MECHANICAL CHARACTERIZATION OF GLASS FIBER REINFORCED COMPOSITE CONTANING NANOPARTICLES

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ABSTRACT - Composites play a vital role in aerospace, land transportation and consumer goods due to their high specific strength and stiffness, leading to reduction in the mass of moving objects. Glass epoxy composites are a unique material for some of the important applications such as pressure vessels of commercial applications, Storage of chemicals, sewage treatment plants, chemical industrial applications.

In the present investigation, a small amount of Cloisite 30B®Cloisite 15A® are to be considering and these nano powders direct mixing to disperse into a mono-component epoxy resin and used as matrix for advanced composites with woven roving glass fiber reinforcements. These nano powders are added directly into the epoxy resin and uniform dispersion is to be carry out by using mechanical stirrer. In manufacturing of the hybrid nano composite with glass fiber, and epoxy polymer in the ratio of 1wt %, and 2 wt% percent of volume are to be considering. These different wt% nano-phased polymer-based nanocomposites plates are to be manufacturing by moulding in vacuum as per ASTM standards. Fatigue tests are to be conducting under constant amplitude, both under tension–tension and three points bending loadings.

1. INTRODUCTION

Fiber-reinforced polymer is a composite material made of a polymer matrix fortified with filaments. The filaments are typically fiberglass, carbon or aramid, while the polymer is normally an epoxy, vinyl ester or polyester thermosetting plastic. FRPs are regularly utilized as a part of automotive, marine, and development commercial ventures. The strength properties of FRP aggregately make up one of the essential purposes behind which structural engineers select them in the configuration of structures. ***Kanny et al*** [1] prepared a hybrid laminate composite comprising nanoclays, glass fiber and epoxy polymer using VARIM. The effect of nanoclay on compressibility, structure and mechanical properties of laminates was examined. The nanoclay content was varied from 0 wt.% to 5 wt.% in laminates. The result showed that the resin flow speed continuously decreased with corresponding increase of clay content. ***Sivasaravanan et al*** [2] observed an increase in impact strength with addition of nano clay in epoxy matrix. The test was performed by izod testing machine, it was found that addition of 5 wt.% of nano clay shown very good results compare to other percentage of nano clay. Average value of 5 wt.% of nano clay was 10.75 J/m when compared to other combination of nano composite materials. ***Dolati et al.*** [3] observed increase in impact damage resistance of epoxy resin reinforced with nanoclay particles. The XRD and TEM analyses confirmed the exfoliation and intercalation of the nanocaly particles and the dispersion of nanoclay in the epoxy resin system for samples containing 0 wt.% and 1.5 wt.% nanoclay. The result of investigating sample without nanoclay showed that placing the layers at an angle of $45°$ in the fiber orientations, resulted in improvement with respect to the impact. ***Azeez et al.*** [4] studied that the final morphology, physical, chemical and barrier properties of the nanocomposites were influenced by processing method, clay modifier and curing agents. Epoxy clay nanocomposites showed remarkable improvement in tensile, flexural and fracture toughness properties. ***Agubra et al*** [5] studied that significant clay exfoliation was achieved by combining the magnetic stirring and shearing mixing followed by three roll milling to disperse montmorillonite nanoclay in the epoxy matrix. A combination of ultrasound sonication and three roll milling on the other hand, resulted in poor clay exfoliation while the sonication process degraded the polymer network, which adversely affected the nanocomposite final properties relative to the unmodified E-glass/epoxy polymer. ***Sharma et al.*** [6] performed tensile and bending tests on nanocomposites showed that with the addition of nanoclay up to 3 wt.%, the tensile strength increased and then decreased at a loading of 5 wt.%. The flexural strength increased with addition of nanoclay up to 5 wt.%. The hardness of the nanocomposites also increased with increasing nanoclay content. ***Dorigato et al.***[7] prepared epoxy-clay nanocomposites by using of organo-modified clays, to evaluate their potential use as matrices for structural long-fiber composites with improved properties ***Manjunatha et al*** [8] studied the tensile fatigue behavior of silica nanoparticle-modified glass fiber reinforced epoxy composite. The epoxy resin was a standard diglycidyl of Bisphenol A with an epoxide. The GFRP composite laminates were manufactured by resin infusion under flexible tooling technique. An anhydride-cured thermosetting epoxy polymer was modified by incorporating 10 wt.% of well-dispersed silica nanoparticles. The fatigue life of 10 wt.%

silica nanoparticle-modified bulk epoxy is about three to four times higher than that of neat epoxy. **Daud et al** [9] investigated three-phase glass fiber reinforced composites (GFRP) consisting of traditional woven glass fiber and polyamide-6 (PA6) matrix dispersed with organically modified layered silicates. The fabrication of GFRP with different weight percentages of layered silicates was successful when the matrix contains less than 5 wt.% of the layered silicates. The improvement due to the high aspect ratio and high stiffness of the layered silicates was illustrated through the matrix-controlled properties of the GFRP. The results showed that the GFRP with 5 wt.% layered silicates offer the largest improvement of approximately 30% increase in both flexural strength and compressive strength at elevated temperatures. **Quaresimin et al.**[10] worked on the effect of three different commercially available nano-modifiers on the mechanical properties of an epoxy/anhydride unidirectional carbon fiber reinforced laminates. The polymeric matrix consisted of a blend of the diglycidyl ether of Bisphenol A and the epoxy novolac resin. The work had shown that tensile modulus exhibited little difference between the unmodified laminates while a modest decrease was observed for the tensile strength for the vapors grown carbon fiber and nanoclay modified systems. **Lam et al.** [11] studied the hardness of nanoclay/epoxy composites with different amounts of nanoclay particles. Microscopic observation using SEM was conducted to measure the cluster size of the nanocomposites. It was found that the hardness of the nanocomposites increased with increasing nanoclay content. It was also seen that there was an optimal limit. **Yasmin et al.** [12] found that the compounding of clay nanocomposites with a shear mixing was highly appealing. It produced good dispersion of clay particles within a short period of time and also disordered intercalates with an average basal spacing higher than 5 nm regardless of clay content. The elastic modulus of the nanocomposites was found to increase with increasing concentration of clay and a maximum of 80% improvement was observed for an addition of 10 wt.% of clay.

From the review of literature, it is found that comparison of mechanical properties of glass fiber reinforced composite incorporating the two different nanoclays Cloisite 30B® and Clositie 15A® has not been reported.

2. FABRICATION & EXPERIMENTATION

2.1 Fabrication of specimens

2.1.1 Materials

All of the specimens were made by E-glass fibre unidirectional of 301 g/m^2 that were obtained from Saetex India Private Limited. Epoxy resin MGS® RIM 135 was used as the matrix material, and Amine based hardener MGS® RIM H 134 was used with the selected epoxy resin; both are manufactured by Hexion. Two nanoclays Cloisite 30B® and Cloisite 15A® were brought from Southern Clays Limited.

2.1.2 Mixing of nanoclay into epoxy

The resin is taken in glass beaker and nanclay is added to it. Homogenizer is used for breaking agglomerates of nanoclay in small sizes and uniformly mixing in epoxy resin. A stirrer setup as shown in Fig. 2.1 was used for mixing. The mixtures were stirred at 20000 rpm for 5 minutes.

Fig. 2.1 Homogenizer used for mixing of nanoclay

2.1.3 Ultrasonic mixing after mechanical stirring

After homogenization of epoxy/nanoclay mixture, ultrasonic mixing of epoxy/nanoclay using an ultrasonic probe is

done. Sonication was done for evenly dispersion of nanoparticles into liquids and better intercalation of epoxy into nanoclay. The degassing was performed by keeping the mixture into ultrasonic bath for 10 minutes as shown in Fig.2.2

Fig. 2.2 Dispersion of nanoclay into epoxy resin with probe

2.1.4　Mixing of epoxy/nanoclay solution with hardener

　　After ultrasonic, the solution is mixed with hardener. Mixing volume ratio of solution with hardener was 100:38. After mixing, mechanical stirring up to 5 minutes was done.

2.1.5　Manufacturing of fiber reinforced composite

　　Epoxy modified with Cloisite 30B® and Cloisite 15A® (1 wt.% and 2 wt.%) is used with E- glass unidirectional fibers to manufacture two plies laminate having [±45⁰] stacking sequence using vacuum assisted resin infusion moudling as shown in Fig.2.3.The laminate was cured at 120 ℃ for 7hrs and then left at room temperature for 24 hrs.
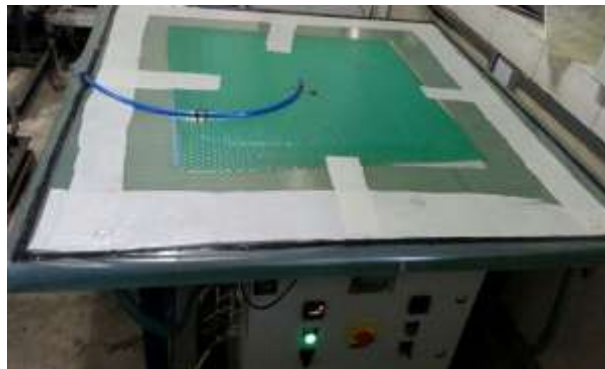

Fig. 2.3 Manufacturing of fiber reinforced composite

2.2 Specimens specifications

　　The specimens had been cut and prepared as per the ASTM standards D3037/3039 and D790 for tensile and bending tests respectively. The dimensions of specimens are shown below.

Table 2.1 Specimens specifications for testing

Parameters for specimens	Specimens for tensile testing	Specimens for flexural testing
Length	250 mm	5 mm
Width	25.4 mm	12.7 mm
Thickness	1.5 mm	1.5 mm

2.3 Testing methods

2.3.1 Tensile testing

A universal tensile testing machine shown in Fig. 2.4 and Fig. 2.5 was used for the testing of the GRP specimens for its tensile strength. The test specimens had been prepared according to ASTM D-3039 standard. The specimens were tested until they break indicating the peak load and ultimate stress value they can bear at required time period to estimate the degradation in the same machine.

Fig.2.4 UTM testing machine

Fig. 2.5 Specimen positioning

Three-point flexural test

   Three-point bending tests of specimens were carried out in using Zwick / Roell. The test specimens had been prepared according to ASTM-D-790 standard.

Fig. 2.6 Three-point flexural test machine

Fig. 2.7 Specimen positioning

2.3.2   Micro hardness test

        Micro hardness test (shown in Fig. 2.8) was conducted on specimens with different clay loadings

Fig. 2.8 Micro hardness equipment

Fig. 2.9 Indent on specimen

The load applied was 50 gm and VHN values were determined by applying this load by using a calibration distance of 50 units in Quantinet software as shown in Fig. 2.9 used for image analyzing. The dwell time used during load application was 20 seconds. An indent is formed in diamond shape used for calculating VHN as shown in fig. above.

2.3.3   Scanning electron microscope (SEM)

Scanning electronic microscope shown in Fig. 2.10 was used to test GFRC specimen microstructure. The dimensioning of specimens was done according to block size of machine. The polishing of specimens was done by using gold coating equipment as shown in the Fig.2.11. The polished specimens were used to observe the microstructure of specimens at different magnification.

Fig. 2.10 SEM machine   Fig. 2.11 Gold coating equipment

SEM micrographs are helpful in viewing the micro-structure of material, hence showing any change in physical structure of material and showing any defects like cracks, voids generated after loading of clay and hygrothermal degradation of the material. These are also helpful in calculating the area fraction of fiber and epoxy in the given specimens and the changes occurring.

3. RESULTS AND DISSCUSSION

The prepared specimens were studied for their morphological and mechanical properties, as discussed in the following section.
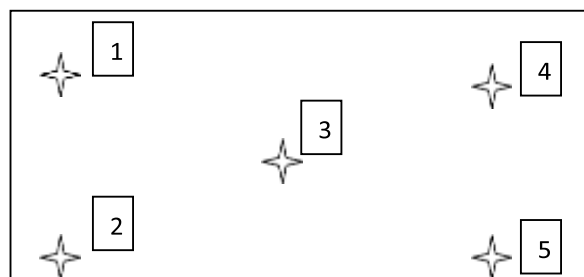
3.1 Micro-hardness

Fig. 3.1 Loading points on specimen

The micro-hardness of specimens at different clay loading was measured. The table 3.1 shows the experimental measurements of micro hardness of the composites with different nanoclay contents. An average hardness was calculated by 5 indentation measurements of each type of samplesTable 3.1 Micro hardness values for different clay loading specimens

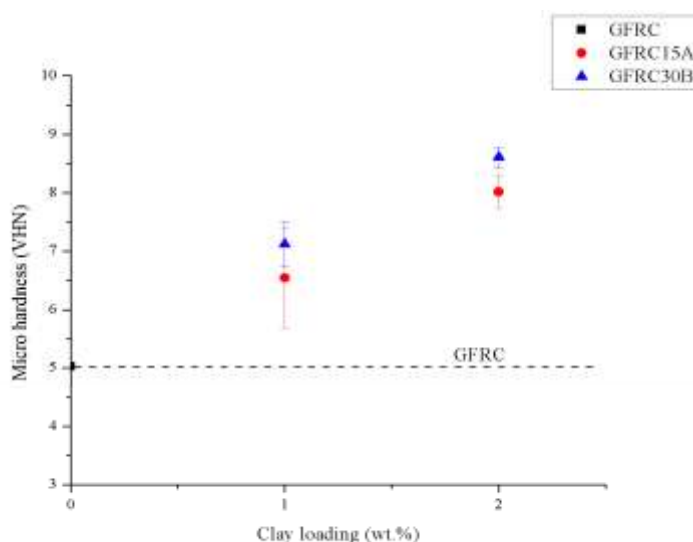|		Micro hardness values			
loading points	wt.% clay	1 wt.% clay Cloisite 15A®	1 wt.% clay Cloisite 30B®	2 wt.% clay Cloisite 15A®	2 wt.% clay Cloisite 30B®
point 1	4.861	5.316	6.739	8.494	8.672
point 2	4.471	6.361	7.048	7.811	8.843
point 3	4.539	6.351	7.015	8.036	8.394
point 4	5.875	7.502	7.759	7.760	8.480
point 5	5.369	7.165	7.036	7.972	8.672
Average	5.023	6.539	7.120	8.015	8.612

Fig. 3.2 Micro hardness test results

The average hardness of Cloisite 30B® at all concentration is more than the average value of Cloisite 15A®. The micro-hardness of nanocomposite increased with increase in nanoclay loading. The highest value at micro-hardness obtained at 2 wt.% is 8.612 with Cloisite 30B® nanoclay.

5.1 Flexural test

The results obtained by conducting three-point flexural tests on nanocomposites using Zwick/Roell universal testing machine are shown below in Table 5.2. Addition of different nanoclays has resulted in improvement of flexural strength. Maximum enhancement in flexural strength got at 2 wt.% with Cloisite 30B® nanoclay was 59% in comparison to neat epoxy glass fiber specimens. Also, the gap between flexural strength of Cloisite 30B® and Cloisite 15A® got widened at 2 wt.% nanoclay loading as shown in Fig. 3.3.

Table 5.2 Results of flexural test

Specimens name	Specimens No.	Flexural strength (MPa)	Average Flexural strength (MPa)
0 wt.% clay	1	134	152
	2	171	
	3	151	
% clay Cloisite 15A®	1	172	168.6
	2	165	
	3	169	
% clay Cloisite 30B®	1	174	

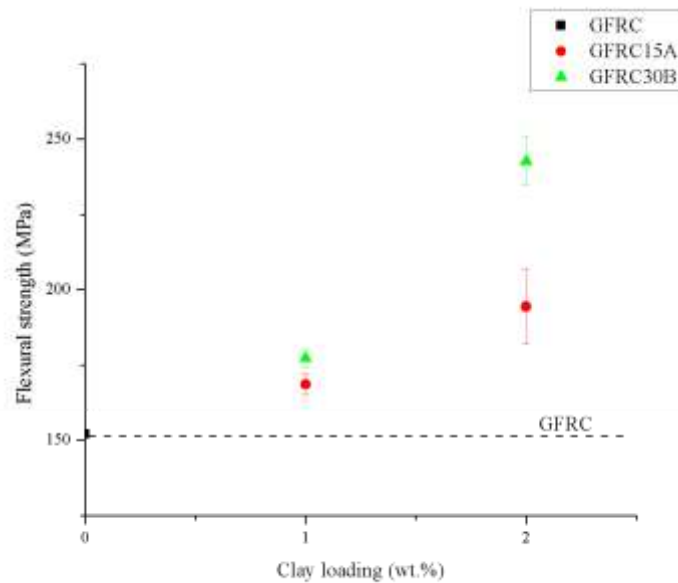|	2	178	177.4
	3	180	
% clay Cloisite 15A®	1	181	194.4
	2	196	
	3	206	
% clay Cloisite 30B®	1	235	242.6
	2	251	
	3	242	

Fig. 3.3 Flexural strength test results

5.2 Tensile test

The results obtained by conducting tensile tests on nanocomposites using Zwick/Roell universal testing machine are indicated below in Table 3.3.

Table 5.3 Results of Tensile test

Specimens Name	Specimens No.	Tensile modulus (MPa)	Tensile strength (MPa)
0 wt.% clay	1	625	73.6
	2	546	63.6
	3	542	69.2
1 wt.% clay Cloisite 15A®	1	731	50.1
	2	748	50.1
	3	765	50.5
1 wt.% clay Cloisite 30B®	1	812	66.6
	2	806	66
	3	783	63.4
2 wt.% clay Cloisite 15A®	1	858	67
	2	890	74.2
	3	853	61.6

	1	964	66.3
2 wt.% clay Cloisite 30B®	2	976	69.4
	3	970	67.4

From Fig. 3.4a, it is observed the tensile modulus of nanocomposite increased with increasing concentration of nanoclay. The improvement in tensile modulus can be attributed to the good dispersion of nano size clay particles. Fig. 3.4b indicate the changes in tensile strength with clay content. Unlike the tensile modulus, nanocomposite of both clay content shows lower tensile strength than that of pure epoxy which are in accordance with research carried out by by *Yasmin et al. [13] and Zerda et al.[14].*
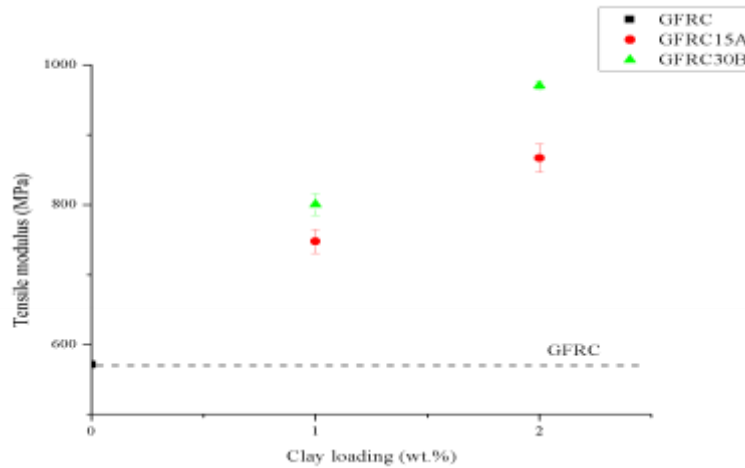

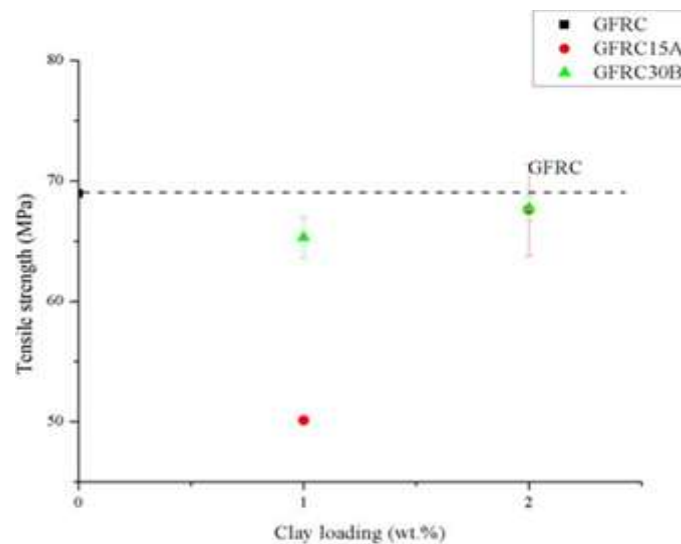
Fig.3.4a Tensile modulus test results

Fig.3.4b Tensile strength test results

The failure of nanocomposite at strengths lower than pure epoxy might be thought to be process related. The aggravating of nanoclay in an epoxy matrix with homogenizer and ultrasonication produced viscous and foamy material and made degassing quite difficult. This in turn may leave some voids inside the nanocomposite. It might be thought that under tensile loading cracks can initiate from these tiny voids and cause specimens failure.

5.3   Scanning electron microscope (SEM)

Samples were mounted onto sample holder and fixed with adhesive carbon tape. Fig. 3.5 and Fig. 3.6 illustrates the top surface morphology of nanoclay composite flat sheet. Voids are observed in Fig. 3.5a. Non circular bright regions shows correspond to clay agglomerates from are observed from SEM micrograph. It is observed clay particles are dispersed in the epoxy matrix and nanoclay is also distributed in the outside zone of glass fiber bundles in which epoxy resin flows.
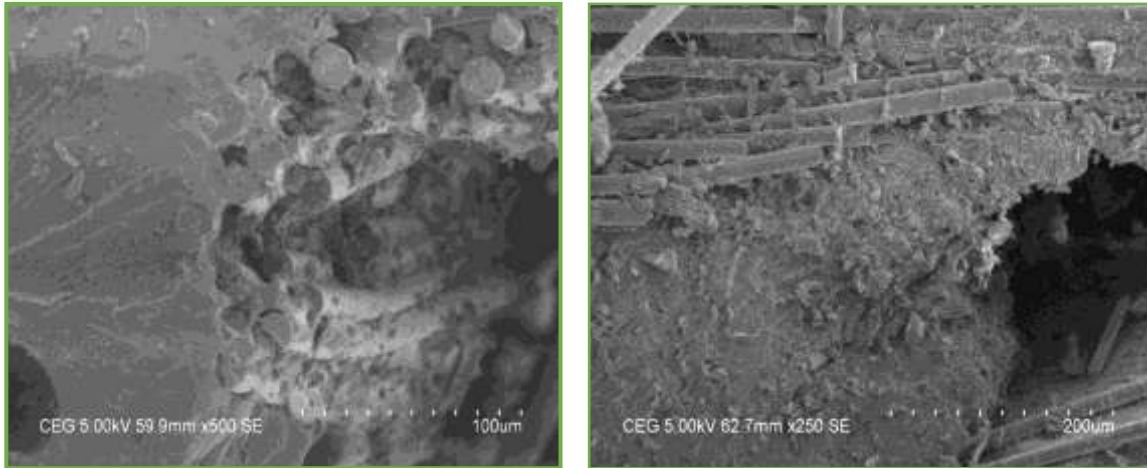

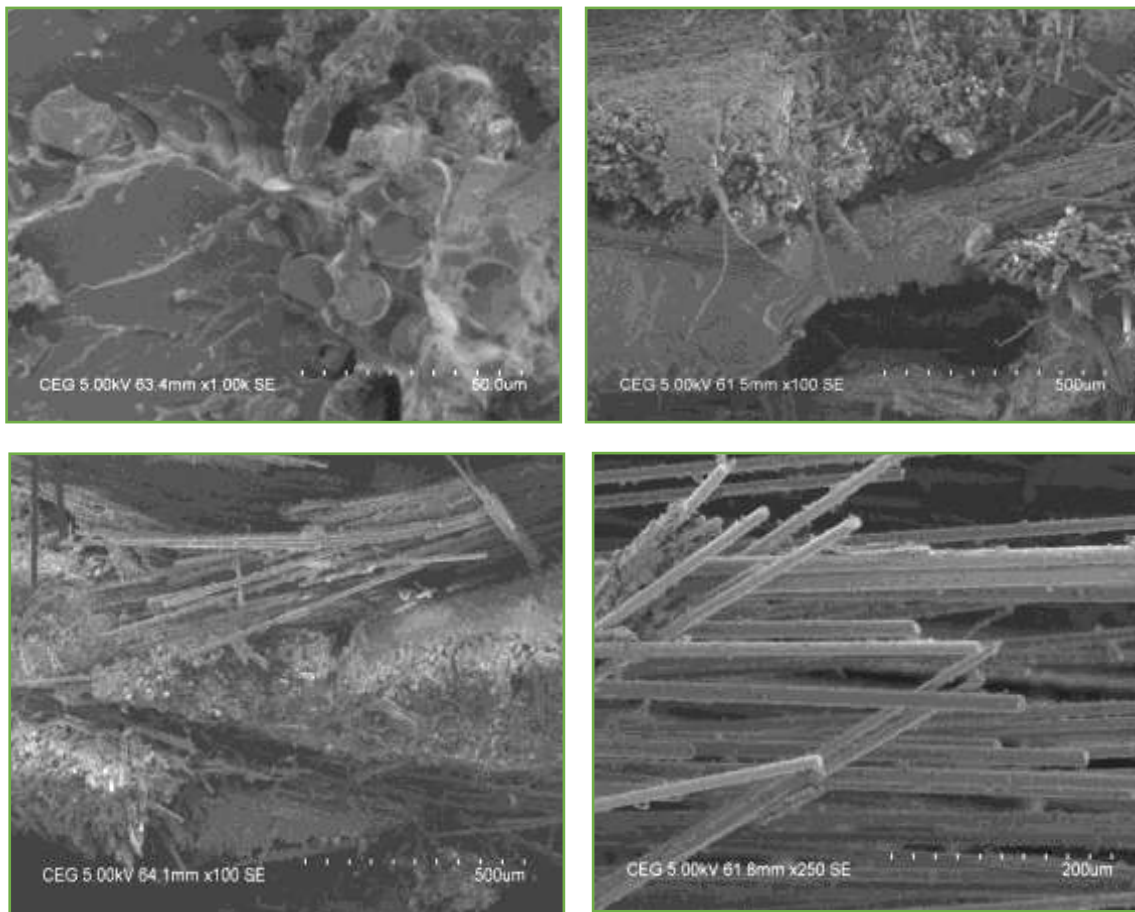Fig. 3.5 SEM image of specimens having nanoclay Cloisite 15A® (a) 1 wt.% and (b) 2 wt.%

Fig. 3.7 SEM images of (a) 1 wt.% nanoclay Cloisite 30B® and (b) 1 wt.% nanoclay Cloisite15A®((c)) 1 wt.% nanoclay Cloisite 30B® / Cloisite 15A® (d) 0 wt.%

6.1 Conclusion

In this work the E glass fiber reinforced epoxy-nanoclay composites are manufactured using vacuum assisted resin infusion moulding. Addition of nanoclays resulted into improvement in mechanical properties of fiber reinforced composites (microhardness, flexural strength, tensile modulus). 2 wt.% Cloisite 30B® nanoclay reinforced specimens showed the best combination of properties. The apparent lower or no enhancement in tensile strength of glass fiber reinforced nanocomposite over glass fiber reinforced composite can be attributed to clustering of nanoclay or to occasional occurrence of micro size voids in microstructure, especially at the interphase of nanoclay and matrix.

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