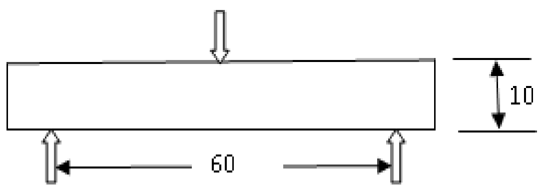

Fig. 3 - Composite sample for Flexural test

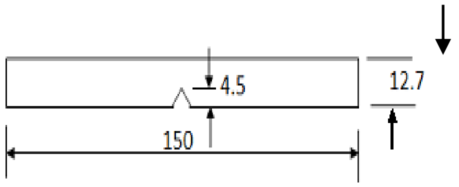

Fig. 4 - Composite sample for Impact test

4.2 SEM examinations

The surface of the specimen was examined by scanning electron microscope (Hitachi Model S-3400N) PC-Based Variable Pressure Scanning Electron Microscope. A thin film of gold was coated on the specimen by sputtering technique to enhance the conductivity.

5. Results and discussion

5.1 Density and Void content

The composite specimens with corresponding theoretical and measured densities and volume fraction of voids are presented in Table 3 and Figure 5.It is observed from Table 3 that sample without filler (EGM0) has least void content as compared with other samples. Porosity in the composites formed due to air-filled cavities becomes unavoidable during fabrication process. Higher void content usually indicates higher sensitivity to water dispersion and weathering and reduces fatigue strength (Agarwal and Broutman, 1990) [19]. However, it is also observed that presence of voids is an unavoidable phenomenon, when the composites are prepared by simple hand-lay-up technique.

Table 3 Theoretical and measured densities of composite samples

Composite samples	Theoretical density (gm/cc)	Measured density (gm/cc)	Void Content (%)
EGM0	1.40	1.42	1.40
EGM1	1.44	1.48	2.70
EGM2	1.48	1.54	3.89
EGM3	1.52	1.61	5.59

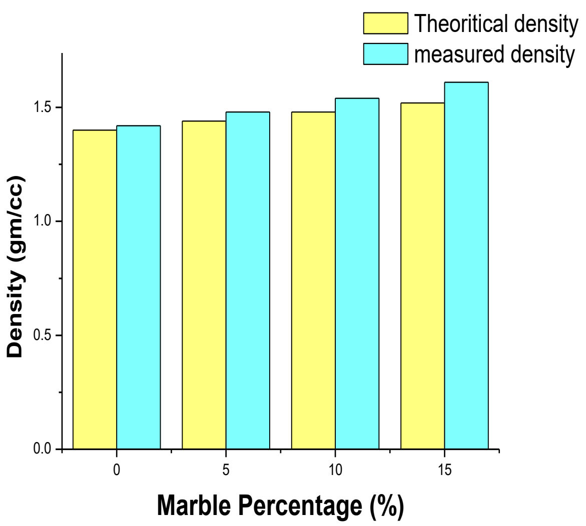

Fig. 5 - Theoretical measured densities of composite samples

5.2 Micro hardness, Tensile Strength, Flexural strength and Impact strength

The hardness values of the prepared composite specimens are represented in Table 4. It can be noticed that the micro-hardness is marginally affected by the addition of marble filler into the composites. Sample EGM2 with 10 wt.% marble powder exhibits maximum hardness value of 49H_V among all the composites as shown in **Figure 4.** Generally, mean hardness of the composite is expected to increase on addition of oxide ceramic filler like marble particulate. However, the experimentally measured values are not remained consistent with this theory. The insignificant effect of marble fillers on hardness of composites may be due to the presence of voids and pores.

Tensile properties were determined by an Instron 3369 machine at room temperature with a gauge length of 50 mm and a crosshead speed of 10mm/min. The test was repeated five times for each sample, and the average value was recorded. The tensile strength test results for the glass-epoxy composite filled with marble powder ranging from 0wt% to 15wt% are presented in **Table 4**. The results show that the addition of marble particulate resulted in a significant decrease in the tensile strength of the glass-epoxy (EGM) composite from 363.27 MPa to 281.64 MPa as shown in **Fig. 2**. The tensile strength of filled composites (EGM1, EGM2 and EGM3) is less as compared to the unfilled (EGM0) composite. Similar behaviors are observed by the previous researchers using glass-epoxy-rice husk composites [10]. The decreased in tensile strength of the composites was observed due filler content (5 wt% to 15wt %) leading poor adhesion between matrix, reinforcement and filler. This behavior is mainly due to the increase of void content on filler addition that resulted in the decrease of the interfacial bond strength between the filler particles and the resin matrix. Furthermore, the sharp corner of the irregular shaped filler particle induces stress concentration during tensile loading. The fillers cannot carry the load due to this de-bonding, which result in composite strength with increase in filler loading [20].

Thomason et al. suggests that reinforcement (glass fiber) in a composite restrains the deformation of the matrix (resin) which decreases the tensile strain value [21]. Consequently; the tensile modulus of the specimens likely improves. The tensile modulus of marble filled composite is increasing from 5.16 to 7.51 GPa as shown in **Table 4.** The presence of marble filler in glass–epoxy composites reduces the tensile strength and increases the modulus instead of presence of long glass fibers as the reinforcement.

Composite materials utilized in structural applications are inclined to failure, which leads to development of new class of composites with improved flexural properties. Inter laminar shear stress is the stress developed between the two layers of the laminated composites. The inert-laminar shear strength or flexural strength results obtained from three point bend test for the glass-epoxy composite filled with marble powder ranging from 0wt% to 15wt% are presented in Table 4 and shown in Fig.6.The results show that the addition of marble resulted in a gradual decrease in the flexural or inter laminar shear strength of the glass-epoxy (EGM) composite from 387 MPa to 323 MPa respectively as shown in **Figure 6**. The presence of micro-sized filler particles and voids in a composite cause's localization of high stresses. The localized high stress generates relative deformation (de-lamination) between the layers.

Impact load is considered as a type of suddenly applied load with certain initial velocity. The resistance to breakage at this impact load condition is considered as impact strength of the material. In polymer matrix composite materials, the impact strength was specified as a common mechanical property [22]. **Table 4** presents the observed impact energies of different hybrid composite samples. The result shows the impact strength of glass-epoxy composites improves with the addition of marble particulates as filler. In case of marble filler, the improvement in the resistance to impact loading is marginal. In structural design, composite materials are exposed to impact loads. Therefore, the impact strain energy plays important role for selection of new composites for many engineering applications. The results of the present study show that marble powder can be a suitable filler material in fabricating high strength (impact) composites.

Table 4 Composite samples with their mechanical properties

Composite samples	Mean hardness (H_v)	Tensile strength (MPa)	Tensile modulus (MPa)	Flexural strength (MPa)	Impact energy (J)
EGM0	36	364	5.17	386	0.82
EGM1	41	329	6.28	362	0.91
EGM2	48	301	6.73	348	0.97
EGM3	45	280	7.54	322	1.05

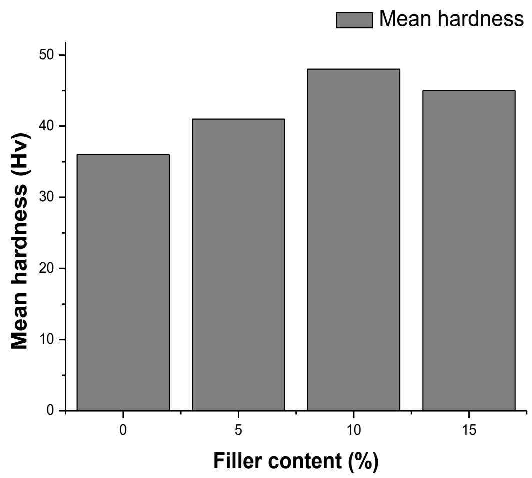

Fig. 5 - Mean hardness of composite samples

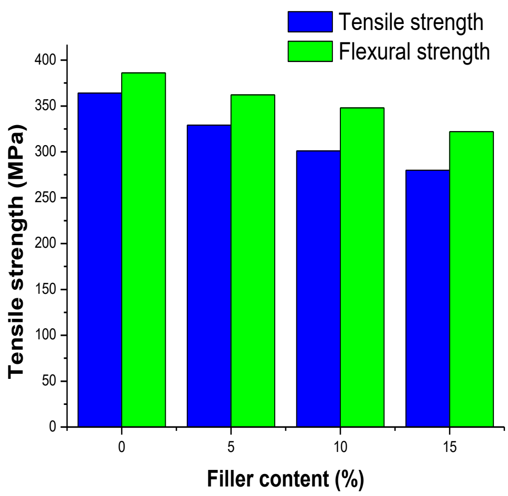

Fig. 6 - Tensile strength and Flexural strength of composite samples

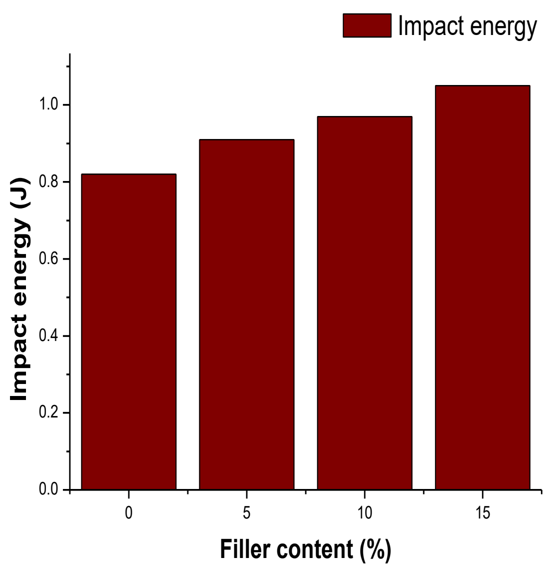

Fig. 7 - Impact energy of composite samples

5.3 Micro-structural observations

Figures 8(a-d) are the SEM micrographs of marble particle-filled glass-epoxy composites after tensile and impact tests. The smooth surface signifies better compatibility of marble powder with the glass-epoxy matrix. After the impact test, the complete fracture of the glass fiber in the specimen is shown in Fig.8 (a-c). The Fig.8(b) shows the fracture behaviour of the present fiber in linear and transverse directions. Similarly, after the tensile test, the broken layers of glass fiber mat are clearly seen in Fig. 8(d). The delaminated layers of the marble-filled glass-epoxy (EGM1) lamina are seen in Fig.8 (a-d) respectively. Fig. 8(c and d) clearly show the presence of marble particle in the composite. The maximum shear stress occurs between the inter layers and a crack running along the mid plane of the lamina causing de-lamination.

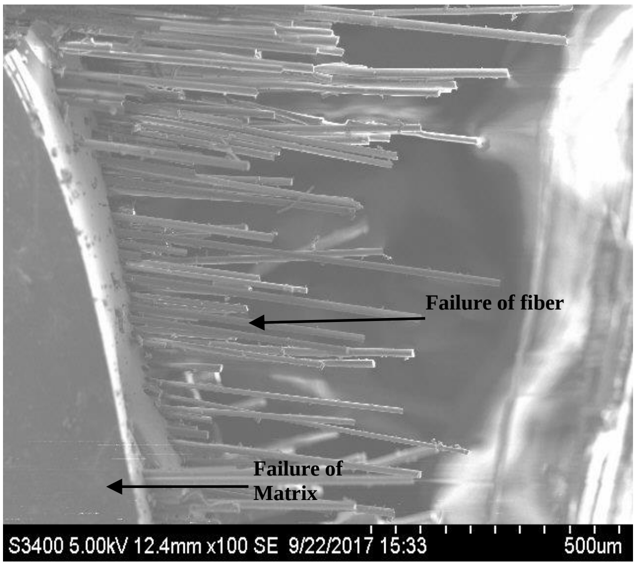

Fig.8.a - Micrograph of EGM0 sample after impact test

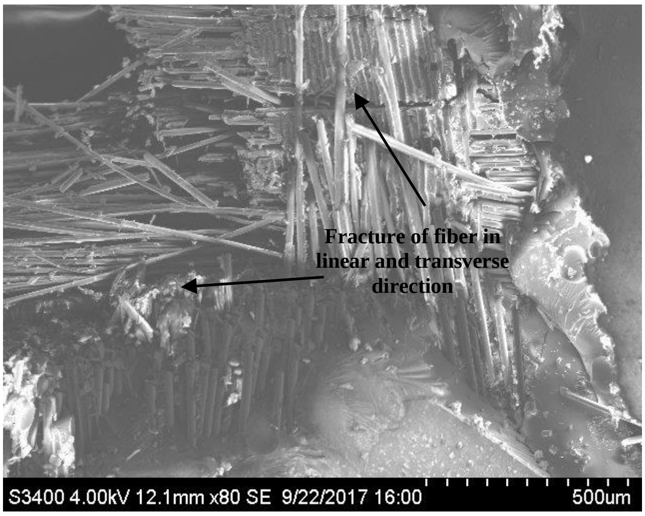

Fig.8.b - Micrograph of EGM1 sample after impact test

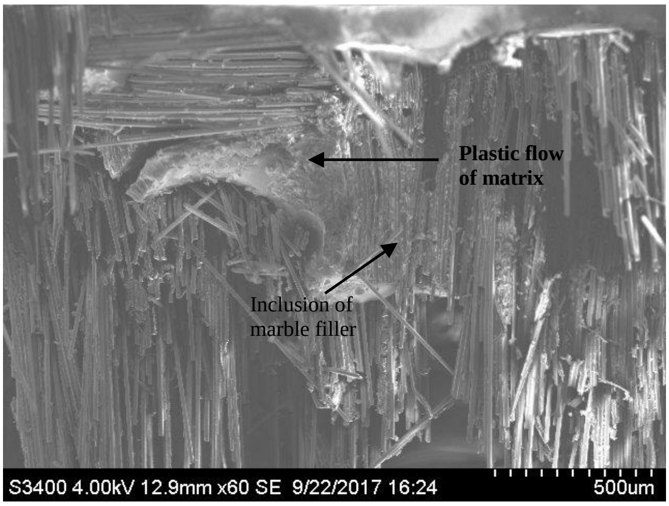

Fig.8.c - Micrograph of EGM2 sample after impact test

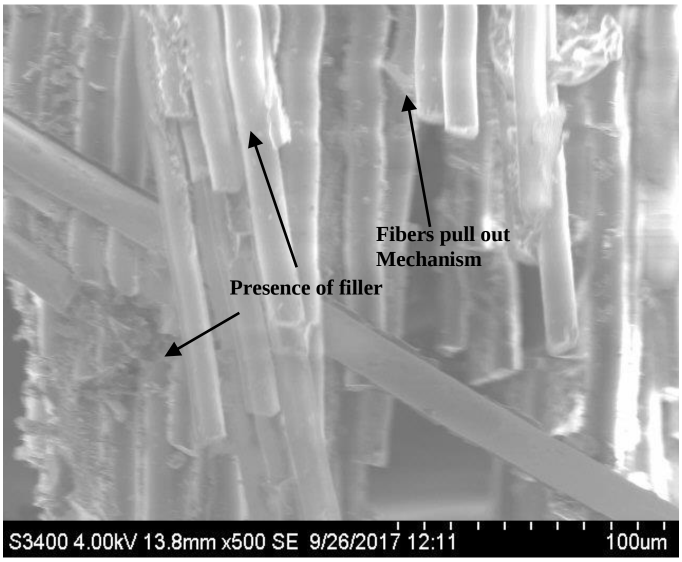

Fig.8.d - Micrograph of EGM0 sample after tensile test

6. Conclusion

The following conclusions can be drawn from our present experimental observations:

Epoxy-glass-fiber filled with marble powder can be fabricated using simple hand lay-up technique.

- From the experimental observation it is found that the composite with 10wt.% marble powder shows better filler characteristics and has a maximum hardness of 49*Hv* for all the composites.
- The unfilled composite specimen (EGM0) shows better tensile strength as compared to filled samples. The tensile strength of composites reduces by the addition of filler.
- It is noticed that the tensile modulus of composites is increasing about 45.84% (from 5.16 to 7.54GPa) by the addition of 15% of filler in unfilled composite (EGM0).
- It is observed that the impact strength of the composites is increasing about 28.04 % (from 0.82J to 1.05J) by the addition of 15% of marble filler.
- The morphological study clearly indicates the fracture behavior of different composites.
- In general, these marble modified hybrid composites may be recommended for low cost construction materials, pipes, automobile parts, artificial limbs, etc.

Further this research may be extended to sliding, erosion and abrasion wear behavior of these composite specimens.

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