



Mechanical and thermal behavior of hybrid nano composite

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Received 16 July 2013; Accepted 21 July 2013

Abstract

The paper focused on the study to improve the mechanical and thermal stability of the composite material by adding fibre reinforced plastic and iron oxide nano fillers. The paper also on study of Nanocomposite material microstructures to find the effective characterization on oxide Nano fillers with epoxy resin and fibre reinforced plastic. Iron oxide nano particles were added in order to increase the strength without affecting the weight of the composite material which has been manufactured with epoxy resin and glass fibre. Varying the percentage of nano filler with epoxy resin we can modify the tensile strength and thermal stability of the composite body for our desire. Composite fabrication deals with two categories in making comparison process. The ordinary resin coated fibre reinforced plastic composite are compared with Nano added resin coated fibre reinforced plastic composite. The significant effect of iron oxide variation will be shown by the tensile and thermal stability test.

Keywords: DMA; mechanical properties; thermal Properties; composite materials, Scanning Electron Microscopy.

PACS: 83.85.Vb, 87.19.R- , 65.80.-g, 78.67.Sc, 68.37.Hk.

1. Introduction

Nanotechnology enable the creation of many new materials and devices with a vast range of applications, such as in medicine, electronics, biomaterials and energy production aviation departments and automotive sectors. The iron oxide nano fillers have wide range application when it is reinforced with the FRP inorganic core. Since the high corrosive resistance of the material, it can be used in marine parts when they subjected high salt bath conditions [1].

The composite material is having less weight and good strength could be used in infrastructure developing areas. Adequate high tensile strength offer the material could be utilized in auto mobile body parts.

The high thermal stability causes material to with stand high temperature above 350°C so it could be implemented as an insulator for the electrical and electronic devices, high heat produced areas such as power plants. The material is also can be used in wind mill blades because of its less weight [2].

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2. Experimental

2.1. Material

The polymeric matrix used was an epoxy resin, 520 f, Elantas beck India ltd. beck house, which is a mixture of 55 wt% bisphenol-A with an average molecule weight of 970 g mol⁻¹ and 45 wt% Epichlorohydrin molecules monomers. The liquid resin has a viscosity of 900 centipoises (Cps) at room temperature. Ferric oxide (iron III oxide), as a Nanoparticles with an average diameter of 50nm and a specific surface area of 44m² were functionalized and used as an Nano filler for the Nanocomposite fabrication [3].

The nano filler was purchased from sigma-Aldrich chemical company. Hardener 758 (curing catalyst or initiator, was purchased from Elantas beck India. Ltd. 3-Trimethoxysilypropyl methacrylate (MPS), Tetrahydrofuran (THF), were purchased from Sigma-Aldrich Chemical Company. All the chemicals were used as received without further treatment.

The glass fibre composite cloth has been purchased from Hi-Tech Insulation Company with an average glass particle of 98% and 440 mesh of particle size all the above materials are used without any further modification or reactions [4].

2.2 Curing Process

Ferric oxide Nanoparticles of 3 wt % were dispersed into 30ml resin. The dispersion was carried out in an ice–water ultrasonic bath for about 1 hour. The Nanoparticles/resin solution was placed into an 85°C oven for 15 minutes under vacuum to remove gases and ensure good dispersion quality.

The Nanoparticle/resin solution was ultrasonically stirred in an ice–water ultrasonic bath until the temperature cooled. Then 2.0wt% catalysts (initiator) were added into the Nanoparticles/resin solution, which was stirred and degassed for 2 minutes. The mixed viscous solution was applied on a wax coated glass layer for layer formation with the glass fiber [5]. The number of layers depends on thickness of composite needed. 12 layers of glass fiber used and stick with each other by the hand layer making method used in this research is shown in Fig. 1.

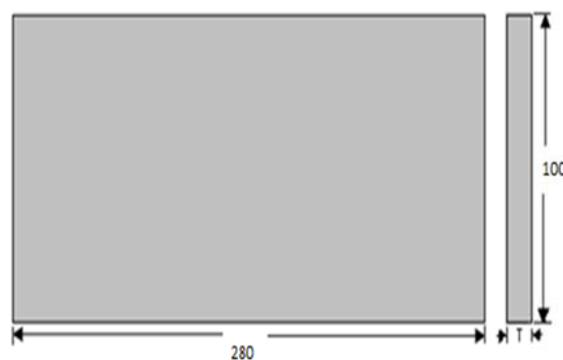


Fig. 1: Sample Hybrid Nano composite.

The curing process was performed at 85 °C for 1 hour and post cured at 120°C inside the vacuum hot oven the resulting composite was allowed to cool to room temperature naturally.

After applying the solution the setup is free for 2-6 hours for curing. Fig. 2 shows the sub layer assembly of the resin coated composite. The curing process should be effective because the resin will react with the fiber reinforced plastic by step by step level alone since we are adding four layer of fiber reinforced plastic.

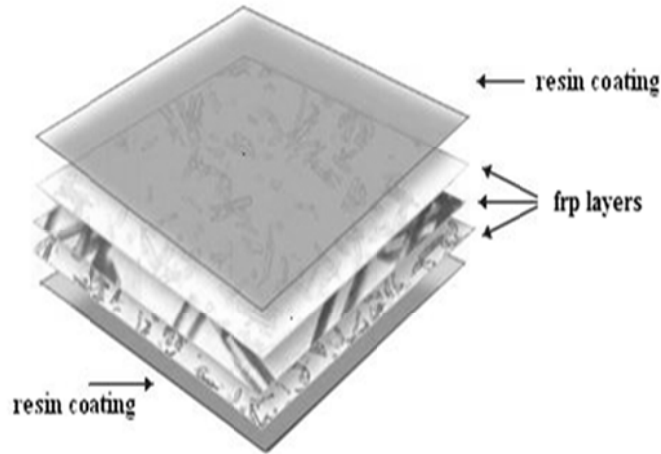


Fig. 2: Sub Layers of Hybrid Composite Body.

2.3 Tensile Testing

Tensile test is a kind of mechanical property that displays the quality of the manufacturing process and determines the further usage of the hybrid composite. For tensile test the special kind of specimens has been manufactured after the thin sheet fabrication to get true value of ultimate tensile strength. The ASTM standards are act as vital role and all the specimens are produced based on ASTM D 638.

Brittle materials, such as concrete and carbon fiber, are characterized by failure at small strains. They often fail while still behaving in a linear elastic manner and thus do not have a defined yield point. Because strains are low, there is negligible difference between the engineering stress and the true stress.

Testing of several identical specimens will result in different failure stresses; this is due to the Weibull Modulus of the brittle material. The ultimate tensile strength is a common engineering parameter when design brittle members, because there is no yield point [6].

The testing was carried out by normal universal testing machine with attached computer programmed software to draw the stress-strain curve based on the inputs and the maximum loaded conditions were found out the values of stress and strains will be plotted separately and the ultimate tensile strength can be found out from those values.

The shape of tensile specimen is normally "Dog bone shape." It can be develop after the thin sheet fabrication and it can be developed by any kind of conventional process like machining and grinding process for linear and the arc formation in the dog bone shape. The values of the dog bone shape will follow the forth coming specification. The machine used for testing the composite has the maximum loaded capacity of 40 tones.

2.4 Thermal Testing

Differential scanning calorimetry or DSC is thermo analytical technique in which the difference in the amount of heat required to increase the temperature of a sample. The reference is measured as a function of temperature. Both the sample and reference are maintained at nearly the same temperature throughout the experiment.

Generally the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned [7, 8].

Thermal stability is a mechanical test to ensure the hybrid composite at high temperature operation. Differential scanning calorimetry will show the quality of the hybrid composite. For thermal stability test minimum quantity of material (20mg) is required to find the output.

The Glass transition temperature (T_g) and complete decomposition temperature is a two different panels measured by the weight reduction of mass of the specimen and based on the peak formation by the endothermic and exothermic reaction based on the heat supplied. The stability process of the scanning calorimetric operation is used to determine the thermal behavior of the composite. All values of peak and thermal values will carry over by degree Celsius. The NETZSCH DSC 200 F3 Maia[®] differential scanning calorimeter covers a temperature range from -170°C to 600°C . The instruments work according to the heat flow principle and are characterized by a three- Dimensional symmetrical construction with homogeneous heating. Sensors with high calorimetric sensitivity, short time constants and a condensation – free sample chamber in the differential scanning calorimetry cell guarantee high detection sensitivity.

Automatic sample changers for up to 64 samples are available; however the configuration available in the lab covers a temperature range of -70 to 600°C by using Intercooler. Instrument control and data acquisition are accomplished via a 32-Bit MS-Windows software and electronics system.

Data evaluation is carried out by a comprehensive PC software package allowing computation of peak and onset temperatures, inflection points, partial area integration, specific heat, transformation energetic, etc. The tests were performed according to, ASTM D 3418-82(88). The varying parameters described below

- Mass of initial sample : 32.502mg
- Temperature range : 30°C to 500°C
- Rate of heating : $10^{\circ}\text{C}/\text{min}$
- Crucible : Al, pierced lid
- Protective gas : Nitrogen
- Flow rate of gas : 20ml/min
- Time consumption : 45 min (total process)
- Checking parameters : T_g and complete decomposition temp

2.5 Nano Fillers

Iron oxide is the inorganic compound used as nano filler. It is one of the main oxides of iron. The magnetite particle (Fe_3O_4) size varies from 1-100nm. The organic and inorganic combination will effectively carry out by surface modification of inorganic materials such as iron oxide, aluminum oxide to produce the coupling effect between the particle and the resin product.

Fig. 3 shows the microscopic image of Nano particle. The particle size is more significance for determining the output of the Nanocomposite because if its surface area. The particle size has used here is < 50 nm for more surface area in at least one dimension.

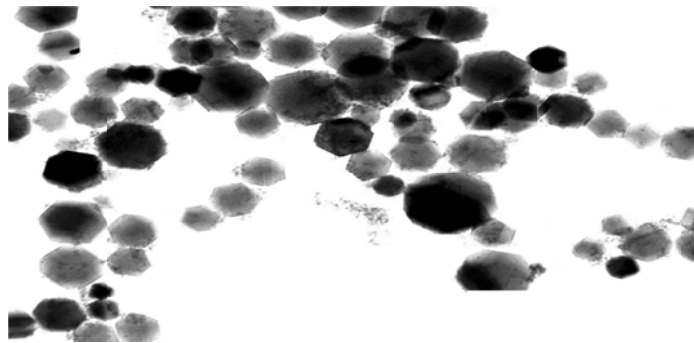


Fig. 3: Electron Microscopic Image of Nano Particles.

Nanoparticles are of great scientific interest as they are a bridge between bulk materials and atomic or molecular structures. A bulk material should have constant physical properties regardless of its size, but at the nano-scale size-dependent properties are often observed. Thus, the properties of materials change as their size approaches the nano scale and as the percentage of atoms at the surface of a material becomes significant.

The particles are having different shapes such as Nano spheres, Nano reefs, Nano boxes, star shapes and long tube like carbon Nano tube. The Nano particles can be visualized and characterized by different kind of electron microscopes like transmission electron microscopy (TEM) for find the particle size, scanning electron microscopy (SEM) for dispersion quality and atomic force microscopy (AFM) for surface level engineering data [9].

3. Results and Discussion

3.1 Mechanical Properties Nano-Composites

The test results have been plotted for further analysis which contains the values like breaking load, ultimate tensile strength. The amount of fiber reinforced plastic composite used with the resin also been plotted, for all samples equal amount of fiber reinforced plastic been used the Table 1 shows the values of different characters of the as-received composite.

Table 1: Test Report for Composite Material

Formulation of samples			Ultimate breaking load (N)	Area in mm ²	Ultimate tensile Strength (MPa)
S.no	Resin matrix	Glass Fiber in Wt (grm)			
1.	Epoxy	28	4900	27.5	179
2.	Epoxy	28	4300	22.5	191
3.	Epoxy	28	4316	23	188

Table 2: Test Report for 5wt% of Nano Composite

Formulation of samples				Ultimate breaking load (N)	Area in mm ²	Ultimate tensile Strength (MPa)
S.no	Resin matrix	Glass Fiber in Wt(grm)	Nano filler wt%			
1.	Epoxy	28	5	6350	26.4	240
2.	Epoxy	28	5	6307	26.5	238
3.	Epoxy	28	5	5865	25.5	230

The Table 2 shows the value of tensile strength varies from 230 MPa to 240 MPa. The adding of 5wt% surface functionalized nano iron oxide particles produced the significant improvement in the tensile strength and the Fig. 4 and 5 shows the stress-strain distribution and the elongation, in two images we could see that the material has offer less yield strength since it is brittle. The value of young's modulus also quite same that shows there is no improvement in the ductility of the material.

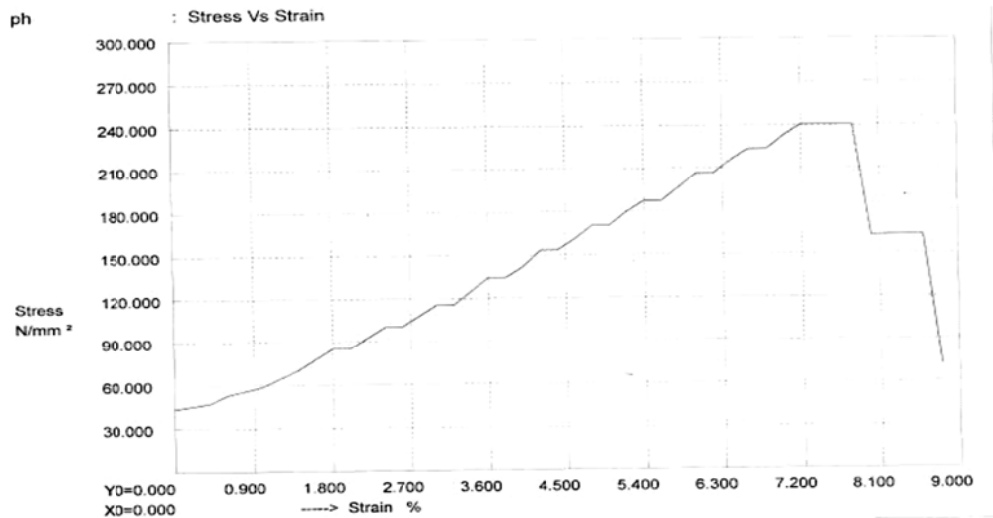


Fig. 4: Stress-Strain Curve for 5wt% Nano Composite.

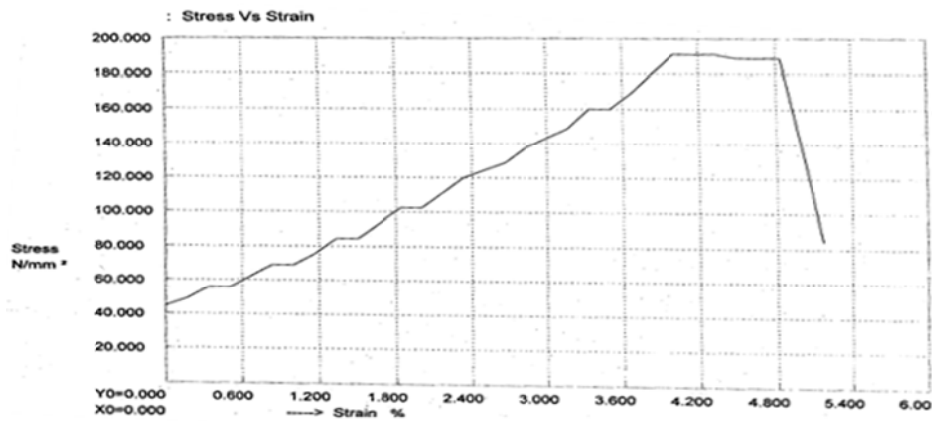


Fig. 5: Stress-Strain Curve for as-Received Composite.

3.2 Mechanical Properties Nano-Composites

The test results have been plotted for further analysis which contains the values like Glass transition temperature (T_g) in °C and Complete decomposition temperature or melting temperature in °C. Table 3 shows the value of T_g and melting temperature.

Table 3: Test Report of Thermal Testing for Composite

Formulation of samples		Glass transition temperature (T _g) in °C	Complete decomposition temperature in °C	Specific heat capacity in J/(g*K)
Resin matrix	Glass fiber in Wt(gm)			
Epoxy	28	63.2°C	322 °C	0.045

The temperature in degree Celsius value been taken in the x- direction and the DSC/(mW/mg) in y- direction with the maximum heat supplying of 500°C. The exothermic glass transition is observed with change in specific heat capacity of 0.045 J/(g*K) by taking the mid value. Fig. 6 shows the glass transition and complete decomposition temperature of the composite the complete decomposition starts from the temperature of 220°C and end with 322°C that shows the materials is having the decomposition rate is nominally high.

Table 4: Test Report of Thermal Testing for Nano Composite

Formulation of samples			Glass transition temperature (Tg) in °C	Complete decomposition °C temperature in °C	Specific heat capacity in J/(g*K)
Resin matrix	Glass fiber in Wt (gram)	Nano filler wt%			
Epoxy	28	5	51.7	358	0.045

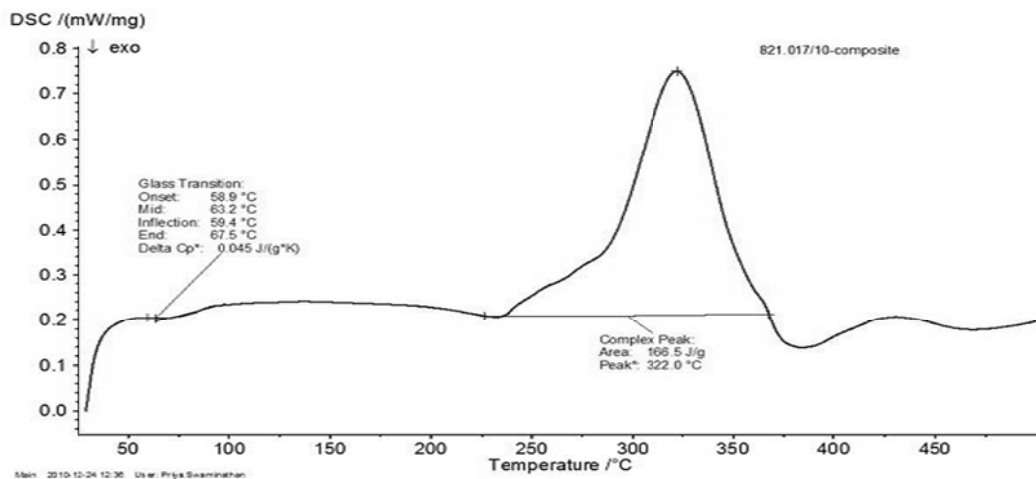


Fig. 6: DSC Graph for As-received Composite.

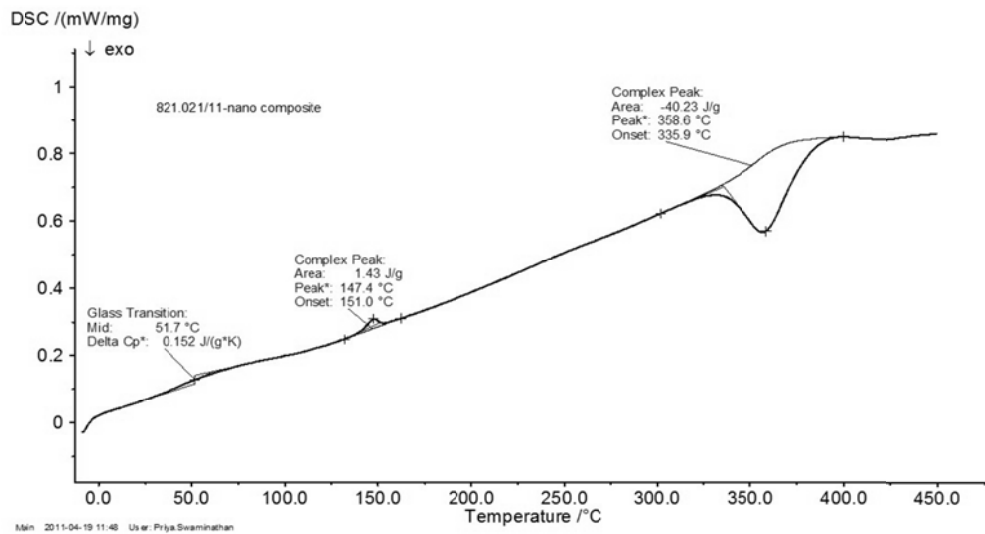


Fig. 7: DSC Graph for 5wt% Nano Composite.

The temperature in degree Celsius value been taken in the x- direction and the DSC/(mW/mg) in y- direction with the maximum heat supplying of 500°C. The exothermic glass transition is observed with change in specific heat capacity of 0.045 J/ (g*K) by taking the mid value. Fig. 7 shows the glass transition and complete decomposition temperature of the nano composite. The decomposition starts from the temperature of 340°C and end with 358°C that shows the materials is having high decomposition temperature.

The test reports and other relevant data of the resin coated non added Nano hybrid composite material have been taken for analyzing the results of the project in the first phase. Based on the tensile test and stress-strain curve the effectiveness of the tensile specimen been studied. The maximum tensile strength of the composite has been observed that 240 MPa with the addition of 5wt% functionalized iron oxide Nano particle and the yield stress has noticed. Since the as received composite has produced only 190 Mpa, the product is brittle and has not display significant effect in the yield stress and necking caused by brittleness. The Nano particle added Nano composite material could offer the substantial improvement in tensile strength in its class. Based on the studies of thermal stability differential scanning calorimetry test the endothermic and exothermic reaction have been absorbed and the glass transition temperature occurs at the temperature of 63.2°C for composite and 51.7°C for Nano composite. The complete decomposition will starts at the temperature of 322°C for as received composite and 358°C for Nano composite material. This shows the material has adopted by good curing process and having good binding energy with the resin and Nano particle and also the glass fiber.

4. Conclusion and Future Work

As per the earlier reports and discussion the adding of Nano particles have done a great job in improving of mechanical properties such as tensile strength, young's modulus, and thermal stability. The improved values of tensile strength from 191MPa to 240 MPa, that is 21% of improvement and the modulus of the Nano composite value haven't been changed that shows the material can produce more durable against the external load. Finally

the thermal stability in that also we could able to see the significant improvement by Nano particle addition hence the complete decomposition temperature improved 10% from 322°C to 358°C.

This values has shown that the adding of Nano particles have improved the mechanical properties. The scored values after adding the Nano particles are the evidence for good and reasonable bonding of Nano particle with resin matrix and glass fiber since the CH₂ group in the iron oxide Nano filler have been reacted with the resin matrix (amine group) and also with the glass fiber in the OH group. That imparts the significant improvement in the Nano composite after adding the iron oxide Nano particles

The same methods can be implemented to reinforce the particles like Al₂O₃, SiO₂, and other metal oxides. Aluminum Nano particle once added the tensile and toughness of the composite change. Similarly SiO₂ is used to improve the thermal stability rate of the composite. Hence the adding of Nano particles will produce substantial improvement in the composite material without changing its physical dimensions.

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