

**Investigation of Thermomechanical properties of FRP
Nanocomposites**

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CERTIFICATE

I hereby certify that the work being presented in the dissertation entitled “**Investigation of Thermomechanical properties of FRP Nanocomposites**” in partial fulfillment of the requirement of the award of the Degree of master of technology and submitted to the Department of Mechanical Engineering of Lovely Professional University, Phagwara, is an authentic record of my own work carried out under the supervision of **Shivaraj Puggal, Professor** Department of Mechanical Engineering, Lovely Professional University. The matter embodied in this dissertation has not been submitted in part or full to any other University or Institute for the award of any degree.

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ABSTRACT

Epoxy-clay nanocomposites have been widely used in application areas like encapsulation of electronic components, industrial coatings, adhesives for bonding, engineering components in aeronautics, protective coatings, high tension electrical insulators, flooring tooling etc. The present work studies the effect of nanoclay addition on the mechanical performance of epoxy based composites. The key issues included synthesis of nanocomposites with changing Nano filler content and their subsequent characterization and evaluation of mechanical properties. Synthesis of nanocomposites comprised of a definite sequence of processing steps involving homogenization and ultrasonication of the clay-epoxy mix, so as to obtain an exfoliated or intercalated morphology. The composites were manufactured using hand layup method and characterized using SEM and XRD. Tensile strength and flexural strength of the clay content samples were improved.

Chapter 1

1 INTRODUCTION

Nanotechnology is which involved in control and manipulation of materials at the nanoscale which is (1 to 100 nanometre (nm)), nanoparticles have high surface to volume ratio of phase and high aspect ratio. There are great variety of nanomaterials which have different range of properties and as many as applications. we can observe this in our surroundings as there are in tiny electron devices, miniature batteries and in some parts of automobiles.

Nanotechnology is divide into various materials such as:

- Nanocomposites
- Nano fibers

1.1 Composite

Composites are materials made from two or more integral materials with significant different physical or chemical properties when united produce a material with different characteristics from the separate components. Composites are made up of separate materials referred as constituent materials. These two materials: matrix and reinforcement. matrix material surrounds and supports the reinforce materials by maintain their relative positions.

1.1.1 Matrix phase

The main phase which have an incessant character is termed as matrix. matrix is generally more ductile and less hard phase.

1.1.2 Reinforced (Dispersion)Phase

There is an additional phase which is imbedded in the matrix in an aperiodic form, secondary phase is called dispersed phase. All the loads acted over the matrix are transfer to this reinforced through interface. It is stronger than the matrix phase so it is all so called reinforced phase. The degree of mechanical behaviour improvement depends on the strong bound between matrix and reinforced interface.

1.1.3 Classification of Composite

- **Natural composites:** there are many natural materials which comes under natural composites e.g. Wood, bones, shells etc.
- **Man Made composites:** These composites formed via merging two or additional materials in certain properties under controlled circumstances e.g. Fiber reinforced composites, concrete and RCC, Reinforced glass etc.

1.1.4 Applications of composite material

- It is used in Aerospace industry
- Used in production of high cost racing cars and bicycle frames.
- Also used in the industrial and engineering structure

1.2 Fibre reinforced polymers (FRP)

The composite material which is made up of polymer matrix strengthened with fibers. These fibers which are frequently used are fiberglass, carbon whereas epoxy is used as polymer. FRP's are used in many different areas which are aerospace, automobile, marine and structure industries. The extent of the strength and elasticity are improved in fibre reinforced plastic depend on the mechanical properties of both fiber and matrix. Strengthening of the matrix happens when the FRP materials displays increase in strength or resistance relative to the strength and resistance of the matrix alone.

1.2.1 Advantages of Fiber Reinforced Polymers:

1. High strength to weight ratio
2. FRP are of low cost
3. Corrosion resistant.

1.3 Polymer Nanocomposites

PNC consist polymer with nanoparticles or nano fillers spread in the polymer matrix. There are different shapes of these polymers such as fibers, spheroids but at least one dimension must be in range of 1-50 nm. These PNC's belong to the group of multi -phase systems (MPS). These systems need precise compounding steadiness of the attained scattering orientation of the dispersed phase.

1.4 Epoxy Nanocomposites

Epoxy nanocomposite is a composite which is in resin form, this is also called as thermoset plastic which is an irreversibly cured from a soft solid or of a resin. This course curing variates the resin to an infusible, insoluble polymer network which is inducted by this action of heat or suitable radiation which often under high pressure. the wide range of epoxy resin in applicable in coating, electrical, automotive, marine, aerospace and civil infrastructure. The epoxy resin refers to the both polymer and curing agent (hardener).

1.5 Clay Nano particles

Nanoclays are nanoparticles of layer's mineral silicates. chemical composition of each clay is different. Clay nanoparticles such as montmorillonite clay are incorporated into polymers to form resulting polymer nanocomposite which possess unique electrical, mechanical and optical properties. This is the most common clay in the family of clay. MMT is different from the vary common types of clay which are available from many years such as talc and mica. It has individual layers of one nanometer thick by about 70nm to 150nm across. This clay can absorb about 20-30 times its volume in water. MMT contains of Nano sized deposits of alumina and silicate slips whereas alumina sheet is squeeze in between two silicate sheets which causes a negative charge. This charge is compensated via metal ions present on surface of layers. MMT clays are normally hydrophilic in character and can be transformed into organophilic via substituting the exchangeable metal ions with alkylamonium cations. This addition of small amount of clay in an organic polymer matrix leads to the improvement of mechanical properties as well as thermal and barrier. organic modification favours the diffusion of polymer

into the interlayer space when the d-spacing increases with the organic modification of clay and it further increase when it is swelled in to epoxy resin.

1.6 Types of Fiber

1.6.1 Carbon Fibre

Carbon fibres are created when the fibers are carbonized at high temperatures. Through further processing by stretching the fibers we can enhance its elasticity. Carbon fibres are in diameter ranging from 9 to 17 μm .

1.6.2 Glass Fibre

Glass reinforced plastic (GRP) are composite materials which are made up of plastic which strengthened with fine glass fiber. As other composite resources each overcoming the deficits of the other, as we can see that the plastic resins are durable in compressive filling and relative feeble in tensile strength whereas glass fiber is durable in tension nevertheless tent not to resist compression. Via combining these two materials GRP becomes a material resist both compression and tensile force.

1.7 Applications of Glass Fiber Reinforced Polymers

- Used mostly in the automotive vehicles such as sport cars, planes, boats etc.
- Vessels and tanks are made using FRP
- Automobile bodies are made FRP

Chapter 2

2 SCOPE OF THE STUDY

- The present study focused on improving the Thermomechanical properties of FRP nanocomposites.
- These FRP has wide range of application fields such as: erosive surroundings, engineering of tennis rackets, frames of bicycles etc.
- There is wide scope of future study such to other properties of composites which are moisture absorption it can be improved as we are using glass fiber which is mainly applicable in roofing sheets, sanitary ware, storage tanks, Pipes.
- Epoxy resins are widely used in advanced composites due to their good impregnation and adhesion to fiber reinforcement resulting excellent mechanical performance. However, these resins still limited in structural applications as an incidental fire event may involve heath risks and loss of mechanical properties so we need to improve the flame retardant properties.
- Recently mechanical and tribological performance of the epoxy matrix is improved by developing the epoxy resin composites modified by nano fillers in the Bearing material of automobiles

Chapter 3

3 OBJECTIVE OF THE STUDY

- Main aim of this present study, to evaluate the mechanical properties of the epoxy - hardener system reinforced with nano filler.
- The overall study is observing the effects of MMT clay addition on the mechanical properties of epoxy based PMNC system (polymer Material and Nanocomposite), as it is a nano-filler in this experiment.
- In this study we include the fabrication of nanocomposites with different concentration levels of nano fillers and evaluate their mechanical properties.
- Microstructure of the nanocomposites can be analysed using X Ray Diffraction techniques, Scanning Electron microscope.
- In this study XRD is used to study the level of intercalation of epoxy resin between clay galleries for different clay loading.
- SEM is used to study the deformation and failure mechanism during tensile and impact testing.

Chapter 4

4 LITERATURE REVIEW

This chapter reviews the previous work done in the areas relevant to this research. The chapter provides an overview of how these methods are related to each other by means of the results of these methods.

T.H. Mahdi, M. V. Hosu (2014) [1]

The mechanical properties of fiber reinforced epoxy modified with MWCNT, MMT Nano clay and Hybrid nanoparticles are used in study. Then Flexure tests are showed is conducted using Zwick-Reoll machine Dynamic. Morphological study conducted with scanning electron microscope. nanocomposites exhibited improvement in storage modulus, loss modulus and glass transition temperature. The results are obtained with enhance properties.

Aidah Jumahata, Costas Soutisb, Jamaluddin Mahmuda, Nurulnatisya Ahmad (2012) [2].

Described about the effect of nanoclay on epikote. The materials used in this study are Montmorillonite clay, curing agent and Benzylidimethylamine (BDMA) accelerator. Electron microscopy is used to study degree of dispersion. Compression tests are conducted to study the outcome of nanoclay. As a result, we found out that nanocomposites propose higher compressive stiffness as compared to straight polymer. We can further improve the study by using three roll mill machine in construction of the nanocomposites.

Farheen Anjuma, Tahir Jami (2014) [3].

In this study about the production of ZnO, GFRP composites by material added of catalyst and accelerator. The high speed disperser is used to mix the materials; the laminates were processed by hot compression molding technique which is done by high temperature melt press which is made of stainless steel. The flexural properties under three-point bending configuration are tested using Universal Testing Machine (UTM). This study proves that It is evident from this study that mechanical and thermal properties are improved.

Mahesh Hosur, S Jeelani (2017) [4].

Study about the Mechanical and viscoelastic properties. The Various techniques adopted to break up the nanoparticles and see proper dispersion of nanoparticles in resin. Strengthened nanocomposites were imperilled to various tests to obtain the mechanical, thermal and viscoelastic properties, after we got the result Flexural strength and modulus increased significantly. Coefficient of thermal expansion decreases by about 30%, Best results were obtained by addition of 0.1% MWCNT/2% MMT binary nanoparticles in epoxy.

T.D. Ngo, P. Tran (2015) [5].

Described the investigation of the effects of nanoclay on the properties of mechanical and fire performance of epoxy glass fiber composite. at first degassing is done then composite materials are mixed using stirring process. Sonication and vacuum infusion process is used. Elemental analysis is carried out by scanning electron microscope. Fire performances test are conducted on samples; heat issue is determined based on feeding of oxygen level. Properties such as creep rupture and durability are tested. Nanoclay filling from 1 to 5wt% were detected to enhance the fire presentation of the epoxy laminates. the results of tensile test are shows the presence of the clay at the levels of settlement percentage contributed to the enhancement in overall properties.

S. Zainuddina, M.V. Hosura, Y. Zhoua, Ashok Kumarb, S. Jeelani (2008) [6].

In this we are going to study about the properties of epoxy composites under difference environmental conditions. MMT is used as the nanoclay which is filled in the composite. Epoxy and hardener are added and mixed with magnetic stirrer, when we mix with this intense air bubbles are produced in the which are taken out with vacuum desiccator chamber, samples are divided in four different sets then they are cooled in room temperature. weight gain analysis is conducted along with flexural testing for it properties. Scanning electron microscopic analysis is conducted on the specimen after coating of gold done to stop charge build up electron absorption. As result of experiment we found out that as epoxy matrix are delicate to environmental conditions especially under wet conditions. Addition of 1-2wt% nanoclay decrease the weight gain and less discoloration was observed in the sample. 2wt% epoxy addition shows that less crack formation and better bonding.

Mahesh V. Hosur (2015) [7].

In this we study about the Enhancement of properties of FRP composites when we modify the matrix properties by adding nanoparticles. 2wt % montmorillonite nanoclay and 0.3wt% nanotubes are added to carbon fiber. above material is mixed with the mechanical mixture and later with ultra sonicated for 30 min. after dispersion epoxy sc-15 part A is mixed with part B with ratio 10:3. then as the results of all the tests conducted which are Flexural, Dynamic mechanical analysis, low velocity impact we come to conclusion as after modification of epoxy matrix with nanoclay we have found the improvement in flexure strength, modulus and strain at failure is almost same. in low velocity impact nanoparticles samples able to absorb higher amount of energy. Composite reinforced with nanoclay has higher storage modulus. low modulus and properties of damping of nanophased composites are also higher than control composites.

S. Singh, V. Sharma (2014) [8].

In this study we are using MMT nanoclay in E-glass unidirectional fiber to manufacture reinforced composite. Epoxy resin is prepared by using mechanical stirrer. sonication is done to disperse nanoparticles. fiber sheet is prepared and mixture is applied on this sheet and set to drying. Tensile and flexural test are conducted with improvement when there is 3-5wt%. when glass fiber composite shows less degradation at 3wt% along with water resistance of epoxy is improved.

Costas Soutis, Aidah Jumahat (2012) [9].

In this we are studying about effect of MMT on the compressive properties of epoxy. the epoxy based nanocomposite is prepared with 1-5% nanoclay. Degree of dispersion and exfoliation is investigated with electron micro scope. Nanocomposite offer higher compressive stiffness when compared to neat polymer.

Manjeet Sekhon, JS Saini, Gaurav Singal and H Bhunia (2015) [10].

In this paper we have determine and compare the strength of pin joints made of glass fibers reinforced laminated epoxy. Nanoclay and Nano TiO₂ (titanium dioxide) it is used to improve

physical properties. Epoxy L-12, Hardener K-12, Accelerator K-13 are used. Homogenizer is used to stirring, Sonication is used to proper disperse of the nanoparticles. Specimen width and diameter of the hole were evaluated. It is found that strength of bearing at pin joints increases with addition of nanoclay.

Kinjal J. Shah, Dinesh O. shash, Toyoko Imae, Atindra D. Shukla (2016) [11].

In this paper hydrophobic organoclays with various quaternary ammonium substitutes are successfully synthesized. The tensile strength and hardness of nanoclay composites was increased with increase in organoclay in the nanocomposites. done to enhance the mechanical properties of UPE-organoclay composite.in this work it has been found that BQASMMT₁₈ enhances the tensile strength, tensile modules, flexural strength. Impact strength is the only parameter which shows a negligible decrease upon addition of organoclays. Hardness of the UPE-Organoclay nanocomposite was increased with increase in organoclay content in the nanocomposites.

Lijun Wang, Jianhui Qiu, Eiichi Sakai and Xiaowei Wei (2015) [12].

The effects of CNT concentration with mechanical and electrical properties of nanocomposites are investigated. As we found that the size of CNT agglomeration was increased by the increased amount of CNT concentration. The crystallinity of PLA (polylactic acid) was greatly increased at the increase of CNT concentration. we can observe a good distribution and dispersion of the CNT in PLA, this having good mechanical and electrical properties.

Avila A, Almir S and Marcelo I (2006) [13].

In this we are investigated the influence of MMT, silicate layers on glass fiber epoxy laminate behavior with x-ray diffraction test. The laminate prepared for this process is glass, epoxy nanoclay, the clay which we use in this process is Nanomer I30E. the process starts with the nanoclay dispersion into the epoxy is 1%,2%,5%,10% respectively. the amount of nanoclay dispersed gets increased there increase in stiffness. When the tests are performed then the results shoes as with the specimen with 5% nanoclay shows good performances rate of energy increase the performance of the laminate decrease.as the low velocity impact test is conducted then is shows that energy absorption rate close to 48% at low energies of 20J,15% increase at 60J, and 4% increase at 80J.the amount of nanoclay for the optimal condition is at 5%.

Chow W, Ishak Mohamad A and Bakar A (2005) [14].

In this process Epoxy, glass fiber and organo- montmorillonite (OMMT) these nanocomposites are prepared by using hand layup method. there are different types of methods which are used on these nanocomposites which are water absorption tests, XRD, Differential scanning calorimetry. The exfoliation characteristics are glass transition temperature are enhanced due to hand layup technique we used to prepare nanocomposites. The resistance properties of epoxy are better by addition of glass fiber and OMMT.

Harish G, Rajmohan T (2013) [15].

In this process we are discuss about the Fiber reinforced polymer (FRP) composite and to improve its mechanical properties of the polymer. Nano Copper Oxide (CuO), polystyrene resin is used in this process.at first CuO is mixed in resin using ultra sonication. then matrix face for glass fiber is prepared using hang layup process. Tensile and compression test are conducted as per the ASTM standards on universal testing machine. SEM is used to scan the samples. A specimen is developed to predict the mechanical properties of GFRP composites.

T.P. Mohan, K. Kanny (2010) [16].

The process we study about fracture toughness of glass fiber reinforced polypropylene, Chopped GFRP and nanoclay filled polypropylene(PP) composite are present. As the nanoclay (cloisite 15A) of 1wt% to 5wt% is occupied in PP matrix and which is subjected to fracture toughness study, then the result which we got as 5wt% nanoclay. It shows that woven fiber PP composite demonstrates grate crack resistance than that of clay filled nanocomposite chopped fiber PP composites. Crack generated rate of the PP nanoclay composite is similar to chopped fiber composites.

Chapter 5

5 EQUIPMENT, MATERIALS AND EXPERIMENTATION SETUP

In this chapter we are going to know about the materials which we are using and the steps in while experiment takes place.

5.1 Materials

Unidirectional E-glass fiber and Epoxy Resin T-23, Epoxy Hardener K-59 are purchased from Navabharth chemicals (India) Private limited. Zinc oxide(ZnO), Montmorillonite Nano Clay, Titanium Carbide Nanoparticle which are of 99% purity also purchased from Nano labs (India) Private limited.

5.2 Equipment

- Homogenizer
- Ultra sonication
- Mechanical stirrer
- Brush
- Metal scrapper
- Unidirectional Glass fiber.

These are some equipment which are needed during experiment.

5.3 Experimentation Set up

5.3.1 Homogenizer

Homogenizer is a process ensure uniform dispersion of the nanoclay through the resin while mixing the base epoxy and nanoclay after measuring the required amount on digital weighting

machine. with the help of these mechanical stirrer we can easily disperse the clay. The speed of this ranges from 3000-5000 rpm, it helps us to work at speeds even with small rotor diameters.



Figure 5.1 Homogenizer.

5.3.2 Ultra sonication

Sonication is a process in which sound energy is applied on the particles in a sample. Sonication is highly effective for processing of the nano materials such as carbon nanotubes, nanoclay metal oxides etc., this sonication runs using AC power. It is used to speed dissolution by breaking intermolecular connections, and to evenly dispersing the nanoparticles in liquid.



Figure 5.2 Sonicator

5.3.3 Tensile testing

A universal testing machine is used for the testing of the FRP specimen for tensile testing. The testing is conducted according to the ASTM standards. The specimens which are tested using this test breaks indicating that it has reached its peak load and ultimate stress value.

5.3.4 SEM (Scanning Electron Microscope)

Scanning electron microscope is used for the observation of specimen surfaces. When the specimen comes in contact with the electron beam then by colliding with the specimen secondary electrons are emitted on the top of specimen surface. The specimen is coated with gold coating before starting the test to prevent charge build up by electron absorption. SEM helps to view the microstructural view of the material.



Figure 5.3 SEM (Scanning Electron Microscope)

5.3.5 X-Ray Diffraction Test

X-ray scattering techniques are from the family of non-destructive analytical techniques. X-ray diffraction test is a test which is conducted to reveal information about the crystallographic structure, physical properties of the materials. X rays are electromagnetic waves with a wavelength in the range of interatomic distances (0.1-10Å). This test is used to find the degree of exfoliation of nanoclay between polymer layers. This technique is depending on Bragg's law which is

$$n\lambda = 2d \sin \theta$$

n is an integer, distance from atomic layers in a crystal is d , λ is wave length of x- ray beam, θ is angle of incidence.

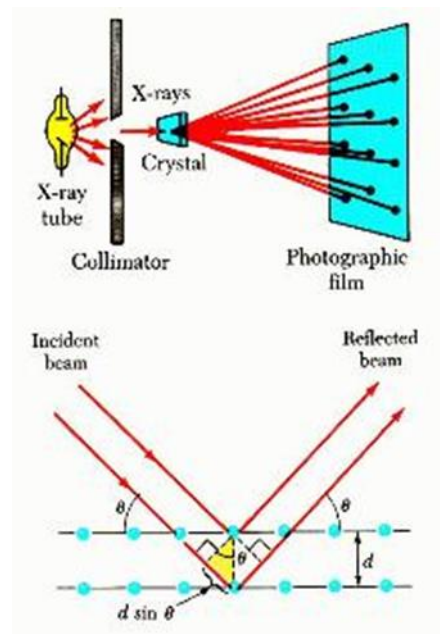


Figure 5.4 X-Ray Diffraction Test Cutting Glass Fiber Sheet

Chapter 6

6 RESEARCH METHODOLOGY

Research methodology is the organized, theoretic analysis of the approaches applies in the study. The theoretical analysis of body of methods and principles related to a division of knowledge. Involves concept like a theoretical model, phases and qualitative and quantitative methods. As an alternative. It gives the theoretical concept for the understanding which method is to be used. Decision method

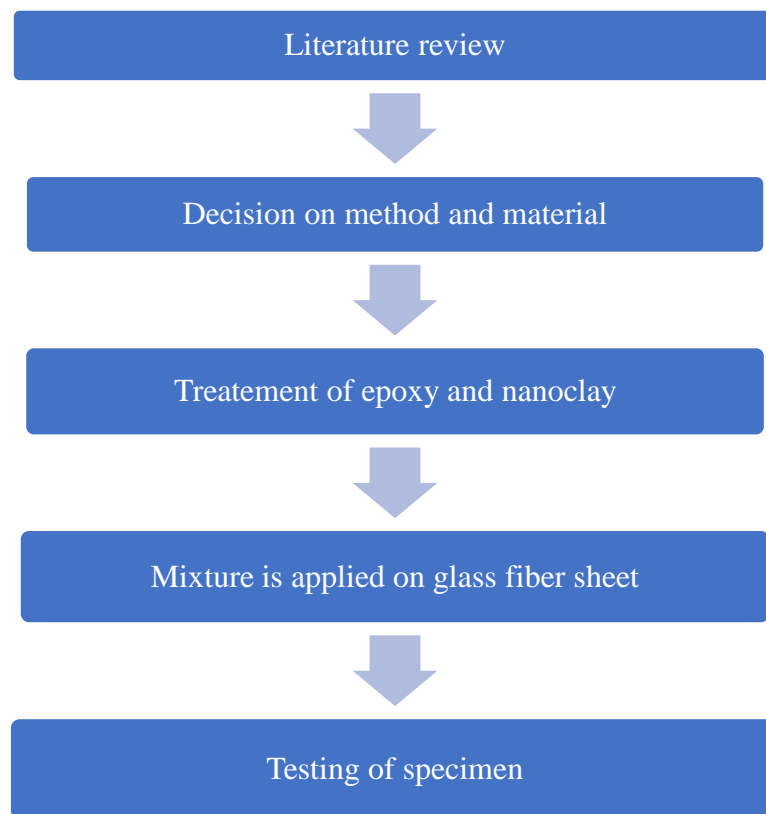


Figure 6.1 Flow chart of the research methodology

6.1 Mixing of nanoclay in to Epoxy:

Nanoclay and the epoxy are measured as per the required amount with the help of digital weighing machine, then both are mixed using mechanical stirrer. We used magnetic hot plate while mixing for proper mixing. Mechanical stirrer helps for better mixing of epoxy and nanoclay.

6.2 Ultra sonication after mechanical stirring

Sonication is a process of applying sound energy to speed dissolution by breaking intermolecular interactions, it is done to evenly disperse nanoparticles in liquids.

6.3 Mixing Epoxy with Hardener

After completion of ultra sonication, the solution is mixed with hardener as per the required ratio 2:1 by volume. After adding the hardener again stirring is done.

6.4 Coating of the Mixture on Glass Fiber Sheet

The mixture which we obtained after mixing nanoclay and Epoxy is applied on the glass fiber sheet uniformly using hand layup method. For this process steel scraper is used for uniform distribution of the mixture. After applying the mixture, the sheet is left to dry.

6.5 Test's conducted on the specimen

After sheet is dried out then we cut the sheet to further testing to know the enhancement of properties by using different methods like tensile testing, Rockwell hardness test, SEM, XRD.

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