OPTIMIZATION OF MATERIAL REMOVAL RATE IN DRILLING CARBON NANOTUBE EPOXY COMPOSITE SPECIMEN BY TAGUCHI METHOD

A Major Project report submitted in partial fulfillment for the award of the Degree of

MASTER OF TECHNOLOGY

IN

POLYMER TECHNOLOGY

Submitted by

VIDHAN SRIVASTAVA
2K15/PTE/10

UNDER THE ESTEEMED GUIDANCE OF

DR. SAURABH MEHTA

Department of Applied Chemistry & Polymer Technology

Delhi Technological University, Delhi-110042

CERTIFICATE

This is to certify that the project report entitled "**OPTIMIZATION OF MATERIAL REMOVAL RATE IN DRILLING CARBON NANOTUBE EPOXY COMPOSITE SPECIMEN BY TAGUCHI METHOD"** submitted by **Vidhan Srivastava (2K15/PTE/10)** in partial fulfillment for the award of degree of Master of Technology in Polymer Technology to Delhi Technological University, Delhi, is a record of the work carried out by him under my supervision. The project embodies the original work by him to the best of our knowledge and has not been submitted to any other degree of this or any other university. The matter embodied in this project report is original and not copied from any source without proper citation.

Dr. Saurabh Mehta
Assistant Professor
Department of Applied Chemistry
& Polymer Technology
Delhi Technological University
New Delhi -110042

Dr. Archana Rani
Head of the department
Department of Applied Chemistry
& Polymer Technology
Delhi Technological University
New Delhi -110042

.

DECLARATION

I **Vidhan Srivastava** hereby declare that the thesis entitled "**Optimization Of Material Removal Rate In Drilling Carbon Nanotube Epoxy Composite Specimen By Taguchi Method"** is an authentic record of research work done by me under the supervision of **Dr. Saurabh Mehta,** Assistant Professor, Department of Applied Chemistry & Polymer Technology Delhi Technological University. This work has not been previously submitted for the award of any degree or diploma of this or any other University/Institute.

Dated   Vidhan Srivastava

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Last but not the least, I owe more than thanks to my family members which includes my parents and an elder sister for their support and encouragement throughout my life. Without their support, it is impossible for me to finish my education seamlessly.

Vidhan Srivastava

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ABSTRACT

This project introduces the application of Taguchi optimization methodology in optimizing the drilling parameters for machining pristine and functionalized multi walled carbon nanotube epoxy composite material to obtain maximum material removal rate. The drilling parameters which are chosen to be evaluated in this study are the diameter of dill bit, cutting speed and feed rate. While, the response factor to be measured is the material removal rate of the composite material. An orthogonal array was set up and signal-to-noise (S/N) ratio was employed to find the optimal levels of drilling parameters for achieving maximum material removal rate (MRR). MINITAB 18 software was used to find out signal-to-noise (S/N) ratio and mean. The combination of optimal drilling parameters for both pristine and functionalized multi walled carbon nanotube epoxy composite has been obtained at speed 440 rpm, feed 0.03 mm/rev and drill bit diameter 12 mm. In this study, Analysis of variance (ANOVA) revealed that speed is the dominant parameter followed by feed and diameter of drill bit for pristine multi walled carbon nanotube epoxy composite while feed is the dominant factor followed by diameter of drill bit and speed for functionalized multi walled carbon nanotube epoxy composite.

Keywords: carbon nanotubes, dispersion, functionalization, drilling, Taguchi.

CHAPTER 1

INTRODUCTION

1.1 Background and Motivation

As the growing need of world is increasing each day, it is therefore becoming impossible for the human to sustain in the present resources that the world have. Thus, the need for the development of new material arises. For development of a new material two things must be kept in mind one is the innovative product design and second is its requirement in use. These things aid to the innovative design of the material needed to accomplish the growing demand of society. Scientists are focussing their research to develop innovative materials as per the use for specific application. Internal or external changes can bring about desired innovation in design of the material. Internal changes include alloying, heat treatment. External changes include reinforcement of fibres, rods etc. with the material which act as matrix. Both of these methods result in an enhanced property of a material. Selection of a material is primarily focused on their mechanical, physical and thermal properties to ensure proper functioning, when several other materials are suitable and no other requirement are to meet then the one which is cheapest must be selected. Today, these aspects are very well accounted by the composite materials as they can be very strong and stiff besides being very light in weight. In composites it is possible to attain combination of properties not achievable with metals, ceramics or polymers alone. Due to the fact that the properties of the composite materials can be tailored, a number of steps required for their fabrication can easily be reduced by employing single step process. The material is designed according to its ultimate requirement. In this thesis a composite material is developed keeping these things in mind and its designated area of application.

1.2 Composite Material

First of all, there is a need to introduce our self with this new category of material known by the name of composite materials. Komundari defined composite materials as a material system which is composed of a mixture or a combination of 2 or more macroconstituents that differ in form and chemical composition and are insoluble in each other" [18] . According to another renowned material scientist, Reinhart: "Composite materials are macroscopic combination of 2 or more distinct materials having discrete and recognizable interface separating them"[19] .

Composite generally consist of a matrix material and reinforcement material. Matrix material is responsible for holding the reinforcement in its place. When load is applied to the composite material, matrix evenly and uniformly distributes the load among the reinforcing element. The reinforcing element on the other hand is a material that provides strength to matrix, it is the major load bearing element. Reinforcement can be classified in following four ways:

- **Particulate**: - Particulate can be in the form of spheres, particles having different faces and shapes.
- **Fibers**: - Fibers are classified as continuous and discontinuous. Continuous fibers run throughout the length of the composite while Chopped fibers are discontinuous fibers.
- **Layers**: - Reinforcement are laid as continuous layers in the matrix.
- **Whiskers**: - Whiskers are basically single crystals that are characterized by their widely varying morphology and dimensions.

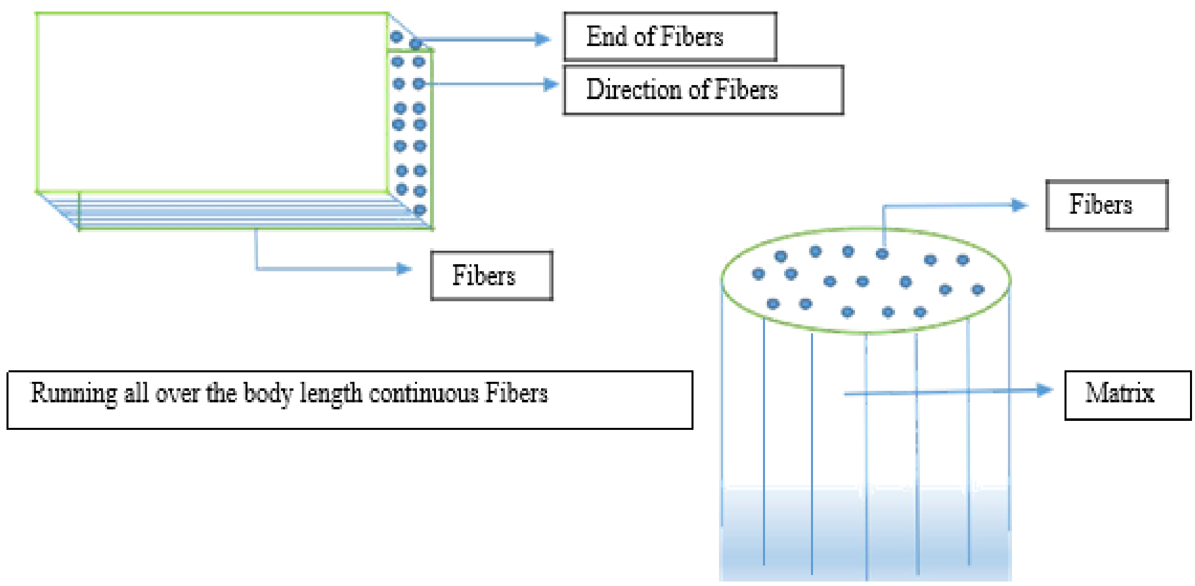


Fig 1: Different arrangement of fibers in a matrix

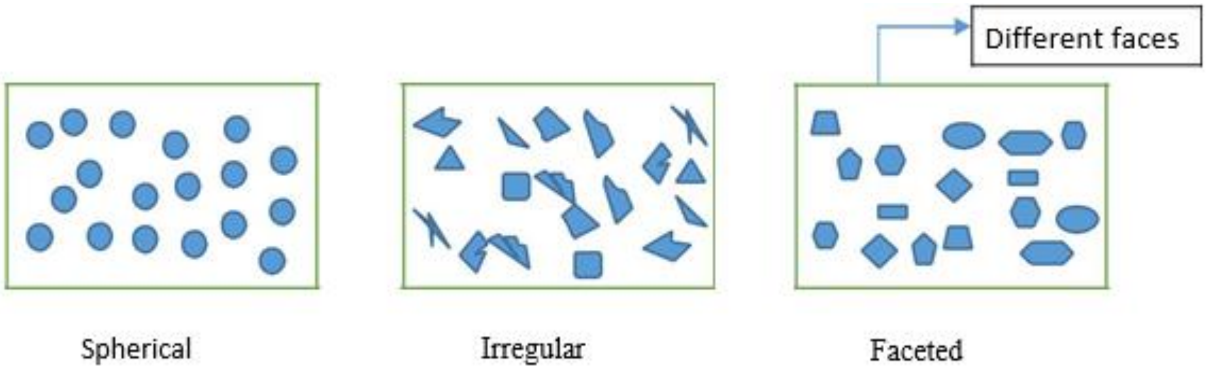


Fig 2: Varoius types of particulate in a matrix

Fig 3: Varoius types of whiskers in a matrix

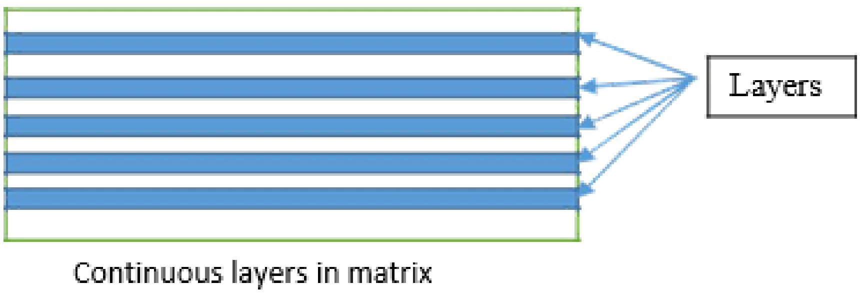


Fig 4: Reinforcement as continuous layer in a matrix

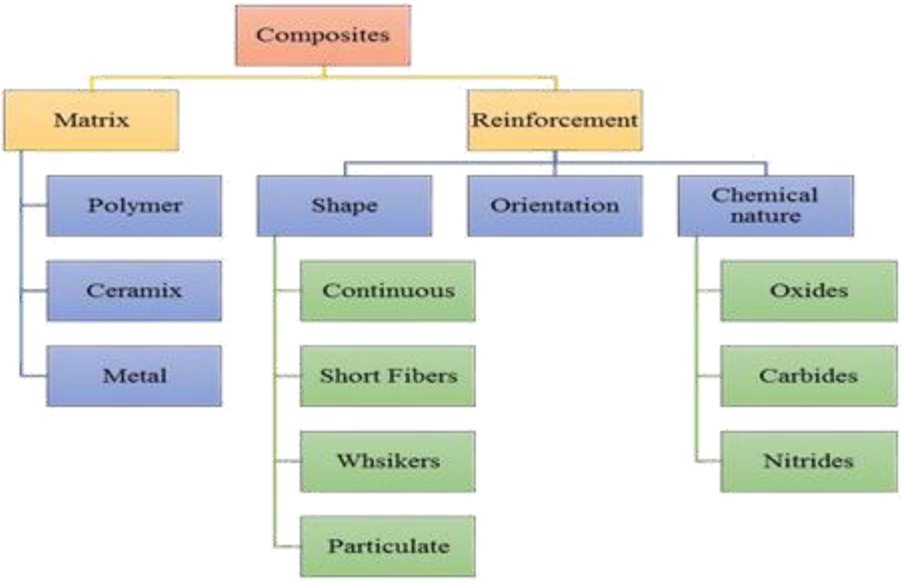


Fig 5: Broad classification of composite as matrix and reinforcement

In Figure 5 broad classification of composite as matrix and reinforcement is given. Further, Matrix material can be classified into three categories they are:

- **Ceramic Matrix**
- **Polymer Matrix**
- **Metal Matrix**

The polymers can be classified as thermoplastic polymer and thermosetting polymer. Thermoplastic polymer are the material that cures reversibly and softens on heating, when heated above the glass transition temperature or melting point and becomes hard after cooling. It has a linear or branched molecular structure. Thermoplastic polymer has high strength and toughness, good durability, chemical resistance, self-lubrication, and transparency. Acrylonitrile Butadiene Styrene (ABS), Polycarbonates (PC), Polyethylene (PE), Polyether ether ketone (PEEK). Polyvinyl Chloride (PVC) etc. are some of the thermoplastic polymer materials. They are used to manufacture dashboard, toys, electrical products, bearings, sheet, hoses, glass frames, ropes, gears etc. are just to name few. Thermosetting polymer material curing takes place irreversibly and it finally becomes permanently hard and rigid after curing. Although these material cannot be melted again but on continuous heating for a long time, these material get degraded and decomposed. Thermosets has a Thermosetting polymers are stiff, tough and durable due to the fact that they have a large three dimensional network of covalent intermolecular bonds commonly called as cross linked structure . They have good thermal and electrical insulation, chemically resistant. Alkyds, Allylies, Bakelite, Epoxies, Phenolic, Polyester, Polyurethane etc are some of the thermosetting polymer material. Applications in which these are used are spectacle lenses, knobs, handles, kitchen utensils, electrical equipment, printed circuit boards, circuit breakers, encapsulation material etc. Figure 6 exhibits different polymers and fillers used in polymer composite.

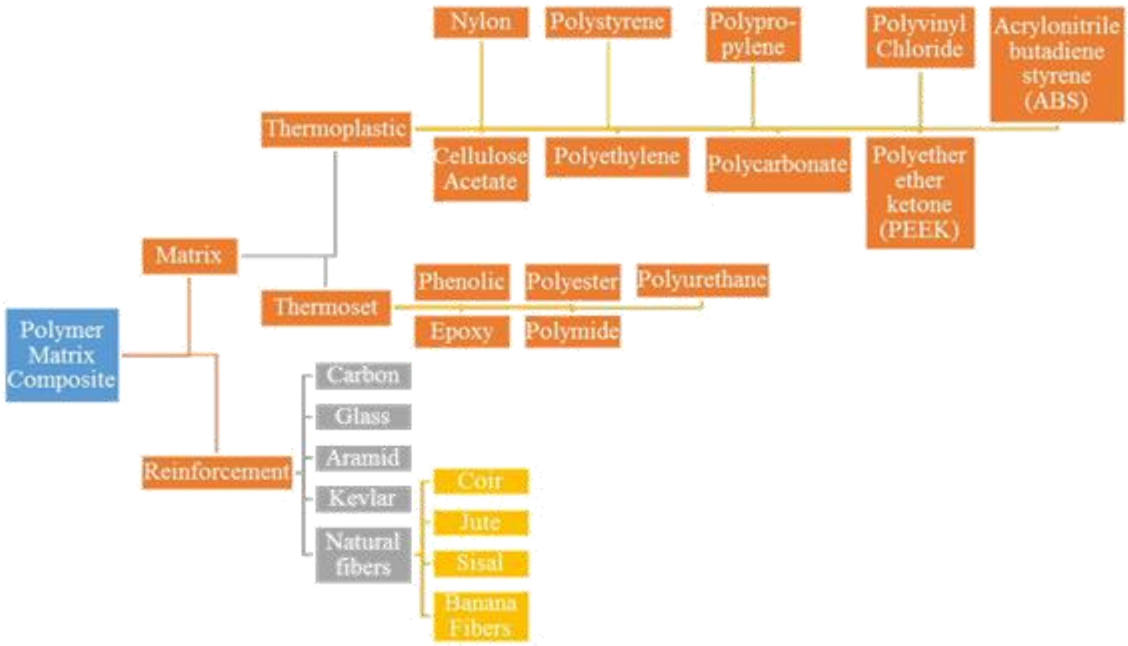


Fig 6: polymer matrix composite

1.3 Nanotechnology

It all began with a famous lecture by physicist Richard Feynman " There's plenty of room at the bottom” at the California Institute of Technology in an American Physical Society meeting (CalTech.) on December 29, 1959. Later, the term nanotechnology was coined by Professor Nario Taguchi. Nanotechnology concerns at the size of about 1 to 100 nm. It includes all structures of nanometer range although at microscopic level they also becomes the part of nanotechnology as they are made up of single atom and are considered as a Nano scale subsystems. So, nanotechnology is the study of enormously small things and its application in various fields of science. Nanostructured materials consist of single phase or multiphase polycrystalline solids whose size may vary up to few nanometers. The use of nanotechnology is growing in various areas as in biology, physics, Chemistry and medicine which leads to discovery of new departments in the field of research like Nano-medicine, Nano-physics, Nano composites, Nano-biotechnology etc. In the present work, experimentation and study has been conducted in the field of Nano composites. Nano composites can be defined as those composite materials which have at least one of the phases or filler dimensions like thickness, width, length in the nanometer range i.e.1 to 100 nm.

1.4 Carbon Nanotubes Fundamentals

Carbon nanotubes act as excellent filler in the field of Nano composites. Lets first discuss the basic building block from which it is made , before going into detail about the carbon nanotubes :-

1.4.1 Carbon

Carbon is a chemical element with symbol C which has an atomic number 6, that means on an atomic orbital it occupies $1s^2$ $2s^2$ $2p^2$ atomic orbitals. Carbon has the ability to get hybridized in sp, sp^2 and sp^3 forms. C2H2 is hybridized in sp^2 form. Fullerene, carbon nanotube, graphene are sp^2 hybridized while diamond is sp3 hybridized. From the view point of our study sp^2 hybridization is important. Figure 7 shows sp^2 hybridization.

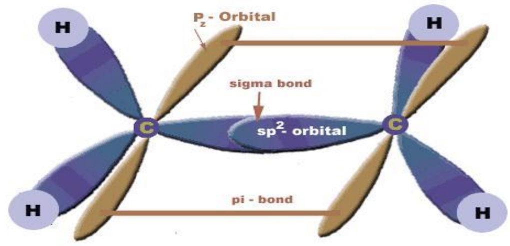


Figure 7: sp^2Hybridization in carbon atom

In this hybridization, one s and two p orbitals are involved, where one electron in s orbital gets promoted to the 2p orbital. Three hybrid orbitals are created which are although equal in energy but has lower energy than p-orbitals on combining the above said three orbitals. 2p orbitals are found to be orthogonal to the planes.

1.4.1.1 Fullerene

Fullerene usually is in the form of hollow sphere. That’s why, these are also called as “Bucky Balls”. They resemble to a soccer ball. Discovery of fullerene dates back in 1985 by the Smalley research group of Rice University while they were performing experiments on vaporization of carbon by laser pulses. Buckminsterfullerene C-60 was named after Richard Buckminster Fuller who was a popular architect. Figure 8 depicts the

Fullerene C-60 molecule that has 60 vertices and 32 faces (20 hexagons and 12 pentagons).

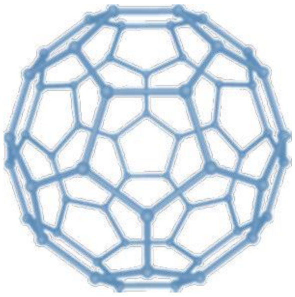
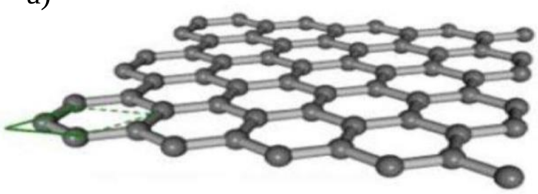


Figure 8: Buckminsterfullerene(C-60 molecule) .

1.4.1.2 Graphene

Graphene is a one atom thick sheet of graphite. It was discovered only in 2004. It has a two dimensional structure and carbon are bonded with sp^2 hybridization. Figure 9 a) shows the graphene sheet that can be thought of one story of a graphite structure (as in Figure 9 b)) cut to form a single layer of graphene.

a)



b)

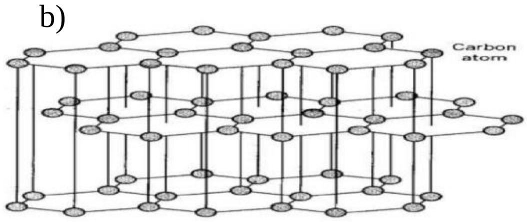


Figure 9: a) Graphene b) Graphite

1.4.1.3 Carbon Nanotubes

Carbon Nanotubes are a long one dimensional structure of carbon. It has seen that carbon atoms are sp^2 hybridized. These can be visualized as a graphene sheet, rolled to form a cylinder as shown. Carbon nanotubes were first synthesized by Sumio Iijima in 1991 [20]. Nanotubes were comprised of several tens diameter of graphite shells, with adjacent shells separation of ~0.34nm and diameter of ~1nm with a very high aspect ratio. It was only after two years that Single wall carbon nanotubes were manufactured by Iijima and Ichihashi [21] and Bethune et al. [22]

Carbon nanotubes possess low mass density , high flexibility and large aspect ratio even up to 1000. These nanotubes are lighter than aluminum, stronger than steel, and more conductive than copper.

Carbon nanotubes basically can be classified in two ways:-

1. Single walled carbon nanotubes
2. Multi walled carbon nanotubes

1.4.1.3.1 Types of carbon nanotubes

1. **Single walled carbon nanotubes (SWCNT)**: - These can be thought as wrapping a one atom thick sheet of graphite (known to the researchers by the name graphene) to form a seamless cylinder. Many SWCNT's have a length of the tubes can be thousand times larger while diameter close to 1nm.

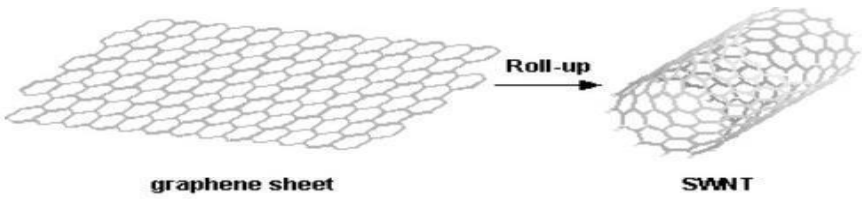


Figure 10: SWNT as visualized from graphene sheet

2. Multi walled carbon nanotubes (MWCNT): - It consists of multiple layers of graphite, rolled on to themselves so as to form a tube. Structure of MWCNT's can be defined by two models which are also shown in Figure 11 and can be visualized.

a. Parchment model: - a single layer of graphene is rolled around itself, which resembles a rolled up newspaper or scroll of parchment

b. Russian doll model: - sheets of graphene are arranged in concentric cylinders.

.

Figure 11 a) Russian doll model b) parchment model for MWCNT's c) scroll of parchment

3. **Double walled carbon nanotubes**: - It can be imagined as a multi walled carbon nanotubes with just two concentric cylinders as shown below in Figure 12.

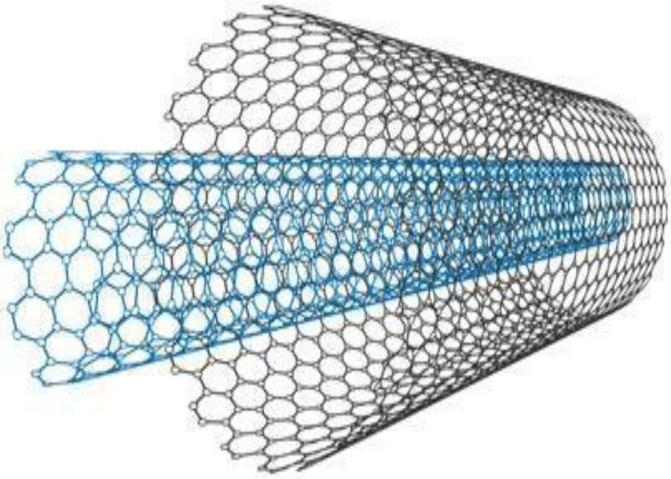


Figure 12: Double walled carbon nanotubes

1.4.1.3.2 CNT structural orientation

Carbon in the carbon nanotubes can be arranged in various types. They have different structural patterns of carbon atoms in it. These orientations of carbon hexagonal structure can be named as like armchair, zigzag, chiral. This is represented in Figure below.

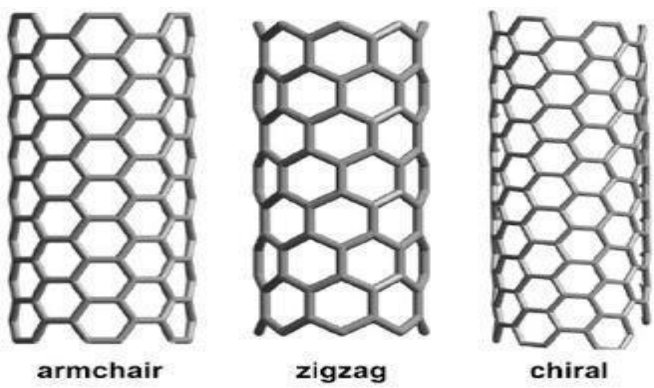


Figure 13: Various orientation of CNT molecule

1.4.1.3.3 Manufacturing of CNT

By following three main methods CNT's are manufactured, they are:-

a) Laser Ablation Method
b) Chemical Vapour Deposition (CVD)
c) Electric Arc Discharge Method

1.5 Chemistry involving functionalization

CNTs functionalization process is crucial as it can in enhance the dispersion and the bonding between the CNTs and the surrounding matrix. This is made possible by providing the specific type of the functional group that will bond with the matrix. For Epoxy matrix following mentioned functional groups are important. These functional group increases the dispersion and interfacial bonding of CNT's in epoxy matrix:-

a) Carboxyl (-COOH) Group
b) Amino (-NH2) Group

Other functional groups are not that successful.
During chemical functionalization, sulphuric acid generates a defective site on the walls of carbon nanotubes. This site is later attacked by nitric acid which result in its oxidization. Thus, on grafting the carboxylic group on the walls of carbon nanotube the sp^2 hybridization is converted into sp^3 hybridization. Because of the functionalization, hydrophobic nature of CNT changes into hydrophilic that results in better dispersion. Composites that are based on chemically modified nanotubes have manifested best mechanical results because functionalization significantly enhances both dispersion and consequently stress- strain transfer. Functionalization is used to increase the dispersion of nanotubes in solvents.

Functionalization is done in order to improve [23] :-

i) Dispersion of CNT in polymer matrix to evenly distribute load.

ii) Strength of covalent bond between CNT and polymer matrix.

For functionalization following points should be kept in mind [24] :-

a) Ends of nanotubes are more reactive than the sidewalls of CNT.
b) Higher the curvature of CNT results in more reactive site.
c) Nanotubes synthesized with defects are highly susceptible to functionalization than those have no defects.
d) Carboxylic acids are extremely useful functional group for many reactions; they can be reacted with alcohols to make esters and reaction with primary amines to make amides
e) Its easy to functionalize smaller diameter nanotubes .

Its bit difficult to mix longer nanotubes with polymer.
Dispersion of CNT in polymer matrix is affected because of following reasons:-

a) Seamless surface of CNT is unable to provide physical interaction between CNT matrix.

b) Surface energy of CNT is significantly different from that of matrices as CNT may not have chemical affinity to organic matrices. Thus, dispersion of CNT's into matrices is biggest obstacle.

There are two major drawbacks of chemical functionalization [25] :-

a) Environmental unfriendly concentrated acids or strong oxidants are often used for CNT functionalization.
Ultra sonication process creates a large number of defects on CNT sidewalls and in some cases CNT's are fragmented into smaller pieces. Carbon hybridization changes from sp^2 to sp^3 which results in severe degradation in mechanical properties.

1.6 Organization of Thesis

Remaining chapters in this are organized as follows:-

Chapter 2: Includes a literature review which provides a summary of knowledge already available by involving the issues of interest. It presents the research works on machining of composite materials and their optimization by Taguchi technique by various researchers.

Chapter 3: Includes a description of the raw materials used in the research and various test procedures. It presents the details of fabrication and characterization of the composites under investigation and also an explanation of the Taguchi experimental design.

Chapter 4: Presents the results that are obtained during various characterization tests and optical microscopy. Further, these results are discussed in detail. Result of drilling operation done on pristine MWCNT composite and functionalized MWCNT composite has been presented by employing Taguchi orthogonal array.

Chapter 5: Provides summary of the findings of this research work, outlines specific conclusions drawn from both the experimental and analytical efforts and suggests ideas and directions for future research

CHAPTER 2

LITERATURE SURVEY

2.1 Literature review

R.Vimal et al. [1] has Modeled and analyzed the Thrust force and Torque in drilling GFRP composites by multifaceted drill using fuzzy logic .The research has been made by taking Glass Fibre Reinforced Plastic using 8 facet solid carbide tool. 3 parameters such as spindle speed; feed rate and drill diameter with each having 3 levels using Taguchi L27 was used. Machine used was ARIX-CNC Machine centre. Thrust force and torque were taken as judging criteria for optimization of input parameters. Fuzzy rule based model was developed to indicate thrust force and torque in drilling of GFRP composites. The results suggested that the model can be effectively used for predicting the response variable by means of which delimitation can be controlled. Davim et al. [3] established an empirical relation between feed rate and cutting velocity with reference to thrust force, cutting pressure, a factor of damage and surface roughness in machining of GFRP composites by using cemented carbide tool. ANOVA has been also performed to investigate the effects of process parameters. El-SonbatyI.et al. [4] examined the effects of process parameters on the thrust force, torque and surface roughness in drilling of fiber-reinforced composite materials. It has been demonstrated from the result that epoxy resin, cutting speed has insignificant effect on thrust force whereas cutting speed and feed has significant influence on surface roughness. Langella et al. [5] investigated the effect of machining parameters such as chisel edge and type of drill on thrust and torque in drilling of composites. Singh et al. [6] approached a fuzzy inference system to predict torque and thrust in drilling of GFRP composites using solid carbide drill bit with eight facet. Experiments conducted by L27 Orthogonal array and ANOVA has been done to investigate the influence of process parameters. Kilickap et al. [7] investigated the effect of cutting parameter that was feed rate, chisel angle of drill tool and cutting speed in drilling of glass fiber reinforced polymer. The primary aim was in this paper minimized the delamination that was produced after drill on GFRP and Taguchi method had used in this research. Latha et al. [8] assessed the effects of drill parameters on thrust in machining of GFRP composites. Kumar et al. studied the influence of factors such as feed rate, cutting velocity, environment condition of the experiment, rake angle of tool and depth of cut in turning of GFRP composites. The optimal machining condition is obtained by Distance Based Pareto Genetic Algorithm. Rahaman et al [9] studied on machinability aspects of carbon fiber reinforced composite. Three types of cutting tool inserts: uncoated tungsten carbide, ceramic and cubic boron nitride (CBN) were used to machine short (discontinuous) and long (continuous) fiber carbon epoxy composites. Ferreira et al [10] studied the performance of different tool materials such as ceramics, cemented carbide, cubic boron nitride (CBN), and diamond (PCD). The results showed that only diamond tools were suitable for use in finish turning. An optimization methodology was used in rough machining to determine the best cutting conditions. It was concluded that the optimization of the cutting conditions is extremely important in the selection of the tools and cutting conditions to be used in the CFRP manufacturing process. Enemuoh et al [11] presented a new comprehensive approach to select cutting parameters for damage-free drilling in carbon fiber reinforced epoxy composite. The approach was based on a combination of Taguchi's experimental analysis technique and a multi-objective optimization criterion. The optimization objective included the contributing effects of the drilling performance measures: delamination, damage width, surface roughness, and drilling thrust force. Palanikumar et al. [12] has utilized grey relation analysis with Taguchi Technique to obtain the optimal machining condition. Experiments were carried out throughL16 4-level orthogonal array in order to investigate the effects of spindle speed, feed on thrust force, delamination factor at an inlet and outlet on composite and surface roughness. Verma et al. [13] proposed fuzzy inference system integrated with Taguchi to assess the favorable machining condition in turning of GFRP composites using HSS tool. The machining evaluation characteristics are taken as Material removal rate and surface roughness. Tsao et al. [14] aimed to reduce delamination in drilling of GFRP composites by active backup force. It has been noticed that applied backup force contributes to reduction of the growth of the delamination at drilling exit by 60– 80%. Krishnaraj et al. [15] analyzed the influence of drilling parameters on thrust force, torque, an eccentricity of hole and delamination of both side of the hole in machining of CFRP composites using ANOVA. The study also used of grey relation analysis method to evaluate the optimal machining condition. In the drilling studies carried out by Sachse et al [16], a manually controlled 10mm diameter angle drill was used with a maximum speed of 1800 min -1. Bello et al [17] specified two diamond drill diameters, ¼'' (6.35mm) and 3/8'' (9.525mm) along with investigating two drill speeds of 725 per minute and 1355 per minute. This study investigated the use of CNT, alumina and graphite-epoxy composites, whereas the studies by Sachse [16] investigated PA6 and PP-silica nanocomposites. Apart from diverse nanocomposites being investigated, the differences in setups for the two studies demonstrate the need for a standardized set of drilling parameters to allow for a feasible comparison.

2.2 Knowledge Gap

From the literature review done above, it reveals the following gap

Because of the fact that nanocomposites are a relatively newly established material, few studies have been produced on the machining of polymer nanocomposites. Machining is an integral part of product development and it is categorized as a secondary manufacturing process. Drilling operation is one of the machining process. Drilling like other machining processes is deeply influenced by many factors, thus it is therefore necessary to evaluate the effect of various parameters on drilling nanocomposites specimen and obtain optimized set of parameters to maximize material removal rate. Till now, from the literature available, only three studies have investigated drilling on nanocomposites. The development and assessment of polymer nanocomposite properties is also considerably scarce within literature.

CHAPTER 3

RESEARCH OBJECTIVE

The purpose of the present research work is:-

1. To confirm the grafting of carboxylic group on to the walls of MWCNT's by FTIR spectrometer.
2. To investigate whether the functionalization condition were smooth enough so as not to disturb the conjugated structure of MWCNT by XRD.
3. To find the optimum parameters of drilling operation by Taguchi design.
4. To study the effect of cutting speed, diameter of drill bit and feed rate on material removal rate on pristine and functionalized multi walled carbon nanotube epoxy composite.

CHAPTER 4

MATERIALS AND METHODS

4.1 Introduction

This chapter gives an insight of all kinds of materials used during the research work and methodologies that are have adopted for the successful completion of research. This provides detailed description about various characterization techniques that are done on the pristine carbon nanotubes and after functionalization on the functionalized carbon nanotubes, nano composites fabrication detail, its mechanical testing , the insight of drilling operation test by using the Taguchi experimental design followed by statistical explanation by analysis of variance (ANOVA).

4.2 Materials

4.2.1 Matrix material

For the preparation of composite materials, matrix material can be of either of three types as disused metal matrix, ceramic matrix or polymer matrix. In this study polymer matrix is chosen as they are readily available, can be easily fabricated , and its properties can be altered according to the need of applications very effectively when compared with the ceramic and metal matrix .For the present study thermosetting polymer matrix is chosen. Most commonly used thermosetting polymers are epoxy, polyester, vinyl ester and phenolic. Among all these materials epoxy has been used widely for many advanced composites. Besides this, epoxy resin with a reinforcement of carbon nanotubes shows excellent mechanical and thermal properties even at high temperature and wide range of viscosity is available with the epoxy. While for carbon nanotube a low viscosity matrix material is needed in order to obtain good dispersion. Due to these advantages with the Multi walled carbon-nanotube epoxy material Lapox L12 is selected. Lapox L12 is unmodified epoxy resin of medium viscosity that can be used with different kinds of hardeners. The decision of selecting a hardener depends not only upon the processing methods to be used but also on the properties required of the cured composite. So, hardener K6 is chosen as it has a low viscosity as it is of prime importance with carbon nanotubes. Due to the fact that it is reactive by nature, it gives a short pot life and rapid cure at ambient temperatures.

Specification of resins and hardener

Hardener K-6

i) Visual appearance pale yellow liquid
ii) Water content 1% max
iii) Refractive index at 25 ºC 1.4940-1.5000

Lapox L-12

iv) Epoxide equivalent gm/eq. 182-192
v) Viscosity at 25ºC 9000-12000 mPa.S
vi) Epoxy value eq. / kg 5.25-5.5

Processing parameters

Lapox L-12 100 parts by weight
Hardener K-6 10-12 parts by weight
Viscosity at 20ºC 5000-8000 mPa.S
Pot life at 20 ºC 0.5-1 hrs.

Lapox L-12 and hardener K-6 can be easily mixed at room temperature. Lapox L-12 is known by the conventional name and its IUPAC name is 4, 4’-Isopropylidenediphenol, oligomeric reaction products with 1-chloro-2, 3-epoxypropane it belongs to the epoxide family . Both the epoxy resin and corresponding hardener are available from polymer processing lab at Applied chemistry deptt. At DTU.

4.2.2 Filler Material

Multi walled carbon nanotubes have been chosen as a filler material, the reason being it's very high aspect ratio, Young's Modulas (over 1TPa) , estimated tensile strength (around 200 GPa) .It is as stiff as Diamond. Because of the reason that carbon nanotubes have a low density for a solid of 1.3-1.4 g/cm^3, its specific strength is the best of known materials. Therefore, these properties are best suited for fabricating reinforced composites. Multi walled carbon nanotubes have been procured from Appied Physics deptt. At DTU. Its specifications as mentioned by supplier are given below:-

Outer Diameter – 11-20 nm

Purity > 97%

Length – 10-30 microns

4.3 Chemical Functionalization of Multi-walled Carbon nanotubes

Inorder to chemically functionalize multi walled carbon nanotubes by carboxyl group, mixture of acid is used which is a combination of nitric acid and sulphuric acid. Sulphuric acid with 98 wt. % concentrations and nitric acid with 69-72 weight percent concentration are taken. 100 ml of mixture acid is taken where Sulphuric acid and nitric acid (Mixture acid) are taken in a 3:1 ratio, 3 parts of sulphuric acid is mixed with 1 part of nitric acid in a beaker and then multi walled carbon nanotube is added to it. After this, the mixture is sonicated in an ultrasonicator for 15 minutes for proper mixing of CNT with the mixture acid. Now the sonicated mixture is poured in a double neck round bottom flask whose one neck has been equipped with thermometer for measuring temperature accurately and other neck is occupied by the condenser arrangement so as to heat the solution under reflux, thus not allowing CNT to escape. We have to heat the solution in a mantle under reflux for 30 minutes while maintaining temperature of 120º C. After heating the mixture it is allowed to cool to a room temperature. Then washing and filtering of MWCNT by the PTFE membrane filter is done. Pore size and material of the filter is chosen carefully as mixture acid may dissolve the material of the filter. Thus, Poly Tetra Flouro Ethylene (PTFE) is selected as the filter material as it will sustain the acid, pore size is selected at 0.22 micron for the CNT to be filtered from the solution and the size of the filter is selected as 25mm diameter according to our set up for filter. Filter is kept in a filter assembly which is connected to a vacuum pump so as to create vacuum for easily facilitating the filtering of CNT. Filtered CNT is then washed several times by the deionized water (5 washing cycles are enough for neutralising the pH level of MWCNT) and pH is measured by litmus paper, it must be neutral for further processing. After this, the filtrate obtained is kept in a pettry dish and put in an oven at 60º C for 24 hours for the CNT to dry. FTIR is performed so to confirm the grafting of carboxyl group onto the walls of MWCNT and XRD is done to check the structure of MWCNT after chemical treatment.

Fig 14: Refluxing set up

4.4 Composite Fabrication

Composite is fabricated by both pristine MWCNT and functionalized MWCNT. By keeping in view the concentration of MWCNT in matrix, MWCNT is taken and mixed with acetone which act as a solvent. MWCNT's were taken in a small beaker,sonicated for 15 minutes, followed by addition of epoxy into the mixture. It is mechanically stirred for 2 minutes followed by sonication for 4 hours, the dispersed mixture thus obtained is then magnetically stirred for 1 hour at 70ºC so as to completely dispersed the MWCNT into the epoxy matrix, this will completely vaporize the acetone present in the mixture which is added so as to obtain better dispersion by decreasing the viscosity of the mixture. After this, the hardener is added into this mixture followed by mechanical stirring for 2 minutes after which it is poured into the mould. Then, samples are allowed to cure at room temperature for 1 day.

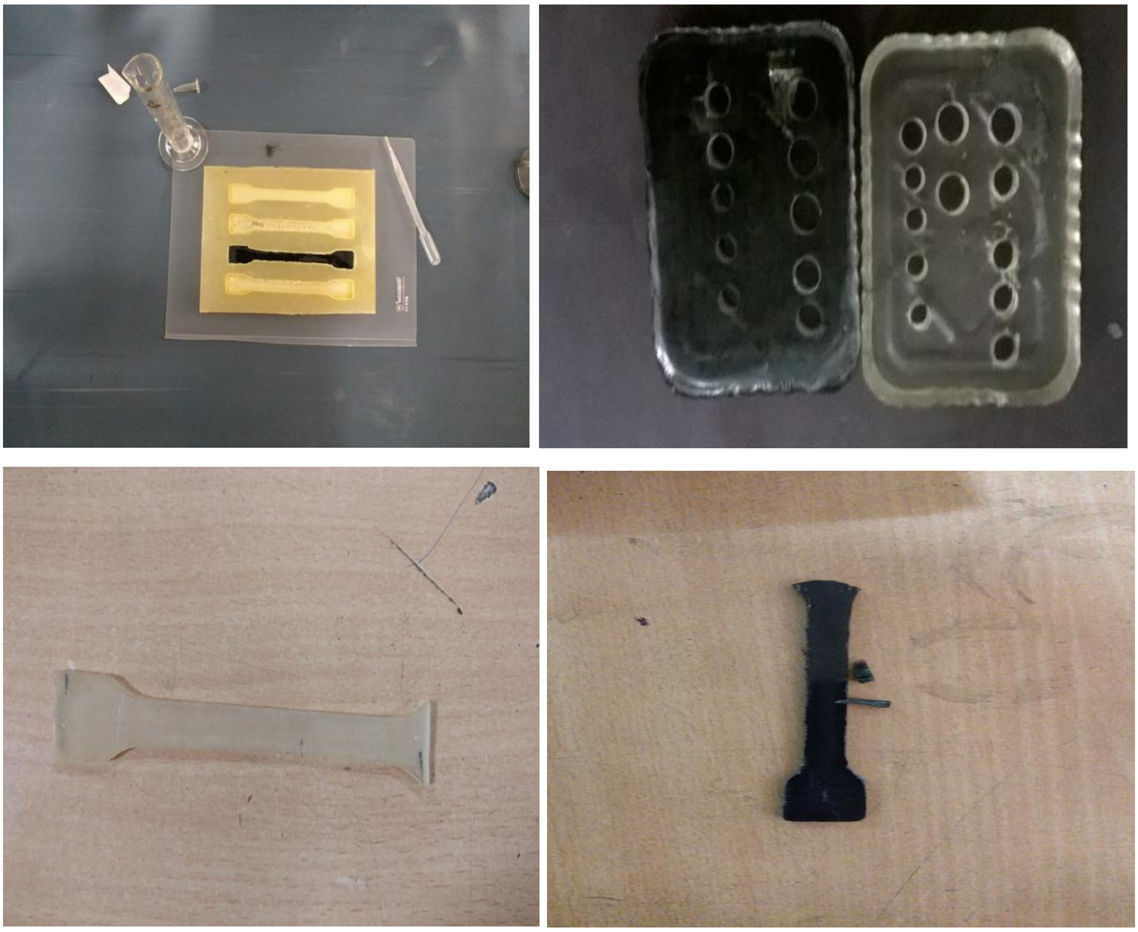
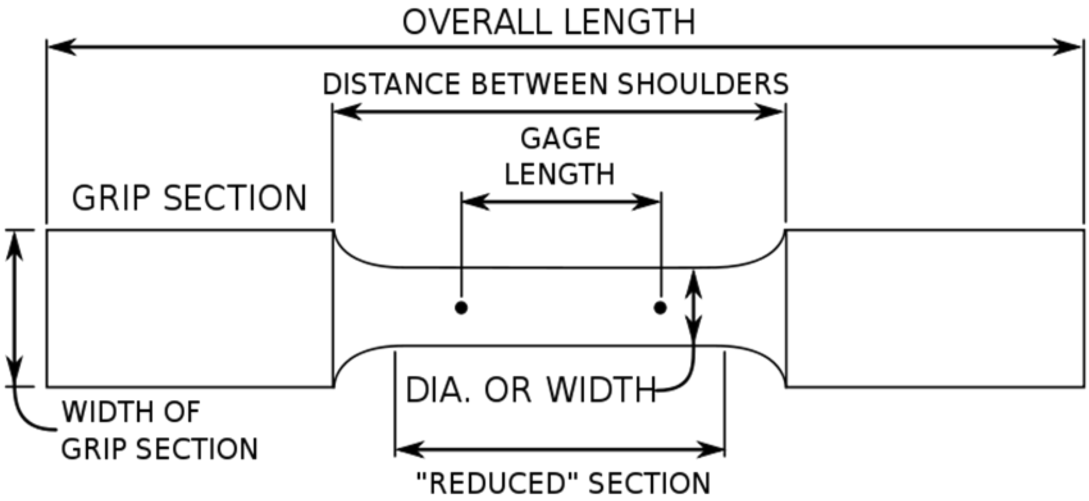


Fig 15: Samples of MWCNT composite and neat epoxy

4.5 Physical Test

4.5.1 Tensile Test

In tensile testing, also known as tension testing, is a fundamental materials science test in which a sample is subjected to a controlled tension until failure. The results from the test are used to select a material for some desirable application, for quality control, and to investigate how a material will react under other types of external forces. There are three properties that can be measured directly from a tensile test, these are ultimate tensile strength, maximum elongation and reduction in area. From these measurements properties like Young's modulus, Poisson's ratio, yield strength, and strain-hardening characteristics can also be determined. Uniaxial tensile testing is required for obtaining the mechanical characteristics of isotropic materials while for anisotropic materials, such as composite materials and textiles, biaxial tensile testing is required.



Equipment used:

Universal Testing Machine (UTM) is the most common testing machine used in tensile testing. This type of machine has two crossheads; one of the crosshead is driven to apply tension to the test specimen while the other one is adjusted for the length of the specimen. They can be either hydraulically powered or electromagnetically powered**.** The machine should have the following capabilities for the test specimen being tested. These are: force capacity, speed, and precision and accuracy. Force capacity refers to the ability of the machine to be able to generate enough force to fracture the specimen. Either the load application is quick or slow, the machine must be able to properly mimic the actual application. Lastly, the machine must be in position to measure the gauge length and forces applied accurately and precisely; for instance, a large machine that is designed to measure long elongations may not work with a brittle material that experiences short elongations prior to fracturing. Alignment of the test specimen in the testing machine is yet another challenge, because if the specimen is not aligned and gripped properly, either at an angle or offset to one side, the machine may exert a bending force on the specimen which is especially bad for brittle materials, as it will dramatically skew the results.

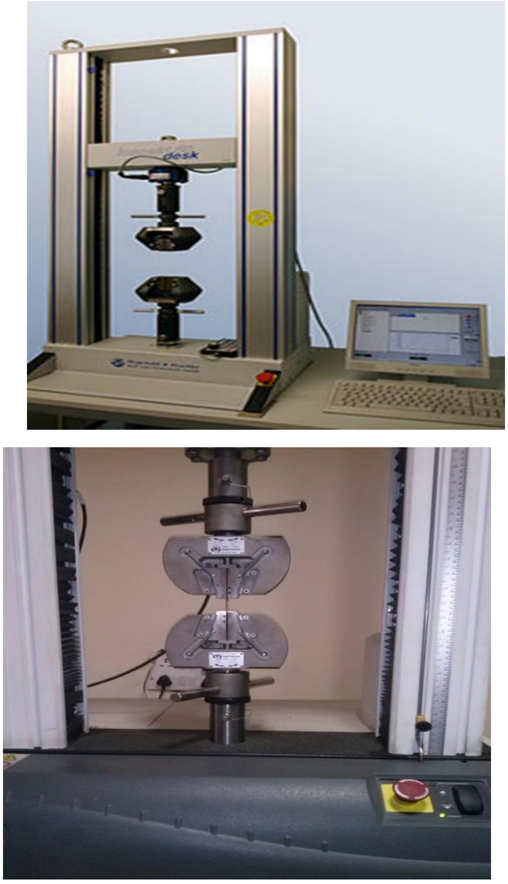


Fig 16: UTM testing apparatus

Test process:

During the tensile testing process , the test specimen the test specimen is placed in the testing machine and tensile force is applied to it until it fractures. During the application of tensile force, the elongation of the gauge section is noted against the applied force. The data is manipulated so that it is not specific to the geometry of the test sample. The engineering strain or elongation measurement can be calculated by using the following equation:

$$\varepsilon = \frac{\Delta L}{L_0} = \frac{L - L_0}{L_0}$$

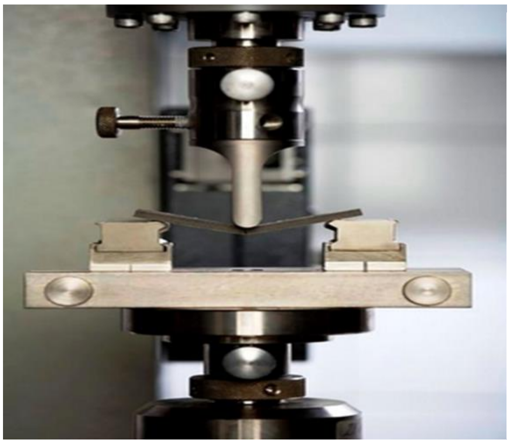
Where ΔL is the change in gauge length, L_0 is the initial gauge length, and L is the final length. Engineering stress, σ, is calculated using the following equation:

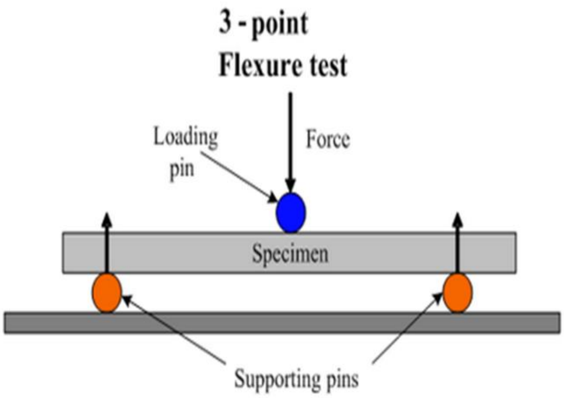
$$\sigma = \frac{F_n}{A}$$

Where F is the force and A is the cross-section of the gauge section. The machine does these calculations as the force increases, so that the data points can be graphed into a stress-strain curve.

4.5.2 Flexural Test

The three-point bending flexural test gives us the values for the flexural stress σ_f , modulus of elasticity in bending E_f, flexural strain ϵ_f, and the flexural stress-strain relationship of the material. Advantage of a three point flexural test is the easy way of the specimen preparation and testing. However, the results being sensitive to specimen strain rate and loading geometry is one of its disadvantage.





Calculation of the flexural stress σ_f

$$\sigma_f = \frac{3PL}{2bd^2}$$ for a rectangular cross section

$$\sigma_f = \frac{PL}{\pi R^3}$$ for a circular cross section

Calculation of the flexural strain ϵ_f

$$\epsilon_f = \frac{6Dd}{L^2}$$

Calculation of flexural modulus E_f

$$E_f = \frac{L^3 m}{4bd^3}$$

In the above formulas the following parameters are used:

- σ_f = Stress in outer fibers at midpoint, (MPa)
- ϵ_f = Strain in the outer surface, (mm/mm)
- E_f = flexural Modulus of elasticity,(MPa)
- P = load at a given point on the load deflection curve, (N)
- L = Support span, (mm)
- b = Width of test beam, (mm)
- d = Depth of tested beam, (mm)
- D = maximum deflection of the centre of the beam, (mm)
- m = The gradient (i.e., slope) of the initial straight-line portion of the load deflection curve,(P/D), (N/mm)
- R = The radius of the beam, (mm)

4.6 Scanning Electron Microscope (SEM)

A scanning electron microscope generates images by scanning the sample by the focused beam of electrons which interacts with the sample and produces various signals that contain various information about the surface topography. In this microscope electron beam is scanned in a raster scan mode. For this research work SEM-JEL-JSM-648 equipment has been used as shown in the Figure. Inorder to prepare sample for SEM examination the surface of the specimen was coated with platinum for 5 minutes prior to the observation at 50 kV after that the surface topography of the multi walled carbon nanotubes was studied.

Fig 17: SEM set up

4.7 Fourier Transform Infrared Spectroscopy (FTIR)

Molecular Vibrational spectrum is considered to be a unique physical property and it is a characteristic of the molecule. IR spectrum acts a finger print and compares the data with the previously recorded reference spectra, in this way it is used as tool to identify the spectrum of unknown materials. Frequency is the term that is used for band/peak position. It is expressed in units of wavenumber (cm^{-1}). Generally average modern infrared instrument records spectra from around 4000 cm^{-1} down to 400 cm^{-1} as defined by the optics of the instrument. That is why, when a spectral region is quoted, the higher value will be quoted first, consistent with the normal left-to-right (high to low cm^{-1}) representation of spectra.

Figure 18 FTIR instrument (Perkin Elmer, USA)

FTIR instrument used here is Perkin Elmer, USA (as shown in Figure 16) which is installed in the Uflex chemical laboratory , pallets of CNT were made with the help of a palletizer.

Figure 19 Palletizer

4.8 X-Ray Diffraction (XRD)

XRD is a tool that is used to identify the molecular and atomic structure of the crystal. XRD is performed not only for identification of phase of crystalline materials but also for obtaining information on the unit cell's dimension. It is regarded as a non-destructive analysis technique which gives the unique fingerprint of Bragg's reflection which is associated with the crystal structure. Structure of the crystal is reflected as layers of atoms arranged in a plane. During scattering of x-rays from a crystal lattice, peaks of scattered intensity are observed which correspond to the following conditions:

The angle of incidence = angle of scattering.

The path length difference = an integer number of wavelengths.

The condition for maximum intensity contained in Bragg's law as written below allow us to calculate details about the crystal structure. However, if the crystal structure is known, it can also be used to determine the wavelength of the x-rays incident upon the crystal.
It is explained by Bragg's law as: -

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

Where, λ is wavelength of x ray.

ϴ is the angle between the incident rays and surface of the crystal. d is spacing between layers of atoms.

Constructive interference occurs when n is an integer. When n is an integer 1, 2, 3 etc.

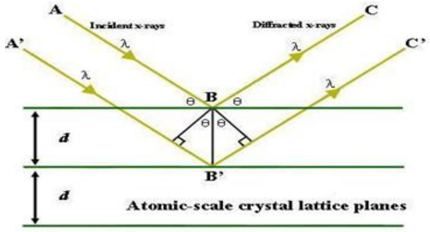


Figure 20: Incident & reflected rays in XRD

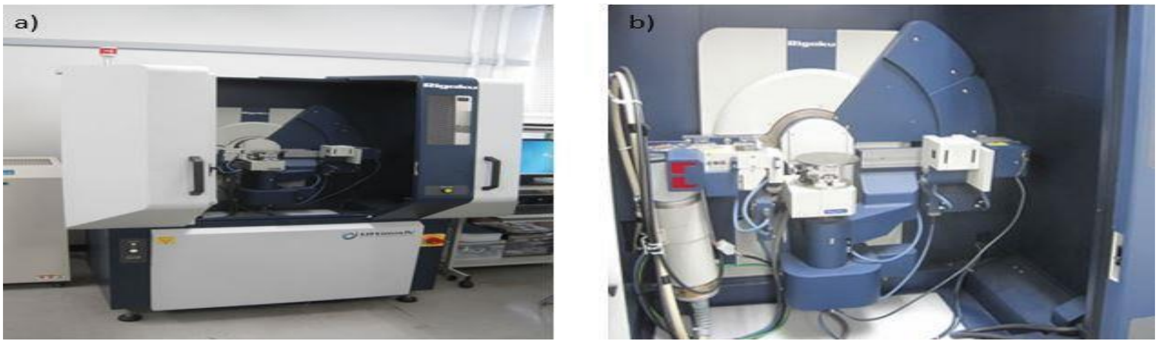


Figure 21 XRD instrument a) Setup of XRD b) Place for holding the slide during the XRD.

X-Ray Diffraction (XRD) is carried out using Rigaku Japan/Ultima-IV equipment as shown above . Both functionalized and pristine MWCNT were taken in a slide and the following parameters were chosen

Source of wavelength - Cu Kα X ray

10º per step per minute
Wavelength - 1.5418Å Range - 5º -90º (2Ɵ)
Step size of 0.02º

4.9 Thermo Gravimetric Analysis (TGA)

This technique observes mass of sample with reference to the temperature as it is heated or cooled. It employs a simple pan which is attached with a precision balance, this pan is kept inside a furnace and weight loss is observed as a function of time. Environment of the sample is filled by sample purge gas which may be inert or reactive, it flows over the sample and exits as exhaust.

In this work, sample is heated from the room temperature to the 700ºC in a nitrogen atmosphere with a gas flow rate of 20ml/minute with a heating rate of 10ºC/minute.

4.10 Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) is performed to measure glass transition temperature which is a temperature at which amorphous solid begins to change into the amorphous liquid. DSC involves heating or cooling a specimen at a constant rate in ºC/minute. It measures heat capacity calculated by measuring amount of heat added or removed from the sample. Heat capacity is directly proportional to the amount of amorphous material that changes from amorphous solid to amorphous liquid. In the glass transition temperature region there is steep increase in the heat capacity . But where glass transition temperature is absent heat capacity increases linearly with temperature. The heating process was scanned from 30-150ºC .Besides this heating rate of 10ºC/min under nitrogen atmosphere was employed. DSC is performed on neat epoxy, pristine MWCNT's composite, and functionalized MWCNT's composite, latter two are prepared at different composition. The prepreg were kept in an Al pan & weight of the samples were taken in between 10 to 30 milligram.

Figure 22: DSC setup and computer attached to it for monitoring the data

4.11 Drilling operation

Drilling is a material removal or cutting process that uses a drill bit to cut a circular hole in solid materials. The drill bit is a rotary multipoint cutting tool. The drill bit is pressed against the workpiece followed by rotation at rates ranging from hundreds to even thousands of revolutions per minute. Because of this act cutting edge exerts force against the workpiece resulting in cutting off chips from the hole. In our experiment radial drilling machine has been used .Radial drilling machine (Specification- batliboi limited,BR61 model, 50-1600 rpm in 16 steps, 0.13-1.4 mm/rev in 8 steps) or radial arm press is a geared drill head that is mounted on an arm assembly that can be moved around to the extent of its arm reach. The most important components are the arm, column, and the drill head. The drill head of the radial drilling machine can be adjusted in height, moved and rotated. The radial drilling machine is one of the most versatile type of drill press. A radial drilling machine can perform countersinking, boring of holes, and grinding off small particles in masonry works. Some drill presses are floor mounted while the most common set-up of radial arm drill presses are those that are mounted on work benches or tables. With the latter kind of set-up, it is quite easy to mount the drill and the work pieces. Since arm can extend as far as its length could allow, there is no need to relocate and position the work pieces. Large work pieces can be easily be mounted on the table with the help of cranes as the arm can be swiveled out of the way. Below mentioned are some of the major parts of the radial arm drilling machine:

- *Column* - is the part of the radial arm drill press which holds the radial arm that can be moved around according to its length
- *Arm Raise* - It adjusts the radial arm’s vertical height along the column
- *On/Off Button* –It is the switch that starts and stops the drill press
- *Arm Clamp* – It secures the column and the arm in place
- *Table* – It is the area where the work pieces are fed and worked on
- *Base* - is the radial arm drill press part that supports the column and the table
- *Spindle* - is the rotated part of the drill press which holds the drill chuck used in holding the cutting tool

- *Drill Head* - is the part of the drill press that penetrates through the material or work piece and drill through the specific hole size
- *Radial Arm* - holds and supports the drill head assembly and can be moved anywhere around its length.

Figure 23 Radial drilling machine

4.12 Taguchi Design

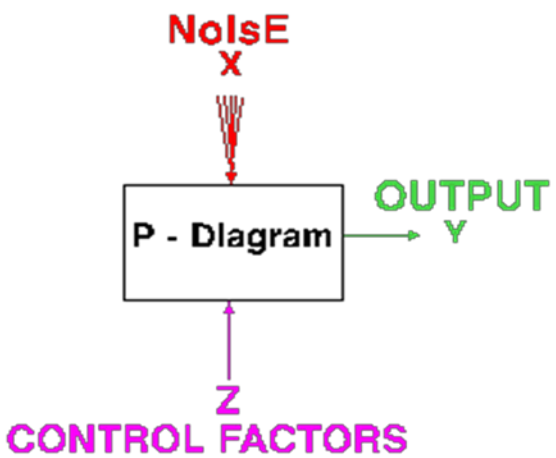
The Taguchi method developed by Genuchi Taguchi is a statistical method used to improve the quality of the product. The conventional design experimentation are very complicated and difficult to use. A large number of experimental work have to be carried out when the number of process parameters increases. To solve this problem, the Taguchi method uses a special orthogonal arrays to study the entire parameter space with only a small number of experiments. Taguchi methods have been widely utilized in engineering analysis and consist of a plan of experiments with the objective of acquiring data in a controlled way, in order to obtain information about the behavior of a given process. The greatest advantage of this method is the saving of efforts in conducting experiments; saving experimental time, reducing the cost and discovering significant factors quickly.

Taguchi's robust design method is a powerful tool for the design of a high quality product. The Taguchi method is a quality tool that helps improve the work efficiently.

It’s important to design the quality in each and every product and their corresponding process. Quality is a measure of the deviation of quality characteristics from its defined target value. Therefore, the objective is to create a design that is insensitive to all possible combinations of uncontrollable factors and is at the time effective and cost effective as a result of setting the key controllable factors at optimum levels. Taguchi offers a simple and systematic approach to optimize a performance, improving quality and reducing the cost. The quality of design can be improved by improving quality and productivity in various activities of company. Those activities concerned with quality include in quality of product planning, product design and process design. S/N ratio and OA are two major tools used in robust design. S/N ratio measures quality with emphasis on variation and OA accommodates many design factors simultaneously.

STATIC PROBLEM

Generally, a process to be optimized has several control factors which directly decide the target or desired value of the output. The technique of optimization involves determining the best control factor levels so that the output hits the target value. Such a problem is categorized as a "STATIC PROBLEM". This is best explained using a P-Diagram which is shown below ("P" stands for Process or Product). Noise however is shown to exit in the process but should have no influence on the output! This is the fundamental objective of the Taguchi experiments i.e. to minimize variations in output in the process even in the presence of noise. The process is then considered to have become ROBUST



P - Diagram for STATIC problems

CALULATIONS FOR S/N RATIO :

The Taguchi method involves a loss function to find out the quality characteristics. Loss function values are converted to a signal-to-noise (S/N) ratio. S/N ratio is defined as the ratio of the mean to standard deviation. Signal represents the square of the mean value of the quality characteristics while noise is the measure of the variability of the characteristics. In general, there are three different quality characteristics equations in S/N ratio analysis, namely "Smaller is the better", "Larger is the better" and "Nominal is the best".

(1) Smaller the better: This S/N ratio is used for all undesirable characteristics like "defects " etc. for which zero is supposed to be an ideal value . Also, when an ideal value is finite and its maximum or minimum value is defined (like maximum purity is 100% or maximum Tc is 92K or minimum time for making a telephone connection is 1 sec) then the difference between measured data and ideal value is expected to be as small as possible. The generic form of S/N ratio then becomes,

N = - 10 Log10 [mean of sum of squares of {measured - ideal}]

(2) Larger the better: This case is opposite to the lower the better case and it is obtained by taking the reciprocals of measured data. This category of S/N ratio is chosen generally when objective function like ―Material Removal Rate‖ needs to be maximized. The general formula for calculating the larger the better S/N ratio is as follows.

N = - 10 Log10 [mean of sum squares of reciprocal of measured data]

(3) Nominal the best: This case arises when a particular specific value is most desired,meaning that neither a smaller nor a larger value is desirable. The formula for determining the nominal the better S/N ratio is as follows;

$N = -10 \log_{10}$ [square of mean / variance]

CHAPTER 5

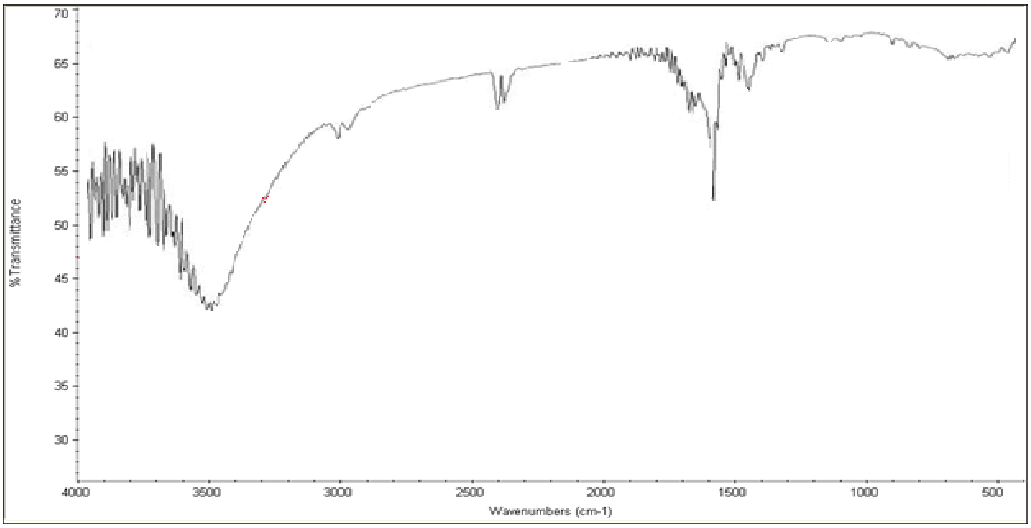
RESULTS AND DISCUSSIONS

5.1 Introduction

This chapter shows and describes the results that were obtained during various characterization tests. Result of drilling operation done on pristine MWCNT composite and functionalized MWCNT composite has been presented by employing Taguchi orthogonal array. Optical microscopy is used for studying fractured surface morphology.

5.2 Tests results for FTIR

For confirming the grafting of carboxylic group FTIR spectrum is analyzed. For the confirmation of grafting of carboxyl group onto the walls of MWCNT there must be the presence of four bonds and these are C-H bond, C-O bond, C=O bond and O-H Bond, the spectrum for these bonds shows stretching at following wave numbers : O-H stretching varies from 3300-2500 cm^{-1} and from 1440-1395 cm^{-1}, C=O stretching varies from 1800-1600 cm^{-1}, C-O stretching varies from 1400-600 cm^{-1}, C-H Stretching varies similar to O-H stretch 3300-2500 cm^{-1} . Carboxylic group is said to be successfully grafted if our spectrum lies in this range



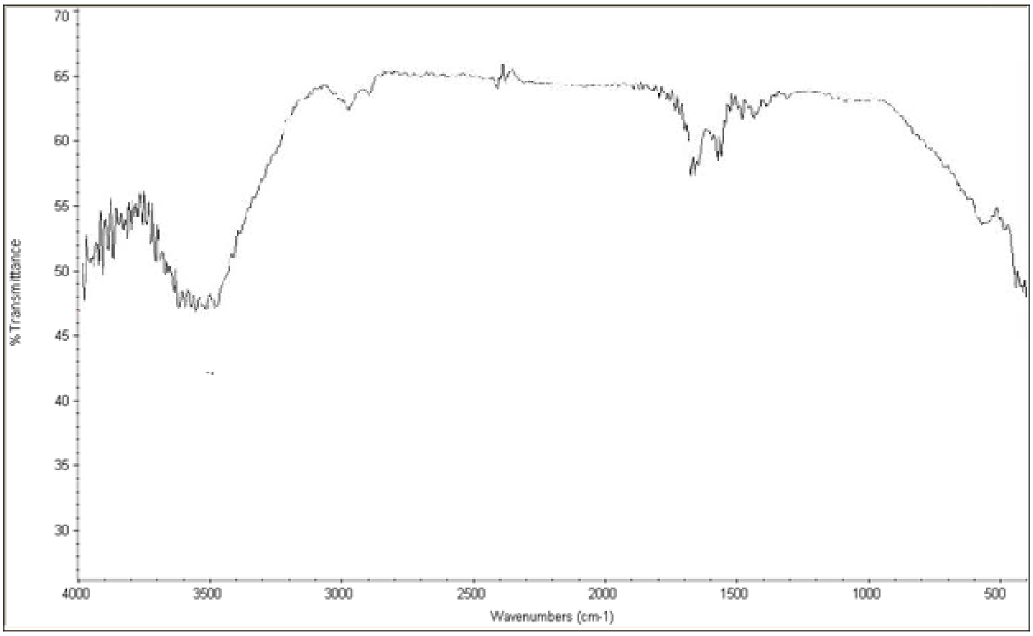


Figure 24 FTIR spectrum for pristine MWCNT and functionalised MWCNT epoxy composite

Above figure shows the FTIR spectrum for pristine MWCNT and the MWCNT sample which is to be declared fuctionalised . The graph for the sample shows the C-H peak intensity at 3011.37 cm^{-1}, 2776.28 cm$^{-1,}$, C-O peak intensity at 1230.62 cm^{-1}, C=O peak intensity at 1729.49 cm^{-1}, 1712.31 cm^{-1}, 1694.58 cm^{-1} and O-H peak intensity at 3011.37 cm^{-1} and 2776.28 cm^{-1}, 1433.49 cm^{-1}, which lies well within the range for confirming the grafting of carboxyl group. Thus, it is found that the following parameters are needed for functionalization – the mixture acid concentration is kept at 3:1, temperature of the mixture is set at 120ºC, time of heating is 30 minutes and 1 g of pristine MWCNT is taken. After this bulk preparation of functionalized MWCNT is done**.**

5.3 Characterization

5.3.1 Surface Morphology using SEM

The failed specimens of flexural tests were further observed under scanning electron microscope to study the influence of CNT functionalization on the failure morphologies. Figure below shows the state of CNT dispersion for both the pristine MWCNT epoxy and Functionalized MWCNT epoxy composites. It is evident from the figure that in the case of pristine MWCNT epoxy composite, relatively larger sized undispersed CNT bundles can be observed whereas uniform dispersion of individual CNTs were seen in case of functionalized MWCNT epoxy composite . Grafting of carboxyl group onto the walls of MWCNT is responsible not only for prevention of formation of agglomerates form but it is also responsible for improving the dispersion of MWCNTs in the nanocomposite which provides improved wetting between CNTs and epoxy. Because of the Improve wetting of CNTs by epoxy, there is better extent of adhesion which results in better interfacial interaction. Although all three materials failed in brittle manner at room temperature but the CNT presence as reinforcement in epoxy matrix modifies the fracture morphology. While for neat epoxy a relatively smooth surface containing few river lines were observed, whereas for both Functionalized MWCNT epoxy and pristine MWCNT epoxy, the fracture surfaces were found to be relatively rough with more number of river lines which is an indication of relatively large deformation of polymer matrix. The possible reason for higher deformation of polymer matrix must have been combined effect of various toughening and strengthening micro mechanisms and induced by the presence of CNT in epoxy. SEM micrographs shown below support the improvement in mechanical property caused by CNT addition in epoxy.

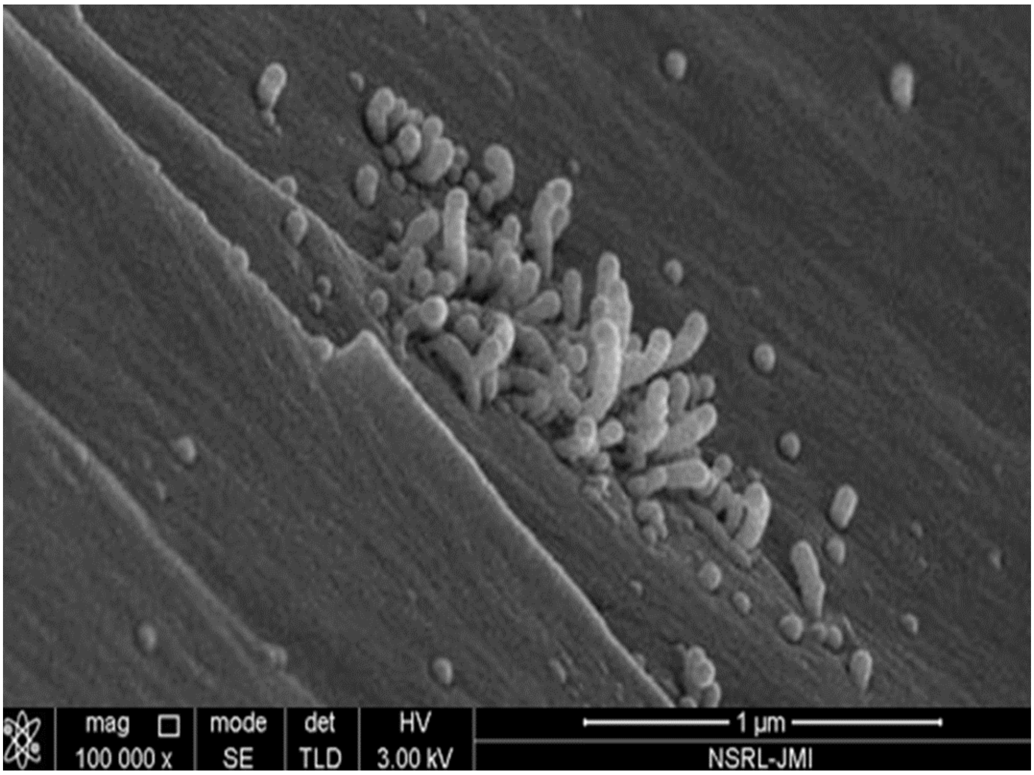


Figure 25 Extent of CNT dispersion in Pristine MWCNT epoxy composite

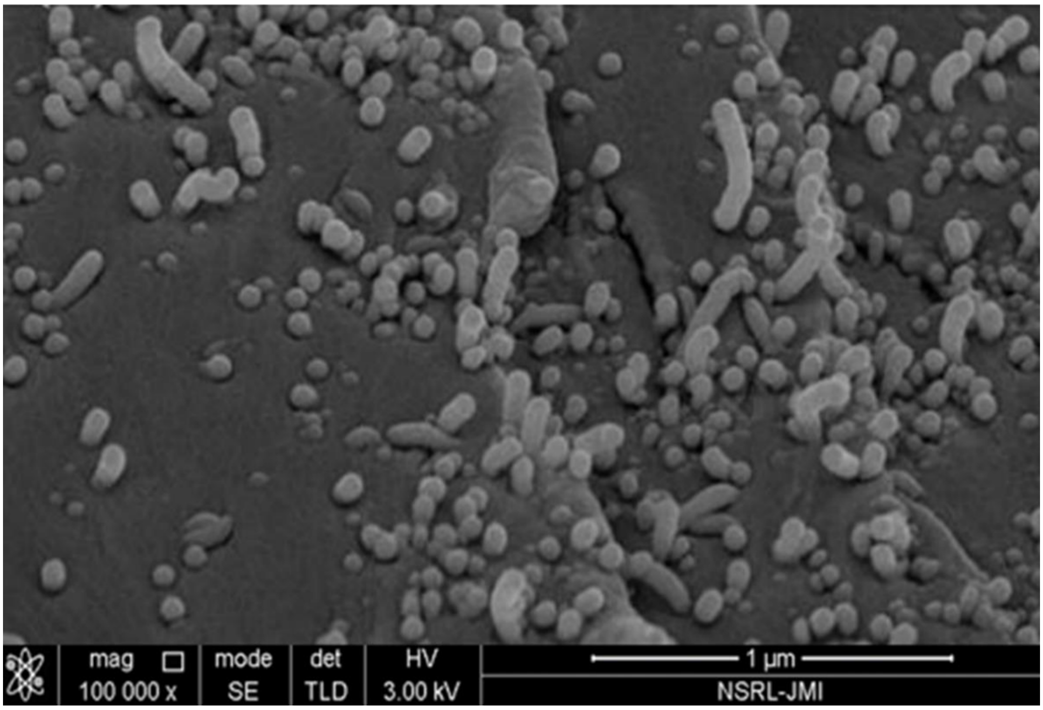


Figure 26 Extent of CNT dispersion in Functionalized MWCNT composite

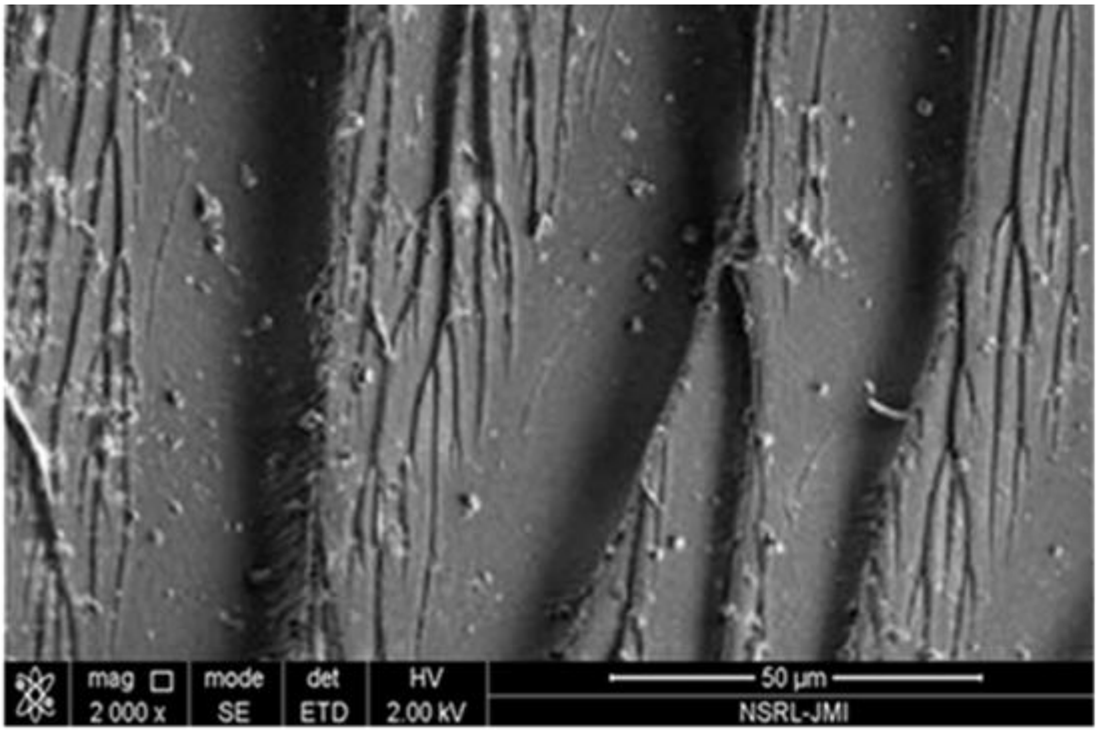


Figure 27 Fracture morphology in neat epoxy sample

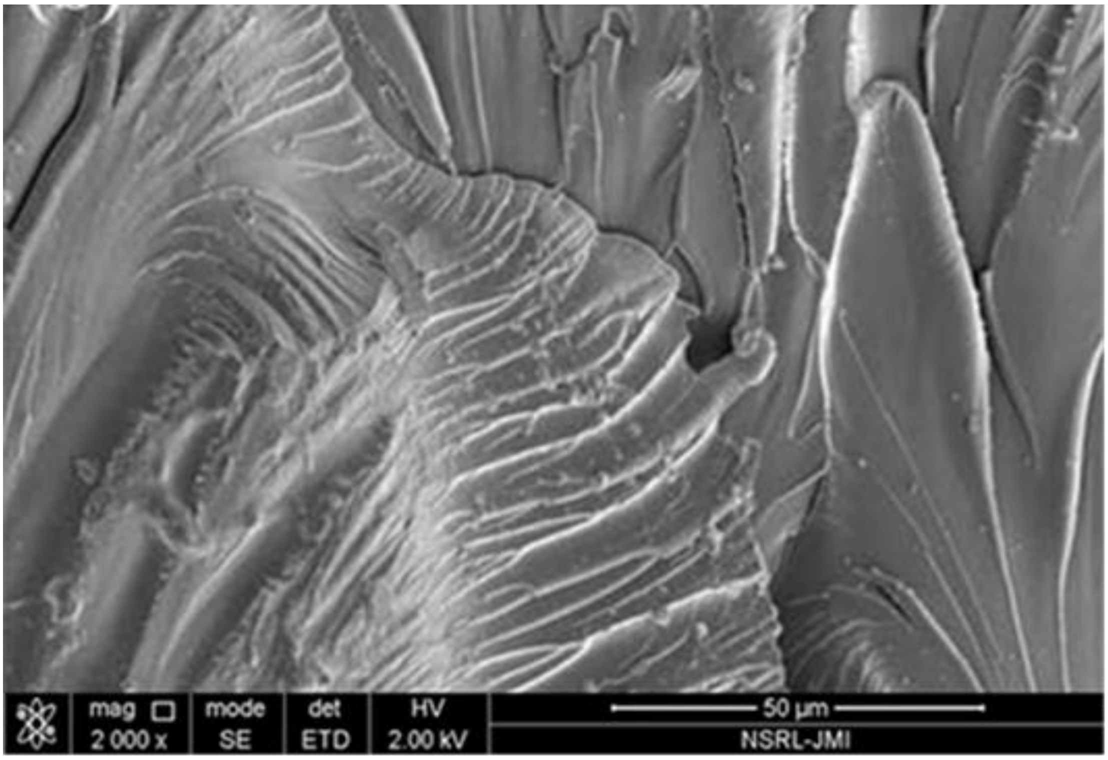


Figure 28 Fracture morphology in Pristine MWCNT epoxy composite

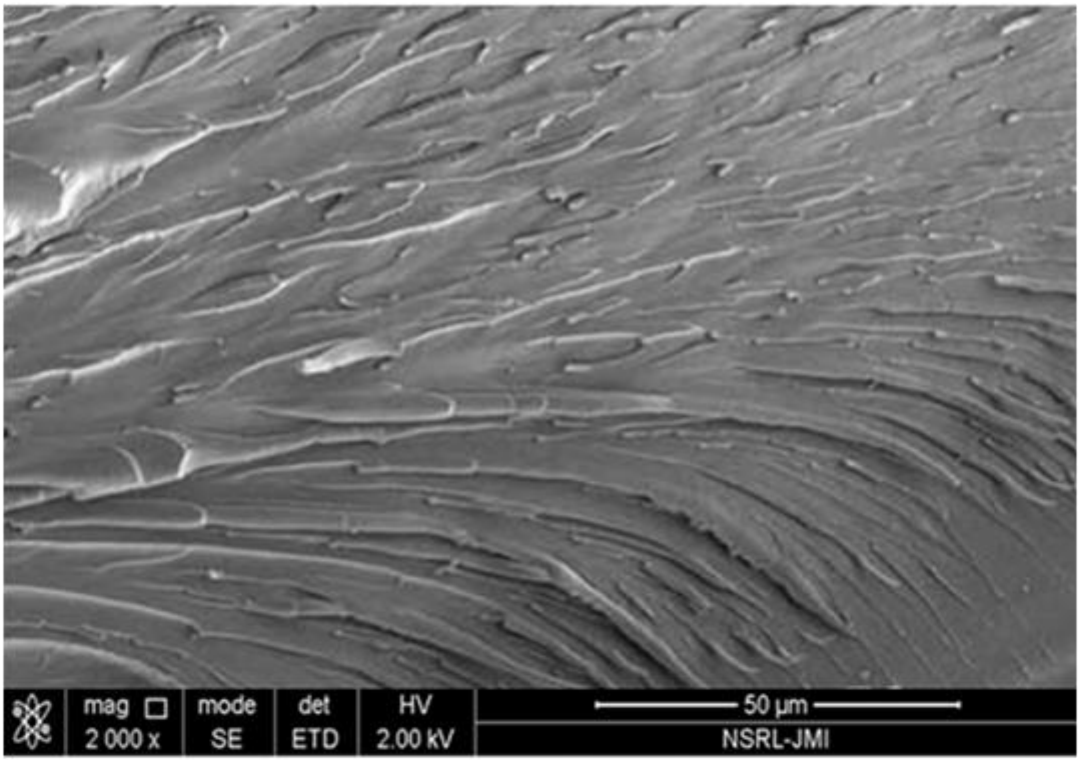


Figure 29 Fracture morphology in functionalized MWCNT epoxy composite

5.3.2 X-Ray Diffraction (XRD)

XRD pattern for pristine MWCNT and functionalized MWCNT epoxy composite are shown in Figure 27 and Figure 28 respectively, the diffraction peaks for pristine MWCNT is at 25.728º, 43.26º, 53.70º, 78.15º and for functionalized MWCNT's is at 25.763º, 42.79º, 52.93º, 78.64º. These diffraction patterns can be attributed to hexagonal graphite structure (002), (100), (004), and (110) . This shows that after the chemical functionalization of MWCNT's the structure of MWCNT's is retained even after the grafting by the carboxyl group on the surface of MWCNT.

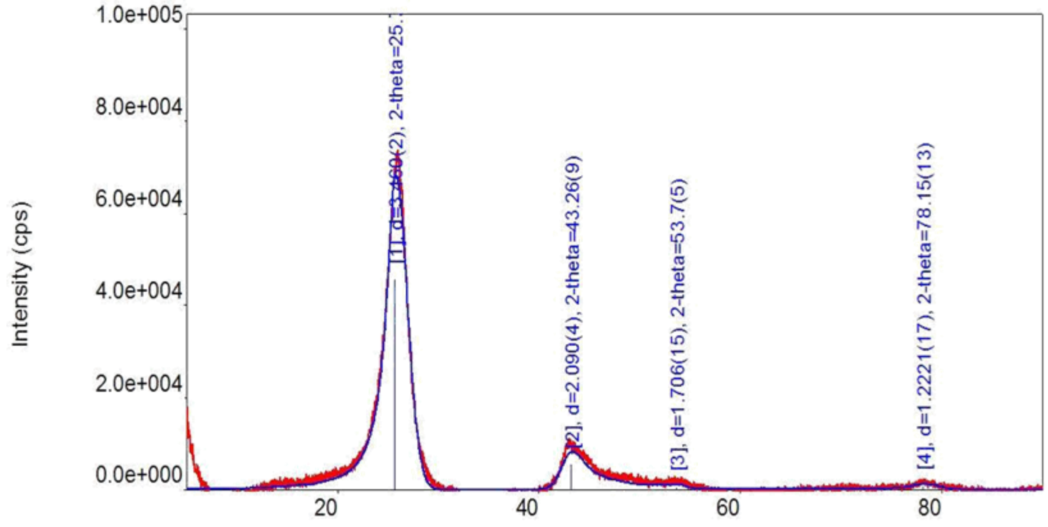


Figure 30 XRD spectrum of pristine MWCNT epoxy composite.

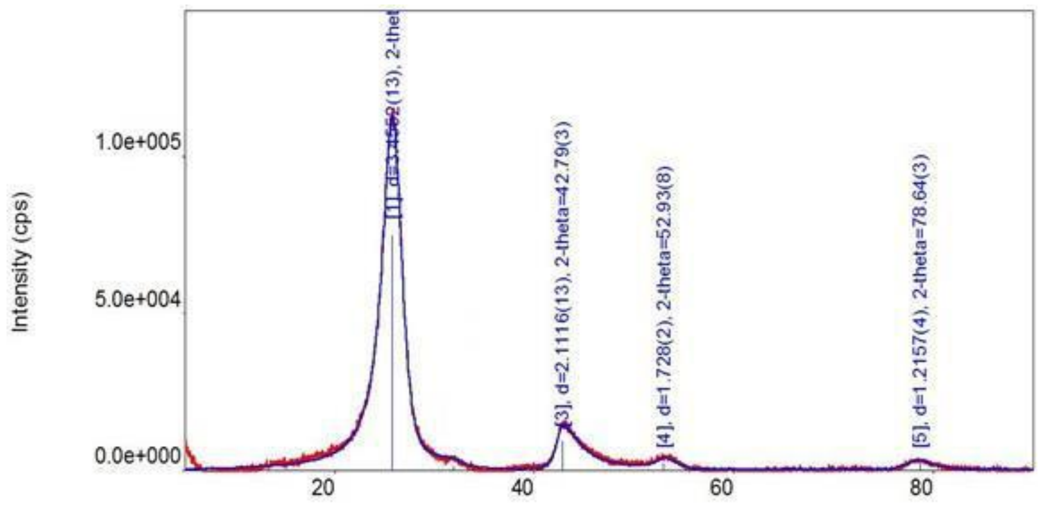


Figure 31 XRD spectrum of functionalized MWCNT epoxy composite.

5.3.3 Thermo Graviemetric Analysis (TGA)

Thermo-gravimetric analysis (TGA) studies were carried out for both the composites samples made with pristine and functionalized multi walled carbon nanotubes with 0.1 wt% to know the thermal stability of these composites. It was observed that surface modification of CNTs can alter the thermal stability of the composite materials. Composites made with pristine MWCNTs, which have only slightly affected the thermal decomposing temperature of epoxy resin whereas the acid- functionalized MWCNTs have a great effect on the onset decomposing temperature. Introducing different acid-functionalized MWCNTs to epoxy resin can increase the initial decomposing temperature of neat epoxy resin. Because of the strong interaction between the epoxy resin and acid-functionalized MWCNTs, the diffusion of small molecules can be retarded under high temperature. The surface modified CNTs could significantly enhance interfacial interaction between the CNTs and matrix in the composites. Functionalization would modify the CNT surface characteristics and enable higher polarity so that CNTs can form covalent bonds with the polymer matrix, resulting in enhanced thermal stability.

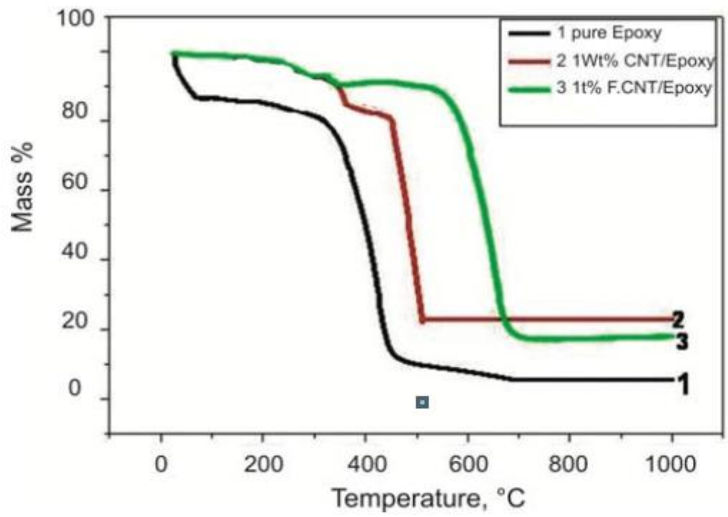


Figure 32:TGA curve for Neat epoxy , Pristine and functionalized MWCNT epoxy composites.

5.3.4 Differential Scanning Calorimetry (DSC)

Tabular data below shows the glass transition onset temperature for pristine MWCNT composite and functionalized MWCNT composite which is observed at different composition..

NEAT EPOXY	65.15 ºC
PRISTINE MWCNT EPOXY COMPOSITE	63.18 ºC
FUNCTIONALISED MWCNT EPOXY COMPOSITE	62.41 ºC

Table 1 Tg of different samples

The Tg of neat epoxy was measured to be about 65.15 ºC. Reduction in Tg to 63.18 ºC was observed due to addition of pristine MWCNT. The reduction in Tg further increased by addition of FMWCNTs and for FMWCNT composite the Tg was about 62.41ºC. The drop in Tg can be explained due to the chemical restructuring of the polymer in the close vicinity of CNT or in the interphase region. This region i.e interphase region can be assumed to be composed of two types of polymeric layers. The first one which is tightly bound to the CNTs due to multi-segment adsorption and this polymer (also called as immobilized polymer) is largely affected by the strong CNTs as they hinder the formation of cross link during curing process. The hindrance in crosslink formation reduces the cross link density in the cured polymer net-work and results in decrease in Tg. The second one is loosely bound polymer layer (or polymer of reduced mobility), which is not severely affected but has significant contribution in the interphase region and possess slightly different structure than the polymer in the resin rich zone and has relatively less contribution in the Tg of the nanocomposite. Functionalization fur-ther improves the dispersion and in turn the volume of net interphase region in the nanocomposite. Higher interphase volume leads to relatively higher volume of tightly bound polymer layers and which further reduces the Tg of the nanocomposite

5.4 Tensile Test

The tensile strength of the CNT reinforced epoxy composite sample was found good and was found increased. Test results were obtained in the form of graph between tensile load and extension, which shows that most of the response is linear, followed by brief nonlinearity before fracture. Tensile strength value as functions of loading direction was derived respectively from the gradient of the linear portion and the maximum stress before failure. The relationship between tensile load and extension for the reinforced epoxy composite is as shown and is compared with the un-reinforced normal epoxy sample. As observed, the tensile strength of Functionalized MWCNT reinforced epoxy composite was greater than pristine MWCNT reinforced epoxy composite at all composition. Although tensile strength of pristine MWCNT reinforced epoxy composite was greater than neat epoxy composite sample. These results are compared through bar chart as shown below.

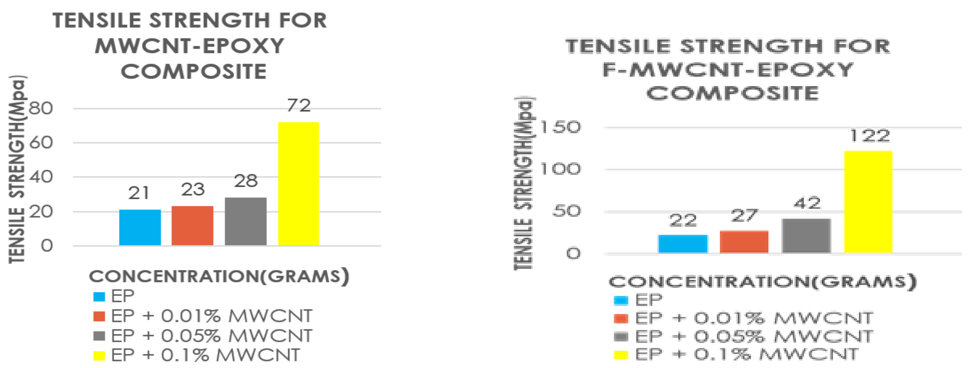


Figure 33: Tensile test results for pristine and functionalized MWCNT epoxy composite

5.5 Flexural Test Analysis

As observed, the flexural strength of Functionalized MWCNT reinforced epoxy composite was greater than pristine MWCNT reinforced epoxy composite at all composition. Although flexural strength of pristine MWCNT reinforced epoxy composite was greater than neat epoxy composite sample. These results are compared through bar chart as shown below

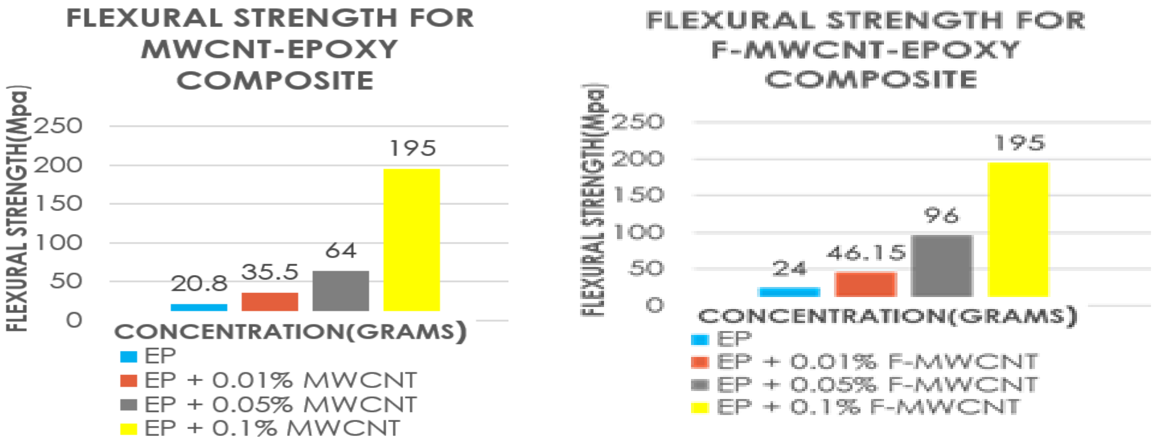


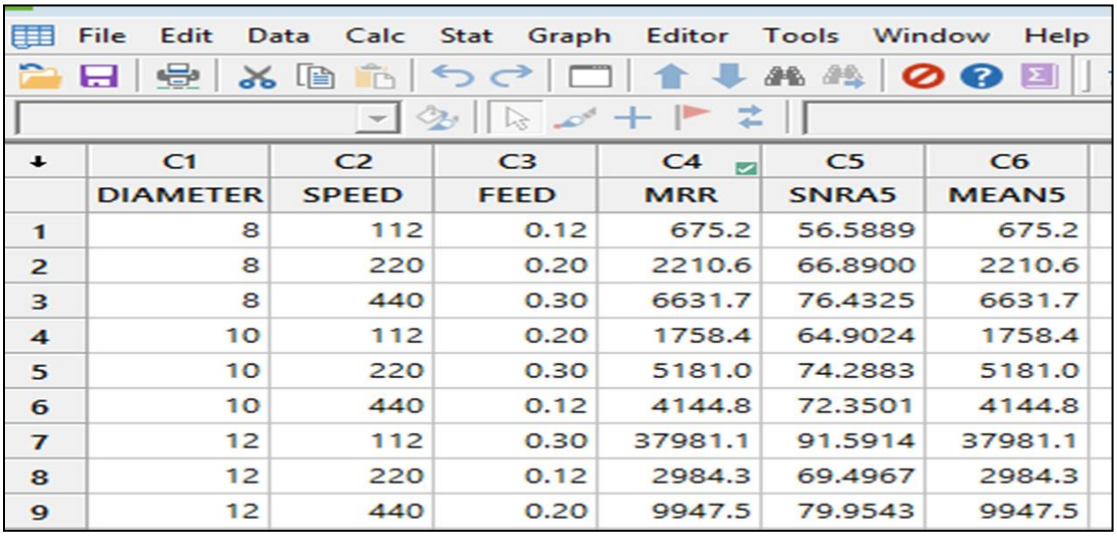
Figure 34: Flexural test for pristine and functionalized MWCNT-EP composite

5.6 Taguchi design

“Larger is better” approach is followed as we have to maximize the material removal rate. Since among the two kind of MWCNT composite, functionalized MWCNT epoxy composite has shown better mechanical properties, it can be considered for structural applications . Thus, in this regard, further machining (drilling in this case) operation was performed on functionalized MWCNT epoxy composite. Figure below shows effect of various control factors on the material removal rate for functionalized MWCNT composite , from various graphs between mean of SN ratios and control factors, it can be concluded that maximum material removal rate rate in drilling comes out to be maximum at 440 rpm, 12 mm drill bit diameter and feed at 0.30 mm/rev.

LEVEL	DIAMETER	SPEED	FEED
1	8	112	0.12
2	10	220	0.20
3	12	440	0.30

Table 2 : Levels of Taguchi design



↓	C1	C2	C3	C4	C5	C6
	DIAMETER	SPEED	FEED	MRR	SNRA5	MEAN5
1	8	112	0.12	675.2	56.5889	675.2
2	8	220	0.20	2210.6	66.8900	2210.6
3	8	440	0.30	6631.7	76.4325	6631.7
4	10	112	0.20	1758.4	64.9024	1758.4
5	10	220	0.30	5181.0	74.2883	5181.0
6	10	440	0.12	4144.8	72.3501	4144.8
7	12	112	0.30	37981.1	91.5914	37981.1
8	12	220	0.12	2984.3	69.4967	2984.3
9	12	440	0.20	9947.5	79.9543	9947.5

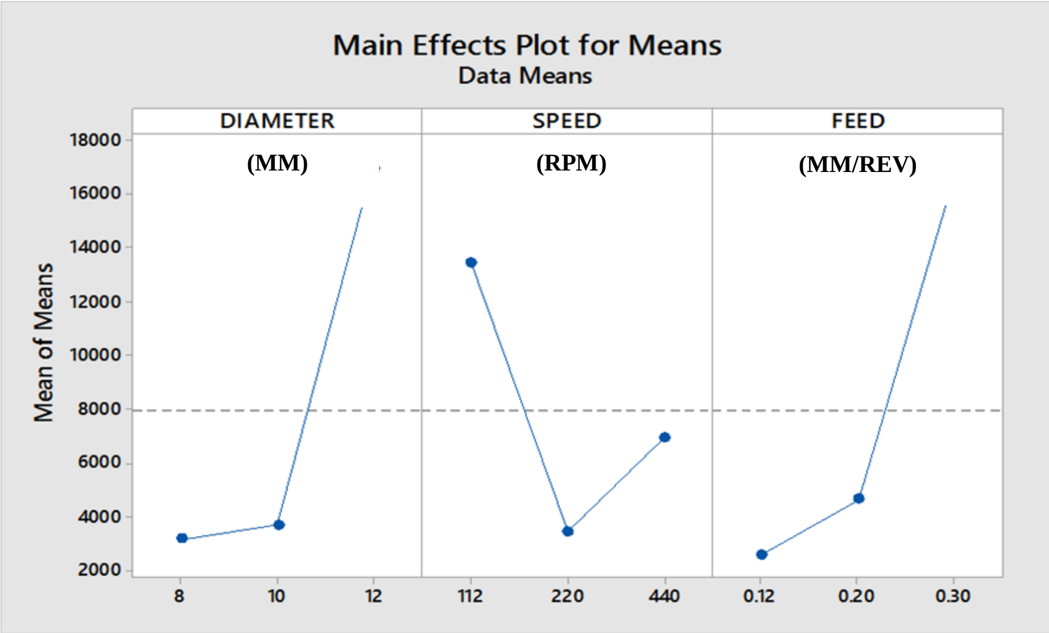
Figure 35: L9 Taguchian array for drilling parameters (Pristine MWCNT-EP composite

LEVEL	DIAMETER OF DRILL BIT	SPEED	FEED
1	66.64	64.36	66.15
2	70.51	70.23	70.58
3	73.68	76.25	74.10
Delta	7.04	11.88	7.96
Rank	3	1	2

Table 3 Response Table for Signal to Noise Ratios

LEVEL	DIAMETER OF DRILL BIT	SPEED	FEED
1	31.72	20.77	26.01
2	36.95	34.59	46.39
3	55.77	69.08	52.04
Delta	24.04	48.31	26.02

Table 4 Response Table for Means



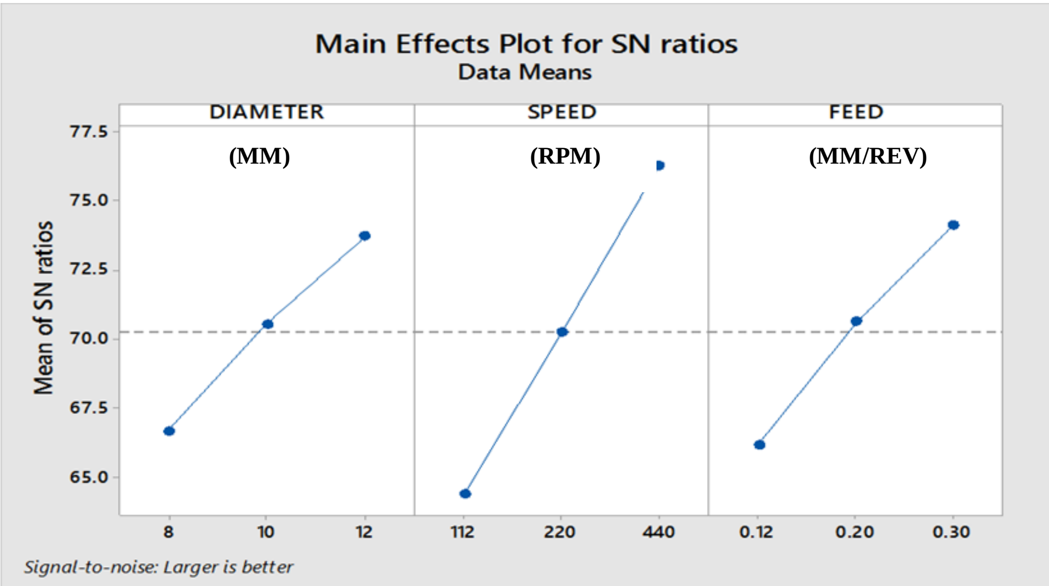


Figure 36: Graphical plot for drilling parameters (Pristine MWCNT epoxy composite)

↓	C1 DIAMETER	C2 SPEED	C3 FEED	C4 MRR	C5 SNRA1	C6 MEAN1
1	8	112	0.12	810.0	58.1697	810.0
2	8	220	0.20	2652.7	68.4738	2652.7
3	8	440	0.30	7958.0	78.0161	7958.0
4	10	112	0.20	2110.1	66.4861	2110.1
5	10	220	0.30	6217.2	75.8719	6217.2
6	10	440	0.12	4973.8	73.9338	4973.8
7	12	112	0.30	41779.2	92.4192	41779.2
8	12	220	0.12	3282.7	70.3246	3282.7
9	12	440	0.20	10942.3	80.7822	10942.3

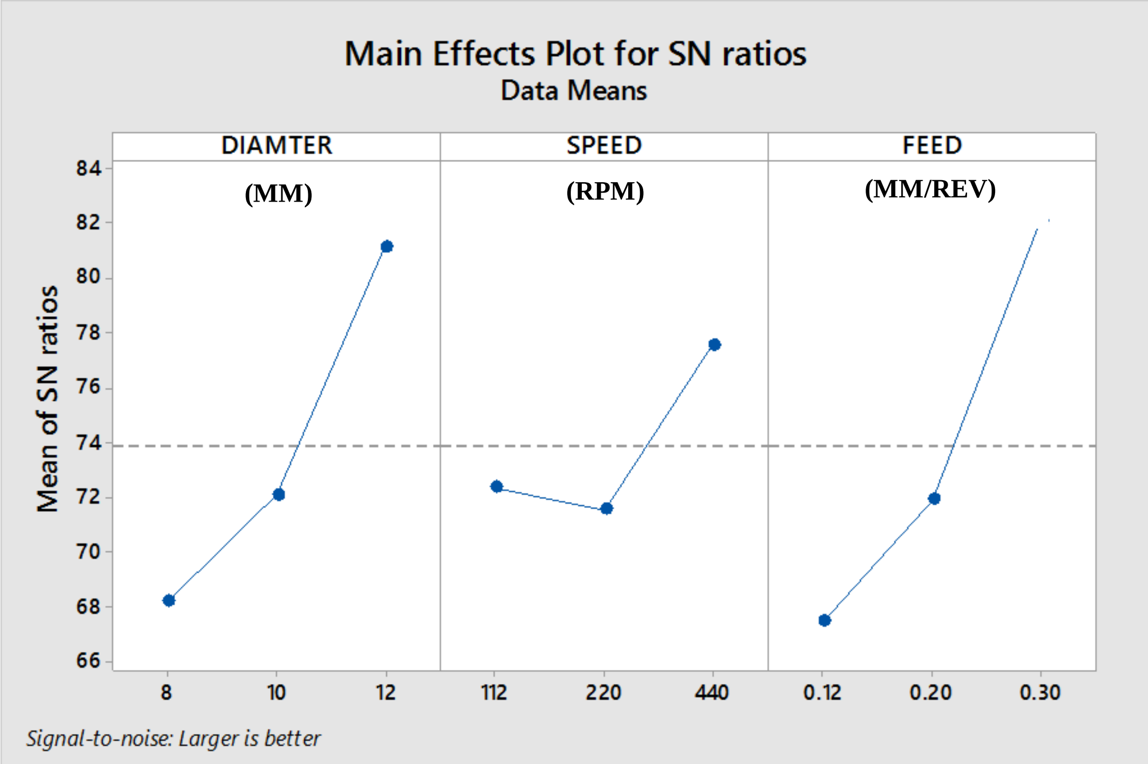
Figure 37:L9 Taguchian array for drilling parameters (Functionalised MWCNT epoxy composite)

LEVEL	DIAMETER OF DRILL BIT	SPEED	FEED
1	66.22	72.36	67.48
2	72.10	71.56	71.91
3	81.18	77.58	82.10
Delta	12.96	6.02	14.63
Rank	2	3	1

Table 5 Response Table for Signal to Noise Ratios

LEVEL	DIAMETER OF DRILL BIT	SPEED	FEED
1	3807	14900	3022
2	4434	4051	5235
3	18668	7958	18651
Delta	14861	10849	15629
Rank	2	3	1

Table 6 Response Table for Means



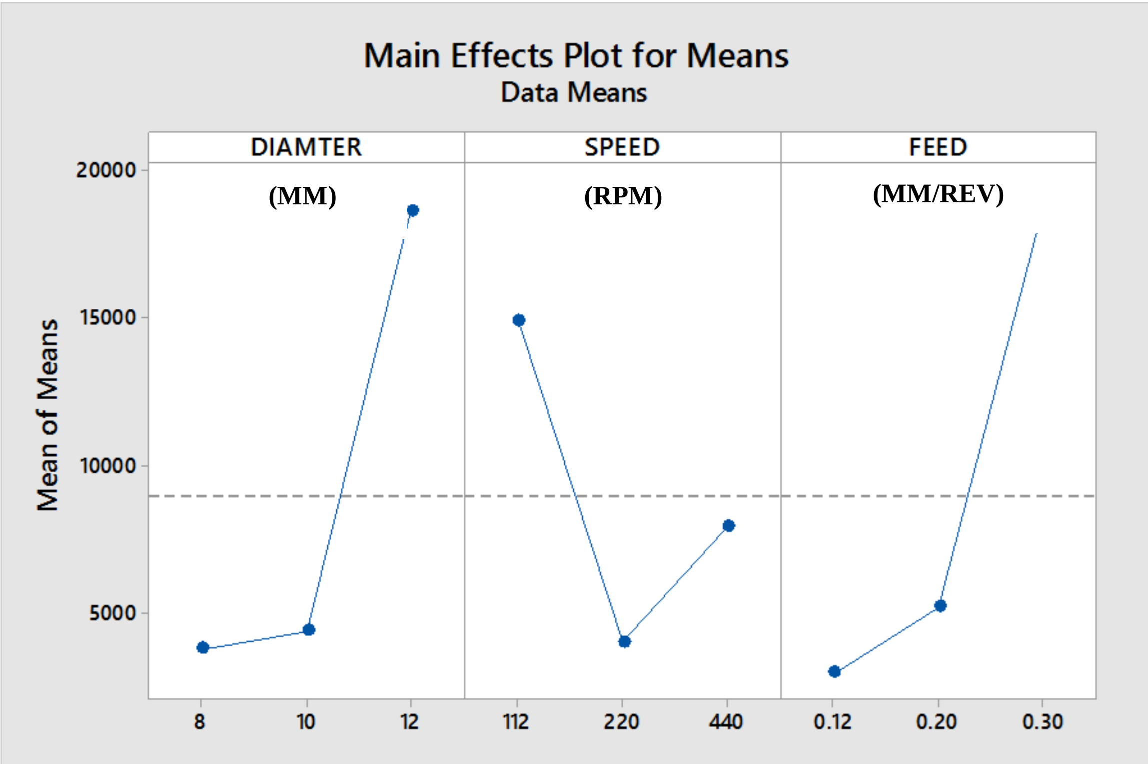


Figure 38: Graphical plot for drilling parameters (Functionalised MWCNT epoxy composite)

5.7 ANOVA and the Effects of Factors

Analysis of Variance has been done from the material removal rate data of functionalized MWCNT composite. In order to find out statistical significance of various factors viz. RPM, drill bit diameter and feed. Table shows the ANOVA result for functionalized MWCNT composite. The last column of the table indicates percentage contribution of the control factors and their interactions on the performance output i.e. material removal rate. Analysis is done for a level of confidence of significance of 5%.

Source	DF	Adj SS	Adj MS	F-Value	P-Value
DIAMETER	1	8669906	8669906	5.13	0.073
SPEED	1	37058274	37058274	21.94	0.005
FEED	1	9695817	9695817	5.74	0.062
Residual Error	5	8446980	1689396		
Total	8	63870977			

Source	DF	Seq SS	Adj SS	Adj MS	F	P
DIAMTER	2	423864078	423864078	211932039	1.65	0.377
SPEED	2	181151735	181151735	90575867	0.71	0.586
FEED	2	429173941	429173941	214586971	1.67	0.374
Residual Error	2	256613349	256613349	128306674		
Total	8	1290803103				

Table 7 ANOVA table for material removal rate (For pristine and functionalized MWCNT reinforced epoxy composites)

REGRESSION EQUATION USING ANOVA: -

MRR = -8601 + 601 DIAMETER + 14.87 SPEED + 14096 FEED

** DF: degree of freedom, ## Seq SS: sequential sum of squares, $ Adj. SS: extra sum of squares $$ Seq MS: sequential mean squares, *** F: F-test, ### P: percent contribution

From Table 7 it can be observed for pristine MWCNT composite speed (p = 0.005) have major influence on material removal rate. The interaction of Diameter of drill bit (p = 0.073) and feed rate (p = 0.062) have relatively less significant contribution. While for

COOH functionalized MWCNT composite feed (p = 0.374) have major influence on material removal rate. The interaction diameter (p = 0.377) and speed (p = 0.586) have relatively less significant contribution

CHAPTER 6

SUMMARY AND CONCLUSION

In design of an application based product its lifecycle is foremost criterion and information about machining properties provides essential evidence towards selection of a product. To this end, MWCNT's were first functionalised by the carboxyl group by the help of chemical functionalization and then are characterized by FTIR, XRD, TGA and SEM. Nano composites is fabricated by sonication and stirring and drilling operation is conducted at various parameters for functionalised Nano composites. The following conclusions are derived from this study:-

1. Parameters for functionalization of MWCNT were found to be at an acid ratio of 3:1 in a 100 ml mixture acid, temperature at 120^{o}C, time during which solution is heated is 30 minute, and 1g of MWCNT is used for the functionalization.

2. FTIR confirmed the grafting of carboxylic group on to the walls of multi walled carbon nanotubes.

3. Condition of chemical functionalization was not harsh as the structure of graphite is retained as confirmed by XRD.

4. For both the samples, that is pristine and functionalized MWCNT epoxy composite, optimum parameters for maximizing material removal rate were obtained at speed 440 rpm, feed 0.03 mm/rev and drill bit diameter of 12 mm. For functionalized MWCNT composite, speed ($p = 0.005$) have major influence on material removal rate. The interaction of Diameter of drill bit ($p = 0.073$) and feed rate ($p = 0.062$) have relatively less significant contribution. While for COOH functionalized MWCNT composite, feed ($p = 0.374$) has major influence on material removal rate. The interaction diameter ($p = 0.377$) and speed ($p = 0.586$) have relatively less significant contribution.

5. Material removal rate at any parameter has been found to be higher for functionalized MWCNT-epoxy composite than of pristine MWCNT epoxy composite. Secondly, tensile and flexural strength for functionalized MWCNT composite is higher than that of pristine MWCNT composite thus functionalized MWCNT composite has higher load resistance. This variation is attributed to the better dispersion of MWCNT in epoxy matrix medium in case of functionalized MWCNT epoxy composite which makes it stiff and harder to exhibit better mechanical and thermal properties.

6.1 Recommendations for Potential Applications

Carbon nanotube reinforced epoxy matrix composite fabricated in this research work found to have adequate potential for wide variety of applications in accordance to machining point of view. Some of them may include manufacturing of light weight sports equipment, wind turbine blades, nozzle, aircraft brake, disk and linings, ultra-light weight aerospace structures, gears, bearings etc.

6.2 Scope for future work

There is a wide scope for future investigators to explore many other aspects of current research work. Some recommendations for future research include: -

1. Study of machining behaviour of functionalized composite by doing functionalization by amide group.

2. Study of response of composite in study to other aspects of machining.

3. Nano composite may be combined with hybrid composite to arrive at variety of new results.

4. For use in industrial mass production, after analysing their cost effects according to their application.

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