Experimental analysis and Degradation and of C-Type GFRP (Glass Fibre Reinforced polymer)

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Abstract: Nanotechnology involves the control and manipulation of materials at the nanoscale. As the surface area of a particle increases, creating more sites for bonding, catalysis or reaction with surrounding materials, resulting in improved properties such as increased strength or chemical or heat resistance. In the present work, epoxy modified with nanoclay cloisite 30B (having concentration 0.5 wt %, 2 wt %, 3 wt % and 4 wt % of epoxy) is reinforced with C-glass chopped strand fiber and Eglass unidirectional fiber to manufacture interply laminates. X-ray diffraction conducted on epoxy clay nanocomposites indicates exfoliation of nanoclay at all level of nanoclay loading. The effect of varying the nanoclay concentration is observed by carrying out tensile, 3-point bending strength test

1. Introduction

This paper introduces the particular type of glass fibre C-Type whose degradation has been done experimentally which involves understanding concept of Fiber-reinforced polymer is a composite material made of a polymer matrix reinforced with fibers [Zafar A, Bertocco F (2012)] The fibers are usually fiber glass, carbon or aramid, while the polymer is usually an epoxy, vinyl ester or polyester thermosetting plastic. They extensively find their applications in industries of manufacturing, automatic controlled and using aero dynamics [Hossain M. K., Imran K. A.(2011)]. They are also used for structure designing due to its inherent structure strength; as it is able to maintain its original length after excessive load testing without inhibit failure characteristics. In order to check its properties, test has been conducted and observed that when specimen has put under axially load, stress finds its direct relation with strain and retains without deformation and possess linear characteristics i.e. elastic behaviour [Zainuddin S, Hosur M.V, Zhou Y(2010)]. During compression, it has been seen that the dependence of response of reinforced fibres is on proposition, interface parameters and capacity of mixture relatively. Even though, the response of fibre varying with elastic strength is bad, but proposed fibre with resin shows good interface

and hardness tests. Test result shows that tensile strength increases as the clay concentration varies from 0 wt % to 3 wt %. Tensile strength is maximum at 3 wt % and further increase in clay concentration shows decrease in tensile strength. Flexural strength increases as the clay concentration varies from 0 wt % to 4 wt %. Micro hardness increases as clay concentration varies from 0 wt % to 2 wt % and then decrease at further loading. Further hygrothermal studies on nanocomposites have been performed in water and NaOH baths. During exposure it is observed that the properties degradation in NaOH is more as compared to water.

Keywords : Nanoclay, C-Type GFRP (Glass Fibre Reinforced Polymer), Microhardness, Nanocomposites.

strength and interaction among the materials are also relatively good [Manjunatha C.M, Taylor A.C (2009)].

Advantages of fiber reinforced polymers

- 1. Very High value of SWR [Quaresimin M., Varley R. J, (2007)].
- 2. High reverence against corrosion. [Berketis K, Tzetzis D, Hogg P.J. (2007)]
- 3. Application based shape can be achieved.
- 4. Very less Gestation cost relatively [Wetzel B, Rosso P. (2006)].
- 5. Response for changing a material is good.
- 6. Low cost of manufacturing.
- 1.1 Types of fiber
- 1.1.1 Glass fiber : The said fiber is composed of very finely placed fibre of Glass and dominant in sales. Glass fiber was invented in 1938 by Russell Games Slayter of Owens-Corning considering for better insulation. [Yasmin A, Luo J.J. (2006)]

Figure – 1 Commercially available glass fibers $[12]$ There are various types of Glass fibres especially E- Glass, S-Glass, R-Glass. But based upon required of application, these composite fibres are use. Likewise E- Glass is used for insulation and mechanically strong where as S- Glass use for high strength but relatively costly. E- Glass is used extensively used in large applications possessive high strength mechanically and good insulation for electrical use. Whenever, there is a requirement of fatigue and chemical overpowered, R-Glass is preferred [Avila A, Almir S (2006)] The typical fiber diameter for glass fiber is 9-17 μm and the specific gravity is about 2.5. The tensile strength of glass fiber is in the order of 2000-4800 MPa and the elastic modulus lies in range of 50-90 GPa, which is of high value. [Lei Wang, Ke Wang (2006)]. 1.1.2 Types of Glass Fibers :-

a) E-glass (electrical) – It is the most commonly used fiber in conjunction with matrix propositions. Its mechanical strength is higher and having lowest basic content among other classes of Fibres. It is available at low cost with relatively poor resistance (in a value of few). Good tensile and compressive strength and stiffness, good electrical properties and relatively low cost, but impact resistance relatively poor. E-glass is the most common form of reinforcing fibre used in polymer matrix

composites[Avila A, Horacio V(2005)].

b) C-glass (chemical) – It is not reactive to chemical compositions. In order to use for water pipes and tanks, they are used for outside layer in very common form of surface tissues[Chow W, Bakar A (2005)]

c) T-glass- Their conventional glass fibres have god strength and chemical inactive properties but this class of glass fibres are developed for aero dynamic applications and for military purposes. They have high value of modulus and having good resistance against wet conditions. They are used with lesser diameter in order to have high shear strength in inter laminar area. The only drawback for this class of fibre is their high price used for these types of applications [Lin Li-Yu, Lee Joong-Hee (2005)]

2. Research problem

The present study has mainly focused upon the synthesis of epoxy layered silicate nano composite as matrix in fiber reinforced composites. In this study, mechanical properties and thermal stability of these FRP laminated nanocomposites has been studied. Also hygrothermal aging studies on these FRPs have been carried out.

3. Experimentation

3.0 Fabrication of specimen

3.1 Materials

Unidirectional E-glass fiber, C-glass fiber and M Brace a two part epoxy resin purchased from BASF Construction Chemicals (India) Private Limited. Organically modified nanoclay Closite 30B purchased from Connell Bros. Mumbai.

3.2 Specimen specifications

Commercially available E-glass fiber mat and chopped strand C-glass fiber had been used for making specimen. The specimen had been cut and prepared as per the assumed dimensions for tensile and bending tests respectively. The assumed specifications of specimens are shown below:

3.3 Specimen dimensions

(a) For bending test

Figure -3 Specimen dimensions for bending test (b) For tensile test

Figure – 4 Specimen dimensions for tensile test

3.4 Cutting glass fiber sheet

For the experimentation, unidirectional E-glass fiber roll having 50cm width having 0^0 fiber orientation woven with polymer fibers and C-glass chopped strand fiber purchased. The sheets were initially cut a square of length 50cm.

Figure -5 Uncoated E glass fiber mat used for making specimen

Figure -6 Uncoated C- glass fiber mat used for making specimen

3.5 Mixing of nanoclay into epoxy (base)

Epoxy base is a blue colour thick fluid. It is quite difficult to mix nano clay into it manually. So we used a mechanical stirrer and an oil bath for proper mixing of nanoclay figure $-5 & 6$. Oil bath was used to heat up the epoxy to desired $(60^{\circ}C)$ temperature for reducing the viscosity of epoxy base. Proper mechanical stirring of epoxy at this stage resulted better dispersion of clay. Different percentage of clay-.5%, 2%, 3% and 4% by weight were added and stirred at 60° C for 2 hours.

Figure -7 Oil bath setup with mechanical stirrer

Ultrasonication after mechanical stirring: Sonicator is being used for using sound energy to agitate the particles with ultrasonic bath or an ultrasonic probe in a process called sonication. This process is required to break interactions between molecular strands and speed up the process of dissolution. Sonication was done for evenly dispersing nanoparticles in liquids. After mechanical stirring of the epoxy solution container was placed into the Ultrasonication bath for up to 2 hours.

Figure – 8 Ultrasonication bath

3.6 Mixing of epoxy base solution with hardener After Ultrasonication the solution is mixed with the hardener in the ratio 5:2 by volume. After mixing mechanical stirring up to 5 to 10 minutes was done. The whole procedure is shown in Figure – 7 & 8.

Figure – 9 mixing of hardener to the base component 3.7 Coating of nanoclay mixed epoxy to glass fiber sheets The mixture was then poured on to the glass fiber mat and applied uniformly using the hand layup method asshown in figure $-10 \& 11$. For this, steel scraper was used to maintain uniformity of the solution. During the process, a special precaution has been taken to make ensure that no bubbles get entrapped; otherwise there would be a porous. After applying epoxy on the both sides of first glass fiber, second fiber sheet was applied on first fiber sheet with and epoxy was applied on second fiber sheet. Finally third fiber sheet was placed on second fiber sheet and epoxy was applied on third fiber sheet. The sheet took overnight to dry. The full curing of sheet was done keeping in mind the temperature value which should be ambient and kept for a week for further process.

Figure – 10 Coating the glass fiber sheet with epoxy solution

Figure – 11 Coated sheets placed for curing

3.8. Cutting of Sheet for Samples

Once the epoxy was fully cured, cut the sheet to actual sample size using the marble cutter shown in figure - 12.

Figure – 12 Marble cutter

3.9 Experimental Set-up

For evaluating performance of glass fiber reinforced polymer (GFRP), they embedded with epoxy matrix and various aging tests have been conducted. The field environment very similar to that of tropical climate had been simulated. The specimens were immersed in two water baths for 30 days.

Figure -13 Setup view of the water baths

After 30 days, the specimens were removed; the tensile and flexural strength was measured to check the degradation in properties of composite material. Both of the water tanks were filled with water. One was of simple water and other tank was containing NaOH 5% by weight of water. Both the tanks were kept at a temperature of 45° C.

3.10 Setup Fabrication:

3.11 Water tanks

The set up involves tanks with insulation and material used for manufacturing is plastic. The approximate capacity of the tank was 60 litres. Both the tanks were filled totally with tap water and set at a temperature of 45°C. NaOH was added to one tank (5% by weight of water). The water which evaporated from the tank was replenished on daily basis during experimentation. Each tank was labelled as per details of experimentation.

3.12 Heating element

The setup in figure -14 was heated with help of commercially available heating rod elements . Each bath was having its own heating rod connected via temperature controller in figure – 15(a) & 15(b). The wattage of rod was 1000KW with single phase connection. As the temperature reached the required value the power supply of rods were cut off by controllers.

3.13 Temperature controller

The objective of this set up was to maintain the bath temperature at specified value till the duration of experiment for day and night on daily basis. So a temperature controller in Figure – 15(a) was connected with each of the bath along with relays cut off. The controller used the proportional-integralderivative (PID) control to maintain the temperature. On the controller display the "Set Value" was given which was the temperature indicated in green and the "Process Value" of temperature was indicated in the red (refer Figure – 15(b), which was the output from the RTD sensor. For the very first time the controller was set to auto-tune mode so that it could adjust itself according to the input variables. Once the bath had attained the set value the controller cut off its supply and after sometime it sensed the temperature if it had gone below set value, it again started heating to obtain the set value.

Figure - 15 (a) Temperature controller (b) Temperature display panel

3.14 Testing methods used in experimentation Tensile testing

A Universal Tensile testing machine shown in Figure 16(a) and Figure 16(b) was used for the testing of the FRP specimen for its tensile strength. The test specimen had been prepared according to assumed dimensions. The specimen 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.

The test specimen had been prepared according to assumed dimensions. The three point bending test results can be taken as indications of strength degradation of composites after they had been hygrothermally treated.

Micro hardness test

Micro hardness test (shown in Figure - 19) was conducted on specimen with different clay loadings to see the effect of clay loading on hardness values.

Figure – 19 Micro hardness equipment

The load applied was 50gm and VHN values were determined by applying this load by using a calibration distance of 50 units in Quantinet software as shown in Fig.1.18 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 figure below.

X-Ray diffraction test

The structure of thin films and materials are somehow complex, and for understanding, different techniques used for revealing information about structure in crystal domain, values of components existing in a material, and physical properties. These techniques and non destructive and used for analysis and primary used for identification of type of structure and inherent characteristics of a material. The basic principle of a technique lies in a fact that when a beam hit any of the object, then incident ray is scattered and due to polarized wave although we can measure wavelength and energy.

Whenever, composition is complex, like in nanocomposites ; diffraction technique is used. XRD will enable the changes that occur to the clay due to the intercalation and/or exfoliation of the epoxy into the clay galleries to be quantified. The dspacing of the intergallery spacing can be determined using Bragg's Law:

$λ = 2$ dsinθ

Where λ is the wavelength of the incidence x-ray source, d is the spacing in question, θ is $\frac{1}{2}$ of 2 θ the Bragg angle or the diffracted angle of the incidence x-ray beam. Below is a schematic of the previously mentioned Bragg's Law (Figure – 20).

Figure – 20 Schematic representation of x-ray diffraction principle and the Bragg's Law

To evaluate the degree of exfoliation in the polymer, XRD measurements were carried out in a Panalytical X-ray diffractometer with Cu Ka radiation $(\lambda=1.54\text{\AA})$ with a scanning speed of 10/min and at 45 kV and 40mA. During the XRD experiments, the samples were analyzed in reflection mode. All XRD scans were through 2 θ of 5⁰ to 15⁰.

3.15 Test matrices

 $Table - 1.3$ specimen details

Specimen	No. of specimens		
Name			Total
	Tensile	bending	specimens
$0 w t$ %	3	3	6
$0.5~wt\%$	\mathcal{R}	3	6
$2 wt\%$	\mathcal{R}	3	6
$3 wt\%$	\mathcal{R}	3	6
$4 wt\%$	3	3	6
Total specimens			30

Table – 1.5 Specimen for accelerated degradation in 45° c NaOH solution (5% by weight of water) Specimen name No. of Total specimens Te bend

4. RESULTS AND DISCUSSIONS

4.1 Microscopic behaviour checking with specimen for micro hardness

Figure – 21 showing different loading points in specimen The micro-hardness of specimen manufactured at different clay loading was measured. The table 1.6 shows the experimental observations of the nanocomposites with different nanoclay contents. An average hardness was calculated by taking measurements at 3 points in each specimen. Figure 21 showed the results of Vickers hardness plotted against nanoclay loading.The maximum hardness had been measured where the nanoclay content reached 2 wt %. A decline of the hardness also appears on further increasing the nanoclay content. The hardness decreased in a significant manner from 7.535 HV (2 wt% of nanoclay) to 5.282 HV (3 wt% of nanoclay). Thus, adding a small amount of nanoclays into polymer-based materials could potentially enhance hardness of the material with the nanoclay content less than 5 wt%. However, it was also reasonable to believe that it should have an optimal limit depending on choice of constituent material and processing conditions .It was suspected that the nanoclays might retard the chemical reaction, and so cause incomplete curing process of the composites. For all specimens with high nanoclay content, the matrix might not be fully cured.

4.2 X-ray Diffraction Test

The X-ray Diffraction experiments were conducted on the samples having different nanoclay loading. X-ray diffractometer gives the values of d-spacing and 2θ for different samples of epoxy clay nanocomposites. An increase

Table – 1.6 Micro hardness Values

 $\overline{1}$

Figure – 24 showing XRD result of sample with 4% clay The absence of peaks in diffraction pattern indicates formation of exfoliated nanocomposites at all levels of nanoclay loading 4.3 Tensile Test

Graph – 2.1 The tensile strength of samples as a function of weight percentage of nanoclay in epoxy.

The fiber reinforced epoxy nanocomposites sample having 3wt% nanoclay loading shows the highest value of tensile stength which is 40% higher than neat epoxy glass fiber reinforced composite. When the movement of molecules is hindered, then hardness of a system increases; which is self explanatory with addition o nanoclay which hinders the movement of molecules. A significant decrease is seen in strength value at clay loading of 4wt%.The variation of ultimate tensile strength as a function of weight percentage of nanoclay in epoxy from 0wt% to 4wt% is shown in Graph -2.1. 4.4 Three-point bending test

Table -1.8 Results of samples from three-point bending test

Graph – 2.2 The flexural strength of samples as a function of weight percentage of nanoclay in epoxy.

Clay Loading by with of resin

An increasing trend of flexural strength value is observed with increase in nanoclay loading upto 4wt%. A 52% improvement in strength of specimen with 4wt% clay loading is observed when compared with neat epoxy-glass composites. The flexural strength of samples against the weight percentage of nanoclay in epoxy from 0wt% to 4wt% is shown in Figure - 22

Tensile testing results of the samples simple and hygrothermal loaded

Table – 1.9 Result of Degradation of Nanocomposites NaOH Tank at 45° C using tensile test

					$\frac{0}{0}$
No.	Sapl	Sample	Tensile	Ten	Decreas
of	e	No.	Modulu	sile	e
Days	Nam		S	Stre	in
	e		(MPa)	ngth(M	Strengt
				Pa)	h
$\overline{0}$	0	Sam	368	63.5	
Day	$wt\%$	ple no.	$\overline{0}$		
		Sam	506	100	

Fig 2.3 Decrease in tensile strength after degradation in NaOH

Graph – 2.4 Percentage Decrease in Tensile strength after degradation in NaOH

After 30 days of water immersion of these samples, there is sharp decrease in tensile strength. The percent decrease in tensile strength value of sample having 0.5 wt% of nanoclay loading is highest among other samples when compared with samples without NaOH immersion. Figure 2.3 & 2.4 shows the effect of NaOH immersion of specimen on tensile strength. A decrease in strength of all specimens is observed.

Table – 1.10 Result of degradation of nanocomposites in water tank at 45° C using tensile test

${\bf N}$ o. of D ays	Sam ple Na me	Sam ple No.	Tensi le Modulus (MPa \mathcal{E}	Tensi le Stren gth(MPa)	$\%$ De crease in Str ength
$\overline{0}$	0wt	Sam	3680	63.5	
Day	$\%$	ple no.			
		1			
		Sam	5060	100	
		ple no.			
		$\mathfrak{2}$			
	0.5	Sam	1210	95.5	
	wt%	ple no.			
		1			
		Sam	1400	81.6	
		ple no.			
		$\mathfrak{2}$			
	2wt	Sam	2320	82.6	
	$\%$	ple no.			

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Graph – 2.5 Decrease in tensile strength after degradation in water

Graph – 2.6 Percentage decrease in tensile strength after degradation in water

After 30 days of water immersion of these samples, there is decrease in tensile strength. The percent decrease in tensile strength value of sample having 3 wt% of nanoclay loading is highest among other samples when compared with samples without water immersion. Graph-2.6 shows the effect of water immersion of specimen on tensile strength. A decrease in strength of all specimens is observed 5.2.2 Bending test results of nanocomposites under hygrothermal loading

Table – 1.11 Results of samples for NaOH Bath at 45° C

using three-point bending test					
					$\%$
$\mathbf N$	Sam	Sa	Elast	Flex	Dec
o. of	ple	mple	ic	ural	rease
D	Na	No.	Modulus	Strength	in
ays	me		(GPa	(MPa)	Stre
			⟩		ngth
$\overline{0}$	0wt	Sa	.817	27.3	
Day	$\%$	mple			
		no. 1			
		Sa	.858	36.5	
		mple			
		no. 2			
	0.5	Sa	1.96	33.1	
	wt%	mple			
		no. 1			
		Sa	1.26	35.1	
		mple			
		no. 2			
	2wt	Sa	1.13	43.8	
	$\%$	mple			
		no. 1			
		Sa	.912	33.7	
		mple			
		no. 2			
	3wt	Sa	.740	51.1	
	$\%$	mple			
		no. 1			
		Sa	.962	38.5	
		mple			
		no. 2			
	4wt	Sa	1.35	43.6	
	$\%$	mple			
		no. 1			

Graph – 2.7 Decrease in flexural strength after degradation in NaOH

Graph – 2.8 Percentage decrease in flexural strength after degradation in NaOH

Table – 1.12 Results of Samples from WATER Bath at 45° C using three-point test

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Graph – 2.9 Decrease in flexural strength after degradation in water

Graph – 2.10 Percentage decrease in flexural strength after degradation in water

From tables 1.10 and 1.11, the NaOH immersion of 3 wt% specimen results in 79.58% decrease in flexural strength but the water immersion of 3 wt% specimen results in 8.8% decrease in flexural strength which shows that there is tremendous decrease in flexural strength of samples immersed in NaOH as compared to samples immersed in water.

5. CONCLUSION AND FUTURE SCOPE

Epoxy modified with Closite 30B® has been used for preparing an interply hybrid laminated nanocomposites. In these nanocomposites unidirectional E-glass fiber was sandwiched between chopped strand C-glass fiber. Five different nanoclayepoxy compositions (0.5 wt%, 2 wt%, 3 wt% and 4 wt %) were prepared in order to show the variation in mechanical property and thermal stability. For samples taken under consideration,

different equipments with separate characteristics were chosen to understand the behavioural characteristics and stability of the nanoclay-epoxy samples. For bending test and tensile test a Zwick/Roell universal testing machine was used and for micro hardness Vickers' hardness tester was used, the ultimate tensile strength, flexural strength and Vickers' hardness values were obtained. From the microstructural analysis instruments such as X-Ray Diffractometry (XRD), the positions of the diffraction peaks of the samples were obtained.

From the Vickers' hardness results of the nanoclay-epoxy samples, the 2wt% nanoclay sample was the hardest among all the compositions with the largest HV value of 7.535. Compared with that of pure epoxy sample of HV 4.308, the increment is 43% in Vickers' hardness value. And 3wt% nanoclay sample showed a drop in the hardness with value of HV 5.282. This might be attributed possible aggregation of nanofiller clay at some sites. From the XRD results, the absence of peaks in diffraction pattern indicates formation of exfoliated nanocomposites at all levels of nanoclay loading. From the tensile results, 3wt% nanoclay loading specimen showed the highest value of tensile strength which is 40 % higher than neat epoxy glass fiber reinforced composite. The addition of nanoclay improves the strength of the epoxy samples which shares the stress imposed on the polymer chains. The individual platelets have much higher mechanical properties as compared to polymer matrix. From the flexural results, when compared with neat epoxy-glass composites 52% improvement in strength of specimen with 4wt% clay loading was observed.

The durability at 45° C studies were conducted on nanocomposites by exposing to water and NaOH for a period of 30 days then evaluating the mechanical property degradations. Mechanical properties were found to degrade with increase in time. Degradation occurs faster in NAOH.

7.FUTURE SCOPE

- 1. To see the effect of nanoclay on FRP`s, experiments can be repeated by changing the types of glass fiber.
- 2. The experiments can be performed on polyester as matrix system, since with this matrix the barrier properties of composites can be enhanced.
- 3. For chemical resistance study, base can be changed.
- 4. The duration of current experiments can be increased to see the effect in long term.
- 5. Epoxy of different structure& properties can be used

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