The Effect of Nano TiO2 Filler Weight on the Mechanical Properties of Chopped Strand Mat Reinforced Epoxy Composite

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Abstract

In this study the specimens were prepared using glass fiber chopped strand mat, epoxy resin and Nano Titanium dioxide fillers by the hand layup technique. The Nano titanium dioxide fillers are incorporated in different weight ratios like 1%, 2%, up to 6% in the fiber reinforced epoxy composite. The prepared samples are prepared to the ASTM standards for each test. The mechanical properties like tensile strength, bending strength, impact resistance, and hardness value and vibration analysis were carried out in order to study the behavior of material under various loads.

Keywords: *Nano TiO2, Mechanical properties, Methodology, Epoxy, Composites*

1. Introduction

In the field of engineering the property and characteristic of any materials plays a crucial part. Minimum requirement to build a composite are a couple of different constituents having various properties and the constituents are distinguishable. Here both the main constituents are referred to as matrix and reinforcement. Generally the matrix material has contains a bonding property which supports the reinforcement material. Reinforcement provides strength and stiffness to the material. More often, reinforcement provides hardness strength and stiffness compared to matrix and is in order to be in the kind of fiber. In composite, matrix comes under continuous phase and which is possibly a polymer, ceramic or even metallic in nature. Typically the objective is to blend the materials and also to have the advantages of both single elements. The most common man made composite was the mud combined with straw to create a brick for the objective of construction. Alloys are generally homogeneous but the composites are heterogeneous in character.

The proposed work is to prepare an E-glass fiber reinforced polymer composite with TiO2 Nano powder as filler substance by using hand lay-up method. This can be an innovative category of polymer composite with E- glass fiber reinforcement where Nano TiO2 as a filler. Epoxy based composite with Nano TiO2 powder filled composite will be tested to find the mechanical property of prepared samples. The study on flexural, tensile, density, impact, inter laminar shear strength (ILSS), free vibration test have done on the prepared polymer composites will be conducted. The material is developed for various needful applications in industries, aerospace as well as navel infantry.

2. Materials

There are different methods to prepare composite materials. Some of the common methods are open molding process, bag molding, filament winding, pultrusion, injection molding, blow molding etc. Hand layup is a type open molding process, which is the simple and easiest method to prepare composites.

The material and methodology of preparing the composite is explained in this chapter. Different combination of specimens are prepared and cut in to the ASTM standards.

2.1. Materials required

Materials required are as follows.

1. Chopped strand mat (CSM) glass fiber
2. Epoxy with hardener
3. Nano titanium dioxide (Tio2) filler
4. Poly vinyl alcohol (PVA) mold releasing agent

2.1.1. Chopped strand mat (CSM) E-Glass Fiber: Chopped Strand Mat is a non-woven reinforced material. It is manufactured by spreading continuous filament roving of 50mm in length, distributed it at random uniformly held together with powder or emulsion binder. Depending on the different composition of binder, chopped strand mat is divided into powder chopped strand mat and emulsion chopped strand mat, which is used extensively for hand lay-up process, and secondly for the winding, molding, continuous system board and other technology.

Table 1. Properties of glass fiber

Properties	Glass fiber
Type	Chopped strand mat
GSM	300
Colour	White
Density	2.55 g/cc

2.1.2. Epoxy matrix: SYNPOL 450XX / hardener SYNPOL 140 epoxy resins are reaction products of Bisphenol-A and Epichlorohydrin. The resin containing average two epoxy groups per molecule that can react with curing agent to produce cross linked polymer. Epoxy systems are often chosen against other systems when chemical resistance is an important factor. Cured epoxy system will perform excellent depending on the curing agent, resin diluents, and additives chosen.

Table 2. Properties of Epoxy

Properties	SYNPOL 450XX	SYNPOL 140	Units
Viscosity at 250 C	45-55	50	poise
Density at 250 C	1.07	1.07	g/cc
Solvent	xylene	-	-
% of non-volatiles	75	100	%

2.1.2. Filler material: In this present Nano Titanium dioxide of 15nm produced by The Kerala Minerals and Metals Ltd (KMML) is used as filler material. Titanium dioxide (TiO2), also referred to as Titanium, is a substance as old as the earth itself. Titanium dioxide is readily mined in its purest form from beach sand.

Raw Ilmenite is chemically processed to remove impurities such as iron, leaving the pure, white pigment available for use. Manufacturing of Titanium dioxide is done by chloride process. The chloride process produces TiO2 products by reacting titanium ores with chlorine gas. The chlorides of impurity metals are removed from Titanium Tetra Chloride (TiCl4) through various processes to complete the recovery of TiCl4. It is further purified by distillation to obtain pure Titanium Tetra Chloride in the liquid form. Titanium Tetra Chloride is vaporized, pre heated and oxidized with oxygen to produce raw Titanium Dioxide at a high temperature. The raw Titanium Dioxide is then classified and surface treated with various chemicals, filtered and washed to remove the salts. The application of TiO2 are in different fields like paints, printing ink, plastic, paper, rubber, ceramic industry etc.

Table 3. Properties of Titanium Oxide

Properties	Nano TiO2
Nano titanium dioxide %	97
pH	7
particle size	15 nm
Moisture %	1.75-2
density	0.5 g/cc

2.1.4. Poly vinyl alcohol: The Release Agent PVA has two solvents, methylated spirits and water. This solution is a film-forming agent, which upon drying will form a release film. PVA is used as an additional aid for release with mold release waxes in the situations like release of fiberglass molds from plugs or patterns under low temperature conditions or where there is concern of under cure in the plug. Here in this experiment PVA is used to reduce the defect and to get required dimensional accuracy for the specimen.

3. Methodology

The project aims to analyze the properties of prepared composite that is the composite produced with chopped strand mat glass fiber reinforced with epoxy matrix and Nano TiO2 as Filler with varying weight %. Thus seven different composite laminates are produced by altering weight % of Nano TiO2 filler.

3.1. Sample preparation

Using raw materials such as fiber, matrix and filler materials, the required samples are manufactured. According to the volume fraction of fiber, resin and filler materials are selected. By using mould component, laminates are prepared. The different variety with different composition of the six samples tabulated in Table 4.

Table 4. Filler Percentage in Specimen

Specimen	Specification
A	0 % filler
B	1 % Nano TiO2 filler
C	2 % Nano TiO2 filler
D	3 % Nano TiO2 filler
E	4 % Nano TiO2 filler
F	5 % Nano TiO2 filler
G	6 % Nano TiO2 filler

Calculation of constituents used for preparing the samples

Table 3. Constituents used for preparing the samples

Percentage	Volume of TiO2	Mass of TiO2	Volume of matrix	Mass of reinforcement	Mass of matrix	Number of layers
%	mm^3	grams	mm^3	gram	grams	Numbers
0	0	0	250000	382.5	107	15.94
1	2500	1.25	247500	378.675	105.93	15.78
2	5000	2.5	245000	374.85	104.86	15.62
3	7500	3.75	242500	371.025	103.79	15.46
4	10000	5	240000	367.2	102.72	15.3
5	12500	6.25	237500	363.375	101.65	15.14
6	15000	7.5	235000	359.55	100.58	14.98

3.2. Setup used for preparing components

Hand layup technique is the easy way to prepare the laminate in the present study. An open contact mould with one side open, by this it is easy to produce high strength with low or medium volume composite. The die consists of four holes which are used to reliving the air while compressing. The press used is a form of hydraulic type which is commonly used in automobile lifting purpose. So the seating arrangement is done with the help of mild steel plate of 5mm thickness. At the top of the lifting hub one plate is attached which helps to the proper seating of female dies. In the setup the male die is attached at top portion of the setup normally facing downwards with the help of 10mm steel bolts. Once the setup completes the female die of 250x250x30 mm is placed on the mild steel plate fixed on the press.

3.3. Steps involved in fabrication of composite

Step1: Primary set up
Here the initial preparation has to be made so that once the layup started it should finish within 15 minutes. So arrange all the requirements like weighing of filler, matrix and countdown of layers etc. before lay up of matrix check whether the die is neat or the holes are properly cleaned.

Step2: Glass fiber (BD Glass) cutting
After adding hardener the matrix cures faster; due to this cutting of glass fiber must done before starting of layup. So that it is very easy to arrange once the cutting done, by this we can get a good composite specimen.

Step3: Surface preparation
After cutting of glass fiber before placing it into the mould we need to ensure about whether the surface of the die is clean, even at all the edges. After that apply PVA to male and female part of the die and place polythene sheet over it.

Step4: Filler and epoxy mixing for surface preparation
Mix the percentage of filler material to the epoxy resin with proper mixing. Mechanical stirrer is used for proper mixing if filler with matrix, minimum 15 min to stir. Once the filler mix with resin add percentage of hardener mix well and start layup.

Step5: Fiber arrangement
Once complete all the primary setup place one layer of fiber cloth and it should be aligned or parallel to the surface of the mould. Make sure that the fiber threads should not come out to left or right.

Step 6: Matrix lay up
Once aligning fiber pours the matrix material on first layer of fiber so that the layer should get wetted. After completion of first layer move on to second layer and continue until the sixteenth layer to complete.

Step 7: General inspection
After completing the sixteenth layer make sure that the matrix to be distributed uniformly and add some quantity at the corner of the laminates. Once completing all the layup procedure add a polythene sheet over the laminate and place the total die on the surface plate of the jack.

Step 8: Squeezing
Once the layup is completed the female die is to be placed on the squeeze setup. Make sure that the squeeze lock nut to be well tightens. Now using a lever, pump the jack so that the female die should sit properly on male die and allow it to cure for one day that means 24 hours.

Step 9: Removal of composite board
Finally the cured composite board is removed out from the mould box. The steps listed are repeated in the preparation of all the specimen plates, by varying the % of Nano TiO2 filler.

4. Sample preparation by cutting fabricated plates

The samples prepared are cut according to ASTM standard. At initially the dimensions are marked on the fabricated plate so that it is easy to get a required shape. The plates are cut using wire cutting machine to conduct flexural, tensile, impact, density and vibration experiments.

The machine is a type of reciprocating and it cuts the material as the feed given by manually. As wire passes the composite is hold properly with both the hands so that the required shape can be achieved.

5. Results

Composite laminate with 0, 1, 2,3,4,5 & 6 weight percentage of Nano TiO2 of size 15nm has been prepared. As per the ASTM standards the mechanical and vibration characterization are done. The results obtained from various tests are tabulated and variations of properties are mentioned by plotting graphs.

5.1. Density of the Composite

The densities of prepared samples are calculated as per the mass by volume method. The density of samples with different filler percentage are shown in figure 1.

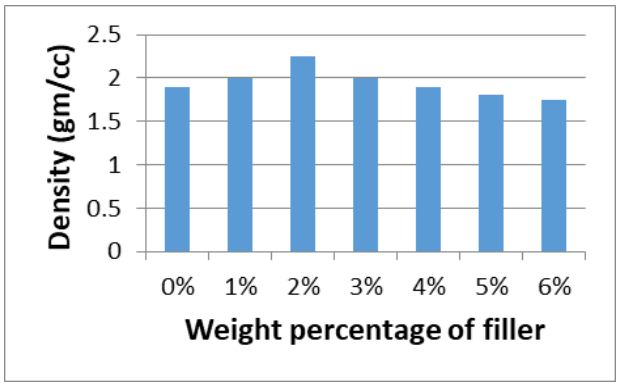

Figure 1. Density of samples at different filler percentages

The addition of nano TiO2 filler led to increase in the density up to up to 2 wt % of Tio2 filled epoxy composite and then gradually the density decreases with increase in the filler percentage. This decrease in density may due to the presence of some internal void development during the time of pouring and variation of filler percentage may the reason for variation in the density for various samples.

5.2. Impact Test

The impact test is carried out as per ASTM D256-56 on each samples and its value is shown in the figure 2

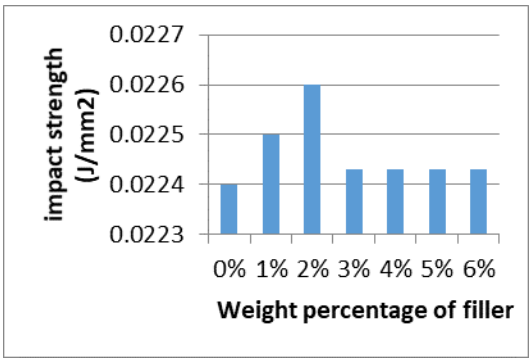

Figure 2. The impact strength for the samples with different filler combination.

From the graph it shows that sample filled with 2 percentage Nano TiO2 filler have the high impact strength compared to other specimens. But further increase in filler percentage not improves the impact strength value. This may due to increased brittleness formed by voids present in the material.

5.3. Hardness Test

Hardness test was conducted by using Rockwell's hardness test. The figure 5.3 graphically indicates the hardness of various samples filled with different weight percentage of nano TiO2 filler.

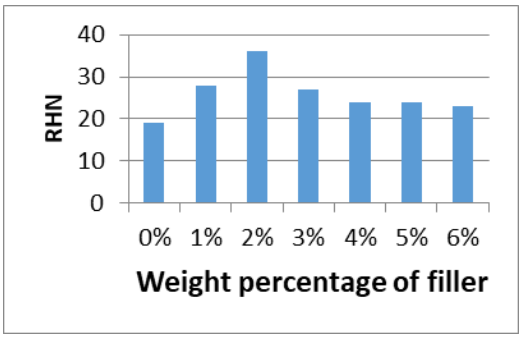

Figure 3. Rockwell hardness number at different filler percentages

It can be see that the hardness of TiO2 filled composite shows better hardness compared to pure epoxy composite. The samples 2 wt % nano TiO2 filled composite shows maximum hardness number and then further increase in filler percentage reduces the hardness gradually. The decrease in hardness may due to porosity in the samples.

5.4. Flexural Test (3 Point Bending)

According to ASTM D2344/D2344M the flexural test for samples with different filler percentages are conducted. The result of flexural strength is shown in the figure 4.

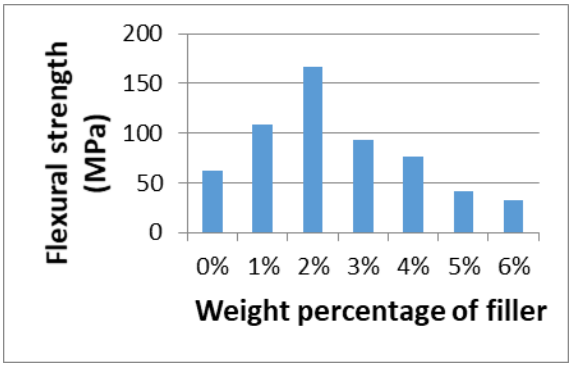

Figure 4. Flexural strength of specimens at different filler percentages

From this results we can observe that flexura;l strength and flexural modulus is maximum for 2 wt % nano TiO2 filled composite. It is clear that TiO2 filled composite have higher flexural strength than pure epoxy composite. But while icrease in the filler percentage the filler percentage the flexural strength and flexural modulus decreses linearly, it may due to poor adhesiveness of epoxy while adding more TiO2

5.5. Inter Laminar Shear Strength (ILSS)

The inter-laminar shear strength for the composite laminate for different variation is carried as per ASTM 2344. The variation of ILSS is based on maximum bending load.
The chart 5 shows ILSS against the weight percentage of filler.

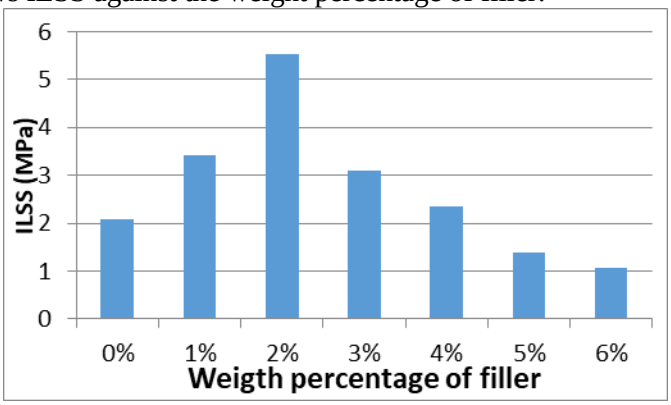

Figure 5. ILSS against the weight percentage of filler

From the figure we can observe that the addition of filler increases up to a certain limit and then decreases gradually when adding more percentage of filler. From this result we can we can say that up to 2 wt % filled composite the bonding is strong and further increase in filler reduces the strength of bonding of constituents in the prepared composite.

5.6. Tensile test

The tensile test is conducted for each sample as per ASTM D3039/ D3039M-08, by considering these standards the gauge length and load is calculated for testing the specimens.

The ultimate tensile strength of the composite specimen is shown in figure 6

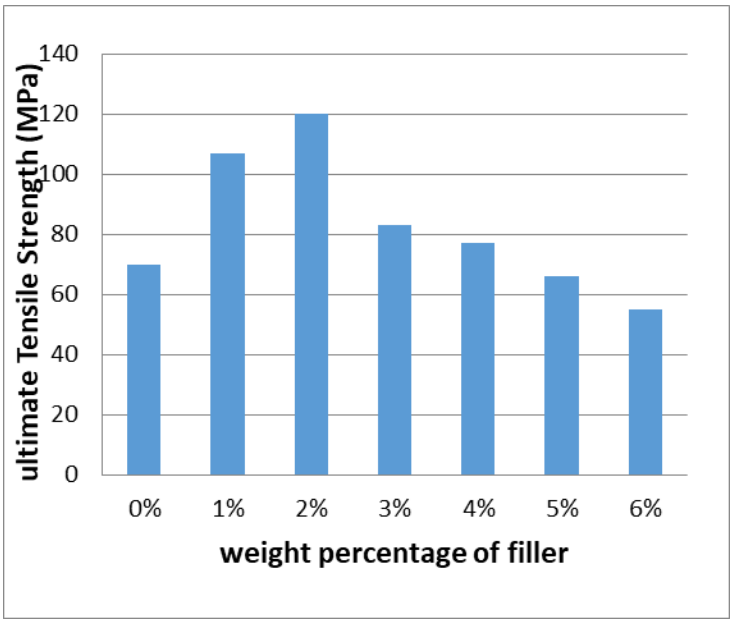

Figure 6. Ultimate tensile strength v/s Filler percentage

6. Conclusions

Present work details about the fabrication and characteristic of a new material of chopped strand mat E glass fiber reinforced epoxy composite filled with titanium dioxide of 15nm size followed by hand layup technique. The physical and mechanical characteristics of prepared specimens were analyzed.

•The density value of sample varied according to the increase in filler percentage.

•It is observed that the flexural and the inner laminar shear strength is increased by the addition of 2% of Nano titanium dioxide filler

•The impact, hardness and tensile strength of the specimen are improved while adding Nano titanium dioxide compared to the pure epoxy composite. So it can be conclude that the presence of filler of certain amount is giving better results.

•In the vibration study it is observed that natural frequency of the specimen increase with the decrease in specimen length.

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