MECHANICAL PROPERTIES OF GLASS FIBER REINFORCED EPOXY NANOCOMPOSITES: EFFECT OF DIFFERENT NANOCLAYS

A

Dissertation

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MASTER OF ENGINEERING

in

CAD/CAM & ROBOTICS

Submitted By

Kabal Singh Bhangu

(Roll No. 821181003)

Under Guidance of

Mr. Bikramjit Sharma
Assistant Professor
Department of Mechanical Engg.
Thapar University, Patiala

Dr. Rajeev Mehta
Professor & Head
Department of Chemical Engg.
Thapar University, Patiala

DEPARTMENT OF MECHANICAL ENGINEERING

THAPAR UNIVERSITY, PATIALA-147004, INDIA

(Declared as Deemed-to-be university u/s 3 of the UGC Act, 1956)

July, 2014

Dedicated to my father

CERTIFICATE

This is certify that the work done in this dissertation entitled **"MECHANICAL PROPERTIES OF GLASS FIBER REINFORCED EPOXY NANOCOMPOSITES: EFFECT OF DIFFERENT NANOCLAYS"** submitted in partial fulfillment of requirements for the award of master of Engineering degree in CAD/CAM & Robotics in Department of Mechanical Engineering of Thapar University, Patiala, is an authentic record of work carried out by me under the guidance of **Mr. Bikramjit Sharma,** Assistant Professor, Department of Mechanical Engineering and **Dr. Rajeev Mehta**, Professor and Head, Department of Chemical Engineering Thapar University, Patiala.

The matter embodied in this dissertation has not been submitted in part or full to any university or institute for the award of any degree.

Date: 10/7/14

Kabal Singh Bhangu

This is certified that the above statement made by the student is correct to the best of my knowledge and belief.

Date: 10/7/14

Bikramjit Sharma.

Mr. Bikramjit Sharma
Assistant Professor
Department of Mechanical Engg.
Thapar University, Patiala

Rajeev Mehta

Dr. Rajeev Mehta
Professor & Head
Department of Chemical Engg.
Thapar University, Patiala

Countersigned by:

Dr. Ajay Batish
Professor and Head
Department of Mechanical Engg.
Thapar University, Patiala

Dr. S.K. Mohapatra
Senior Professor and Dean
of Academic Affairs
Thapar University, Patiala

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Kabal Singh Bhangu

ABSTRACT

In this work, E glass fiber reinforced nanocomposites are manufactured with epoxy matrix modified with two different nanoclays: Cloisite 30B® and Cloisite 15A®. The nanoclays are dispersed in epoxy resin in different concentrations (1 wt.% and 2 wt.%). Two ply fibers (±45^0 stacking sequence) reinforced composites are manufactured using modified epoxy by vacuum assisted resin infusion moudling (VARIM). The baseline data for comparison is generated by carrying out test on neat epoxy glass fiber composite i.e. without nanoclay. X-ray diffraction indicates that an intercalated nanoclay epoxy composite is obtained. Elastic modulus, flexural strength and micro-hardness are improved with incorporation of nanoclays. Highest improvement in flexural strength, tensile modulus and micro-hardness obtained at 2 wt.% Cloisite 30B® nanoclay. The flexural strength and tensile modulus of E glass epoxy composite increased by 59% and 69.8% with 2 wt.% Cloisite 30B® nanoclay. The improvement in the properties may be attributed to the high aspect ratio, contact surface and reinforcing effects of nanoclays.

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NOMENCLATURE AND ABBREVIATIONS

S.No.	Nomenclature	Abbreviations
1.	GFRP	Glass fiber reinforced composites
2.	VLSI	Very large scale integration
3.	PNC	Polymer nanocomposites
4.	OMMT	Monomorillonite organoclay
5.	FRP	Fiber reinforced polymers
6.	PU	Polyurethane
7.	VARIM	Vacuum assisted resin infusion moulding
8.	MMT	Manomorillonite
9.	TEM	Transmission electron microscope
10.	XRD	X-ray Diffraction
11.	ASTM	American Society for testing and materials
12	GFRC15A	Glass fiber reinforced nanocomposite with Cloisite 15A®
13	CFRP	Carbon fiber reinforced composites
14	GFRC30B	Glass fiber reinforced nanocomposite with Cloisite 30B®
15	DMA	Dynamic mechanical analysis

CHAPTER 1 INTRODUCTION

Nanotechnology includes the control and manipulation of materials at the nanoscale (molecule sizes from 1 to 100 nanometer, where 1 nm measures up to 1 billionth of a meter) to create new materials and structures that have novel properties because of their little size (expended surface range) is a point of incredible momentum research. Materials with peculiarity on the scale nanometres (10^{-9}meter) frequently have properties significantly not quite the same as their mass-scale counterparts. At nanolevel, a few mixes change from inert to active, from electrical insulator to conductors, from delicate to extream. They can get to be stronger, lighter and more resistant. These changed properties are what represent the unbounded potential application of nanoparticles. One of the central point, which change these properties, is expand in the degree of surface area to volume. As the surface area increases exponentially, making more locales for holding, catalysis or response with surroundings material, bringing about enhanced properties, for example, increased strength or chemical or heat resistance. Henceforth, because of the high surface-to volume proportion connected with nanometre sized particles it is conceivable to control the major properties of materials through the surface/size impact. There is an incredible variety of nanomaterials and their scope of properties and conceivable applications has all the earmarks of being enormous, from additional normal small electronics gadgets, including scaled down batteries, to biomedical uses and as components of parts of auto. Notwithstanding this, in the field of VLSI, nanocomposites might be utilized as electromagnetic protecting between interconnects. Also the way that nanoparticles have measurements underneath the visible wavelength of light, i.e. cannot disperse light, which is the reason the ensuing nanoproduct is transparent, and which has different implications likewise.

Nanotechnology is directed towards the formation of various nanomaterials such as:

- Nanocomposites
- Nanofiber
- Nanoparticulate fillers
- Nanoporous

1.1 Composite

Composite materials are built materials produced using two or more constituent materials with essentially diverse physical or chemical properties that when joined, produce a material with qualities not quite the same as the individual components. The individual components stay separate and different within the completed structure. Composites are made up of individual materials alluded to as constituent materials.

There are two classes of constituent materials: matrix and reinforcement. No less than one part each one sort required. The matrix material encompasses and backing the reinforcement materials by keeping up their relative positions. The reinforcements impart their uncommon mechanical and physical properties to improve the matrix properties.

1.1.1 Composites phases:

- **Matrix**
  The essential phase, having a consistent character, is called matrix. Matrix is generally more flexible and soft phase. It holds the dispersed phase and shares a heap with it.
- **Dispersed (strengthening)**
  The second phase (or phases) is imbedded in the matrix in a spasmodic structure. This is called dispersed phase. Dispersed phase is typically stronger than the matrix, hence it is in some cases called reinforcing phase.

1.1.2 Properties of composites rely on

- Constituents properties
- Dispersed phase (molecule size, dispersion, orientation)
- Phase amount

1.2 Applications of composite materials

- Extensively used in space technology and production of aerospace components (tails, wings etc.)
- Used in the production of sport goods e.g. racing car bodies and bicycle frames etc.
- Used for general industrial and engineering structures
- Used in high speed and fuel efficient transport vehicles

1.3 Introduction to fiber reinforced polymers (FRP)

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. A material's strength is represented by its capacity to manage a heap without extream deformation. At the point when a FRP specimen is tried in axial tension, the reaction of FRP to axial compression is dependent on the relative extent in volume of filaments, the properties of the fiber and resin, and the interface bond strength. FRP's reaction to transverse elastic stress is truly relaint on the properties of the fiber and matrix, the interaction between the fiber and matrix, and the strength of the fiber-matrix interface. By and large elasticity in this course is exceptionally poor.

Advantages of fiber reinforced polymers:

- High strength to weight ratio
- Corrosion resistant
- Can be tailored for the application
- FRP has a low cost considering other materials
- Cost of installation versus replacement is low
- Cost of installation time (both direct and indirect) is also low.

1.4 Nanocomposites

A nano-composite is as a multiphase solid material where one of the phases has one, two or three dimensions of short of 100 nanometer (nm), or structures having nano-scale rehash removes between the distinctive phases that make up the material. In the broadest sense this definition can incorporate permeable media, colloids, gels and copolymer, yet is all the more normally taken to mean the strong consolidation of a mass matrix and nano-dimensional phase(s) varying in properties because of dissimilarities in structure and science. The mechanical, electrical, thermal, optical, electrochemical, catalytic properties of the nano-composite will vary particularly from that of the part materials. Size points of confinement for these impacts have proposed, <5 nm for catalytic activity, <20 nm for making a hard magnetic material soft, <50 nm for refractive index changes, and <100 nm for achieving superparamagnetism, mechanical strengthening matrix dislocation movement.

Nanocomposites are found in nature, for instance in the structure of the abalone shell and bone. The utilization of nanoparticle-rich materials long originates before the understanding of the physical and chemical nature of these materials. It was examined the starting point of profundity of shade and the resistance to acids and bio-corrosion of maya blue paint, ascribing it to a nanoparticle system. From the mid 1950's nanoscale organo-clays have been utilized to control stream of polymerresults (e.g. as paint viscosifiers) or the constitution of gels (e.g. as a thickening substance in cosmetics, keeping the arrangements in homogeneous form). By the 1970's polymer/nano composites was the subject of course readings, despite the fact that the expression "nanocomposites" was not in like manner utilization.

In mechanical terms, nanocomposites vary from ordinary composites because of the extraordinarily high surface to volume degree of the reinforcing stage and/or its astoundingly high proportion. The reinforcing material could be made up of particles (e.g. minerals), sheets (e.g. exfoliated clay stacks) or fibers (e.g. carbon nanotubes). The zone of the interface between the matrix and reinforcement phase(s) is normally order of magnitude more then for ordinary composite materials. The matrix material properties are essentially influenced in the region of the reinforcement.

The large amount of reinforcement surface area means that a relatively small amount of nanoscale reinforcement can have an perceptible impact on the macroscale properties of the composite. For instance, including carbon nanotubes enhances the electrical and thermal conductivity. Different shorts of nanoparticulates may bring about improved heat resistance or mechanical properties, optical properties, dielectric properties, for example stiffness, resistance and strength to wear and damage. As a rule, the nano reinforcement is scattered into the matrix during preparing. The rate by weight of the nanoparticulates presented can stay low (order of 0.5% to 5%) because of the low filler prelocation, edge particularly for the most usually utilized non-spherical, high perspective degree fillers (e.g. nanometer-meager platelets, for example, clays.

Advantages of nano-composites:

- Good tensile and flexural strength for the same dimension of plastic component.

- Less weight and same performance
- Enhanced mechanical strength
- Enhanced gas barrier properties
- Enhanced chemical resistance

1.5 Polymer nanocomposites

For many fields of the modern industry appreciable improvement of physicomechanical properties of polymeric materials is desirable. Nowadays, the most perspective decision of this problem is a modification polymer with nanostructure modifiers. Polymer nanocomposites (PNC) are polymers (thermoplastics, thermo sets, elastomers) that have been reinforced with small quantities (less than 5% by weight) of nano-sized particles. Molecular interaction between polymer and nanofillers do not posses. Uniform dispersion of nanoparticles in polymer matrix produces ultra- large interfacial area per volume between the nanoparticle and the host polymer. This immense internal interfacial area and the nanoscopic dimension between nanopartical fundamentally differentiate PNCs from traditional composites and filled plastics. Introduction to nanoparticles to polymer matrix ensure significant property improvements with very low loading levels. Traditional micro particles additives require much higher filler concentration to achieve similar results. The most commonly used nano fillers are Manomorillonite organoclays, Carbon nanofiber, Carbon nanotubes, Metallic nanoparticles and others.

Mechanical properties

- High adhesion of nanoparticles to polymer matrix result in the enhanced strength of nanocomposites relative to conventional composite.
- Small size of nanoparticles ensures small size of pores in the case of exfoliation of a matrix from filler particles. It results in the strength increase too.
- Introduction of small amount of nanoparticles to polymer significantly enhance the adhesion of polymer to different substance.

1.6 Epoxy nanocomposites

In 1946, the first mechanically created epoxy resin was acquainted with business. From that point forward, the utilization of thermosetting polymers has consistently expanded. The extensive variety of epoxy resin applications incorporates: electrical, auto, coating, aerospace, marine and civil infrastructure and additionally tool manufacture and pipes and vessels in the chemical factory. Because of their good adhesive, low density and mechanical properties, epoxy resin turned into a guaranteeing material for superior applications in the vehicle business, ordinarily as composite materials, for example fiber composite In the avionic business, epoxy-composites material could be found in different piece of the body and structure of military and common aircrafts, with the quantity of uses on the ascent. A not long past approach to enhance and expand polymer properties in the aviation commercial ventures is through the scattering of nanometer-scaled fillers in the polymer lattice. A critical number of scholastic and mechanical undertakings have explored the potential outcomes to further enhance epoxy resin (and at times composites or other twofold frameworks) through the technique of creating nanocomposites. The term 'epoxy resin' alludes to both the polymer and its cured resin/hardener framework. The previous is a low atomic weight oligomer that contains one or more epoxy groups for every atom (more than one unit for every particle is obliged if the resultant material is to be cross-interfaced). The trademark amass, a three-member ring known as epoxy, epoxide, oxirane, glycidyl or ethoxyline group is very strained and hence exceptionally responsive. Epoxy resins might be cross-joined through a polymerization response with a hardener at room temperature (latent reaction). Curing agents are utilized for room temperature cure are typically aliphatic amines, whilst utilized higher temperature, higher execution hardener are aromatic amines and corrosive anhydrides. An expanding number of particular curing agents, for example,poly-functional amines, polybasic carboxylic acids, mercaptans and inorganic hardener are additionally utilized. These results in distinctive, customized properties of the last polymer matrix. When all is said in done, the higher temperature cured resin systems have enhanced properties, for example higher glass transition temperatures, strength and stiffness, contrasted with those cured at room temperature.

1.7 Nanoclays

Clay as nanoparticals such as smectic clays (e.g. montmorillonite) are incorporated into polymers to form resulting polymer nanocomposite, which may possess unique electrical, mechanical and optical properties. The presence of clay as filler is expected to strengthen the mechanical properties of polymer matrix even upon lower loading of filler. Fiber retardancy, barrier resistance and ion conductivity are also expected to be influenced by loading of clay as filler. As their name proposes, polymer/clay nanocomposites contain organic/inorganic mixture polymer matrices containing platelet-formed clay particles that have sizes of the order of a couple of nanometers thick and a few hundred nanometer long. Due to their aspect degree and high surface territories, the clay particles, if appropriately scattered in the polymer matrix at a stacking level of 1 to 5 wt.%, confer special blends of physical and chemical properties that make these nanocomposites alluring for making films and coatings for a variety of industrial applications. With respect to the unmodified polymer, the polymer/clay nanocomposites may display upgrade in strength, modulus, and toughness, tear, radiation, fire resistance, and lower thermal expansion and permeability to gases while holding a high level of optical transparency. The most well known kind of nanoclay is montmorillonite (MMT), a layered aluminosilicate in the smectite group of clay.

1.8 Structure and properties of nanoclays

Silicate comprises of small crystalline particles made up of aluminosilicates of different compositions, with conceivble iron and magnesium substitutions by alkalis and alkaline earth elements. The essential silicon-oxygen unit is a tetrahedron, with four oxygen atoms encompassing the focal silicon. The tetrahedra are connected to form hexagonal rings. This example rehashes in two dimensions to structure a sheet. Aluminum, in fusion with oxygen, strucuresan octahedron, with the aluminum at the inside, and the octahedral connection to structure an all the more nearly packed 2D sheet as indicated in fig. 1.1.

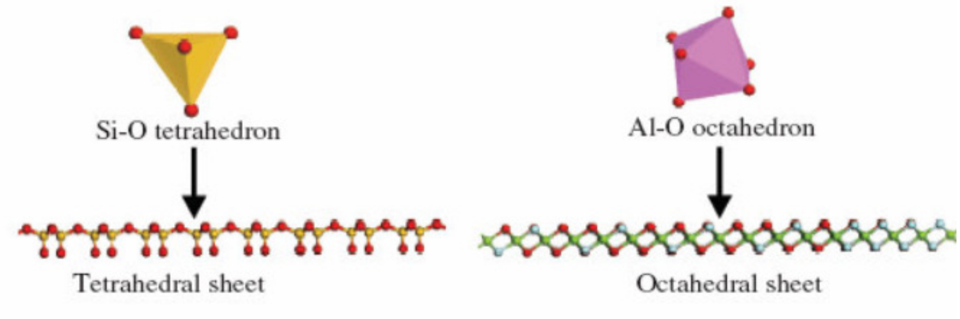


Fig. 1.1 Structure of tetrahedral and octahedral sheets [27]

Two basic types of silicate structures (1: 1 and 2: 1). Kaolinite is an example of 1:1 type of non-swelling dioctahedral silicate. In 2:1 layered silicates two tetrahedral layers surround one octahedral layer, and the oxygen atoms are shared, as an example in montmorillonite. Silicates used to preparing layered silicate/polymer nanocomposites which belong to the 2:1 layered structure type. MMT is a standout amongst the most intriguing and generally investigated silicates refer to the 2:1 layered silicates for polymer nanocomposites due to the weak bonding (Van der Waals) between layers. The chemical formula of MMT silicate is as follows:

$Na_{0.33} (Al_{1.67} Mg_{0.33}) Si_4O_{10} (OH)_2\, nH_2O$

The crystal structure of montmorillonite consists of two silica tetrahedra fused to an edge shared octahedral sheet of either alumina or magnesia as shown in fig. 1.2. Each layer is composed of a sheet of aluminum or magnesium octahedra sandwiched between two sheets of SiO4 tetrahedra, which has a unit cell structure consisting of 20 oxygen atoms and 4 OH groups.

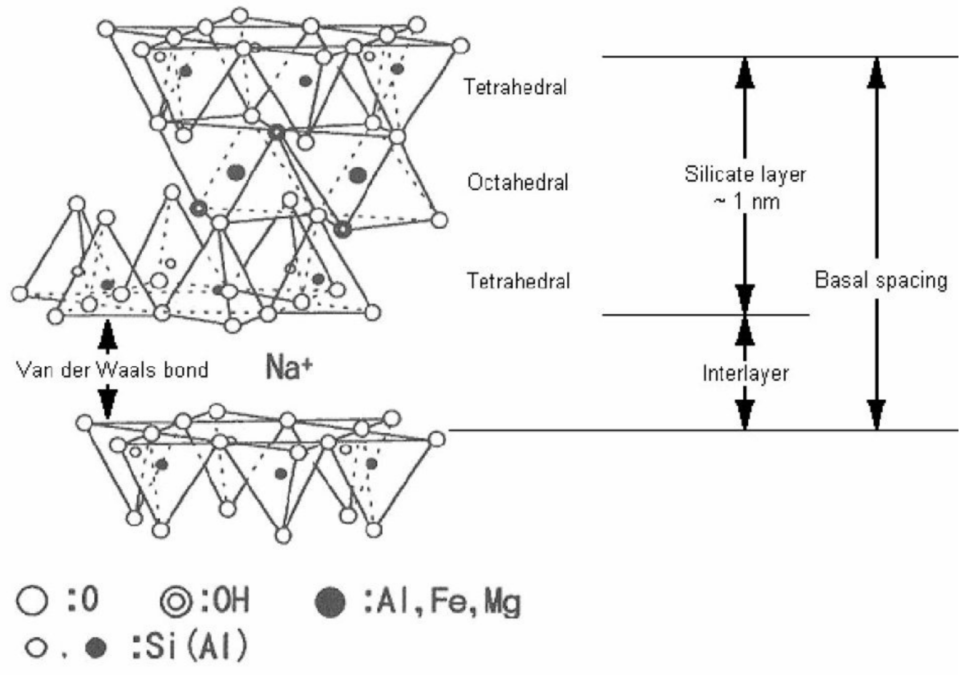


Fig. 1.2 Schematic illustration of atomic arrangement in a typical MMT layer [27]

Hundreds or thousands of these layers are stacked together with weak Van der Waals forces to form a silicate particle. Silicate is needed to be organophilic in order to disperse then in monomer phase. So silicate should become organophilic by treatment with suitable modifiers.

1.9 Organically modified nanoclays

Montmorillonite is the most common type of layered silicates used in polymer nanocomposites due to its swell able layered structure. However, the charged nature of the silicate sheets in the silicate makes the silicate sheets incompatible with hydrophobic polymers. The lack of affinity between hydrophilic silicate and hydrophobic polymer tends to agglomeration of the mineral within the polymer matrix. In this case, pre-treatment of the silicate is necessary. Pristine layered silicates (MMT) usually contain hydrated Na^+ or K^+ cations. They can be replaced through ion exchange reaction with cationic surfactants, including amino acids, organic ammonium salts or tetra organic phosphoniums to render the normally hydrophilic silicate surfaces as organophilic. This reaction is illustrated schematically in fig. 1.3. The most popular cationic surfactant is alkylammonium ion because it might be effectively traded with the ions situated between the silicate layers. Contingent upon the layer charge thickness of the silicate, the alkylammonium ions recevie diverse structures between the silicate layers. The adusted silicate becomes organophilic known as organosilicates (OMMT) and it gets to be more perfect with organic polymers. The purpose of the pre-treatment is to increase the interlayer spacing as well as to provide better compatibility with polymer.

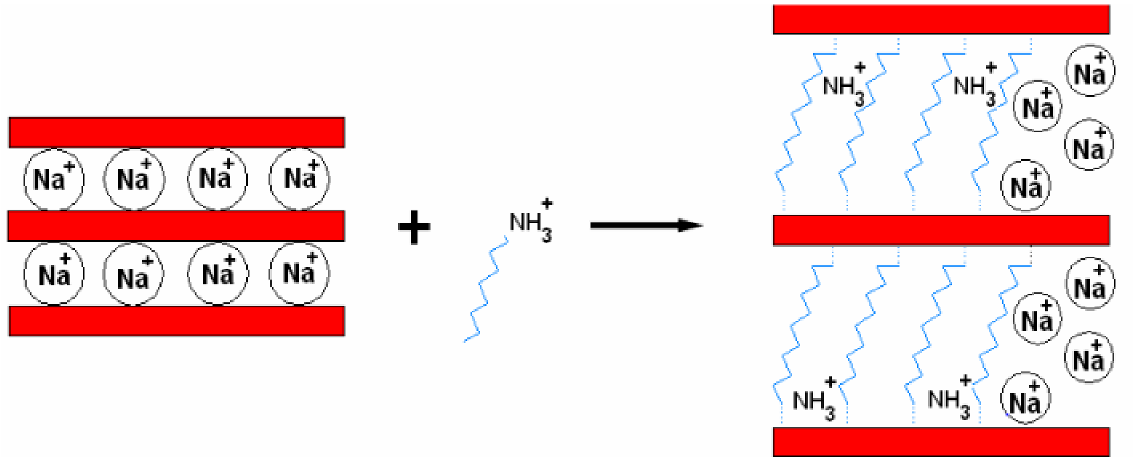


Fig. 1.3 Modification of clay surfaces through ion exchange reaction by replacing the Na^+ cations with cationic surfactant [27]

The properties of OMMT nonoclays studied in this work are given in table 1.1

Table 1.1 Properties of Cloisite 15A® and Cloisite 30B®

Properties	**Cloisite 15A®**	**Cloisite 30B®**
Organic modifier	2M2HT	MT2EtOH
Modifier concentration	125 meq/100g clay	90 meq/100g clay
% Moisture	<2%	<2%
% Weight loss on ignition	43%	30%
Color	off white	off white
Density	1.66 g/cc	1.98 g/cc
d_{001} spacing	31.5Å	18.5 Å

1.10 Morphology of nanocomposites

At the point when layered silicates are organically modified and scattered inside a polymer, the separating stretches to take into consideration the intercalation of polymer between the unit layers. Contingent upon the way of the layered clay and polymer matrix and the level of connections between them, three separate sorts of polymer nanocomposite morphology are observed. These are: intercalated, exfoliated and phase separated.

- **Phase separated:** Layered silicates exist in their unique collected state with no intercalation of the polymer matrix into the exhibitions (fig. 1.4a). For this situation, the particles demonstration as microscale fillers. Their properties stay in the same level as seen in conventional microcomposites.
- **Intercalated:** When polymer resin is embedded into the exhibiton between the adjoining layers, the dispersing stretches, and it is known as the intercalated state (fig. 1.4b). In the intercalated structure, matrix polymer molecules are presented between the ordered layers of silicate bringing about an increase in the interlayer separating, yet keeping up the order.
- **Exfoliated:** In an exfoliated nanocomposite, the individual nm scale thick silicate layers are differentiated and scattered in a ceaseless polymer matrix with average distances between layers relying upon the silicate concentration (fig. 1.4c). At the point when the layers are completely divided, the silicate is thought to be exfoliated. Exfoliated

nanocomposites enhance particular properties better than intercalated one, that are influenced by the level of scattering and resulting interfacial zone in the middle of polymer and silicate nanolayers.

Also, incompletely intercalated or exfoliated composite morphology might additionally be acquired. In this generally happening case, the exfoliated layers and intercalated clusters are randomly circulated in the matrix. The last structure of silicate composite has an extensive range of variations, contingent upon the level of intercalation and exfoliation. X-ray diffraction estimations are utilized to portray the intercalation and exfoliation structures. Appearance in the low angle region showed intercalated composite, however if the peaks are disappear completely, this shows complete exfoliation.

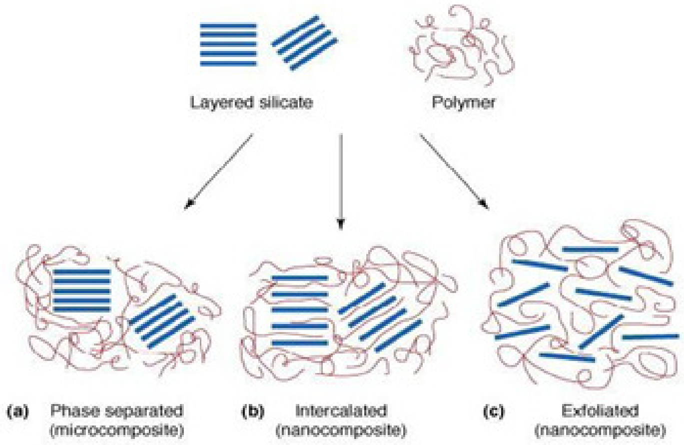


Fig. 1.4 Different morphology of layered polymer nanocomposites [27]

1.11 Types of fiber

1.11.1 Carbon fiber

Carbon fiber, separately graphite fiber, carbon graphite is a material containing fibers having dia about 5–10 μm and combined mostly of carbon atoms. The carbon atoms are joined together in crystals that are more or less aligned parallel to the long axis of the fiber. The crystal alignment gives the fiber increased strength-to-volume ratio. Many of thousand carbon fibers are combined together to create a tow, which may be utilized by itself or woven into a fabric. The properties of carbon fibers, for example, high stiffness, high tensile strength, low weight, high chemical resistance, high temperature tolerance and low thermal expansion, make them very popular in aerospace, military, and motor sports, along with other competition sports. These are relatively expensive when comparison with similar fibers, for example, glass fibers or plastic fibers. Carbon fibers are usually joined with other materials to form a composite. In it the composite consists of two phases; a matrix and reinforcement. In CFRP the reinforcement is carbon fiber, which provides the mechanical strength. The matrix is usually a polymer resin, for example, epoxy, to bind the first phase together. When combined with a polymer resin and molded it forms carbon fiber reinforced plastic which has a very high strength-to-weight proportion, and is extremely strong although somewhat brittle.

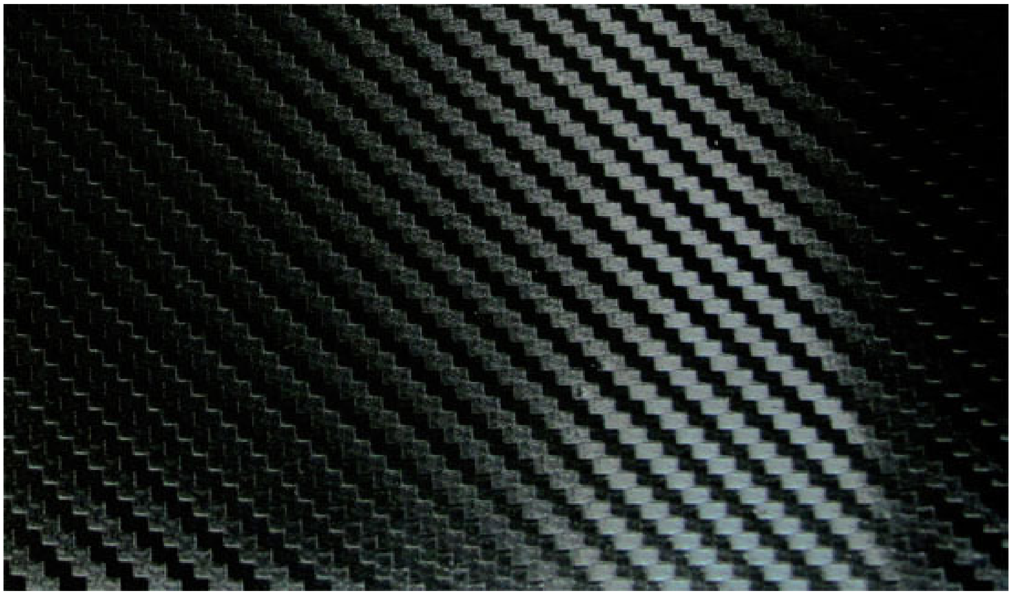


Fig. 1.5 Carbon fiber [25]

1.11.2 Glass fiber

FRP plastics use material glass strands; textile fibers are not quite the same as other forms of glass fibers utilized for insulating purposes. Textile glass fibers start as shifting combos of SiO_2, Al_2O_3, B_2O_3, CaO, or MgO in powder structure. These mixtures are then heated through an immediate melt procedure to temperatures around 1300 °C, after which dies are utilized to extrude fibers of glass fiber in distance across extending from 9 to 17 µm. These fibers are then wound into bigger strings and spun onto bobbins for transportation and further handling. Glass fiber is by long shot the most mainstream intends to strengthen plastic and in this manner appreciates an abundance of generation methodologies, some of which are pertinent to aramid and carbon fibers also owing to their imparted fibrous qualities. Diverse sorts of glass fiber are indicated in the fig. 1.6.

Fig. 1.6 Commercially available glass fibers [26]

The sorts of glasses utilized for structural reinforcements are as :-

- **E-glass** (electrical) - lower alkali content and stronger than A glass (soluble base). Great malleable and compressive strength and stiffness, great electrical properties and generally ease, however affect resistance moderately poor. E-glass is the most well-known manifestation of reinforcing fiber utilized within polymer matrix composites.
- **C-glass** (chemical) - best resistance to chemical assault. For the most part utilized within the type of surface tissue in the external layer of laminates utilized as in chemical and water pipes and tanks.
- **R, S or T-glass** – maker's exchange names for identical fibers having higher tensile strength and modulus than E glass, with better wet quality maintenance. Higher inter-laminar shear stress and wet out properties are attained through more diminutive fiber diameter. Created for aviation and defence commercial enterprise, and utilized within some hard ballistic reinforcement applications. This component, and low generation volumes mean generally high cost.

Glass fiber is available in the following forms

- Continuous fiber
- Chopped strands
- Woven fabric.

Applications of glass fiber reinforced polymers

- Sailplanes, sports cars, karts, body shells, boats, kayaks, flat roofs, Lorries, wind turbine blades.
- Pods, domes and architectural features where a light weight is necessary.
- Bodies for automobiles.
- FRP tanks and vessels: FRP is used extensively to manufacture chemical equipments and tanks and vessels.
- UHF-broadcasting antennas are often mounted inside a glass-reinforced plastic cylinder on the pinnacle of a broadcasting tower.
- Engine intake manifolds are made from glass fiber reinforced PA 66.
- Automotive gas and clutch pedals made from glass fiber reinforced PA 66 (DWP 12-13)

Some typical properties if fibers are given below in table 1.2.

Table 1.2 Comparison of typical properties for some common fiber

Materials	Density (g/cm^3)	Tensile strength (MPa)	Young modulus (GPa)
E-Glass	2.55	2000	80
S-Glass	2.49	4750	89
Alumina	3.28	1950	297
Carbon	2	2900	525
Kevlar 29	1.44	2860	64
Kevlar 49	1.44	3750	136

1.12 Vacuum assisted resin infusion moulding

Vacuum assisted resin infusion molding is a composite assembling procedure to create fantastic huge scale parts. In this procedure, dry preform fabrics are set in an open mold and a plastic vacuum bag is put on the highest point of the mold. The mold is joined with a resin source and a vacuum pump. The fluid resin implants into the reinforcing fiber because of the vacuum drawn through the mold. Curing and de-molding steps take after the impregnation methodology to complete the product. The parts of the mixture procedure used in this work are outlined in fig. 1.7. The capacity of the every part, given in the fig, 1.7, amid assembling could be compressed as:

- **Vacuum bagging film** is fixed to the edge of the mould with vacuum sack sealant tape to make a closed framework.
- **Double side sack sealant tapes** are utilizes to give a vacuum tight seal between the bag and the surface of tool.
- **Release film** is ordinarily set specifically in contact with the preform fiber. They separate the preform fiber from the distribution medium. Release films are regularly perforated to guarantee that any trapped air or volatiles, which may compromise the nature of the preform, are evacuated.
- **Release fabrics** and peel plies are set against the surface of the preform fiber. They are woven items which are strong and have gread heat resistance. Release films confer a gleam finish on the cured laminate, though peel plies and release fabrics leave an impression of the weave design. Peel plies give a clean, uncontaminated surface for resulting bonding or painting.
- **Tool release material** is utilized to discharge the item from instruments effortlessly and acquire a smooth surface completion. For this reason, liquid discharge operators are used. In specific circumstances Teflon films can additionally take care of tool porosity issues.
- **Resin conveyance** medium called a profoundly permeable layer set on the highest point of the preform spreads the resin rapidly over the horizontal degree of the part.
- **Breather** fabric is non-woven fabric permit air and volatiles to be expelled from inside the vacuum bag all through the cure cycle. They additionally ingest resin present in composite layups.

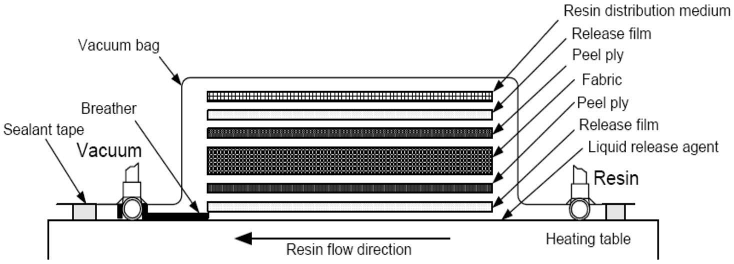


Fig. 1.7 Schematic illustration of the vacuum assisted resin infusion moudling process [24]

Avila et al. (2007) demonstrated that the dissolution of nanoclay particle, into epoxy system lead to an intercalated nanostructures, for concentrations up to 5% and mixed nanostructures, intercalated and immiscible nanosystems for 10% concentration. The nanocomposites mechanical properties were evaluated considering tensile, indentation and low-velocity impact tests. It was also demonstrated that the indentation power law was also applicable to the nanocomposites studied. When the low-velocity impact results were analyzed, they showed an increase on energy absorption close to 48% for low energies, 20 J, 15% increase for medium–high energies, 60 J, and 4% for high energy, 80 J. The presence of intercalated nanoclays into laminates leads an enhancement on stiffness and an increase on impact resistance/fracture toughness and changes into failure mechanisms. A competing mechanism between stiffness and fracture toughness was noticed, as the 10% nanoclay content laminate did not perform as well as the 5% condition. As the amount of intercalated nanoclay content varied from 0% to 10%, the optimum condition for low-velocity impact seems to be around 5%.

Kanny et al. (2014) 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. Unfilled epoxy resin showed uniform viscosity across the entire laminate processing length. Nanoclay filled resin showed two types of viscosities, initial resin mixture viscosity (gi) and time dependent gel viscosity (gg). Both gi and gg values were continuously increased as nanoclay content continuously increased in resin. FTIR results indicated that the organoions of the clay act as catalyst during epoxy resin curing, and increased the degree of cure and resulted in increased viscosity. Maximum improvement in was observed in 5 wt.% nanoclay filled laminate. The structural analysis (TEM and XRD) indicated that the nanocomposite formed exfoliated structure up to 3 wt.% nanoclay and above 3 wt.% formed intercalated structure. Due to the intercalated structure above 3 wt.% nanoclay, the material resulted in decreasing trend of tensile and DMA properties. Maximum improvements in these properties were observed at 3 wt.% nanoclay filled laminate due to exfoliated structure. At 3 wt.% nanoclay filled laminate, about 9%, 21% and 15% increase of strength, modulus and elongation respectively was observed when compared with unfilled laminates. The outcome of this work provides basic flow characteristics of the nanoclay filled epoxy resin and suggests that designers to select infusion points carefully to produce optimally designed structures during nanoclay filled laminate processing.

Sivasaravanan et al. (2014) 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.

Lin et al. (2006) prepared hybrid laminate composite comprising nanoclays, glass fiber and epoxy ternary composites successfully using the VARTM process. The nanocomposite showed good impregnation when unidirectional glass fibers were aligned in the longitudinal direction. When the glass fibers were placed in the transverse direction, air bubbles were readily entrapped during resin flow, negatively affecting the properties of the nanocomposite. Fairly uniform distributions of clay were observed for both directions of resin flow, along and perpendicular to the fiber direction. XRD and TEM revealed the intercalation structure of the clays in the epoxy resin, which greatly influenced the molecular mobility of the epoxy. The mechanical properties of the ternary composites deviated slightly with the direction and location of the glass fibers.

Dolati et al. (2013) 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.

Hegde (2009) investigated influence of different percentage of nanoclay and different dispersion agents on the microstructure, morphology and mechanical properties of nylon 6 and polypropylene nanocomposites products. Compared to polypropylene, slightly better dispersion of platelets was observed in nylon 6. Nylon 6 blown films and fibers could be processed with nanoclay additives. Different nanocomposite morphologies were observed for different matrix (nylon 6 and polypropylene) and process (film, fiber, nonwovens and injection molded composites).

Azeez et al. (2013) 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. Thermal stability and barrier properties were significantly improved by the incorporation of clay particles to epoxy systems. Water permeation of the nanocomposite were reduced considerably.

Thiagarajan et al. (2014) prepared the nanocomposite by varying consentient of nanocaly of 0% to 4%. The XRD results clearly show that the particles was uniformly dispersed and obtained intercalation structure. The 1% nanoclay sample showed that the random distribution of nanoclay over epoxy. The 3% nanoclay sample showed that good interfacial bonding with fiber.

Kornmann et al. (2005) synthesized epoxy-layered silicate nanocomposites based on anhydride cured epoxy and octadecylamine modified fluorohectorite and used as matrix in glass-fiber-reinforced laminates. X ray diffraction analysis and transmission electron microscopy indicate that the silicate layers were dispersed in the form of swollen aggregates with an interlamellar spacing of 9 nm. Environmental scanning electron micrographs of the laminates had broken at cryogenic temperatures shows that silicate layers had a particular affinity for the glass fibers surface, probably increasing the interfacial properties of the fibers. Flexural tests on the laminates indicated that the presence of silicate layers in the epoxy matrix leads to a flexural strength improvement of 27%. According to dynamic mechanical measurements, the presence of organosilicate caused a decrease of the glass transition temperature. The glass transition temperature decrease is apparently responsible for the larger water uptake observed in the nanocomposite and the corresponding laminates at 50 $^{\circ}$C.

Yasmin et al. (2003) 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. The observed higher tensile strength with better degassing also confirms the importance of degassing in reducing the number of nano- to micro-size voids.

Sharma et al. (2012) 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. Durability studies conducted on nanocomposites in water and alkaline medium for a period of one month showed degradation in mechanical properties of all specimens. The decrease in tensile and flexural strength was however less in specimen with nanoclay modified epoxy matrix in comparison to specimen with neat epoxy matrix. The water resistance properties of epoxy were improved by the addition of both glass fiber and nanoclay, which may be attributed to the increasing of the tortuosity path for water penetration.

Dorigato et al. (2012) 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. Rheological measurements revealed that the viscosity increased due to the presence of clay lamellae was proportional to the filler content. XRD indicate formation of an intercalated structure for all the samples. Fracture toughness was substantially improved by the clay addition, especially in the case of 5 wt.% Cloisite 10A-filled sample. The investigation of the fiber-matrix interfacial adhesion through the single-fiber micro-debonding tests revealed a marked (about 10-30%) increase of the interfacial shear strength for the nanofilled samples. A lowering of the fiber-matrix contact angle values has been observed when the organo-modified clays were added to the epoxy resin.

Lam et al. (2005) 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. This might be due to the size of the clusters reaching a crucial limit and therefore the reinforcing function of the nanoclays decreased. Interlaminar shear test showed the short beam shear strength of the epoxy decreased after adding few percents of nanoclay particles. A shiny surface of the clusters, captured from the fractured samples also revealed that a weak bonding interfacial existed between the cluster and matrix, which led to poor short beam strength of the composites.

Zulfli et al. (2012) observed that the glass fiber reinforced epoxy composites containing various loading of OMMT showed a balance of flexural modulus, flexural strength, impact strength and fracture toughness at 4 wt.%. loading of OMMT The improvement in the mechanical properties attributed to the exfoliation-ability, reinforcing-ability, and interfacial bonding-ability of OMMT layered silicate. The thermal stability of epoxy/glass fiber composites was greatly enhanced by the addition of OMMT loading up to 4 wt.%, due to the presence of exfoliated OMMT platelets which hindered the diffusion of volatile decomposition products from the epoxy matrix.

Agubra et al. (2013) 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.

Quaresimin et al. (2008) 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 hardener was a hexa-hydrophthalic anhydride. The nanoclay used here was Cloisite 30B. The organoclay was dispersed through a shear mixing process. Hand-layup method was used to prepare the specimens. 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. The result being that the ‘‘effective’’ clay concentration in the interlayer resin rich regions was much higher than the nominal nano-additive concentration.

Manjunatha et al. (2010) 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. The fatigue life of the GFRP composite with 10 wt.% silica nanoparticle modified epoxy matrix is about three to four times higher than that of the GFRP with the neat epoxy matrix. The suppressed matrix cracking and reduced crack growth rate due to the particle debonding and plastic void growth mechanisms appears to contribute for the observed enhancement of the fatigue life in the GFRP with the nanoparticle-modified matrix.

Bozkurt et al. (2007) investigated the mechanical and thermal properties of non- crimp glass fiber reinforced clay/epoxy nanocomposites. E-glass non-crimp fabrics, epoxy thermosetting resin and Na^+ montmorillonite (MMT and OMMT) clay particles were used to fabricate specimen. The clay/epoxy mixture was mechanically stirred for about 1 hour at room temperature followed by ultrasonication for 20 min in order to further exfoliate the clay within the resin. Hand Layup technique was used to impregnate and laminate the composite structures. Clay/epoxy nanocomposites were synthesied with different concentrations of MMT and OMMT clay. X-ray analysis of surface treated and untreated clays reveal that the modification of clay increased the d-spacing of clay layers that ease the dispersion of clay particles in the matrix. The tensile strength and modulus values remains almost constant by the addition of MMT and OMMT up to 6 wt.% clay contents as compared to those fabricated without clay addition. However, further addition of MMT and OMMT reduced the strength values. On the other hand, tensile modulus values are reduced by the addition of OMMT while they remained constant with MMT addition at 10 wt.% clay loading. Flexural strength and modulus values are increased with the addition of MMT and OMMT, up to 6 wt.% of clay loading. Maximum improvement in flexural strength and modulus was obtained at 6 wt.% clay content and by the addition of nanoparticulates the flexural strength and modulus were improved by 16% and 13%, respectively. With the addition of OMMT (10 wt.%) particles to the matrix, fracture toughness of laminates increased by 5%. Addition of clay significantly reduces the flammability of polymer composite. The average extends of burning and average time of burning was reduced by 55% and 77%, respectively due to the addition of 10 wt.% MMT into epoxy. This improvement was even higher with surface treated clay (OMMT) due to its better dispersion in the polymer matrix.

Daud et al. (2009) 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. On the other hand, the in-plane shear properties of laminates revealed that the layered silicates help improved both the in-plane shear strength and modulus appropriately. By utilizing a nanocomposite matrix, improvement of stiffness and strength, as well as thermal and barrier properties was obtained without any change in processing temperature of the fiber composites

Bakar et al. (2013) investigated the effect of sonication amplitude and mixing time on the mechanical and thermal properties of an epoxy nanocomposite. At short mixing times, the impact strength increased with increasing amplitude and also with decreasing time at a fixed maximum sonication amplitude of 260 µm. Maximum impact strength values were obtained with a maximum ultrasonic amplitude of 260 µm and a mixing time of 10 min. Impact strength decreased with increasing amplitude at longer mixing times. The deterioration of the mechanical properties was confirmed for long ultrasonic dispersion times. At higher fixed amplitude, the values of flexural stress increased and then decreased with increasing mixing time. The flexural stress value increased with increasing the sonication amplitude due to most probably the increase of the epoxy resin degree of cross-linking. The effect of mixing time was more pronounced on the deflection temperature under load than the sonication amplitude. Similar to impact strength, the highest temperature was obtained with the shortest mixing times and highest amplitude. The fracture surfaces of the epoxy nanocomposites mixed for 5 min were rough with significant plastic deformation zones and nanoparticles embedded in the polymer matrix, explaining thus the improvement of the epoxy matrix mechanical properties.

Haque et al. (2003) manufactured fiber reinforced polymer nanocomposites using vacuum assisted resin infusion process. TEM observation of epoxy–clay system reveals that silicate phase was stacked alternately with polymer chain forming basic lamellar type microstructures of the nanocomposites (intercalated). The micrographs also showed regions where it was not strictly of lamellar morphology, rather a group of differently oriented parallel silicate and polymer layers bundled together (phase separated). The thermal properties such as onset of decomposition and glass transition temperatures were determined by Thermo Gravimetric

Analysis (TGA) and Dynamic Modulus Analyzer (DMA). The onset temperature of decomposition of nanocomposites was seen to be increased approximately 20 °C to 22 °C and simultaneously, the glass transition temperature (Tg) was also observed to be slightly increased (3 °C to 4 °C) than the conventional composites. The mechanical properties of fiber reinforced polymer nanocomposites have been evaluated. Interlaminar shear and flexural strength of S2-glass/epoxy–clay nanocomposites were seen to be increased almost 44% and 24%, respectively by adding 1% clay into the system. The fracture toughness of the same was also seen to be increased almost 23%. The results indicated that the thermomechanical properties mostly increased at low clay loadings 1–2% by weight but decreased at higher clay loadings 5 wt.% and above.

Basara et al. (2005) studied from scanning electron micrographs, that clay particles have a tendency to form agglomerates as the clay content increases. A higher degree of agglomeration was occurs in nanocomposites with Cloisite Na^+. As a result of this, mechanical properties of the nanocomposites with Cloisite Na^+ was not significantly improved. Cloisite 30B exhibits better dispersion than Cloisite Na^+. From XRD patterns, it was observed that, in nanocomposites with 3 wt.% Cloisite 30B, d-spacing expands from 18.4 Å (the initial value of pure Cloisite 30B clay) to 38.2 Å, indicated intercalation. In nanocomposites with 3% Cloisite Na^+, the d-spacing expands from 11.7 Å to 15.4 Å, exhibited a lower degree of intercalation in comparison to nanocomposites with Cloisite 30B. In accordance with this observation, DSC data showed that the Tg value increased from 73 °C in the neat epoxy to 83.5 °C and 75 °C in the nanocomposites with 9 wt.% Cloisite 30B and Cloisite Na^+, respectively. The impact strength values of the nanocomposites exhibit marked improvement in both types of clays. Addition of 0.5 wt.% Cloisite 30B improved the impact strength by 137%. On the other hand, addition of 0.5 wt.% Cloisite Na^+ improved the impact strength by 72% with respect to the impact strength of the neat resin. The mechanical properties of nanocomposites with Cloisite 30B was improved more than those of nanocomposites with Cloisite Na^+.

CHAPTER 3 RESEARCH PROBLEM

3.1 Gaps in literature

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. To full fill this gap the following work is purposed.

3.2 Research problem

Mechanical properties of glass fiber reinforced epoxy nanocomposites: Effect of different nanoclays.

3.3 Objectives of work

Evaluations of mechanical properties of fiber reinforced epoxy nanocomposites and study of effects of two different nanoclays.

1. Improvement in mechanical properties of fiber reinforced composite by addition of nanoclays in matrix.
2. A study of dispersion of nanofiller in epoxy matrix.

CHAPTER 4 FABRICATION & EXPERIMENTATION

4.1 Fabrication of specimens

4.1.1 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.

4.1.2 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. 4.1 was used for mixing. The mixtures were stirred at 20000 rpm for 5 minutes.

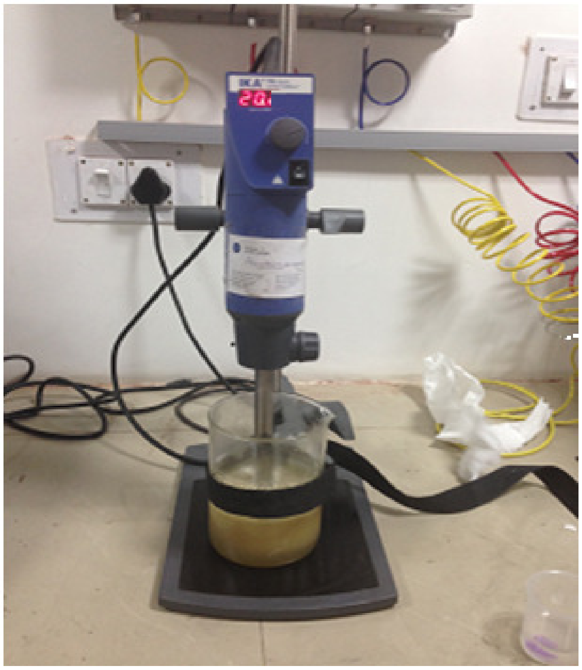


Fig. 4.1 Homogenizer used for mixing of nanoclay

4.1.2 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.

Fig. 4.2 Dispersion of nanoclay into epoxy resin with probe

4.1.3 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.

4.1.4 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 [$\pm 45^0$] stacking sequence using vacuum assisted resin infusion moudling .The laminate was cured at 120 $^{\circ}$C for 7 hrs and then left at room temperature for 24 hrs.

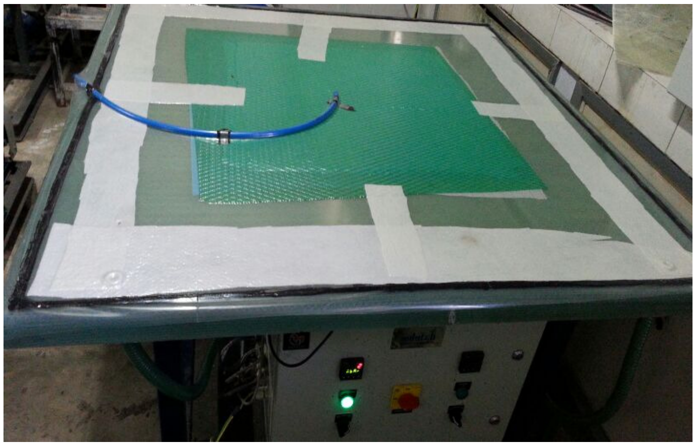


Fig. 4.3 Manufacturing of fiber reinforced composite

4.2 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 4.1 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

4.3 Testing methods

4.3.1 Tensile testing

A universal tensile testing machine shown in fig. 4.4 and fig. 4.5 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.

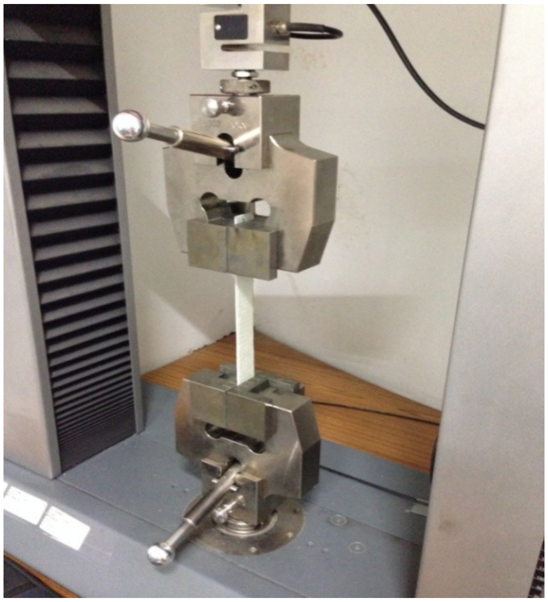


Fig. 4.4 UTM testing machine **Fig. 4.5 Specimen positioning**

4.3.2 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. 4.6 Three point flexural test machine **Fig. 4.7 Specimen positioning**

4.3.3 Micro hardness test

Micro hardness test (shown in fig. 4.8) was conducted on specimens with different clay loadings.

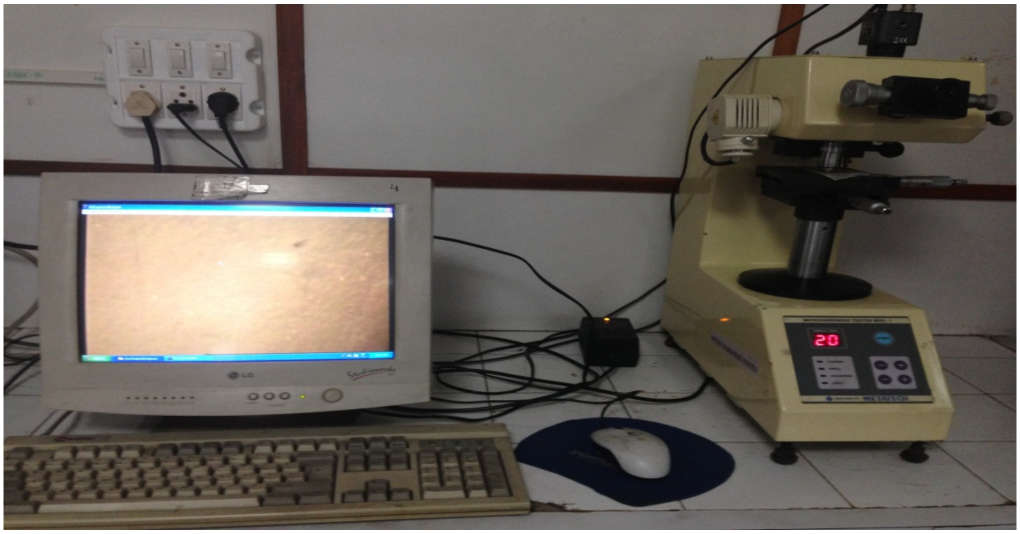
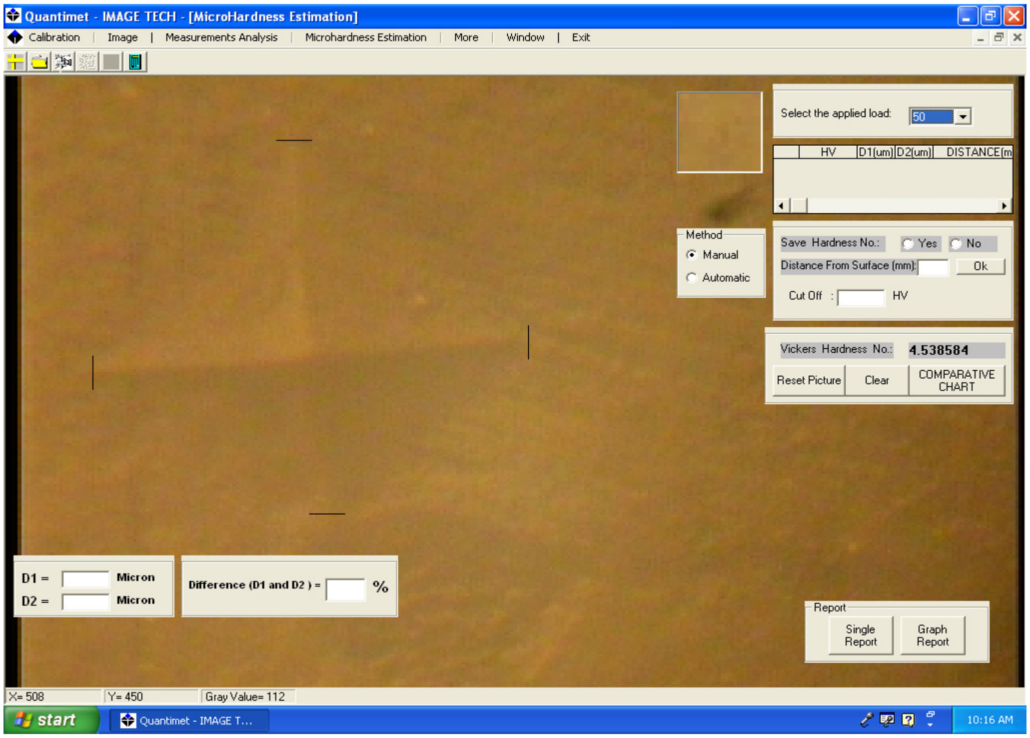


Fig. 4.8 Micro hardness equipment



Select the applied load: 50

Method
● Manual
○ Automatic

Vickers Hardness No.: 4.538584

X= 508 Y= 450 Gray Value= 112

Fig. 4.9 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. 4.9 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.

4.3.4 Scanning electron microscope

Scanning electronic microscope shown in fig. 4.10 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. 4.11. The polished specimens were used to observe the microstructure of specimens at different magnification.

Fig. 4.10 SEM machine

Fig. 4.11 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.

4.3.5 X-Ray diffraction

X-ray scattering techniques are a group of non-destructive analytical methods which reveal data about the crystallographic structure, chemical composition, and physical properties of materials and thin films. These techniques are focused around observing the scattered intensity of an X-ray beam hitting a sample as a function of incident and scattered angle, polarization, and wavelength or energy.

X-ray diffraction was utilized as a part of this study to research the crystallographic structure of the epoxy nanocomposites. 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 d-spacing of the intergallery spacing can be determined using Bragg's Law:

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

Where λ is the wavelength of the incidence x-ray source, d is the spacing in question, θ is ½ of 2θ the Bragg angle or the diffracted angle of the incidence x-ray beam. Below is a schematic of the previously mentioned Bragg's Law (fig. 4.12)

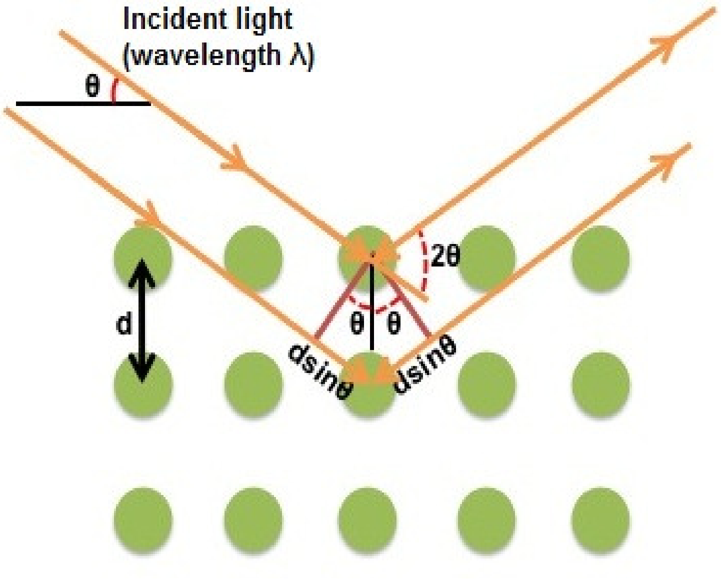


Fig. 4.12 Schematic representation of x-ray diffraction principle [29]

To evaluate the degree of exfoliation in the polymer, XRD measurements were carried out in a Panalytical X-ray diffractometer with Cu Kα radiation (λ=1.54Å) with a scanning speed of

1^0/min and at 45 kV and 40mA. During the XRD experiments, the samples were analyzed in reflection mode. All XRD scans were through 2θ of 2^0 to 10^0.

4.4 Test matrices

Table 4.2 Initial test specimens

Clay loading	No. of specimens		Total specimens
	Tensile	Bending	
0 wt.% clay	3	3	6
1 wt.% clay Cloisite 15A®	3	3	6
1 wt.% clay Cloisite 30B®	3	3	6
2 wt.% clay Cloisite 15A®	3	3	6
2 wt.% clay Cloisite 30B®	3	3	6

CHAPTER 5 RESULTS AND DISSCUSSION

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

5.1 Micro-hardness

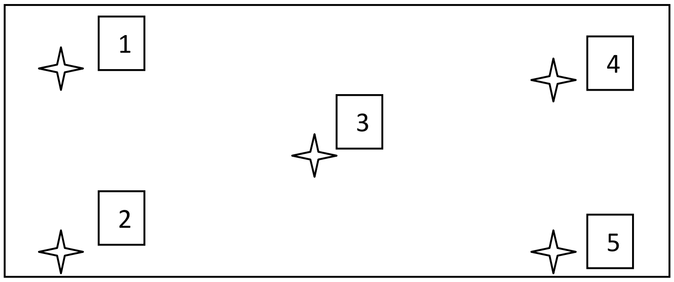


Fig. 5.1 Loading points on specimen

The micro-hardness of specimens at different clay loading was measured. The table 5.1 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 samples

Table 5.1 Micro hardness values for different clay loading specimens

Clay loading ⇨ **loading points** ⇩	**Micro hardness values**				
	0 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

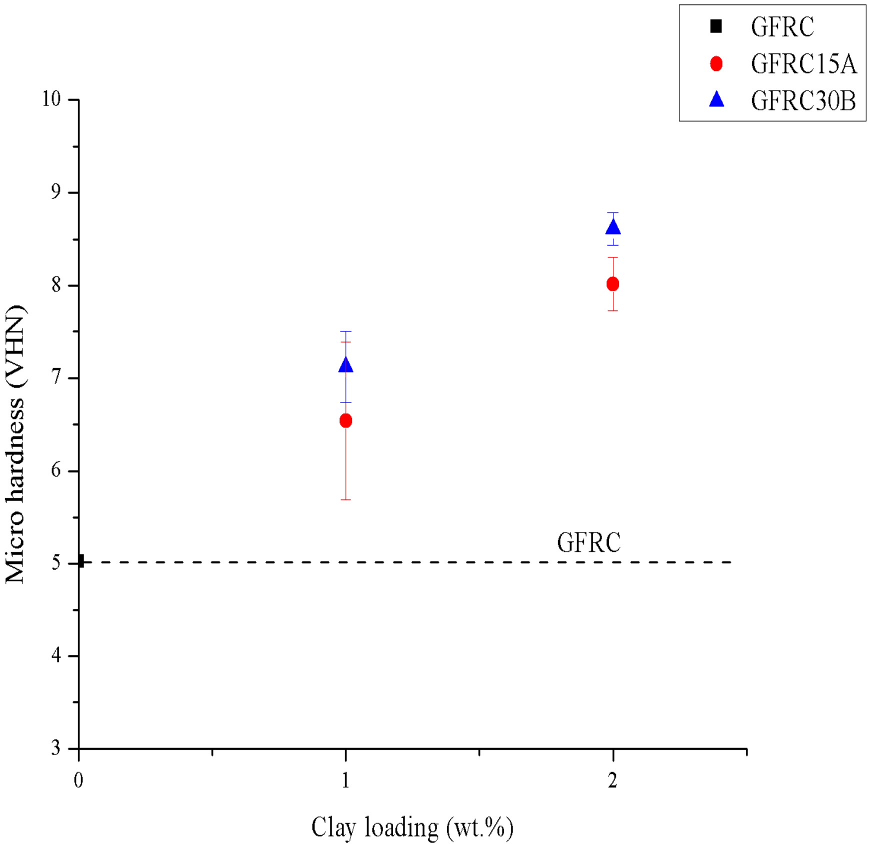


Fig. 5.2 Micro hardness test results

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.

5.2 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. 5.3.

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	
1 wt.% clay Cloisite 15A®	1	172	168.6
	2	165	
	3	169	
1 wt.% clay Cloisite 30B®	1	174	177.4
	2	178	
	3	180	
2 wt.% clay Cloisite 15A®	1	181	194.4
	2	196	
	3	206	
2 wt.% clay Cloisite 30B®	1	235	242.6
	2	251	
	3	242	

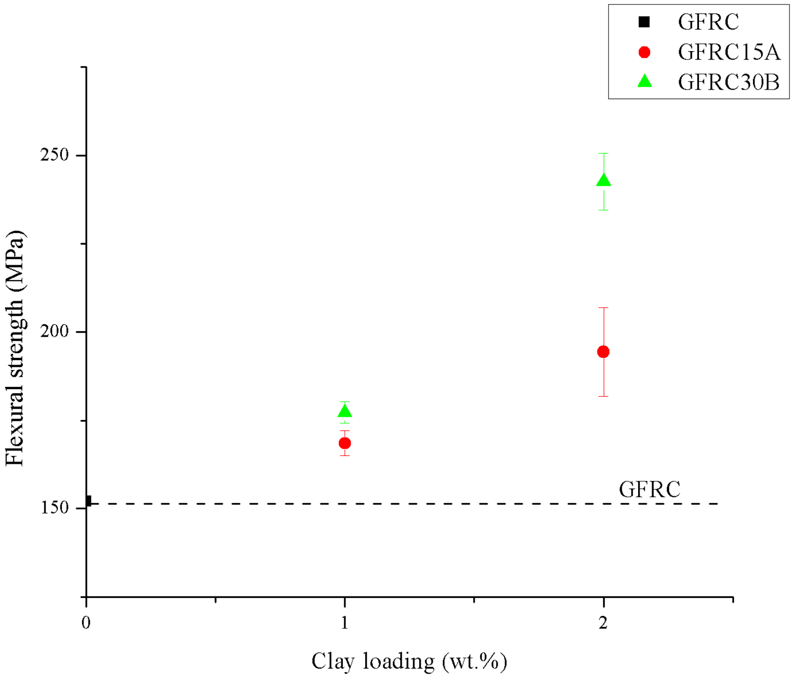


Fig. 5.3 Flexural strength test results

5.3 Tensile test

The results obtained by conducting tensile tests on nanocomposites using Zwick/Roell universal testing machine are indicated below in table 5.3.

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

From fig. 5.4a, 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. 5.4b indicate the changes in tensile strength with clay content. Unlike the tensile modulus, nanocomposite of both clay content show lower tensile strength than that of pure epoxy which are in accordance with research carried out by by Yasmin et al. [10] and Zerda et al. [23].

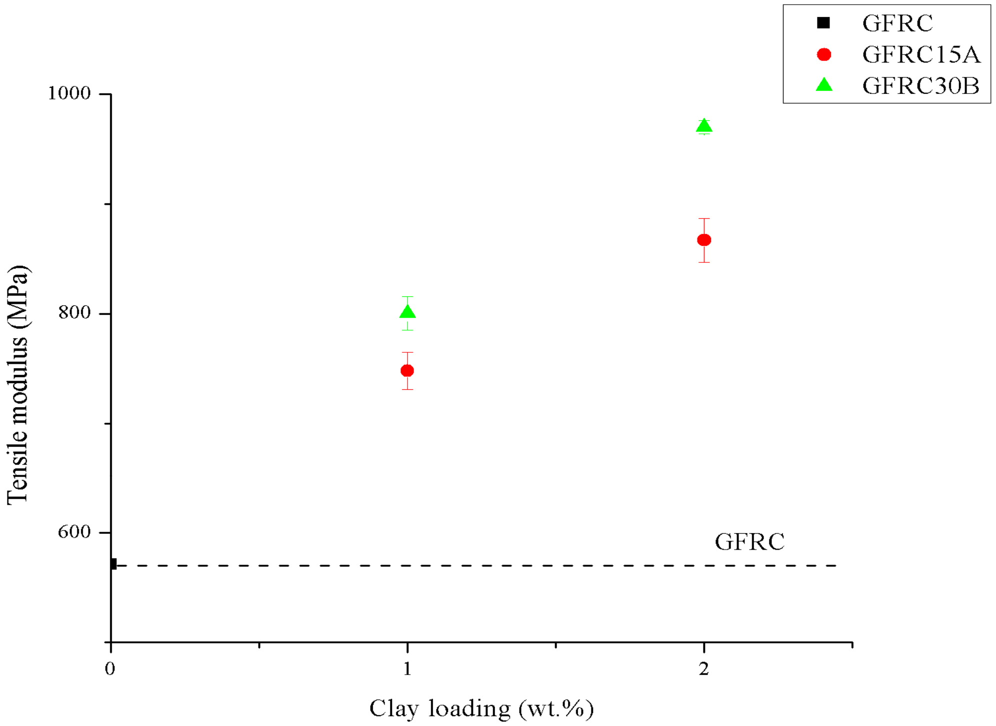


Fig. 5.4a Tensile modulus test results

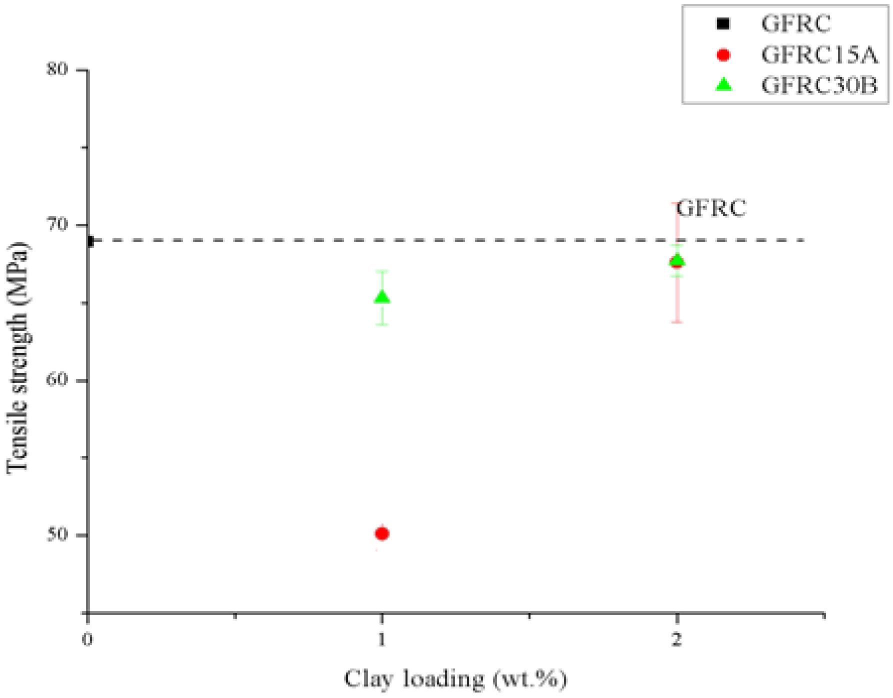
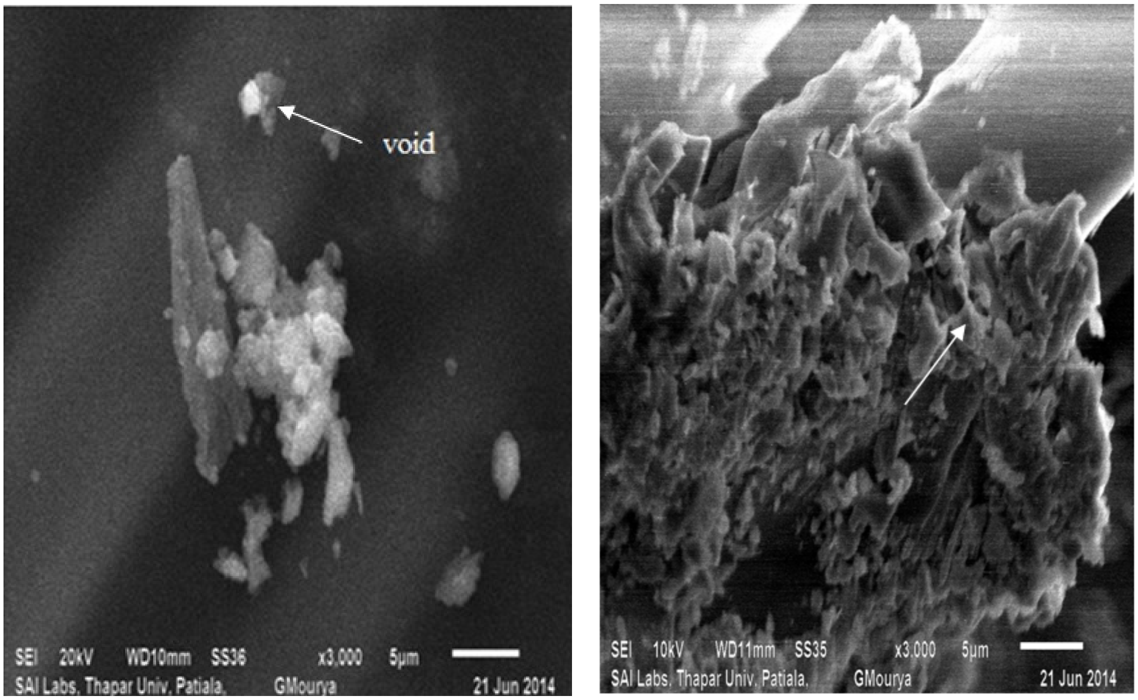


Fig. 5.4b Tensile strength test results

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 inturn 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.

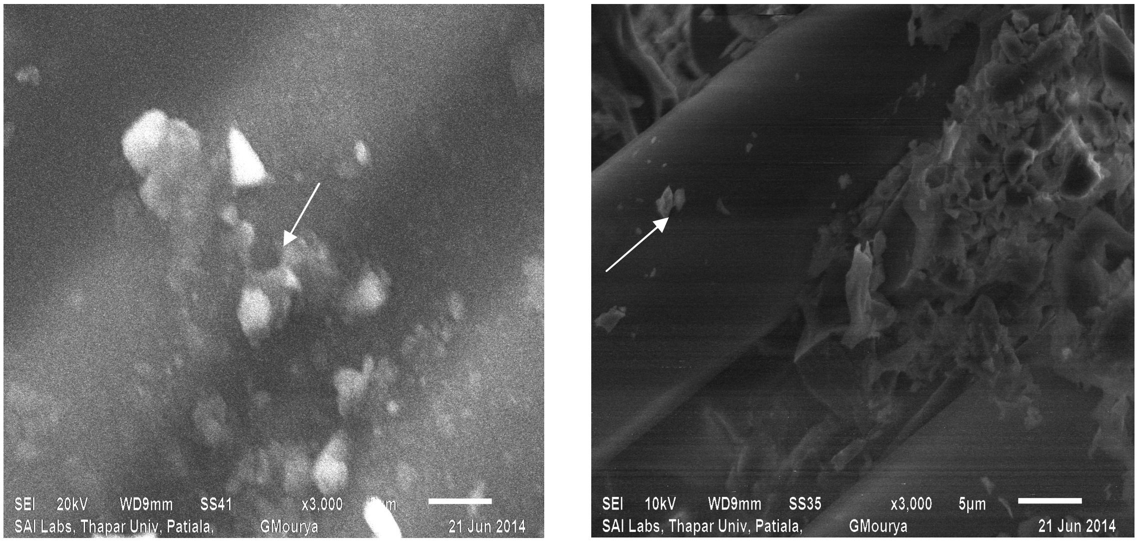
5.4 Scanning electron microscope (SEM)

Samples were mounted onto sample holder and fixed with adhesive carbon tape. Fig. 5.5 and fig. 5.6 illustrates the top surface morphology of nanoclay composite flat sheet. Voids are observed in fig. 5.5a. 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.



(a) **(b)**

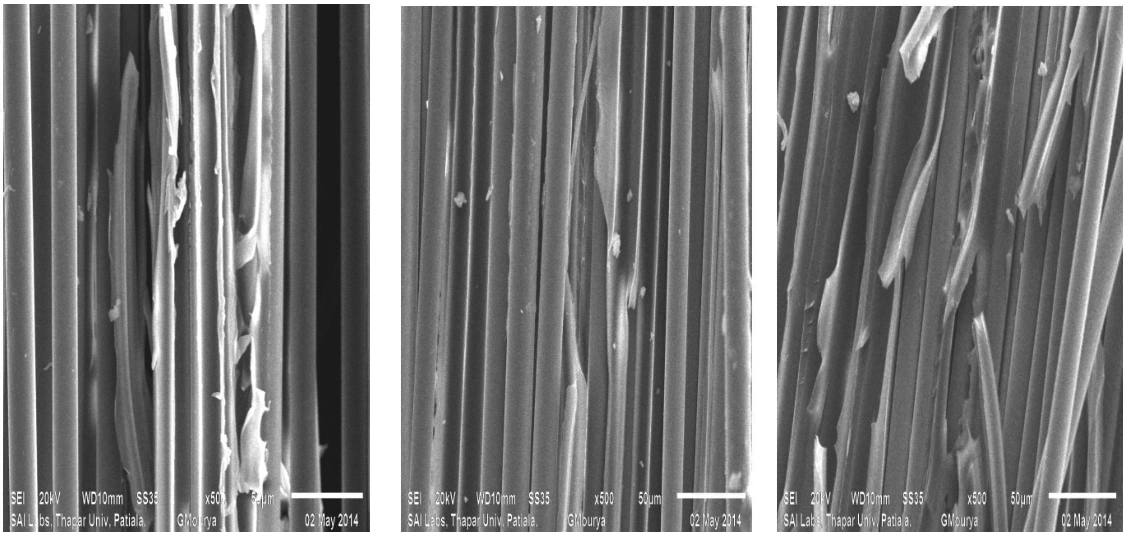
Fig. 5.5 SEM image of specimens having nanoclay Cloisite 15A® (a) 1 wt.% and (b) 2 wt.%



(a) (b)

Fig. 5.6 SEM image of specimens having nanoclay Cloisite 30B® (a) 1 wt.% and (b) 2 wt.%

In neat epoxy glass fiber composite smooth crack surface are framed because of weaker interfacial bonding in laminates as shown in fig. 5.7a. In contrast with an addition of nanclay a rough fracture surface are formed as shown in fig. 5.7b, c. The fracture modes indicate that an interface between nanoclay and epoxy is formed in these composites.



(a) (b) (c)

Fig. 5.7 SEM images of (a) 0 wt.% (b) 1 wt.% nanoclay Cloisite 30B® and (c) 1 wt.% nanoclay Cloisite 15A®

5.4.1 Particle size measurements

In image J software, after having set the scale and the threshold (the threshold was set to a uniform value for the whole image), the “Analyse Particles” features was used to generate a table of particles area and aspect ratio (output as major axis and minor axis). This then exported into Microsoft excel, were statistical analysis and histogram plotting were performed. It is assumed that particle distribution is calculated by taking ratio of major and minor axis. Fig. 5.8 shows a SEM image and associated particle size histogram (386 non touching particles counted by Image J),

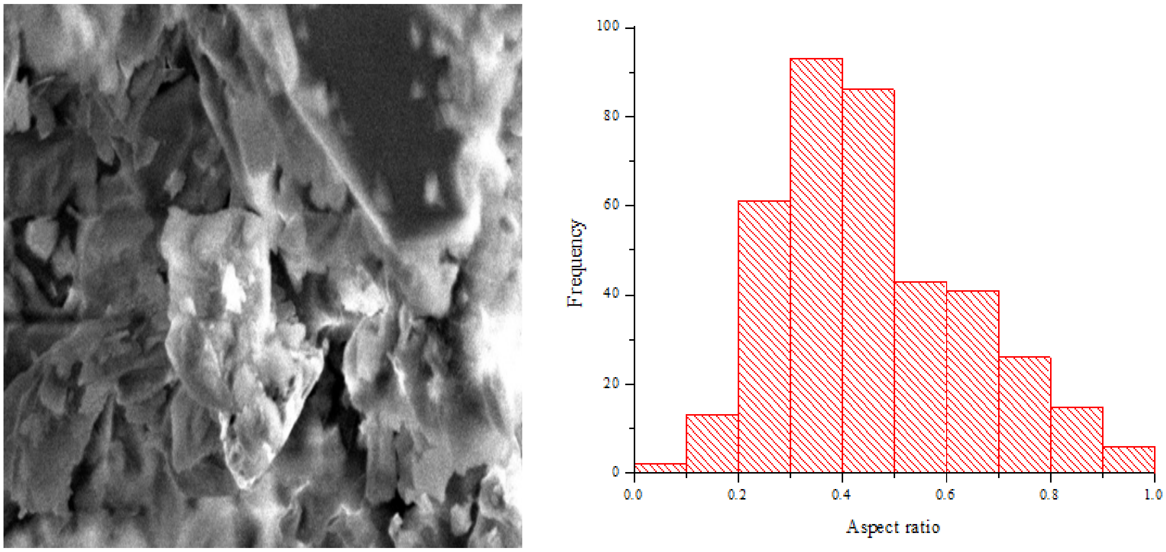


Fig. 5.8 SEM image and associated particle size histogram

5.5 X-ray diffraction

Comparison of interlayer distance between clay platelets (table 5.4) indicates that intercalated nanocomposites

Table 5.4 d_{001} spacing

	d_{001} spacing	
Clay loading	Cloisite 15A® [31.5 Å]	Cloisite 30B® [18.5 Å]
1 wt.%	43.6 Å	23.3 Å
2 wt.%	44.5 Å	24.12 Å

Compared to d_{001} spacing of 31.5 Å in Cloisite 15A®, the d spacing in the 1 wt.% and 2 wt.% increases to 43.6 Å and 44.5 Å. This suggests formation of intercalated morphology. Similar increase in d-spacing is observed for 1 wt.% and 2 wt.% Cloisite 30B®, suggesting an intercalated morphology.

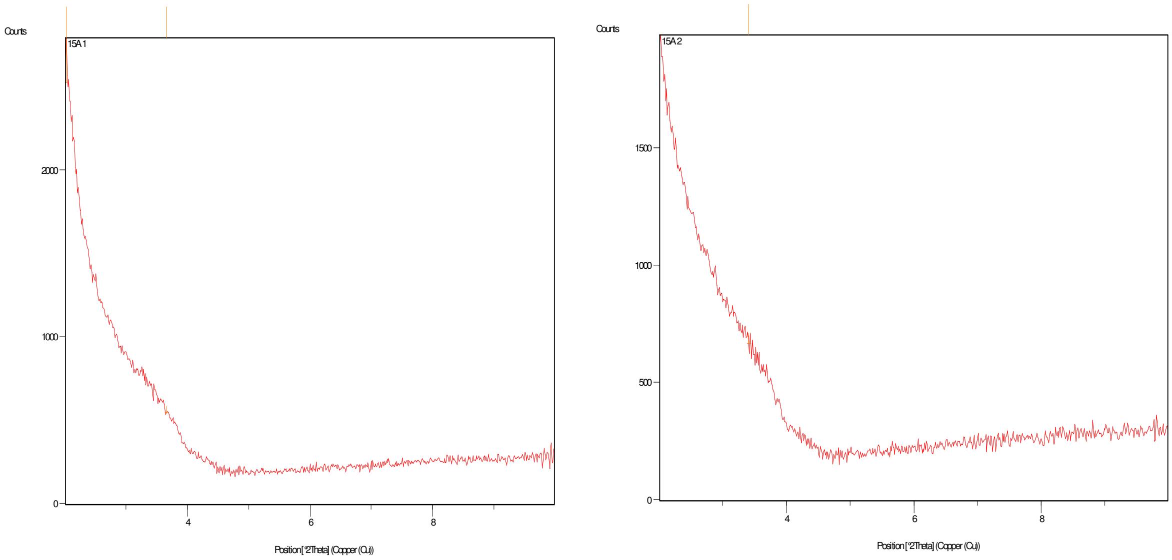


Fig. 5.9 XRD results of specimens with 1 wt.% and 2 wt.% loading of nanoclay Cloisite 15A®

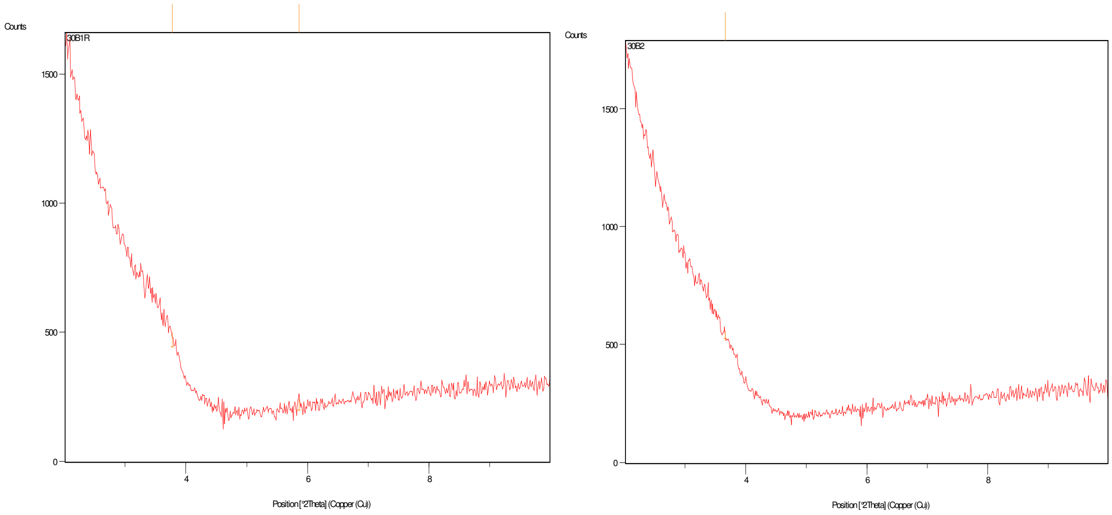


Fig. 5.10 XRD results of specimens with 1 wt.% and 2 wt.% loading of nanoclay Cloisite 30B®

CHAPTER 6 CONCLUSION AND FUTURE SCOPE

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.

6.2 Future scope of work

1. Experiments may be performed on laminates by stacking woven glass fibers.
2. Experiments can be repeated by changing the type of nanofillers.
3. Wear properties can also be ascertained.

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