

# **CARBON FIBER REINFORCED EPOXY COMPOSITES: EFFECTS OF NANO REINFORCEMENTS**

**A  
Thesis report**

**Submitted in partial fulfillment of the requirement  
for the award of degree**

**MASTER OF ENGINEERING  
in  
CAD/CAM & ROBOTICS**

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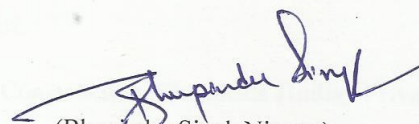
*Dedicated to my parents*

## CERTIFICATE


This is to certify that the work done in this thesis report titled “**Carbon Fiber Reinforced Epoxy Composites: Effects of Nano Reinforcements**” submitted in partial fulfillment of requirement for the award of Master of Engineering degree in CAD/CAM & Robotics in the Mechanical Engineering Department of Thapar University, Patiala, is an authentic record of work carried out by me under the guidance of **Mr. Bikramjit Sharma**, Assistant Professor, Mechanical Engineering Department and **Dr. Rajeev Mehta**, Associate Professor and Head, Chemical Engineering Department, Thapar University, Patiala.


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

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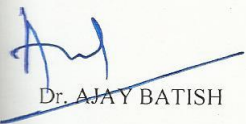
  
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This is to certify that above declaration made by the student concerned is correct to the best of our knowledge and belief.

  
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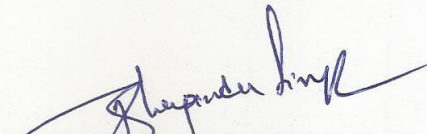
I am highly grateful to the authorities of Thapar University, Patiala for providing this opportunity to carry out the Thesis work.

I would like to express a deep sense of gratitude and thank profusely to my thesis guide **Mr. Bikramjit Sharma** for their sincere & invaluable guidance and suggestions which inspired me to submit seminar report in the present form. I am highly thankful to **Dr. Rajeev Mehta**, H.O.D, Chemical Engineering Department, Thapar University, Patiala for his invaluable guidance and making the work enjoyable and challenging.

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My special thanks are due to my family members and friends who constantly encouraged me to complete this study.

  
**BHUPINDER SINGH NIRMAN**

## ABSTRACT

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In present work, fiber reinforced nanocomposites have been manufactured using carbon fiber as reinforcement and epoxy modified with small quantities of multiwall carbon nanotubes(0.1 wt%, 0.5 wt% of resin) , Cloisite 30 B<sup>®</sup> nanoclay (2 wt%, 3 wt% of resin) and a hybrid composition (0.1 wt% MWCNT and 3wt% nanoclay, 0.5 wt% MWCNT and 3wt% nanoclay of resin). For uniform dispersion of nanofillers in epoxy matrix, mechanical stirring and ultrasonic mixing was done. The morphology of nanocomposites was studied using XRD and TEM. Two ply 0° fiber reinforced composites were manufactured using modified epoxy by hand layup method. Tensile and bending tests were performed on nanocomposites as per ASTM standards. It was found that the hardness of the nanocomposites increased with increasing MWCNT and nanoclay content. However, it was also seen that there was an optimal limit. The maximum value of hardness was found in the 0.5wt% of MWCNT and 3wt% of nanoclay samples. Tensile strength and flexural strength of the 2 wt% and 3 wt% nanoclay and content samples were improved. The maximum tensile strength was found in the 3 wt% nanoclay samples. The maximum tensile strain and flexural strength was found in the 0.5 wt% MWCNT samples.

## TABLE OF CONTENT

<b>S.No.</b>	<b>TITLE</b>	<b>PAGE No.</b>
	CERTIFICATE	I
	ACKNOWLEDGEMENT	II
	ABSTRACT	III
	TABLE OF CONTENT	IV-V
	LIST OF FIGURES	VI-VII
	LIST OF TABLES	VIII
	NOMENCLATURE AND ABBREVIATIONS	IX
<b>CHAPTER 1</b>		
1.0	INTRODUCTION	1
1.1	NANOCOMPOSITES	2
1.2	POLYMER NANOCOMPOSITES	4
1.3	EPOXY NANOCOMPOSITES	6
1.4	CLAY NANOPARTICLES	6
1.5	CARBON NANOTUBES	8
1.6	CARBON FIBER	10
<b>CHAPTER 2</b>		
2.0	LITERATURE REVIEW	12
<b>CHAPTER 3</b>		
3.1	GAPS IN LITRATURE	20
3.2	RESEARCH PROBLEM	20
<b>CHAPTER 4</b>		
	<b>EXPERIMENTATION</b>	21

4.1	FABRICATION OF SPECIMEN	21
4.2	EXPERIMENTAL SET-UP	22
4.3	TESTING METHODS USED IN EXPERIMENTATION	28
4.4	TEST MATRICES	33
<b>CHAPTER 5</b>		
	RESULTS AND DISCUSSIONS	34
5.1	MICRO HARDNESS	34
5.1.1	SPECIMEN FOR MICRO HARDNESS TESTING	34
5.1.2	X-RAY DIFFRACTION TEST	36
5.2	TENSILE TEST	40
5.3	3-POINT BENDING TEST	43
5.4	TRANSMISSION ELECTRON MICROSCOPY (TEM)	45
<b>CHAPTER 6</b>		
6.1	CONCLUSION	46
6.2	SCOPE OF FUTURE WORK	46
	<b>REFERENCES</b>	47

## LIST OF FIGURES

FIGURE NO.	TITLE	PAGE NO.
1.1	CLASSIFICATION OF COMPOSITES MATERIALS	2
1.2	NATURAL POLYMER NANOCOMPOSITES	3
1.3	THREE IDEALISED STRUCTURES OF POLYMER-CLAY COMPOSITE	7
1.4	SINGLE AND MULTIWALLED CARBON NANOTUBES	9
1.5(a)	ROLL OF CARBON FIBER	11
1.5(b)	MATTED CARBON FIBER	11
4.1	UNCOATED CARBON FIBER MAT	21
4.2	MULTIWALLED CARBON NANOTUBES	22
4.3	MECHANICAL STIRRER AND OIL BATH SETUP	23
4.4	ULTRASONICATOR	24
4.5	DISPERSION OF MWCNT INTO ETHANOL	25
4.6	MANUFACTURING OF FIBER REINFORCED NANOCOMPOSITES	27
4.7	MARBLE CUTTER	27
4.8	SPECIMEN DIMENSIONS FOR BENDING TEST	28
4.9	SPECIMEN DIMENSIONS FOR FLEXURAL TEST	28
4.10	ZWICK/ROELL UNIVERSAL TESTING MACHINE	29
4.11(a)	THREE POINTS BENDING TEST SETUP	30
4.11(b)	SPECIMEN POSITIONING	30
4.12(a)	MICRO HARDNESS TEST EQUIPMENT	30

4.12(b)	INDENT ON SPECIMEN	31
4.13(a)	SCHEMATIC REPRESENTATION OF X-RAY DIFFRACTION PRINCIPLE	32
4.13(b)	SCHEMATIC REPRESENTATION OF X-RAY DIFFRACTOMETER PRINCIPAL	32
5.1	LOADING POINTS IN SPECIMEN	34
5.2	MICRO HARDNESS VALUES AT DIFFERENT CLAY LOADING	35
5.3	XRD RESULT OF SPECIMEN WITH 2 WT% OF NANOCLAY LOADING	37
5.4	XRD RESULT OF SPECIMEN WITH 3WT% OF NANOCLAY LOADING	37
5.5	XRD RESULT OF SPECIMEN WITH 0.1 WT% OF MWCNT LOADING	38
5.6	XRD RESULT OF SPECIMEN WITH 0.5 WT% OF MWCNT LOADING	38
5.7	XRD RESULT OF SPECIMEN WITH 0.1 WT% OF MWCNT AND 3 WT% OF NANOCLAY LOADING	39
5.8	XRD RESULT OF SPECIMEN WITH 0.5 WT% OF MWCNT AND 3 WT% OF NANOCLAY LOADING	39
5.9	TENSILE STRENGTH OF SPECIMENS	41
5.10	TENSILE STRAIN OF SPECIMENS	41
5.11	FLEXURAL STRENGTH OF SPECIMENS	44
5.12	FLEXURAL MODULUS OF SPECIMENS	44
5.13	TEM IMAGES OF NANOCOMPOSITEs	45

## LIST OF TABLES

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<b>TABLE NO.</b>	<b>TITLE</b>	<b>PAGE NO.</b>
TABLE 4.1	SPECIMEN SPECIFICATIONS FOR TESTING	28
TABLE 4.2	INITIAL TEST SPECIMENS	33
TABLE 5.1	MICRO HARDNESS VALUES FOR SPECIMENS	35
TABLE 5.2	TENSILE PROPERTIES FOR DIFFERENT CLAY/CNT COMPOSITION	40
TABLE 5.3	3-POINT BENDING PROPERTIES FOR DIFFERENT CLAY/CNT COMPOSITION	43

## NOMENCLATURE AND ABBREVIATIONS

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<b>GFRP</b>	glass fiber reinforced polymer
<b>VLSI</b>	very large scale integration
<b>PNC</b>	polymer nanocomposites
<b>MMT</b>	montmorillonite clay
<b>OMMT</b>	montmorillonite organoclay
<b>CNF</b>	carbon nanofibers
<b>CNT</b>	carbon nanotubes
<b>SWNT</b>	single-walled carbon nano tube
<b>MWNT</b>	multi-walled carbon nano tube
<b>WAXD</b>	wide angle x-ray diffraction
<b>PAN</b>	polyacrylonitrile
<b>SEM</b>	scanning electron microscope
<b>TEM</b>	transmission electron microscope
<b>CTE</b>	coefficient of thermal expansion
<b>VGCF</b>	vapor grown carbon fiber
<b>CAI</b>	compression after impact

# CHAPTER 1

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## 1.0 INTRODUCTION

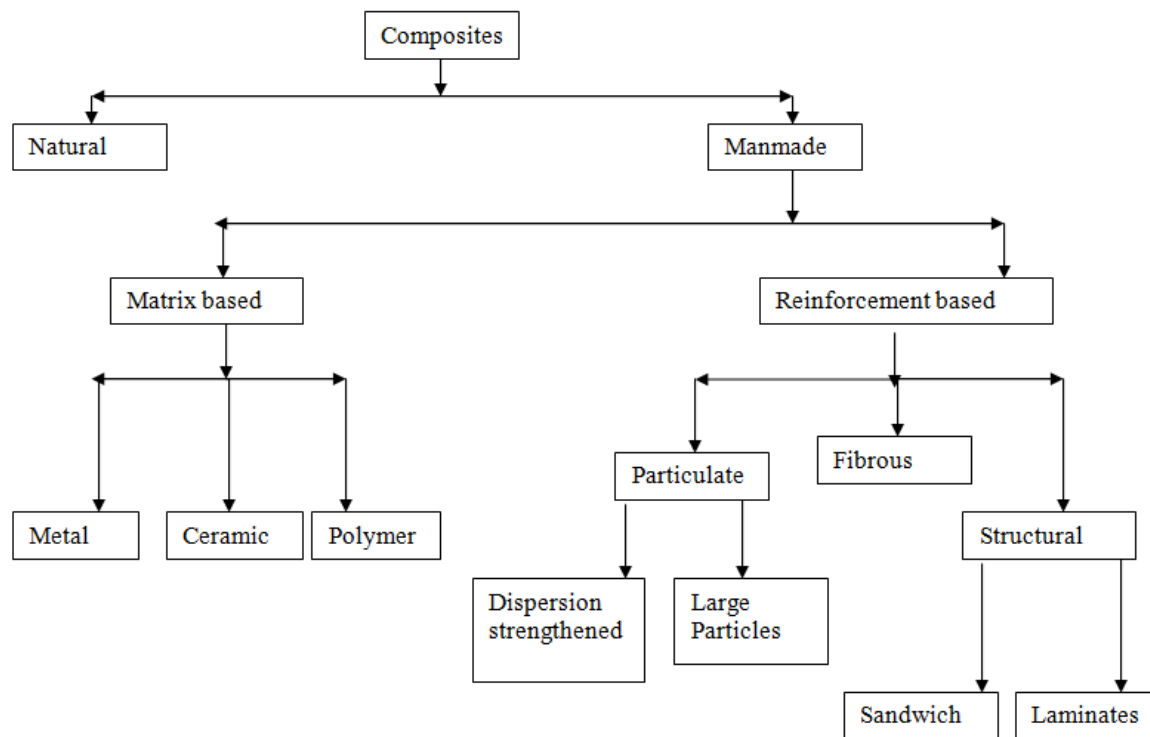
Nanotechnology is the design, characterization, production and application of structures, devices and systems by controlling shape and size at nanometers. Nanotechnology is the manipulation of matter on an atomic and molecular scale. Nanotechnology works with materials, devices, and other structures with at least one dimension sized from 1 to 100 nanometer, where 1 nm equals 1 billionth, or  $10^{-9}$ , of a meter. Materials with dimensions on the nanometres scale ( $10^{-9}$  meter) often have properties dramatically different from their bulk-scale counterparts [Sakaki, 1994]. Nanotechnology may be able to create many new materials and devices with a vast range of application such as in medicine, electronics, biomaterials and energy production. At nano level, some compounds transform from inert to active, from insulator to conductors, from fragile to tough. They can become lighter, stronger and more resistant. These transformed properties are what account for the infinite potential application of nanoparticles. The major factors of alter these properties, is the increase in the ratio of surface area to volume. Hence, due to the high surface-to volume ratio associated with nanometre sized particles it is possible to control the fundamental properties of materials through the surface/size effect. There is a variety of nano materials and range of their properties and possible applications appears to be enormous, from extra-ordinary tiny electronics devices, including miniature batteries, to biomedical uses and as components of parts of automobiles. In addition to this, in the field of VLSI, nanocomposites can be used as electromagnetic shielding between interconnects.

Nanotechnology is helpful in formation of various nano materials such as:

- Nanocomposites
- Nanofiber
- Nanoparticulate fillers

## 1.1 Nanocomposites

Nanocomposites are the new class of materials that are the combination of two phase materials in which one of the phase dispersed in the matrix has dimension on nanometre scale. Traditional composites materials are made by using the 10-20% of reinforced materials. Nanocomposites can show significant improvement on the addition of 1-2% of reinforcement materials.



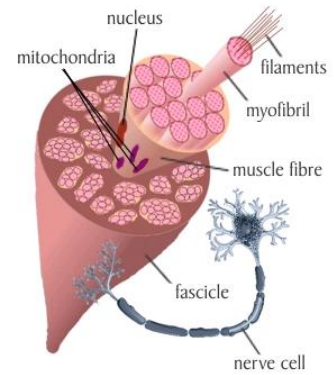
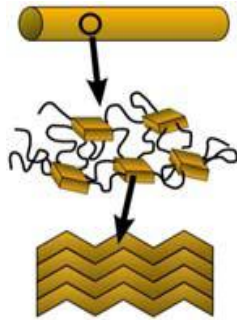
**Fig. 1.1: Classification of composite materials [30].**

Nanocomposites are also commonly found in nature, for example in the structure of abalone shell and bone. The use of nanoparticle-rich materials long predates the understanding of the chemical and physical nature of these materials. Nature also shows some of the most amazing forms of nanocomposites. For example, inside a typical spider silk (as strong as steel on a weight basis), one finds assembled crystalline particles separated by amorphous protein linkages. Mother of pearl or nacre is another natural nanocomposite formed by regularly stacked and layered calcium carbonate platelets bonded together by proteins. Resembling fiber reinforced composites, wood consists of nanoscale cellulose fibers bonded together with lignin and other carbohydrate constituents. In all these examples, nature has demonstrated enormous success over

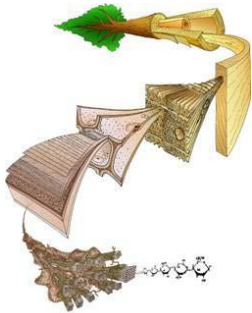
eons of evolution by utilizing bio-matrix materials to bond and support organized nano-particles or nano-fibrils to attain superior or specialized properties.



Spider silk



Structure of muscle



Wood

**Fig.1.2: Schematic views of natural polymer nanocomposites: spider silk, muscle, wood [27]**

The nanocomposite differs from the conventional composites due to their exceptionally high surface to volume ratio of reinforcing phase or its exceptionally high aspect ratio. The composite materials are made up of particles, sheets or fibers. Jose-Yacaman *et al.* (1996) investigated the origin of the depth of color and the resistance to acids and bio-corrosion of maya blue paint, attributing it to a nanoparticle mechanism. From the mid 1950s nanoscale organo-clays have been used to control flow of polymer solutions (e.g. as paint viscosifiers) or the constitution of gels (e.g. as a thickening substance in cosmetics, keeping the preparations in homogeneous form). By the 1970s polymer/nano composites were the topic of textbooks, although the term "nanocomposites" was not in common use. Ajayan *et al.* (1992) found that with polymer

nanocomposites, properties related to local chemistry, degree of thermo set cure, polymer chain mobility, polymer chain conformation, degree of polymer chain ordering or crystalline can all vary significantly and continuously from the interface with the reinforcement into the bulk of the matrix.

The reinforcement must have dimensions in nano-scale, for it to be considered as nanocomposites material.

### **Advantages of nanocomposites:**

- Improve mechanical strength.
- Improved gas barrier properties for the same film thickness.
- Reduced weight for the same performance.
- Higher chemical resistant.

### **1.2 Polymer nanocomposites**

The term “polymer nanocomposites” describes the number of multicomponent system, in which the primary component is the polymer and the filler material has at least one dimension below 100 nm. Polymer nanocomposites are generally light in weight and require low filler loading and are easy to process. Rapid advancements in nanocomposites technologies realized the new classes of nanoscale fillers continue to emerge. Nanoparticles commonly dispersed in polymers are: Layered silicate clay, carbon nano fibers, carbon nanotubes and graphite nanoflakes. The nano particles listed above are different in aspect ratio, morphology, chemistry and aggregate size. The nanoparticles chosen for dispersion are dependent on application. Nanomaterials require that the nano particles are well dispersed throughout the matrix. Due to dispersion of nano scale inorganic particles into organic resin materials, three distinct morphologies such as phase separated, intercalated and exfoliated regions are typically observed in nanocomposites structures. Among those, intercalated and exfoliated structures are mostly desirable for improving the performance of the material. The most commonly used nanofillers are:

1. Montmorillonite organoclays (OMMT)
2. Carbon nanofibers (CNFs)
3. Carbon nanotubes (CNTs)
4. Metallic nanoparticles
5. Others

*Mechanical properties:*

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

*Optical properties:*

- Nanocomposites are optically more transparent in comparison to conventional composites.
- Special optical effects.

*Magnetic properties:*

- In some case composites with magnetic nanoparticals have the magnetoresistence and magnetic permeability much higher than in the case of conventional composites.
- It is possible to make composites with magnetic threshold concentration smaller than electrical percolation threshold.

### **1.3 Epoxy Nanocomposites**

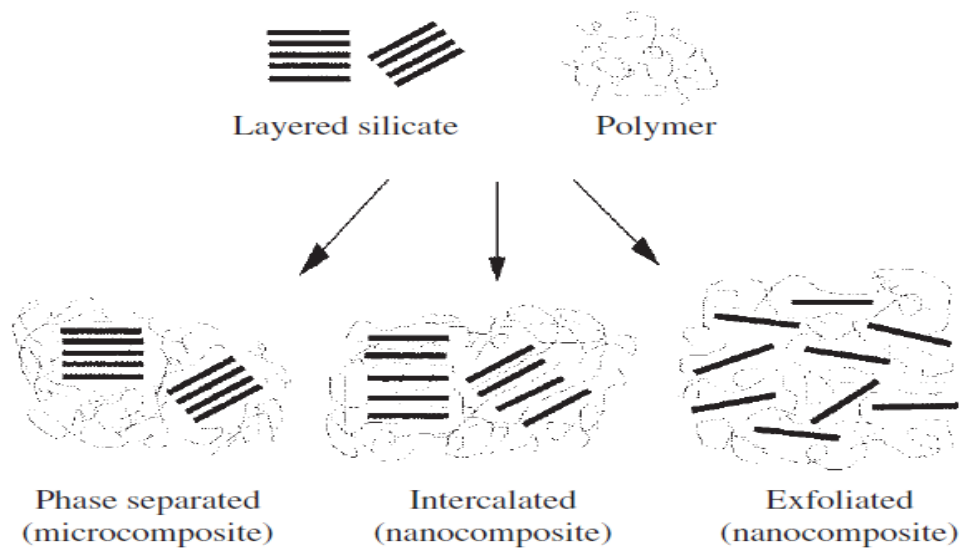
Epoxy is a thermoset material which is widely used in many industries. The applications of epoxy resin includes: coating, electrical, automotive, marine, aerospace and civil infrastructure as well as tool fabrication and pipes and vessels in the chemical industry. Due to low density of epoxy resin, which is around  $1.3 \text{ g/cm}^3$  and good adhesive and mechanical properties, epoxy resin became a promising material for high performance applications in the transport industry, usually in the form of composite materials such as fiber composite or in honeycomb structures. In the aerospace industry, epoxy-composites material can be found in various part of the body and structure of military and civil aircrafts, with the number of applications on the rise. The term 'epoxy resin' refers to the polymer and its cured resin/hardener system. The former is a low molecular weight oligomer that contains one or more epoxy groups per molecule (more than one unit per molecule is required if the resultant material is to be cross-linked). The characteristic group, a three-member ring known as epoxy, epoxide, oxirane, glycidyl or ethoxyline group is highly strained and therefore very reactive. Epoxy resins can be cross-linked through a polymerization reaction with a hardener at room temperature or at elevated temperature. Curing agents used for room temperature cure are usually aliphatic amines, whilst commonly used higher temperature, higher performance hardener are aromatic amines and acid anhydrides. An increasing number of specialized curing agents, such as poly-functional amines, polybasic carboxylic acids, mercaptans and inorganic hardener are also used. All of these results in different, tailored properties of the final polymer matrix. The cured resin systems of higher temperature have improved properties, such as higher glass transition temperatures, strength and stiffness, compared to those cured at room temperature.

### **1.4 Clay nanoparticles**

Clay as nanoparticles such as smectic clays (e.g. MMT) are incorporated into polymers to form resulting polymer nanocomposites, which may possess unique electrical, mechanical and optical properties. The clay as filler is expected to strengthen the mechanical properties of PU even upon lower loading of filler. Fiber retardancy, barrier resistance and ion conductivity are expected to be influenced by loading of clay as filler. Polymer/clay nanocomposites comprise

organic/inorganic hybrid polymer matrices containing platelet-shaped clay particles that have sizes of the order of a few nanometers thick and several hundred nanometers long. Partly because of their aspect ratios and high surface areas, the clay particles, if properly dispersed in the polymer matrix at a loading level of 1 to 5 wt. %, impart unique combinations of chemical properties and physical that makes these nanocomposites attractive for making films and coatings for a variety of industrial applications. Relative to the unmodified polymer, the polymer/clay nanocomposites may exhibit improvements in strength, modulus, and toughness, tear, radiation, fire resistance, and lower thermal expansion and permeability to gases while retaining a high degree of optical transparency.

According to the structure three different types of clay-polymer composites can be distinguished (Fig. 1.3).



**Fig. 1.3: Three idealized structures of polymer-clay composites [27].**

- When the matrix polymer chains are unable to penetrate between the layers of the silicate particles a conventional composite is formed.
- Intercalated structures are formed when one or more polymer chains intercalate between the layers. Hereby the interlayer spacing is increased but the ordered layer structure of the clay particles is retained as can be observed by wide angle X-ray diffraction (XRD).
- In exfoliated composites the clay particles are completely delaminated and the silicate layers do not show any periodicity in their arrangement.

The most common type of nanoclay is montmorillonite (MMT), a layered alumino-silicate in the smectite family of clay. Clay minerals such as talc and mica that have been used as fillers for years, MMT can be delaminated and dispersed into an individual layers only one nanometer thick by about 70 nm to 150 nm across. Dispersing clay in the polymer is very difficult and therefore a compatibilizing agent is commonly used. Compatibilizing agent is a molecule constituted of one hydrophilic and one organophilic function. The compatibilizing agents allow dispersing layered silicates in polymers by substitutions of the metallic cations between the silicate layers.

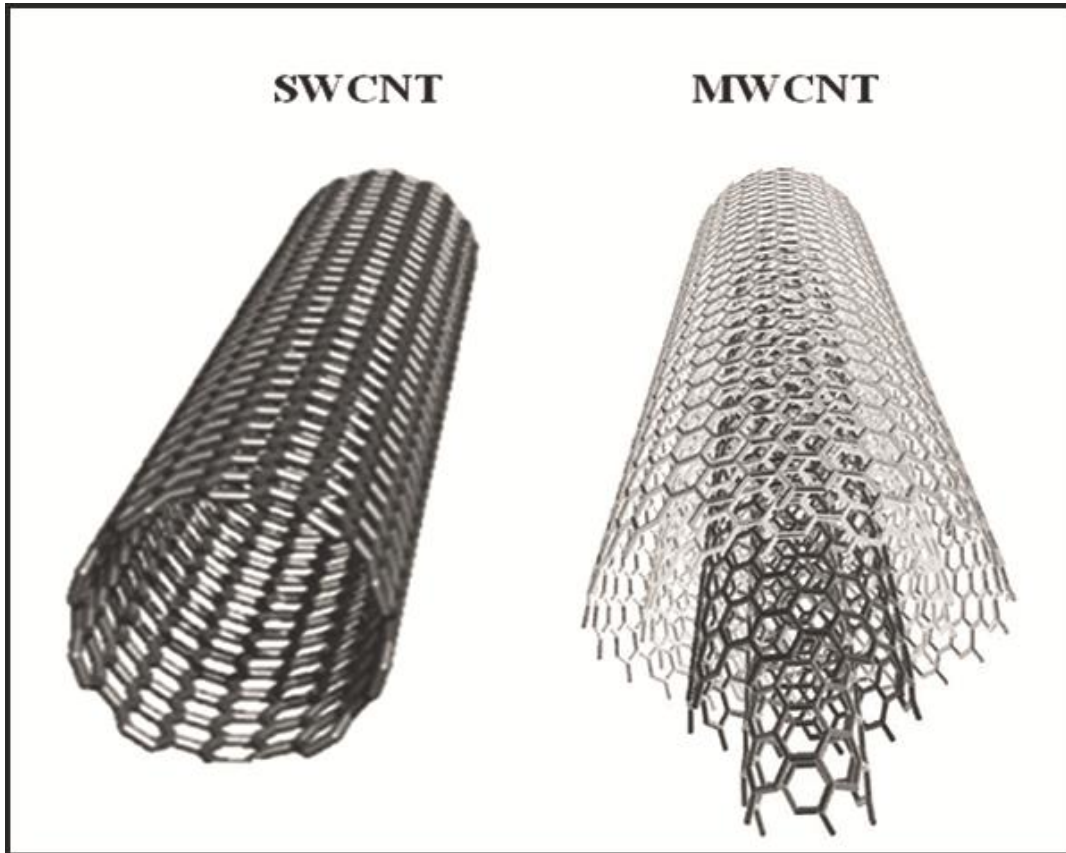
### **1.5 Carbon nanotubes**

Carbon nanotubes have long been synthesized as products of an action of a catalyst on the gaseous species originating from the thermal decomposition of hydrocarbons. Carbon nanotubes (CNTs) have been the focus of considerable research since Iijima confirmed their structure in 1991. The first evidence that the nanofilaments produced in this way were actually nanotubes having an inner cavity. They can be found in the transmission electron microscope micrographs published by *Radushkevich* and *Lukyanovich* in 1952.

A carbon nanotube (CNT) is a seamless hollow cylindrical structure with a diameter of 1–2 nm, comprising a hexagonal network of carbon atoms, one or both ends of the nanotubes being capped with a hemispherical fullerene molecule. In general, CNTs take two forms, namely single-walled nanotubes (SWNTs) and multi-walled nanotubes (MWNTs). SWNTs are single cylindrical layers of this carbon atoms network, whereas MWNTs consist of several layers in the form of concentric cylinders bounded by weak Vander Walls forces. Due to the covalent bonding between the carbon atoms similar to that of graphite, CNTs exhibit a high surface area to volume ratio and hence very high tensile strength (typically 100–600 GPa), elastic modulus (typically 1–5 TPa ) and compressive strength. They are extremely flexible due to their ability to reversibly absorb large stresses by deforming through twisting and buckling motions without fracturing.

Carbon nanotubes have been the focus of a lot of research work for nearly two decades now. Considering this investment of time and money, relatively few nanotube applications have reached the market yet. The basic structure of both SWNT and MWNT is shown in Fig 4. The basic structure of both SWNT and MWNT is derived from a planar graphene sheet. A SWNT can be imagined to be a sheet that has been wrapped into a seamless cylinder. Multiwall

nanotubes are essentially multiple SWNTs of different sizes that have formed in a coaxial configuration. Although MWNTs are typically tens of nanometers in diameter. The spacing between the layered shells in the radial direction of the cylindrical nanotube is approximately 0.34 nm.



**Fig. 1.4: Single walled Carbon nanotube and Multi walled Carbon nanotube[11].**

### **Functionalization of CNT**

For a nanocomposite, a good dispersion of the filler within the host matrix is highly desirable. At the same time it is also important to stabilize the dispersion to prevent reaggregation of the filler. These tasks are particularly challenging in case of nanofillers since the extremely large surface area leads to a strong tendency to form agglomerates. CNTs are very well known to form aggregates during compounding and hence various techniques have been used to overcome this problem, like use of sonication or mechanical mixing during the fabrication of nanocomposite

which generally help in dispersing CNTs. But the most effective way to resolve this problem is surface functionalization of CNTs. Surface functionalization helps in stabilizing the dispersion, since it can prevent re-aggregation of nanotubes and also leads to coupling of CNT with polymeric matrix. Coupling between CNT and polymer matrix is also very important for efficient transfer of external stress to nanotubes. In recent years, various methods have been developed for surface functionalization of CNT which includes functionalization of defect groups, covalent functionalization of sidewalls, non-covalent functionalization, e.g., formation of supramolecular adducts with surfactants or polymers. Although surface functionalization leads to significant improvement in CNT dispersion and stress transfer but this method also causes deterioration of intrinsic properties of CNTs. Covalent functionalization often lead to a tube fragmentation and the non-covalent functionalization results in poor exfoliation. Alteration of CNT properties lead to poor reinforcement and conductivity. Hence, it becomes obvious that dispersion and stabilization are not simple issues and compromises have to be made depending on the applications.

### **Applications of MWNTs and SWNTs**

Potential applications of carbon nanotubes are predicted for electrical, electronic, mechanical, and chemical applications, such as field emission, flat-panel display source, quantum wires, X-ray source, contact needle of atomic force microscopy, hydrogen storage, gas sensors, capillarity-induced filling of metal, and fullerene peapods. Highly crystallized MWNTs and SWNTs produced by arc discharge should have more potential for electronic and mechanical applications. Application-based research of SWNTs and MWNTs is continuously increasing worldwide.

### **1.6 Carbon Fiber**

Carbon fiber is another fiber reinforcement used in FRP. One of the most common methods of manufacturing carbon fiber is the oxidation and thermal pyrolysis of polyacrylonitrile (PAN), to give so called PAN-based carbon fibers. This material consists of thin fibers about 5-10  $\mu\text{m}$  in diameter and comprises mostly of carbon atoms. The carbon atoms are bonded together in microscopic crystals that are mostly aligned parallel to the long axis of the fiber. This alignment makes the fiber show very high tensile properties. Carbon fibers have lower density but higher

tensile strength and elastic modulus as compared to glass fibers. Due to these properties carbon fiber is an ideal reinforcement for composite materials used in aircraft components, high-performance vehicles, sporting equipment, wind generator blades, and other high demand, high performance applications. Roll and the mat of carbon fiber is shown in the figure (Fig 5,6). However, increased performance comes at a price since carbon fiber costs much more than glass fiber.



**Fig. 1.5(a): Roll of carbon fiber [26].**



**Fig 1.5(b): Matted carbon fiber [26].**

In this chapter an extensive literature review of the subject is presented.

**Sharma *et al.* (2012)** analyzed that in addition of layered silicate nanofillers in the polymer matrix has led to improvements in the elastic moduli, strength, heat resistance, decreased gas permeability and flammability. In this work epoxy modified with Cloisite<sup>®</sup> 30B nanoclay (at 1, 3 and 5 wt% of resin) and E-glass unidirectional fibers are used to prepare fiber reinforced nanocomposites using hand lay-up method. The nanocomposites have been characterized by XRD and SEM. XRD results show that the interlayer spacing between the clay platelets increased significantly indicating that the polymer is able to intercalate between the clay layers. The mechanical properties are measured by carrying out tensile, hardness and flexural tests and values are compared with those found for fiber reinforced neat epoxy composites. The tests show that an addition of nano-clay up to 3 wt% increases tensile strength and micro-hardness and there is a decrease in values with further clay addition up to 5 wt%. The flexural strength increased significantly with clay loading and the highest value is observed for specimens with 5 wt% of clay. 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.

**Ashrafi *et al.* (2011)** used single-walled carbon nanotubes (SWNT) to develop nano-modified carbon fiber/epoxy laminates. A functionalization technique based on reduced SWCNT was employed to improve dispersion and epoxy resin-nanotubes interaction. A commercial prepregging unit was then used to impregnate unidirectional carbon fiber tape with a modified epoxy system containing 0.1 wt% functionalized SWNT. Mechanical mixing and sonication were done. Incorporation of 0.1 wt% of SWCNT resulted in a 5% reduction in impact damage area, a 3.5% increase in compression after-impact strength, 13% increase in mode I fracture toughness and 28% increase in mode II interlaminar fracture toughness. SWNT are more effective in enhancing the mechanical performance.

**Hossain et al. (2011)** investigated the effect of seawater on the degradation of mechanical properties of conventional and nanophased carbon-epoxy composites. Epoxy resin was modified using 1 wt%, 2 wt%, and 3 wt% nanoclay. Carbon-epoxy composites were fabricated by vacuum assisted resin transfer molding process and compared with neat samples with and without exposure to seawater. Nanoclay was dispersed into matrix by using magnetic stirring. Mechanical characterization performed through three point bending tests showed that 2 wt.% nanoclay loading was optimum. Flexural strength and modulus were increased by 25% and 12.5%, respectively, compared to neat system for samples not exposed to seawater. Flexure samples exposed to the seawater for 30, 60, and 180-day periods revealed that samples with nanoclay retained better mechanical properties compared to neat samples. After 30-day exposure to seawater, there was no significant reduction in the tensile strength and modulus. However, flexural strength was reduced by 10.24%, 7.08%, 5.28%, and 7.13% for neat, 1 wt.%, 2 wt.%, and 3 wt.% nanoclay-infused samples, respectively, after the samples were exposed to seawater for 180-day. At the same time flexural modulus was reduced by 12.61%, 7.16%, 4.59%, and 6.11%, respectively. From scanning electron microscopy (SEM) studies, it was found that failure occurred due to delimitation and initiated from the compression side. Nanophased composites exhibited better bonding between fiber and matrix. SEM micrographs also revealed that both unconditioned and conditioned nanophased epoxy produce relatively rougher fracture surfaces compared to neat samples. Optical microscopy study revealed no significant physical change in outer surfaces of the samples conditioned up to a 90-day period.

**Kim et al. (2011)** investigated the effect of carbon nanotube (CNT) modifications on the flexural and wear behaviors of multiscale carbon/CNT/epoxy composites in this study. Carbon/epoxy woven composites and two types of multiscale carbon/CNT/epoxy composites were fabricated by incorporating woven-type carbon fibers into epoxy matrices modified with 2 wt% acid-treated and silane-treated multi-walled carbon nanotubes (MWCNTs). Mixing of CNTs in epoxy was done by a magnetic stirrer. The flexural moduli and strengths of carbon/epoxy composites improved by an addition of CNTs, and were better in silane treated CNTs/carbon/epoxy nanocomposite. The wear properties of carbon/epoxy composites were also improved by the addition and surface modification of CNTs.

**Davis et al. (2011)** studied the carbon fiber reinforced epoxy composite laminates for improvements in quasi static strength and stiffness and tension–tension fatigue cycling at stress-ratio (R-ratio) = +0.1 through strategically incorporating amine functionalized SWNT at the fiber/fabric–matrix interfaces over the laminate cross-section. In comparison to composite laminate material without carbon nanotube reinforcements there are modest improvements in the mechanical properties of strength and stiffness. A potentially significant increase is demonstrated for the long-term fatigue life of these functionalized nanotube reinforced composite materials. In this study amine functionalized SWNTs incorporated at the fiber/fabric–matrix interfaces of a carbon fiber reinforced epoxy composite laminate material showed improvements in the tensile strength and stiffness and resistance to T–T fatigue damage. At operating load levels fatigue life is projected to be several decades beyond that of the neat laminate material. Optical, scanning electron microscopy and Raman spectrometry confirmed the effectiveness of this strategy and the noted improvements in strength, stiffness and fatigue durability of the nanocomposites laminate materials.

**Singh et al. (2010)** reports on the durability of epoxy-clay nanocomposites upon exposure to multiple environments. Nanocomposites are fabricated by mixing the clay particles using various combinations of mechanical mixing, high-shear dispersion, and ultrasonication. Clay morphology is characterized using X-ray diffraction and transmission electron microscopy. Specimens of both neat epoxy and the epoxy-clay nanocomposite are subjected to two environmental conditions: combined UV radiation and condensation on 3-hour repeat cycle and constant temperature-humidity, for total exposure duration of 4770 hours. Both materials lose mass under exposure to combined UV radiation and condensation due to the erosion of epoxy by a synergistic process. Surprisingly, the epoxy-clay specimens exhibit greater mass loss, as compared to neat epoxy. Mechanical testing shows that either environment does not significantly affect the flexure modulus of either material. On the other hand, both materials undergo degradation in flexural strength when exposed to either environment.

**Sager et al. (2009)** studied the interfacial shear strength of carbon nanotube coated carbon fibers in epoxy by using the single-fiber composite fragmentation test. The carbon fibers were coated with carbon nanotubes (CNT) using thermal chemical vapor deposition (CVD). The purpose of the CNT coating was to potentially produce a multifunctional structural composite. 71%

Improvement in interfacial shear strength with an addition of nanotube coating. Ultimate tensile strength and tensile modulus values decreases 37% in the case of the radically aligned MWNTs and by 30% in the case of randomly oriented MWNTs.

**Quaresimin *et al.* (2007)** analyzed 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 was Cloisite<sup>®</sup> 30B. The organoclay was dispersed through a shear mixing process. Hand-layup method was used to prepare the specimens. The work showed that tensile modulus exhibited little difference between the unmodified laminates while a modest decrease was observed for the tensile strength for the VGCF (vapors grown carbon fiber) and nanoclay modified systems. The reason was that the “effective” clay concentration in the interlayer resin rich regions was much higher than the nominal nano-additive concentration.

**Shang-Lin *et al.* (2007)** performed experimental investigation of nanocomposites coatings for healing surface flaws of glass fibers and improving alkali-resistance. He found that, with low fraction of nano-reinforcements, the nanostructures and functionalized traditional glass fibers show significantly improved both mechanical properties and environmental corrosion resistance. The most remarkable mechanical strength improvement is found for glass fibers with nanotubes coatings, corresponding to the highest healing efficiency factor. No apparent strength variation appears for nanoclay coated fiber subjected to alkaline attack, which indicates that, the influence of moisture solvent uptake and concentration on mechanical properties decreases when the organoclay is dispersed in coating polymer. Overall, the hybrid nanocoatings cause improved fiber strength, corrosion resistance, and interfacial properties.

**Bekyarova *et al.* (2007 a)** explored functionalized single-walled carbon nanotubes (SWNTs) as nano reinforcement for carbon fiber/epoxy composites. SWNTs functionalized with carboxylic acid groups (SWNT-COOH) are dispersed in epoxy and used for infiltration of carbon fabric (CF) by the vacuum-assisted resin transfer molding technique to fabricate SWNT-COOH/epoxy/CF composites. Mechanical tests demonstrate that the incorporation of SWNT-COOH improves the mechanical performance of the composites and produces a 40% enhancement of the shear strength at a SWNT-COOH loading of 0.5 wt %. Carboxylic acid

functionalized SWNTs were of high purity, low metal content, and high nanotube length to diameter aspect ratio. The reinforcing role of the SWNTs is associated with the improved SWNT-epoxy interface due to cross linking of the carboxylic acid groups in the SWNTs with the epoxy matrix.

**Bekyarova *et al.* (2007 b)** report an approach regarding the development of advanced structural composites based on engineered multiscale carbon nanotube-carbon fiber reinforcement. Electrophoresis was utilized for deposition of nanotubes on carbon fibers vacuum-assisted resin transfer molding. Electrical conductivity improved with an introduction of single wall carbon nanotubes while it remained unaffected with introduction of multi wall carbon nanotubes (MWCNT). The 30% enhancement of interlaminar shear strength in MWCNT- fiber/epoxy hybrid composite as compared to that of carbon fiber/epoxy composites was achieved.

**Shen *et al.* (2007)** achieved functionalization with different amino groups of multi-walled carbon nanotubes. Nanotube-reinforced epoxy nanocomposites were prepared by mixing amino-functionalized multi-walled carbon nanotubes with epoxy resin. Differential scanning calorimetry, thermo-gravimetric analysis and bending tests were used to investigate the thermal and mechanical properties of the nanocomposites. The results showed that different kinds of amino-functionalized multi-walled carbon nanotubes increased the flexural strength and tensile strength of the nanocomposites.

**Wang *et al.* (2006)** investigated the effects of hydrothermal ageing on the thermo-mechanical properties of high performance epoxy and its nanocomposite. In this work epoxy-clay nanocomposite samples containing 2.5 wt% of clay were prepared through a “slurry-compounding” approach. The cured samples were immersed in distilled water at 60°C for different periods of time before subjecting to characterization. The hydrothermal effect on the thermal/mechanical properties of neat epoxy and epoxy-clay nanocomposite was studied. The moisture uptake significantly affects the modulus at high temperature, and the tensile strength. On the other hand, at low temperature, the modulus and fracture toughness were not strongly influenced. As the moisture content increased, there was a reduction in strain at break for the epoxy-clay nanocomposite while that of the neat epoxy remained constant. This effect was attributed to epoxy-clay interface debonding induced by water and formation of water cluster fillers that acted as defects in the composite.

**Yasmin et al. (2006)** prepared the clay/epoxy nanocomposites by shear mixing method and used the 1-10wt. % of clay to prepare the samples. The epoxy matrix was reinforced with MMT clay particles to fabricate clay/epoxy nanocomposites. They used bisphenol A as epoxy resin and methyl tetrahydrophthalic anhydride as the hardener. Two types of clay nanoparticles were used as the reinforcement, one was Nanomer I.28E and other was Cloisite<sup>®</sup> 30B. In this study Cloisite<sup>®</sup> 30B showed a homogeneous dispersion of nanoparticles throughout the cross-section compared to Nanomer I.28E/epoxy. As the clay content increased, the strain to failure decreased. It was also found that the modulus of the nanocomposites increased monotonically with increasing clay content. For 10wt.% of clay, the Cloisite<sup>®</sup>30B/epoxy showed an increase of 53% over the neat resin, where as the other showed an increase of about 22% at room temperature. The observation further confirmed the direct relation between the degree of exfoliation and the mechanical properties of these nanocomposites. Addition of clay also reduced the coefficient of thermal expansion of pure epoxy.

**Kaushik et al. (2006)** prepared an epoxy–short glass fiber composite by directly blending two-pack system of Araldite (CY-230) and hardener (HY-951) with short glass fibers. The short glass fiber content was varied from 2% to 10% by weight of the total matrix. These composites were then characterized for morphology, and resistance toward various chemicals. Tensile strength and flexural modulus increased with an increase in fiber content and was maximum for 10% fibers. The study could not be extended beyond this fiber fraction because of limitations in processability. Scanning electron micrographs reveal good bonding and uniform dispersion of glass fibers within the matrix. According to absorption studies, absorption increases with increase in fiber fraction and it was maximum for DMF. The samples were completely destroyed in concentrated sulfuric acid, pyridine, and nitric acid.

**Chow et al. (2005)** analyzed the water absorption and hygrothermal aging behavior of organomontmorillonite (OMMT) reinforced polyamide 6/polypropylene (PA6/PP ratio = 70/30), with and without maleated PP (MAH-g-PP), at three different temperatures (30, 60, and 90°C). The water absorption of the PA6/PP nanocomposites obeyed the Fickian law behavior. It was found that the equilibrium moisture content and the diffusion coefficients were dependent on the OMMT loading, MAH-g-PP concentration and immersion temperature. Tensile modulus and strength of PA6/PP nanocomposites deteriorated after exposure to hygrothermal aging. Water

acted as a plasticizer for the PA6/PP matrix and silicate layer of OMMT. The nanocomposites showed excellent retention ability and recovery properties under any immersion temperature. The MAH-g-pp enhanced the resistance of the nanocomposites against water immersion and also improved the resistance against hygrothermal attack.

**Chun-Ki Lam *et al.* (2004)** experimentally studied the hardness and inter-laminar shear properties of nanoclay/epoxy composites with different amount of nanoclay content, which formed different sizes of nanoclay/epoxy clusters after mixing in an extruder. The results showed that the micro-hardness of the composites could be enhanced even when a small amount of nanoclay was added to the epoxy. However, there was an optimal limit after which the hardness was dropped with an increase in the nanoclay content. Microscopic observation on the 18 fracture surfaces showed that the size of the clusters varied with the amount of nanoclay used in the composites.

**Gojny *et al.* (2004)** report an approach to nanocomposites consisting of double-wall carbon nanotubes (DWCNTs) and an epoxy matrix, which was produced by a standard calendaring technique. A standard shear-mixing technique was applied in order to disperse double-wall nanotubes in an epoxy resin. The prepared nanocomposites were investigated by transmission and scanning-electron microscopy in order to receive information concerning the achieved dispersion and the CNT-epoxy interface. The use of calendars seems to be an appropriate and mild technique to exfoliate and disperse carbon nanotubes in epoxies. The addition of only small amounts of carbon nanotubes leads to improved mechanical properties. In comparison to a carbon black filled epoxy, the nanotubes/ epoxy composites showed an increased tensile strength, fracture toughness and Young's modulus under retention of the ductility. A further increase of the mechanical properties of nanotube/epoxy-composites was achieved by a variation of the processing parameters. The qualitative observation of a better dispersibility and an improved interface of amino-functionalized nanotubes in an epoxy matrix and the obtained mechanical properties reveal the relevance of a functionalization of CNTs.

**Sinha Ray *et al.* (2003)** discussed the academic and industrial aspects of the preparation, characterization, materials properties and processing of polymer/layered silicate nanocomposites. Smectites are a valuable mineral class for industrial applications because of their high cation exchange capacities, surface area, surface reactivity and adsorptive properties. These composites

exhibited improved mechanical properties compared to conventional composites. The composites exhibited a remarkable increase in thermal stability, as well as self-extinguishing characteristics for flammability, such that the flammability of pristine polymers significantly reduced after nanocomposite formation with layered silicate.

**Thostenson *et al.* (2002)** synthesized carbon nanotubes on the surface of carbon fibers and the fiber/matrix interfacial properties were assessed using the single-fiber fragmentation test. Carbon nanotubes were grown directly on carbon fibers using chemical vapor deposition. Single fiber epoxy matrix composites were moulded. Presence of carbon nanotubes at the fiber/matrix interface improved the interfacial shear strength of the composites as observed in single fiber fragmentation test.

**Wang *et al.* (2001)** prepared polymeric nano-composites by melt intercalation method. The nanoclay was mixed with polymer in twin-screw extrusion. The clay-spacing in the composites was measured by X-ray diffraction (XRD). The morphology of the composites and its development during the extrusion process were observed by scanning electron microscopy (SEM). Melt viscosity and mechanical properties of the composites and the blends were also measured. It was found that the clay spacing in the composites is influenced greatly by the type of polymer used. Also the addition of the nanoclay can greatly increase the viscosity of the polymer when there is a strong interaction between the polymer and the nanoclay. The mechanical test showed that the addition of 5-10 wt% nano-clay largely increases the elastic modulus of the composites. The water absorption of nylon 6 is decreased with the presence of nano-clay. The effect of nano-clay on polymers and polymer blends was also compared with Kaolin clay under the same experimental conditions.

**3.1 Gaps in literature**

From the literature review it is found that previous work has mainly focused on the synthesis of fiber reinforced epoxy nanocomposites, study of morphology and unique properties of these new classes of materials. There is an important gap in the previous research work that no study in which both types of nanofillers (nanoclay and MWCNT) have been studied.

**3.2 Research Problem**

Nanoclay and nanotubes when added to fiber reinforced polymer individually lead to an improvement in several properties to different extents. It would be very interesting to find out what happens when both of them are added in different proportions to fiber reinforced polymers.

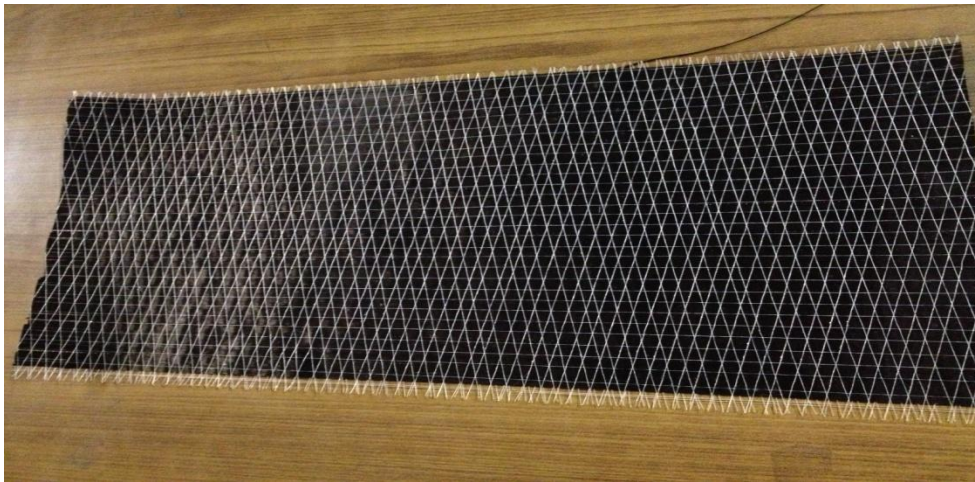
## 4.1 Fabrication of specimen

### 4.1.1 Materials

Unidirectional carbon fiber mat and two part epoxy resin purchased from Huntsman store Delhi. Organically modified nanoclay Cloisite 30B<sup>®</sup> was purchased from Southern clay products, Mumbai. Multiwall carbon nanotubes were purchased from Reinste Nano Ventures Private Limited, New Delhi.

#### 4.1.1.1 Carbon fiber Mat

For the experimentation, unidirectional carbon fiber with 0° fiber orientation and woven with polymer fibers was cut to fabricate composites (Fig. 4.1).



**Fig. 4.1: Carbon fiber mat used for making specimen**

#### 4.1.1.2 Multiwall carbon nanotube

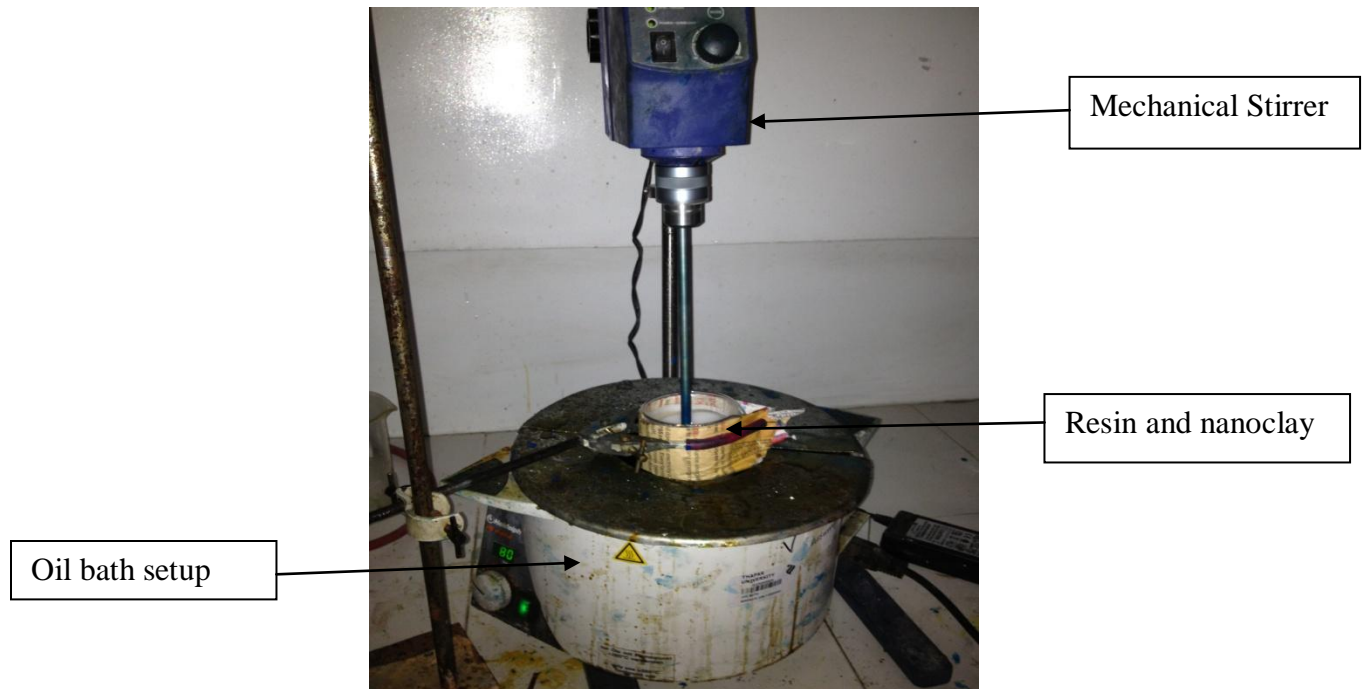
Multiwall carbon nanotubes commercially available with 95% purity and 10-20 nm dia and of 200 nm length were used for the experimentation.



**Fig. 4.2: Multiwalled Carbon Nanotubes**

#### 4.2.1 Mixing of nanoclay into epoxy (base):

- **Mechanical stirring:** Epoxy base is a transparent thick fluid. It is quite difficult to disperse nanofillers into it without vigorous stirring. An oil bath and remi-stirrer setup as shown in figure.4.3 was used for mixing. The oil bath was used to heat the epoxy to the desired (80°) temperature, so that the viscosity of epoxy base is reduced. High speed stirring of epoxy/nanoclay mixture resulted in a better dispersion of clay.



**Fig. 4.3: Mechanical stirrer and an oil bath used for proper mixing of nanoclay**

Different weight percentages of clay- 2wt% and 3% were added and stirred at a temperature of 80° C for 2 hours.

- **Ultrasonic mixing after mechanical stirring:** Sonication is an act of applying sound energy to agitate particles in a sample, for various purposes. In the laboratory, it was carried out using an ultrasonic bath. Sonication was done for evenly dispersion of nanoparticles into liquids and better intercalation of epoxy into nanoclay galleries. Ultrasonic mixing was performed for 2 h.



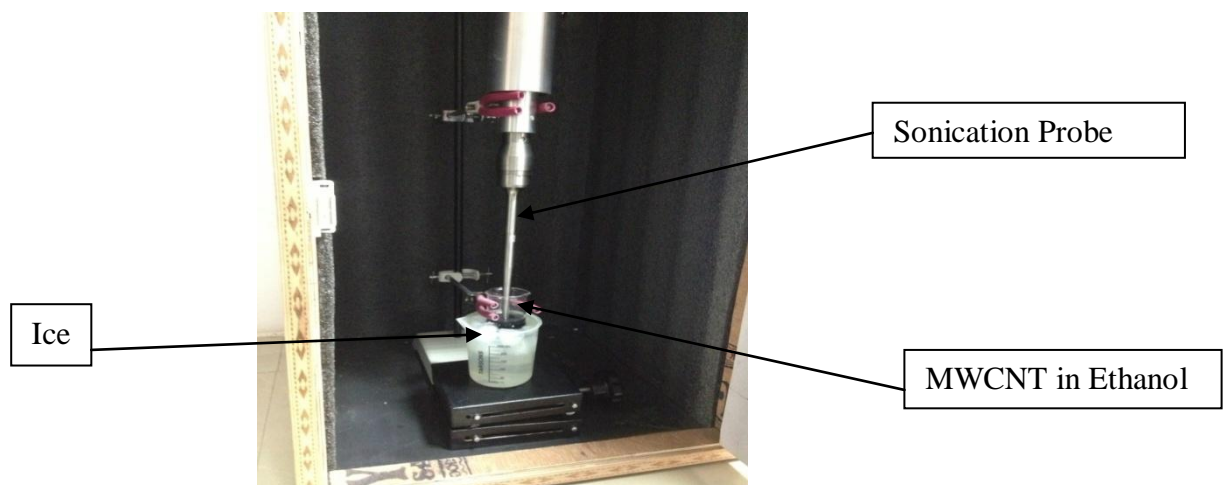
**Fig. 4.4: Sonication being done for evenly dispersing nanoparticles**

#### **4.2.2 Mixing of epoxy base solution with hardener**

After ultrasonication, the solution is mixed with the hardener in the ratio 10:1 by volume. After mixing, mechanical stirring up to 15 minutes was done.

#### **4.2.3 Mixing of MWCNT into epoxy (base):**

MWCNT's in different weight percentages (0.1 wt.%, 0.5 wt.%) were dispersed in the resin. The necessary quantities were first dispersed in ethanol in an ultrasonic bath at room temperature for 1 h.



**Fig. 4.5: Dispersion of MWCNT into ethanol with Probe**

After mixing the ethanol-based solution of MWCNT's into the resin, the suspensions were stirred for 1 h at 2000 rpm. During the stirring process, the temperature of the resin was kept at 80°C using a silicon oil bath in order to maintain a low viscosity of the resin. The mixtures were then stirred again for 1 h at 2000 rpm. After adding the hardener, the mixtures were stirred at 2000 rpm for 15 min.

#### **4.2.4 Manufacturing of fiber reinforced nanocomposites:**

The mixture was poured on to the carbon fiber mat and applied uniformly using the hand layup method. Care was taken during the process so that no air is entrapped in the resin and a uniform thickness of coating is maintained on the ply.



**Fig. 4.6: Manufacturing of fiber reinforced nanocomposites**

#### 4.2.5 Cutting of sheet for samples

Once the epoxy was fully cured, the sheet was cut to required size using the marble cutter (Fig. 4.7).



**Fig. 4.7: Marble cutter**

#### 4.2.6 Specimen specifications

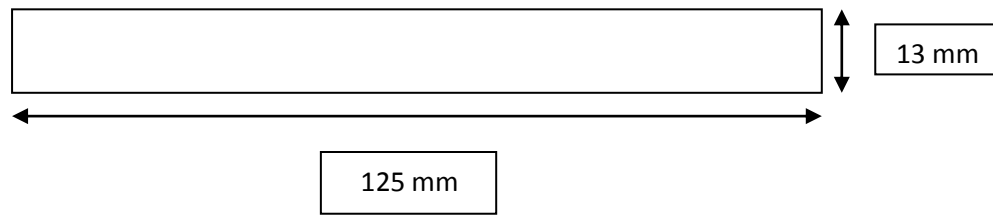
The specimen had been cut and prepared as per the ASTM D3039 and ASTM D790 for tensile and bending tests respectively.

**Table 4.1: Specimen specifications for testing (ASTM D3039 and ASTM D790)**

Parameters for specimen	Specimen for tensile testing	Specimen for flexural testing
Length	125 mm	125 mm
Width	15 mm	13 mm
Thickness	3 mm	3 mm

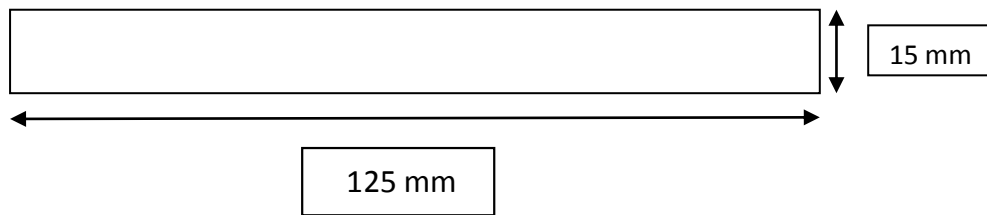
#### 4.2.7 Specimen dimensions

- For bending test



**Fig. 4.8: Specimen dimensions for bending test**

- For tensile test



**Fig. 4.9: Specimen dimensions for tensile test**

### 4.3 Testing methods used in experimentation

#### 4.3.1 Tensile testing

A Universal tensile testing machine is shown in Fig. 4.10 was used for the testing of the FRP specimen under tensile load. The specimens were tested until they break indicating the peak load and ultimate stress value.



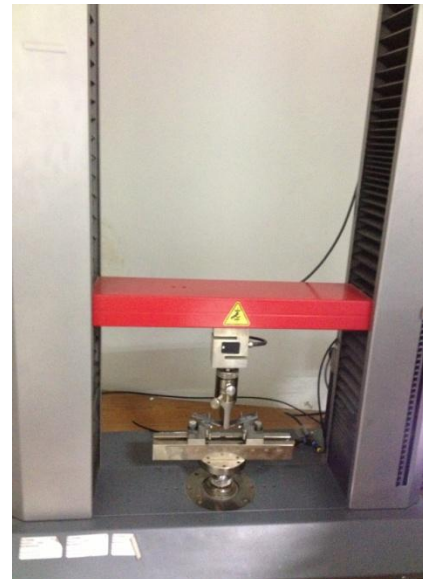
**Fig. 4.10: Zwick/Roell Universal Testing Machine**

### 4.3.2 Three point flexural test

Three point bending tests of specimen were carried out in Zwick/Roell universal testing machine (Fig. 4.11(a) and Fig. 4.11(b)).



**Fig. 4.11(a): Three points bending test setup**



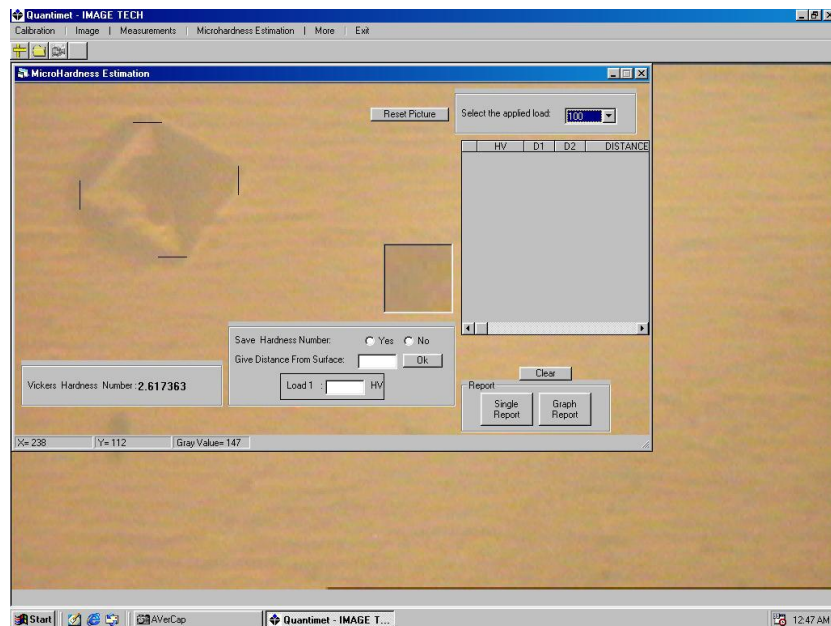
**Fig. 4.11(b): Specimen positioning**

### 4.3.3 Micro hardness testing

Micro hardness test (shown in Fig. 4.12) was conducted on specimen with Vicker's hardness testing setup.



**Fig. 4.12(a): Micro hardness test equipment**



**Fig. 4.12(b): Indent on specimen**

The load applied was 100 gm and VHN values were determined by applying this load by using a calibration distance of 50 units in Quantimet software as shown in Fig. 4.12 used for image

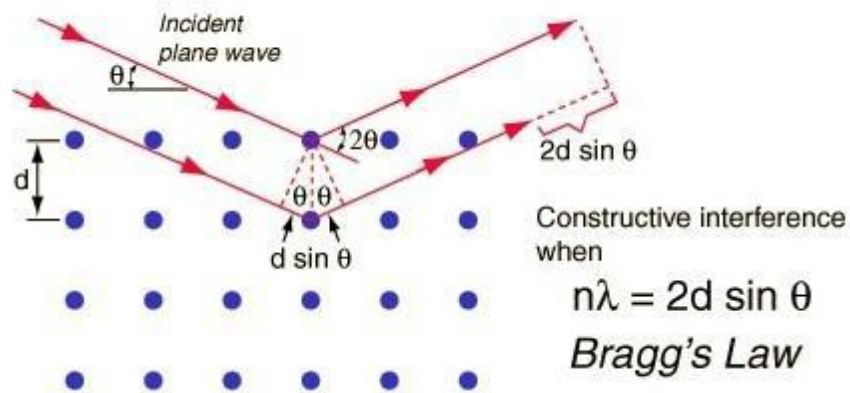
analyzing. The dwell time used during load application was 20 seconds. An indent is formed in diamond shape used for calculating VHN as shown in figure.

#### 4.4.6. X-Ray Diffraction Test

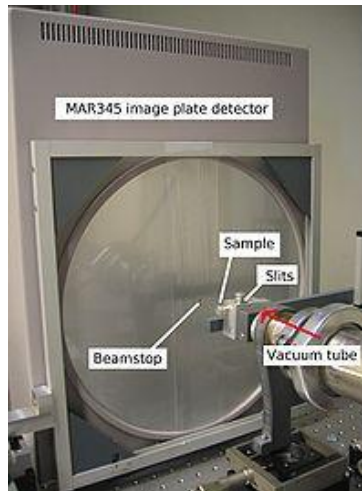
**X-ray scattering techniques** are a family of non-destructive analytical techniques which reveal information about the crystallographic structure, chemical composition, and physical properties of materials and thin films. These techniques are based on observing the scattered intensity of an X-ray beam hitting a sample as a function of incident and scattered angle, polarization, and wavelength or energy. X-ray diffraction was used in this study to investigate 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  $\lambda$  is the wavelength of the incidence x-ray source,  $d$  is the spacing in question,  $\theta$  is  $\frac{1}{2}$  of  $2\theta$  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. 4.13).



**Fig. 4.13(a): Schematic representation of x-ray diffraction principle.**



**Fig. 4.13(b) Schematic representation of x-ray Diffractometer principal**

To evaluate the degree of exfoliation in the polymer, XRD measurements were carried out in a Panalytical X-ray diffractometer with Cu K $\alpha$  radiation ( $\lambda=1.54\text{\AA}$ ) with a scanning speed of 1 $^\circ$ /min and at 45 kV and 40mA. During the XRD experiments, the samples were analyzed in reflection mode. All XRD scans were through  $2\theta$  of  $0^\circ$  to  $32^\circ$ .

#### 4.4 Test Matrices

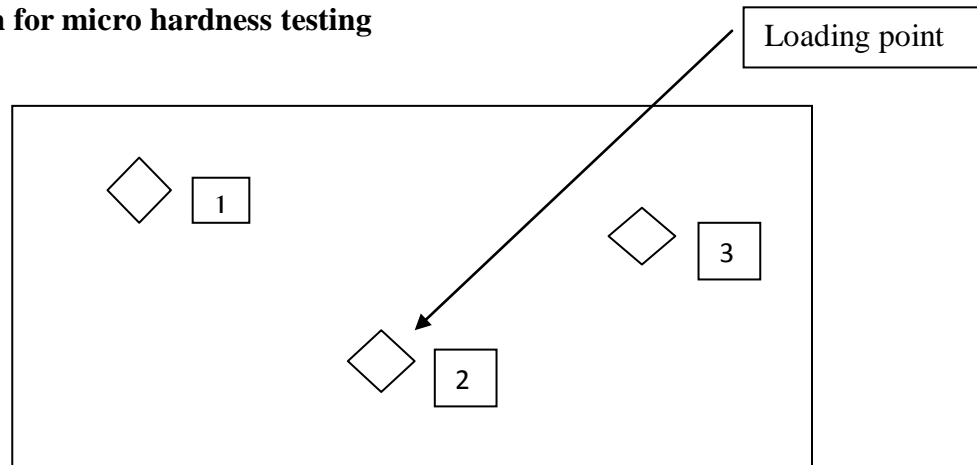
**Table 4.2: Initial Test Specimens**

Specimen Name	No. of Specimen		Total Specimen
	Tensile	Bending	
0 wt% clay	2	2	4
2 wt% clay	2	2	4
3 wt% clay	2	2	4
0.1 wt% MWCNT	2	2	4
0.5 wt% MWCNT	2	2	4
0.1wt% MWCNT and 3 wt% clay	2	2	4
0.1wt% MWCNT and 3 wt % clay	2	2	4

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

## 5.1 Micro hardness

### 5.1.1 Specimen for micro hardness testing

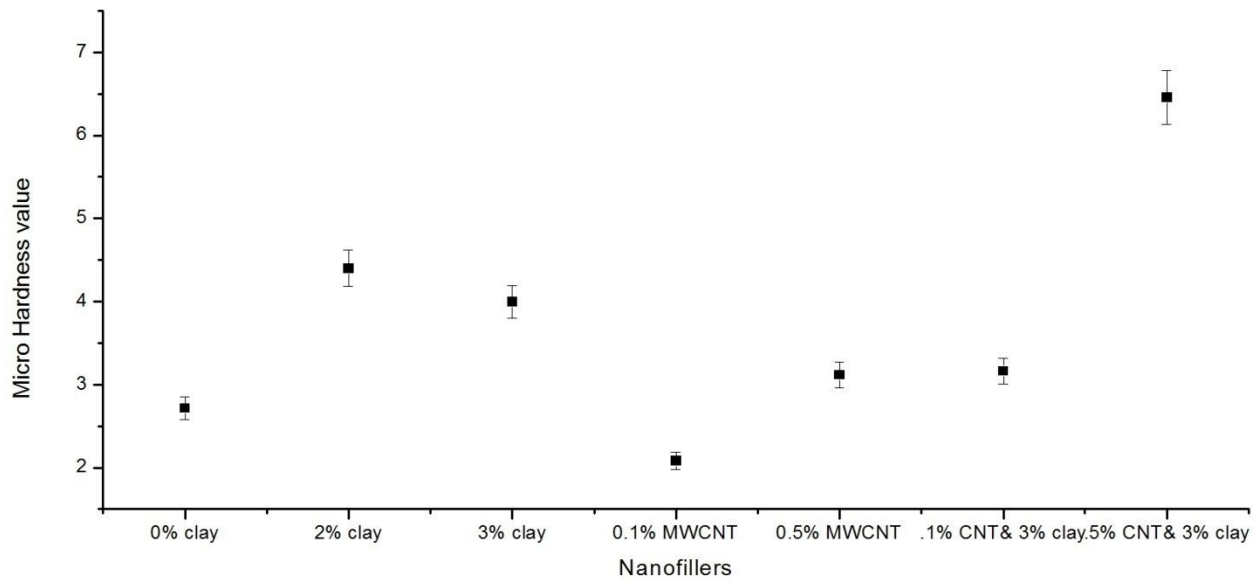


**Fig. 5.1: Loading points in specimen**

The micro-hardness of specimens containing different nanofillers was measured. Table 5.1 shows the experimental measurements of micro hardness of the nanocomposites. An average hardness was calculated from three indentation measurements on a composition. A plot of the results obtained on each type of specimen is shown in fig. 5.2. A 0% specimen is a neat epoxy, fiber reinforced composite.

**Table 5.1: Micro hardness values specimens**

Clay and CNT Loading Points	Micro hardness values						
	0% clay	2% clay	3% clay	0.1% CNT	0.5% CNT	0.1% CNT & 3% clay	0.5% CNT & 3% clay
Point 1	2.657	5.082	3.934	2.012	2.872	3.456	7.833
Point 2	2.695	4.139	4.227	2.201	3.725	2.253	3.920
Point 3	2.794	3.983	3.827	2.032	2.752	3.774	7.632
Average	2.715	4.400	3.996	2.081	3.116	3.161	6.461

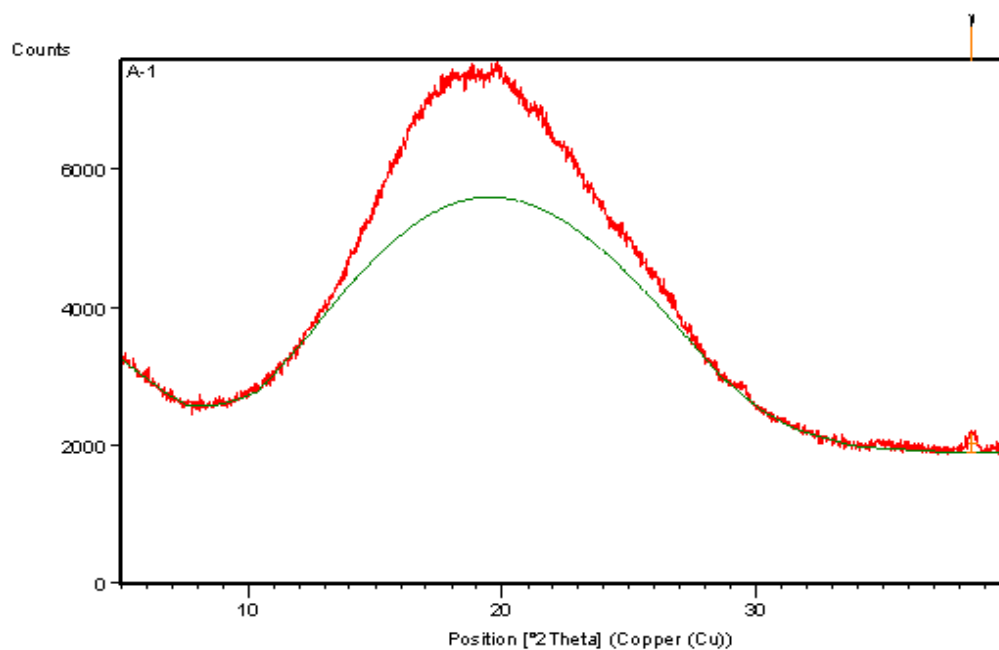


**Fig. 5.2: The Vicker's hardness value of specimen as a function of weight percentage of nanoclay and MWCNT in epoxy resin**

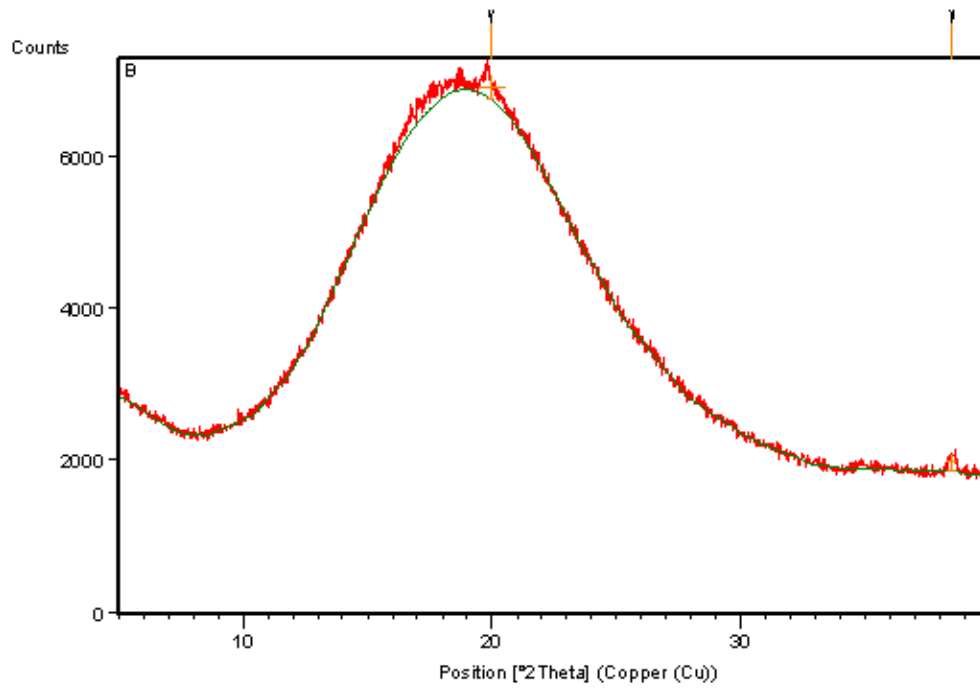
The hardness of specimen increased with addition of 2 wt% of nanoclay. A decline of the hardness also appears on further increasing the nanoclay content after 2 wt %; the hardness decreases in a drastic manner from 4.4 Hv (2 wt% of nanoclay) to 3.99 Hv (3 wt% of nanoclay). An addition of 0.1 wt% of MWCNT resulted in decrease in the hardness below than the neat epoxy composites. The maximum hardness has been measured where the nanotube and nanoclay content is 0.5 wt% of nanotubes and 3 wt% of clay.

### 5.1.2 X-ray diffraction test

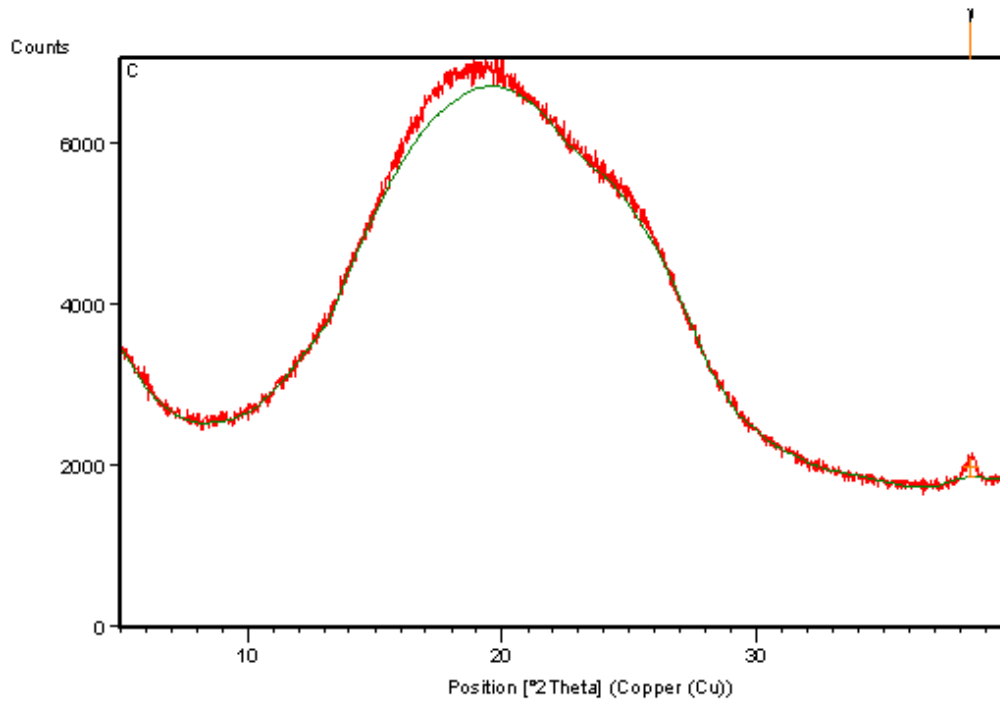
The X-ray diffraction experiments were conducted on the specimens having different nanoclay loadings. X-ray diffractometer gives the values of d-spacing and  $2\theta$  for different specimens of epoxy clay and epoxy clay and MWCNT nanocomposites. An increase in the interlayer spacing leads to a shift of the diffraction peak towards the lower angle. The diffraction peak of Cloisite 30B is at an angle  $2\theta=4.84$  and corresponding d-spacing value is  $d=18.26$ .



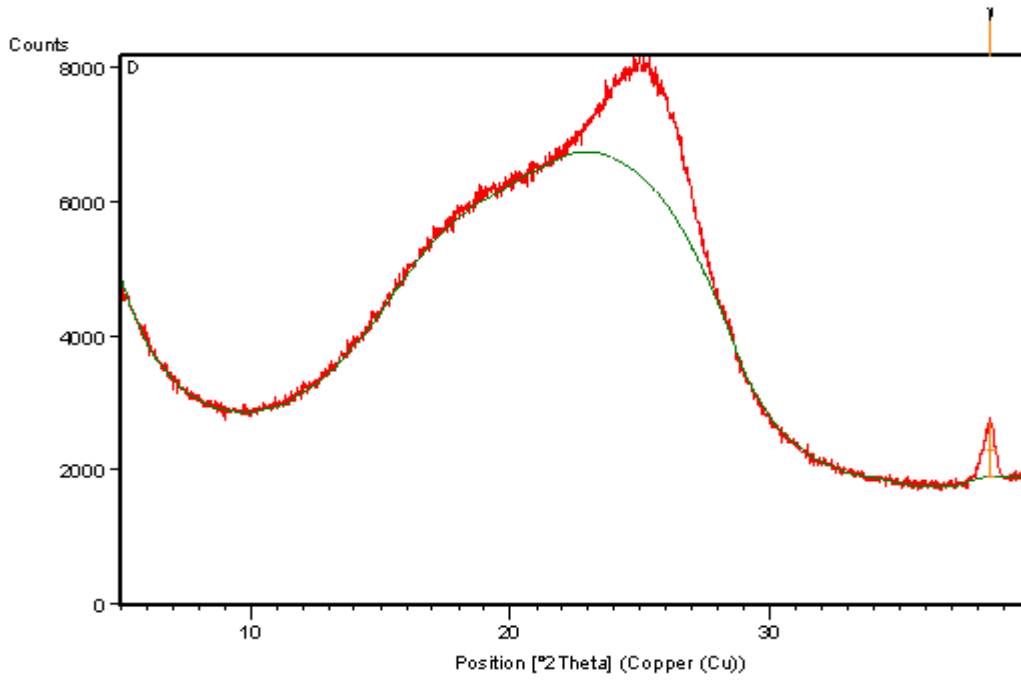
**Fig. 5.3: XRD result of specimen with 2 wt% of nanoclay loading**



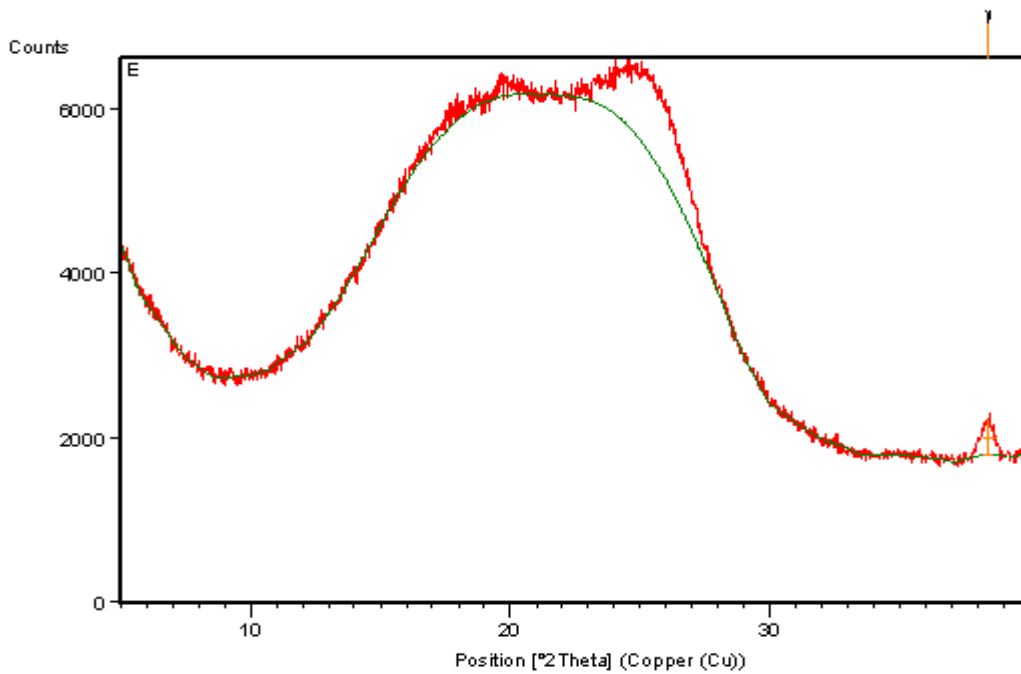
**Fig. 5.4: XRD result of specimen with 3wt% of nanoclay loading**



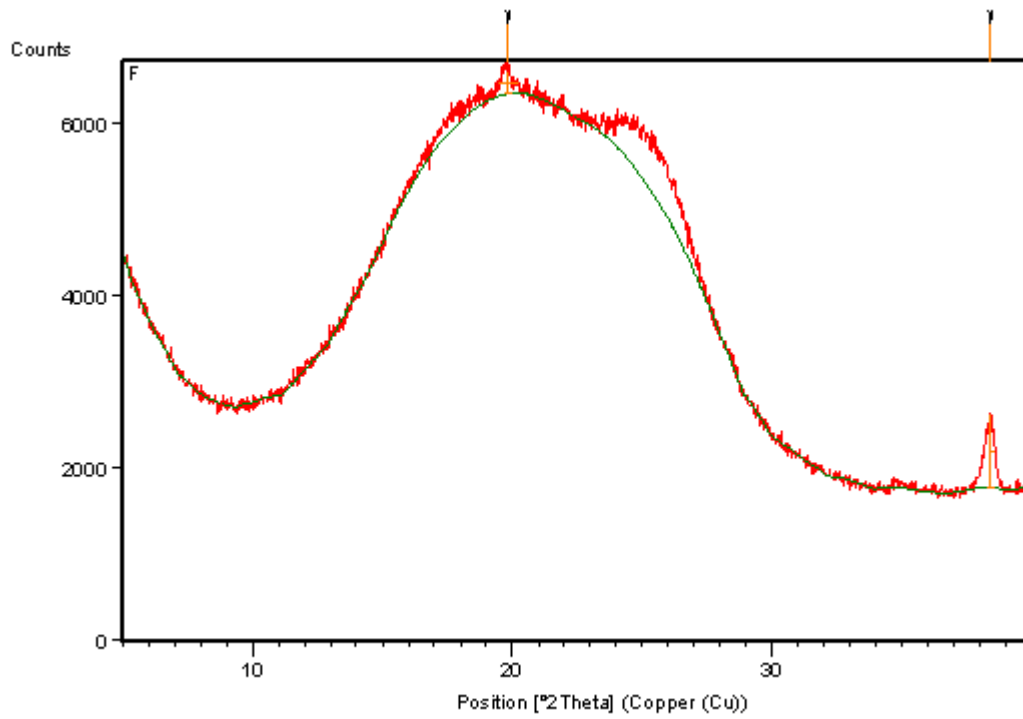
**Fig. 5.5: XRD result of specimen with 0.1 wt% of MWCNT loading**



**Fig. 5.6: XRD result of specimen with 0.5 wt% of MWCNT loading**



**Fig. 5.7: XRD result of specimen with 0.1 wt% of MWCNT and 3 wt% of nanoclay loading**



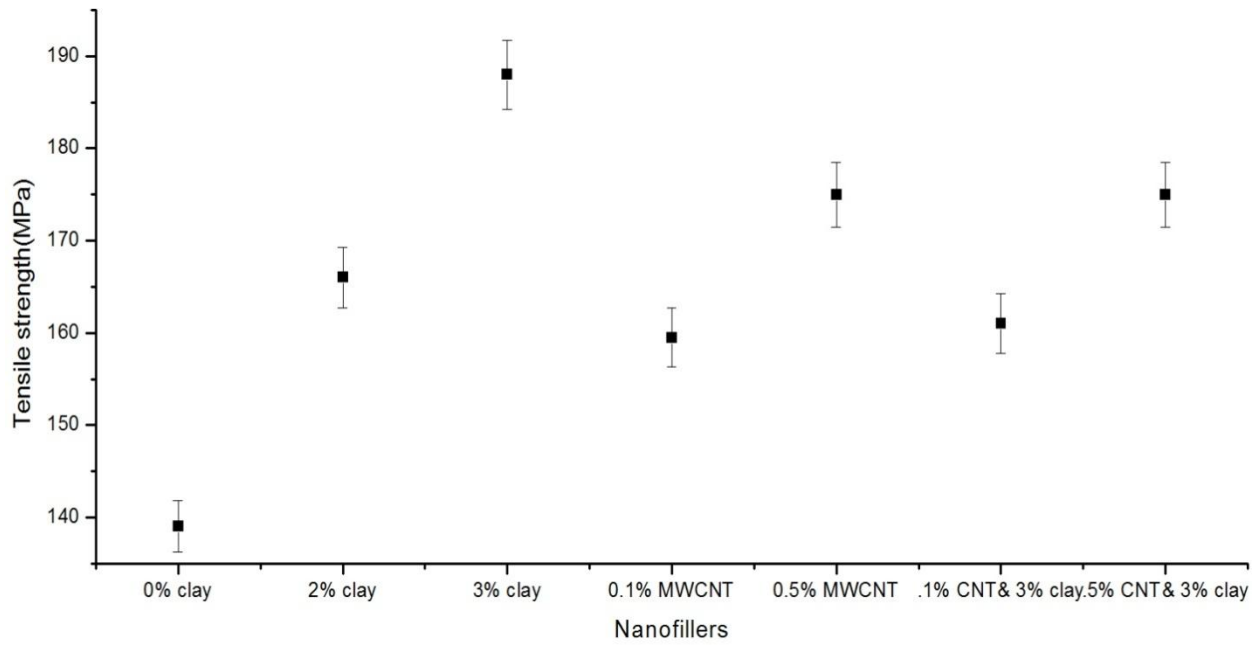
**Fig. 5.8: XRD result of specimen with 0.5 wt% of MWCNT and 3 wt% of nanoclay loading**

## 5.2 Tensile test

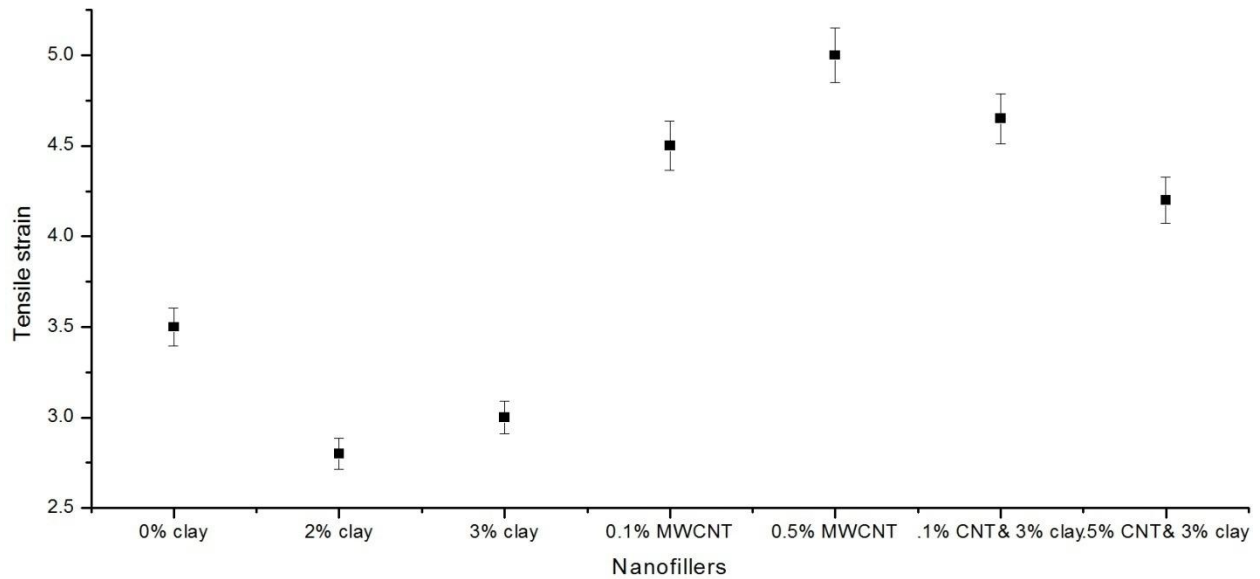
The results obtained by conducting tensile tests on nanocomposites using Zwick/Roell universal testing machine are shown below in Table 5.2.

**Table 5.2 Tensile properties for different clay/CNT composition**

<b>Specimen Name</b>	<b>Specimen No.</b>	<b>Tensile Modulus (MPa)</b>	<b>Tensile Strength (MPa)</b>	<b>Strain (%)</b>
0% clay	<b>Specimen no.1</b>	261	135	3.5
	<b>Specimen no.2</b>	259	143	3.5
2% clay	<b>Specimen no.1</b>	250	158	2.9
	<b>Specimen no.2</b>	172	174	2.7
3% clay	<b>Specimen no.1</b>	282	176	3.1
	<b>Specimen no.2</b>	294	200	2.9
0.1% CNT	<b>Specimen no.1</b>	535	160	4.1
	<b>Specimen no.2</b>	521	159	4.9
0.5% CNT	<b>Specimen no.1</b>	573	170	5.5
	<b>Specimen no.2</b>	714	180	4.5
0.1% CNT&3% clay	<b>Specimen no.1</b>	814	138	4.4
	<b>Specimen no.2</b>	519	184	4.9
0.5% CNT&3% clay	<b>Specimen no.1</b>	747	168	4.2
	<b>Specimen no.2</b>	760	190	4.2



**Fig. 5.9: Tensile strength of specimens as a function of weight percentage of nanoclay and MWCNT in epoxy resin**



**Fig. 5.10: Tensile strain of specimens as a function of weight percentage of nanoclay and MWCNT in epoxy resin**

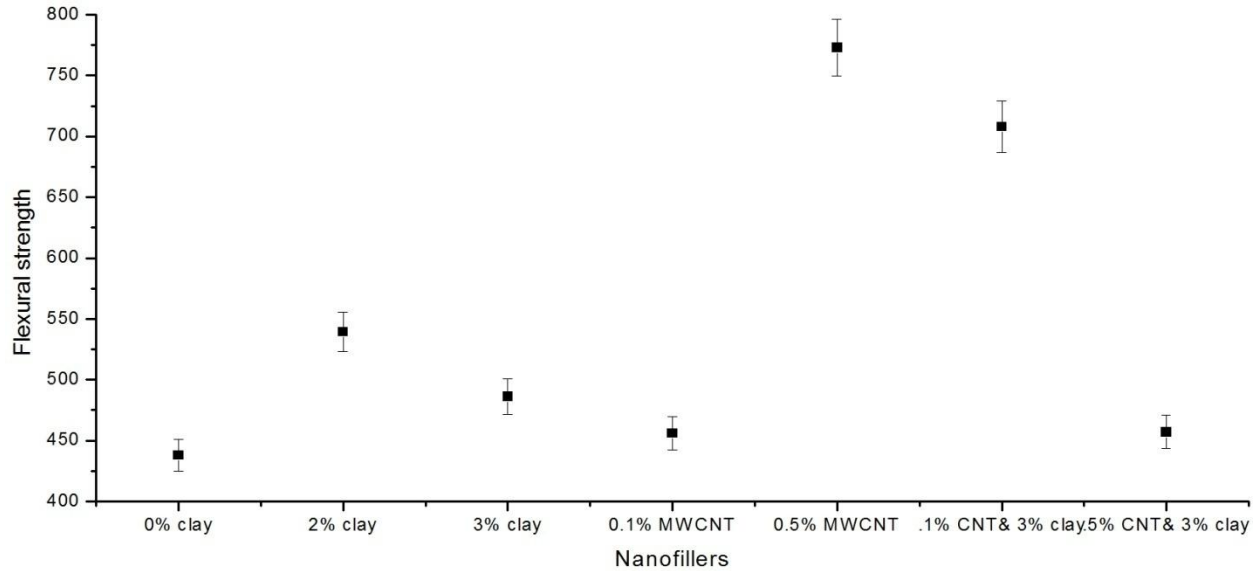
Monotonic increase in tensile strength with increase in quantity of each nanofiller. With addition of 3 wt% of nanoclay into 0.1 wt% of MWCNT specimens, tensile strength and strain values are not significantly affected, but in 0.5 wt% MWCNT specimen strain values have significantly decreased despite no change in tensile strength. The result also indicate that only small amount of MWCNT addition increased the tensile strength significantly.

### 5.3 3-Point bending test

The results obtained by conducting 3-point bending test on nanocomposites using Zwick/Roell universal testing machine are shown below in Table 5.3.

**Table 5.3 Results of 3-Point bending test**

<b>Specimen Name</b>	<b>Specimen No.</b>	<b>Elastic Modulus(MPa)</b>	<b>Flexural Strength(MPa)</b>	<b>Deformation At Max. Force%</b>
0% clay	<b>Specimen no.1</b>	0.28	435	0.81
	<b>Specimen no.2</b>	0.67	441	0.77
2% clay	<b>Specimen no.1</b>	0.47	553	0.60
	<b>Specimen no.2</b>	0.49	526	0.51
3% clay	<b>Specimen no.1</b>	1.90	401	1.40
	<b>Specimen no.2</b>	0.27	571	1.30
0.1% CNT	<b>Specimen no.1</b>	0.60	453	0.90
	<b>Specimen no.2</b>	0.67	459	0.74
0.5% CNT	<b>Specimen no.1</b>	0.41	703	0.60
	<b>Specimen no.2</b>	0.32	843	0.65
0.1% CNT&3%clay	<b>Specimen no.1</b>	0.43	670	0.52
	<b>Specimen no.2</b>	0.67	746	1.40
0.5% CNT&3%clay	<b>Specimen no.1</b>	1.00	489	1.40
	<b>Specimen no.2</b>	0.56	425	1.50

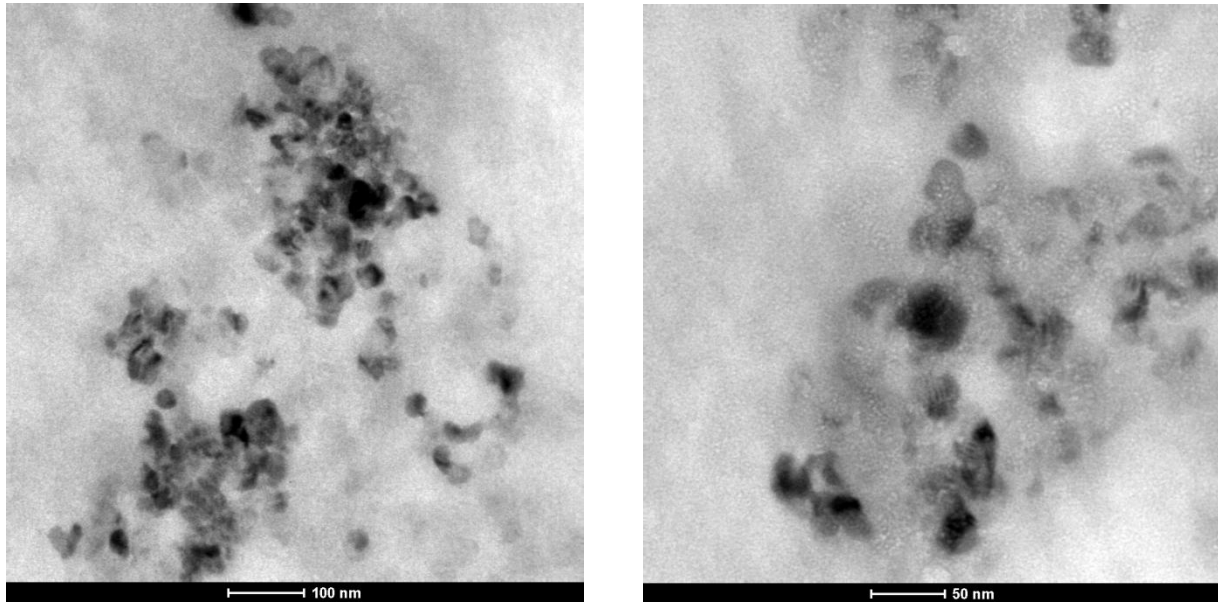


**Fig. 5.11: Flexural strength of specimens as a function of weight percentage of nanoclay and MWCNT in epoxy resin**

Very small quantity of MWCNT increased the flexural strength to a large extent. The maximum value of flexural strength observed is 750 MPa with 0.5 wt% MWCNT's specimen, which is much higher than values obtained from other compositions. Similarly an addition of 3 wt% nanoclay and 0.1 wt% MWCNT's showed an improvement in flexural strength if compared with specimen containing 0.1 wt% MWCNT's and 3wt% nanoclay individually.

#### 5.4 Transmission electron microscopy (TEM)

MWCNT and nanoclay were dispersed in the epoxy resin. Transmission electron microscopy (TEM) was used to characterize the MWCNT and nanoclay in the epoxy resin.



**Fig. 5.13: TEM images of nanocomposites having 0.5wt% of MWCNT and 3wt% nanoclay loading**

**6.1 Conclusion**

In the present work the fiber reinforced composites with modified epoxy matrix were manufactured. Addition of nanofillers at low quantities has resulted in improvement in mechanical properties of fiber reinforced composites. Tensile and Flexural tests performed on nano FRP composites showed improvement in tensile strength, tensile modulus and flexural strength and modulus but a decrease in values of strain before failure. A hybrid composition resulted in a good combination of tensile strength and strain at 0.5 wt% of MWCNT and 3 wt% of nanoclay. The maximum hardness has also been measured for this composition.

Very small quantity of MWCNT's mixed in epoxy resulted in significant improvements in mechanical properties. A decrease in properties at higher loadings of nanofillers was observed which is due to poor dispersion and formation of agglomerates.

**6.2 Future Scope**

1. To study the effect of other types of nanofillers on FRP's.
2. High shear homogenizer may be used for better premixing of nanofillers.
3. The work can be extended for finding out the effect of hygrothermal loading conditions on nano FRP system.

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