1. INTRODUCTION

1.1 Background

In general, nanocomposites exhibit gains in barrier, flame resistance, structural, and thermal properties yet without significant loss in impact or clarity. Because of the nanometer-sized dimensions of the individual platelets in one direction, exfoliated Nanomer Nano clays are transparent in most polymer systems. Nanocomposites also demonstrate enhanced fire-resistant properties and are finding increasing use in engineering plastics. Due to their mechanical properties nanoparticles (NPs) like multiwalled carbon tubes (MWCNTs), carbon Nano fibers (CNFs), Graphene (Ga) are considered to be some of the most promising reinforcing materials for the next generation high-performance Nano composites. Increased mechanical stability in polymer-clay nanocomposites also contributes to an increased heat deflection temperature.

In plastics the advantages of nanocomposites over conventional ones don't stop at strength. The high heat resistance and low flammability of some nanocomposites also make them good choices to use as insulators and wire coverings. Another important property of nanocomposites is that they are less porous than regular plastics, making them ideal to use in the packaging of foods and drinks, vacuum packs, and to protect medical instruments, film, and other products from outside contamination. nanocomposites include improvement in modulus, flexural strength, heat distortion temperature, barrier properties, and other benefits and, unlike typical mineral reinforced systems, they are without the conventional trade-offs in impact and clarity.

Research in the development of polymer based composites for structural applications is gaining prominence in the present scenario. This is because of their unique light weight and high strength properties. Plain polymer alone cannot provide the deserved strength required for the structural applications due to the brittle nature of the plastics. Such drawbacks of polymeric materials can be suitably addressed by reinforcing it with strength fillers at both micro and nano-level. However, continuous effort has been made by several investigators to improve the mechanical properties of polymers by

adopting several reinforcement techniques. This is the need of the hour required for several structural applications such as aviation, marine, automotive and polymer industries. Recently the usage of Nano-materials in polymer-based matrix is gaining tremendous importance due to their distinctive physical and chemical characteristics as compared to conventional strength fillers like carbon fibers, natural fibers, etc. Nano-composites for structural applications have been making inroads into the polymer industries due to their unique properties.

Although nanoparticles are ideal reinforcement materials when compared to their counterpart conventional fillers the main problem associated with their use in polymer matrix depends on the dispersion technique adopted while preparing the Nano-composites. Dispersion is a critical issue which needs to be addressed for practical realization of the Nano-composites for industrial applications. The factor which make nanoparticles an ideal reinforcement is their large surface-to-volume ratio as compared to the other conventional fillers. However, the difficulty in the dispersion of Nano-particles in the polymer matrix is because of the strong interaction provided by Vander Waals forces that exist between themselves. The inter-nanoparticle interactions make it extremely difficult for homogeneous nanoparticles dispersion especially at higher particle dosage levels.

Viscosity of the holding matrix increases with the rise in filler concentration. The increase in the viscosity of the holding matrix can be attributed to the high surface adhesion of polymer molecules to the surface area of Nano-particles resulting in the reduction of polymer chains mobility. With all these difficulties associated in the development of polymer nanocomposites for the required applications this area needs rigorous research before it can be realized for practical scale production. Several dispersion techniques such as shear mixing, mechanical mixing, in-situ polymerization and sonication have been used for dispersing nanoparticles in the polymers.

1.2 Nano-fillers for structural applications:

Due to their exceptional mechanical properties nanoparticles (NPs) like Multiwalled
Carbon Tubes (MWCNTs), Carbon Nano Fibers (CNFs), Graphene (Ga) are
considered to be some of the most promising reinforcing materials for the next
generation high-performance Nano-composites. NPs like MWCNTs, CNFs, Ga on the
other hand has outstanding mechanical properties, making it ideal reinforcing
materials at Nano-scale in polymer based matrix.

1.2.1 Multi Walled Carbon Nano Tubes (MWCNTs)

This section discusses the effect of MWCNTs as Nano-level reinforcement in polymer-
based matrix for structural applications. Nano science attracts a lot of attention from
both scientific community as well as industry. In the case of civil engineering, a
substantial interest can be observed in nan-sized materials, such as Nano-silica fume,
nanofibers and nanotubes. Nanofibers and nanotubes are anticipated to introduce Nano-
reinforcement, which would enable a decrease of the required amount of steel
reinforcement and corrosion problems affecting the concrete structure.

Fig. 1.1 MWCNTS used as filler in the present work

1.2.2 Carbon Nano Fibers (CNFs)

This section discusses the effect of CNFs as Nano-level reinforcements in polymer-based matrix for structural applications. CNFs are an option for effective reinforcement because of their aspect ratio. CNFs have become ideal reinforcement because of their mechanical properties such as specific modulus ~ 500 GPa and specific strength ~3 GPa of individual nanofibers. CNFs reinforced polymer are gaining importance because of these distinguished mechanical properties. Elemental carbon has a no. of allotropes including diamond, graphite and fullerenes. Thought they all consist of elemental carbon; their properties vary widely. This underscores the versatility of Carbon Nano Fibers (CNF's), which are notable for their thermal, electrical, electromagnetic shielding and mechanical properties enhancement.

Fig.1.2 CNFs used as filler in epoxy

1.2.3 Graphene (Ga)

This section discusses the effect of Graphene as nano-level reinforcement in polymer-based matrix for structural applications. Plastics are replacing metals in structural applications. Epoxy is one of the polymer which is curable at the laboratory scale. The mechanical properties of the plastics can be modified by adding different nanoparticles and carbon-based materials. Ga have been considered as one of the effective reinforcement nanoparticles for polymer-based composites owing to their outstanding mechanical properties as well as their high surface area.

Fig.1.3 Graphene used as filler in epoxy

1.3 Use of Nano level reinforcement in polymer-based matrix for Engineering application.

This section discusses the combined effect of Nano-level reinforcements in polymer based matrix for structural applications. In fact, some GA are stronger than steel, lighter than aluminium and more conductive than copper. Theoretical and experimental studies have shown that Ga exhibit extremely high tensile modulus (>1,000GPa) and strength (>5GPa). Due to this unique combination of physical and mechanical properties, Ga have emerged as excellent candidates for use as reinforcing agents in polymeric materials to yield the new generation nanocomposites. However, reinforcement of fillers like Graphene (Nano) in Polymer based Matrix for structural applications has been less investigated.

1.4 Problem Identification:

Recently practical realization of composites for structural applications with use of nano-scale reinforcement is gaining prominence when compared to micro-scale fillers. This is mainly because of the unique blending of mechanical, chemical and physical properties associated with the use of nano-fillers preferably with a characteristic dimension typically below 50 nm. Apart from this, study on the combination of fillers usage at nano level is scantily reported in the literature and this area needs to be rigorously explored to realize the benefits of such reinforcements.

2. LITERATURE REVIEW

Microstructure property relationships are at the heart of modern material science. Nanotechnology is one of the most intense research areas for innovative engineering applications. The development of fillers at the Nano-scale has opened a new field of research in polymer technology. In view of this, detailed literature pertaining to the processing and characterization of polymer-based Nano-composites for varied engineering applications using various Nano-particles such as Multi-Walled Carbon Nanotubes (MWCNTs), Graphene Oxide (GO), Carbon Nano Fibers (CNFs) etc. at Nano level and Carbon Fiber (CF), Kevlar etc. at micro scale reinforcements have been reviewed and discussed.

1) Review of Fiber Reinforced Polymer Composites

Shivakumar S, G. S. Guggari (2011) [1] studied that, polymer-matrix composites (PMCs) have been used for a variety of structural memberships for chemical plants and airplanes, since they have outstanding performances, such as lightweight and good fatigue properties. To hold the long-term durability and to estimate the residual life of the composites under some hostile environments, it is an important issue to clarify the facture and/or the failure mechanism in each service conditions. Degradation of components made from polymeric materials occurs in a wide variety of environments and service conditions, and very often limits the service lifetime. Degradation occurs as the result of environment-dependent chemical or physical attack, often caused by a combination of degradation agents, and may involve several chemical and mechanical mechanisms. The main concern of this review will be to examine the causes of degradation of polymeric components from the completion of fabrication to ultimate failure.

2) Review on the Processing and Properties of Polymer Nanocomposites and Nanocoating's and Their Applications in the Packaging, Automotive and Solar Energy Fields.

Kerstin Müller, Elodie Bugnicourt, Marcos Latorre, et. al. (2017) [2] studied that, for the last decades, nanocomposites materials have been widely studied in the scientific literature as they provide substantial properties enhancements, even at low nanoparticles content. Their performance depends on a number of parameters but the nanoparticles dispersion and distribution state remain the key challenge in order to

obtain the full nanocomposites' potential in terms of, e.g., flame retardance, mechanical, barrier and thermal properties, etc., that would allow extending their use in the industry. While the amount of existing research and indeed review papers regarding the formulation of nanocomposites is already significant, after listing the most common applications, this review focuses more in-depth on the properties and materials of relevance in three target sectors: packaging, solar energy and automotive. In terms of advances in the processing of nanocomposites, this review discusses various enhancement technologies such as the use of ultrasounds for in-process nanoparticles dispersion. In the case of nanocoating's, it describes the different conventionally used processes as well as nanoparticles deposition by electro-hydrodynamic processing. All in all, this review gives the basics both in terms of composition and of processing aspects to reach optimal properties for using nanocomposites in the selected applications. As an outlook, up-to-date Nano safety issues are discussed.

3) Nano clay Reinforced Polymer Composite and Its Application to Hybrid Composite Structures.

Gefu Ji, (2008) [3] studied that, Changes in mechanical properties associated with each mixing combination were examined through a variety of tests, which were low velocity impact test, bending test, compression test, and dynamic mechanical analysis (DMA). Additionally, changes in thermal properties (e.g., thermal diffusivity, glass transition temperature) and fire-retardant properties associate with each mixing combination were also evaluated. The result was compared with pure vinyl ester resin. Finally, an optimum mixing combination was proposed. In order to additionally approve the effect of exfoliated MMT/VE, it was introduced to Advanced Grid Stiffened Fiber Reinforced Polymer (AGS-FRP) tube encased concrete cylinders and to improve their fire resistant behaviour and mechanical property. Nanocomposites have the potential of offering improved physical properties over conventional composites. Mixing technique plays a critical role in transition from conventional composites to nanocomposites.

4) Use of Nanoparticles for Enhancing the Interlaminar Properties of Fibre-Reinforced Composites and Adhesively Bonded Joints.

Davide De Cicco, Zohreh Asaee, Farid Taheri (2017) [4] studied that, at reporting some of the notable works carried out concerning the use of nanoparticles (NPs) as a means of improving the resistance of fibre-reinforced polymer composite materials (FRPs) and adhesively bonded joints (ABJs) to delamination initiation and propagation. Applications of various nanoparticles, such as carbon-based, ceramic-based and mineral-based are discussed. The main properties that have been considered for improving the delamination and fatigue resistance of FRPs are the interlaminar shear strength, fracture toughness, and fracture energy. On the other hand, cohesive and interfacial strengths have been the focused parameters in the works that considered enhancement of ABJs. The reported results indicate that inclusion of NPs in polymeric matrices leads to improvement of various material properties, even though some discrepancies in the results have been noted. Notwithstanding, additional research is required to address some of the issues that have not yet been tackled, some of which will be identified throughout this review article.

5) Mechanical And Tribological Behaviour Of Nanofiller Reinforced Polymer Nanocomposite

Gujjala Raghavendra, (2014) [5] studied that, increasing demand for special materials lead to new inventions, one of the most promising inventions is the concept of composites. We always will strive to understand and modify the world around us and the stuff of which it is made. A new era is opened when the thorough understanding of a particular material and combining the valuable properties of the different materials are taken together to optimize the individual properties. Nowadays, engineering material at the atomic and molecular levels are creating a revolution in the fields of materials and processing. Nanoscale particles are presently considered to be high potential filler material for improving the properties of the existing material. Due to the Nano technology the polymer composites are become more attractive. As surely as polymer composites changed the face of industry twenty-five years ago, polymer nanocomposites will usher in a new era in materials development. Therefore, the present research work has been under taken with an objective to produce Nano-fly ash and alumina Nano particles in the laboratory by utilizing

the method of high energy ball milling and by auto combustion process. It is also planned to use these nanofillers as reinforcing material in polymer composite. The low modulus of glass fibre composites have limited their use in applications where buckling stability or high natural frequency is the criteria. It is also known that natural fibres composite possesses much lower mechanical strength properties than synthetic fibre reinforced composite. Hence the use of natural fibre alone in polymer composite is inadequate in satisfactorily tackling all the technical needs of a fibre reinforced composite. It is reported that if natural fibre is hybridised with a synthetic fibre in the same matrix the properties of natural fibre could be improved by taking the advantage of both the fibres.

6) Nanocomposites: Synthesis, Structure, Properties and New Application Opportunities.

Pedro Henrique Cury Camargo, Kestur Gundappa, et. al. (2009) [6] studied that, Nanocomposites, a high-performance material exhibit unusual property combinations and unique design possibilities. With an estimated annual growth rate of about 25% and fastest demand to be in engineering plastics and elastomers, their potential is so striking that they are useful in several areas ranging from packaging to biomedical applications. In this unified overview the three types of matrix nanocomposites are presented underlining the need for these materials, their processing methods and some recent results on structure, properties and potential applications, perspectives including need for such materials in future space mission and other interesting applications together with market and safety aspects. Possible uses of natural materials such as clay-based minerals, chrysotile and lignocellulosic fibres are highlighted. Being environmentally friendly, applications of nanocomposites offer new technology and business opportunities for several sectors of the aerospace, automotive, electronics and biotechnology industries.

7) Mechanical Properties of Polymer Composites Filled with Nanoparticles

Marc Nogués Piñol (2015) [7] studied that, the recent discovering of new carbon allotropes has opened a new door in the field of Nanomaterials. Among all of them, carbon nanotubes (CNTs) might be the most fascinating material due its unique electrical, mechanical and thermal properties. Polymeric Nanocomposites (PNCs) combine the lightness and low-cost manufacturing of polymers with CNTs high

performance properties, being an attractive alternative to heavier metals or expansive ceramics. Thus, demand for new and stronger materials is constant in the PNCs industry. The aim of this project is to enhance friction and mechanical properties of Epoxy, Polyamide (PA) and Acrylonitrile butadiene styrene (ABS) using a wide range of different nanofillers. 1wt% and 3wt% filled Epilox T19-36/700 epoxy was used for the friction analysis, using the reciprocating Anton Paar Pin-on-Disk Tribometer. One sample face was abraded against a 6mm diameter steel AISI 420 sphere while the other one was tested using the same material. For the mechanical analysis, 0,5wt% carbon soot ABS and PA wires were manually manufactured using an industrial extruder and tested in a QTest Elite 10 tensile machine.

8) Polymer-Nanoparticle Composites: From Synthesis to Modern Applications

Thomas Hanemann, Dorothée Vinga Szabó, (2010) [8] studied that, The addition of inorganic spherical nanoparticles to polymers allows the modification of the polymers physical properties as well as the implementation of new features in the polymer matrix. This review article covers considerations on special features of inorganic nanoparticles, the most important synthesis methods for ceramic nanoparticles and nanocomposites, nanoparticle surface modification, and composite formation, including drawbacks. Classical nanocomposite properties, as thermomechanical, dielectric, conductive, magnetic, as well as optical properties, will be summarized. Finally, typical existing and potential applications will be shown with the focus on new and innovative applications, like in energy storage systems.

9) Mechanical and tribological studies of polymer hybrid nanocomposites
with nano reinforcements

D Lingaraju, K Ramji, M Pramila Devi, U Rajya Lakshmi, (2010) [9] studied that, Developments of nanoparticle reinforced plastics are of growing interest towards the emergence of new materials which enhance optimal utilization of natural resources and particularly of renewable resources. The effects of nanoparticles as fillers in glass–epoxy composite systems on the mechanical and tribological properties have been discussed in this article. The mechanical properties such as tensile strength, impact strength, flexural strength, and hardness have been studied in accordance with ASTM standards. The composites employed in the study have been fabricated using hand lay-up technique. By varying notch radius impact strength is studied. The clay and silica

used in the present system were treated with 3-aminopropyltriethoxysilane. The effect of variants in sliding speed, time and applied load on the wear behavior of polymer nanocomposites is studied by measuring the weight changes and observing the surface features using scanning electron microscope. In the experiments with wear test pin having flat face in contact with hardening rotating steel disc, sliding speed, time and loads in the range of 640–1000 RPM, 300–900 s and 5–25 N respectively was used. It is observed that wear rate increases with increasing applied load, time and sliding speeds.

10) Conventional fillers used in polymer-based matrix:

This section presents the feasibility studies on the use of micro-fillers (both synthetic and natural) in polymer-based matrix for varied structural applications.

Ozdemir et. al., (2015) [10] studied the effects of Nano-particle reinforcement such as carboxylic acrylonitrile butadiene rubber (CNBR-NP) and Nano- acrylonitrile butadiene rubber (NBR-NP) in dicyandiamide epoxy matrix. It was reported in the study that the addition of these fillers resulted in the delamination fracture toughness improvement (GIC) of the CFRP by 250% while its delamination fracture toughness (GIIC) by 80%. During the last few years, green composites are gaining prominence as alternative materials for aerospace, automotive and construction sectors. Green composites are renewable in nature, economical and biodegradable.

Natural fibers especially plant fibers as they are 'carbon positive' and absorb more carbon dioxide than they release (McDonald et. al.2006). Many bio-composites which are catering to domains like automotive, construction and packaging industries (Fowler et. al. 2006).

11) Carbon Nano-tubes (CNTs):

Several investigators have used CNTs as strength filler in polymer-based matrix and carried out critical analysis for their practical applications as presented below.

Martone et. al., (2010) [11] studied the effect of mechanical property such as flexural strength of carbon nanotube (MWCNT) reinforced epoxy matrix. Beyond 0.3wt.% of the fillers used in the holding matrix showed a decrease of the bending modulus of the modified beams when compared to conventional control beams.

12) Graphene:

Several investigators have used graphene as strength fillers in polymer-based matrix and the analysis of the results obtained are presented below.

Cheng et. al., (2015) [12] studied the effect of Nano-filler Graphene oxide (GO) in polymer matrix composed of polyaniline (PANI) and divinylbenzene. The developed composite beams showed higher electrical conductivity (0.02–9.8 S/cm) compared to the plain polymer as well as system when added with only PANI (109–101 S/cm), By 0.45 wt% loading of GO, 29.9% enhancement in bending modulus was observed.

13) Carbon Nano-fibers:

Several investigators have used carbon Nano-fibers as strength fillers in polymer-based matrix and the analysis of the results obtained are presented below.

Ali et. al., (2013) [13] studied the spraying effect of CNF on CF based composites. Compression, tensile, short beam strength tests and flexural were performed to assess the efficiency of CNF accumulation on carbon/epoxy composites. The results obtained led to the change in the mechanical performance by 22-28% in tensile strength, 14-19% in flexural strength, 7-11% compressive strength and 45-55% short beam strength as compared to unmodified beams.

14) Dispersion issues of Nano-particles in polymer-based matrix:

Dispersion plays significant role in the preparation of the Nano-composites and several researches have adopted different methods of dispersion and the analysis of same is presented below.

Arras et.al., (2013) [14] conducted experiments to align MWCNT in polymer composite in one step process. Based on the study they suggested that uniform alignment of CNT can be obtained by melt drawing technique.

2.1 OBJECTIVE

➤ To develop a novel hybrid polymer-based Nano-composites using commercially available Nano-fillers viz.Graphene.

➤ Fabricate polymer-based Nano-composites using different Nano-fillers viz., CNTs, GA, CNFs with varied composition.

➤ Study the effect of the Nano-filler concentration on the mechanical properties of the Nano-composites.

➤ Perform Mechanical tests like Flexural Testing (three point loading), Tensile testing etc.

➤ Making of light weight material for applications like turbine blades, wind mill blades, car interior, drone and aircrafts.

2.2 PROPOSED WORK

The methodology adopted for the study includes:

a) Selection of optimum percentage of NPs (Nanoparticles) required for the development of new hybrid polymer composite material.

b) The experiments were performed as per full factorial design (FFD) to analyze the interaction effects of process parameters.

c) To evaluate standard procedure for the dispersion of NPs in polymer-based matrix and standard procedure for the preparation of the Nano-composite specimens.

d) Development of new hybrid Nano-composite specimens and further structural scale testing and micro-structural scale testing to assess the structural performance of NPs at Nano-level.

3. METHODOLOGY

3.1 Materials

Several researchers have reported use of different polymer systems with various reinforcements at micro, Nano and combination of micro-Nano fillers to develop polymer-based composites. However, reinforcement of fillers like Graphene (Nano) and Carbon Fiber in polymer-based matrix for structural applications has been less investigated. In the present work, mechanical performance of polymer beams reinforced with graphene, carbon fibers, combination of graphene and carbon fibers is studied and the results were compared with that of plain beams such as load versus deflection criteria.

3.1.1 Materials Selected for the study

This section focuses on the materials used in the sample preparation for the development and characterization of the composites under study. It gives the specifications of the tests in terms of mechanical, physical, micro-structural of the modified specimens.

3.1.2 Matrix Material

Base matrix for holding the reinforcement materials may be of diverse kind like metals, ceramics and polymers. Among the different base matrices polymer matrix is intentionally selected because of economic viability, ease of fabrication in complex parts, room temperature fabrication when compared to other base matrix such as metal and ceramic. Polymer classification include either thermoplastic or thermosets. Irreversible chemical transformation of thermoset polymers transforms them in to cross-linked polymer matrix. Complex molecular structures of thermoset resins offer good thermal insulation and electrical properties which are very much necessary for the structural applications. The low viscosity nature of the polymer matrices provides proper fiber wettability, thermal stability and creep resistance. Among the resins such as polyester, epoxy, phenolics and vinyl ester, epoxy resin was selected in this study. Epoxy resin is specifically selected because of its unique nature of excellent bond to a vivid fiber variety, apart from which the modified samples provide superior mechanical and electrical properties at high temperatures. Apart from these they are dimensionally stable for chemical reactions. All these advantages make L-12 to be selected as base

matrix for the present research study. This chemical is an inherent of 'epoxide' family. Epoxy is attributed with the name Bisphenol-A-Diglycidyl-Ether (commonly abbreviated to DGEBA or BADGE). Its molecular structure is represented in Fig 1. It is easily curable at laboratory scale because of the low glass transition temperature when mixed with the hardener tri-ethylene-tetraamine (TETA) which is an aliphatic primary amine commercially known as K-6 (Fig 2). The L-12 epoxy resin (Fig 4) and the corresponding hardener K-6 are obtained from Atul India Ltd. Table 1 show significant properties of epoxy.
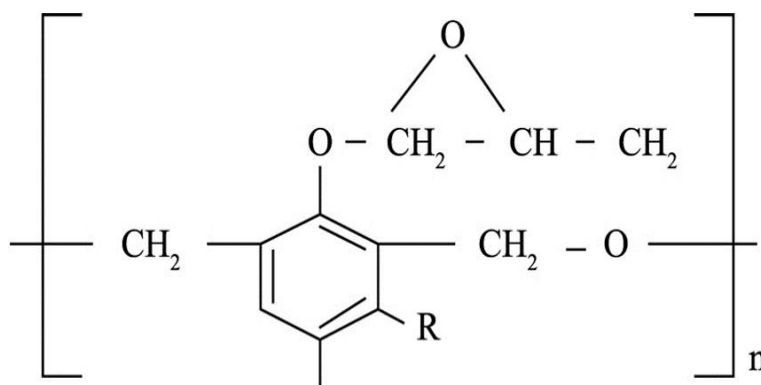
Fig. 3.1. Unmodified epoxy resin chain ('**n**' denotes number of polymerized unit)
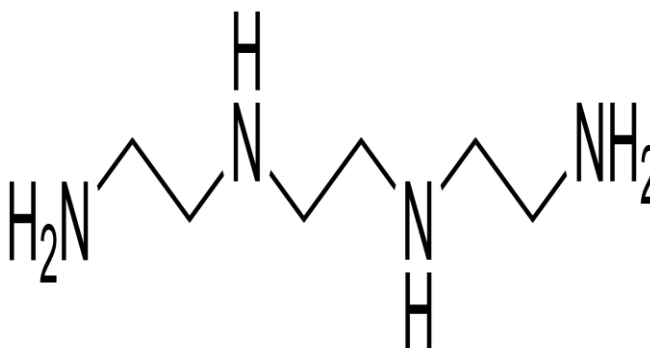
Fig. 3.2: Tri-ethylene-tetraamine (curing agent)

Fig.3.3 Image of Epoxy resin(L-12) and Hardener(K-6)

Table 3.1 Epoxy properties

Characteristic Property	Inferences
SNS Part Number	31200000LPX0002
Type of Product	Epoxy Resin with Hardener
Brand	Lapox
Model No	L-12 / K-6
Temperature (°C)	100ºC
Minimum Curing Time	15-30 mins at 100 ºC
Shear Strength	1.4 Kg millimeter/ min
Pot Life	1/2 -1 hr at 20ºC
Viscosity	9000 -12000 m Pa.s. at 25 ºC

3.2 Multi Walled Carbon Nanotubes: (Filler Material-1)

Multi Walled Carbon Nanotubes (MWCNTs) were procured from United nanotech. The MWCNTs have diameter ranging from 10-30 nm and its length varies from 1-2 microns which were sourced as strength fillers in this research work. These MWCNTs were manufactured through Chemical Vapour Deposition (CVD) technique. The purity of CNTs is 95%. Table 2 shows significant properties of MWCNTs.

Table 3.2 Specifications of MWCNTs

Characteristic Property	Inferences
Manufacturing Process	Chemical Vapor Deposition (CVD)
Diameter	10-30 nm
Length	1-2 microns
Purity	>95% (MWCNT)
Amorphous carbon	< 3%
Residue (Calcination in air)	<2%
Average interlayer distance	0.34 nm
Special surface area	$> 350 \text{ m}^2/\text{g}$
Bulk density	$0.05\text{-}0.17 \text{ g/cm}^3$
Real density	$1\text{-}2 \text{ g/cm}^3$
Charging	2180 (capacity: mA h/g)
Discharging	534 (capacity: mA h/g)
Volume Resistivity	0.1-0.15 ohm.cm (measured at pressure in powder)

3.3 Carbon Nano Fibers: (Filler Material-2)

Carbon Nanofiber (CNFs) were procured from United nanotech. The CNFs have outer diameter ranging from 125-150 nm and inner diameter varies from 50-70nm. The length of the CNF varies from 1-2 microns which were sourced as the filler material in this research. These CNFs were manufactured through Chemical Vapour Deposition (CVD) technique. Table 3.3 shows its significant properties.

Table 3.3 Some important properties of Carbon Nano Fibers

Characteristic Property	Inferences
Product Number	719811
Pyrograph Product Number	PR-25-XT-PS
Bulk Density of Product (lb/ft^3)	(0.5 – 3.5)
Kg/m^3	8.00 – 56.06
*Nanofiber Density (including hollow core) (g/cm^3)	1.4 - 1.6
Nanofiber Wall Density (g/cm^3)	2.0 - 2.1
Catalyst (Iron) Content (ppm)	< 14,000
Outer Diameter, (nm)	125 - 150
Inner Diameter, (nm)	50-70
Specific Surface Area, m^2/g	54
Average pore volume (cm^3/g)	0.120
Average Pore Diameter (angstroms Å)	89.30

3.4 Graphene: (Filler Material-3)

Research grade graphene used in the study was synthesized by joint effort of CVD and chemical graphite exfoliation technique. Graphene used consists of 3-10 layered aggregated sub-micron sheet with a lateral dimension of 5-10 microns with high aspect ratio about 1000 more than 99% carbon content along-with natural presence of other entities. The Graphene used in the present work is shown in Fig 6. Table 4 shows some of its significant properties.

Table 3.4 Some important properties of Graphene

Characteristic Property	Inferences
Formula Weight: 12.01 g/mol	Appearance (Form) Powder
Appearance (Color) Black	Carbon nanofiber- graphitized (iron free)
Iron (Fe) < 100 ppm	Composed of conical platelets D x L 100nm x 20-200μm
Iron (Fe) ≤ 100ppm	Diameter

3.5 Why we choose Functional Graphene?

- We are looking towards Nano composite material which can be better for Structural Application.

- Among all the nanomaterial's functional graphene shows effectively improvement in mechanical properties of base materials.

- Also, it increases stability and improves functionality of material.

3.6 Methodology Adopted in The Preparation of Polymer Composites

This section focuses on the processes used for development and characterization of the different composites under study. The specimens were developed as ASTM standards. It gives the specifications of the tests in terms of mechanical, physical, micro-structural of the modified specimens.

3.6.1 Epoxy Composites filled with Graphene

The GA employed in this work were of industrial grade with a purity of 95 wt% and accordingly a concentration of 0.1, 0.2, 0.3 and 0.4 % by the total weight of the epoxy matrix were used. Hardener added was 10% by the weight of epoxy matrix to initiate the crystallization of epoxy resin. The final mixture was then placed in 40mm x 12mm x 6 mm aluminium moulds for fabrication of the specimens to conduct flexural test. The specimens were then cured in a room for 24 hours before being removed from the moulds. In a similar manner, epoxy-Graphene composites of six replicas of selected compositions (Table 3.8) were made for each of these compositions.

Table 3.5 Epoxy composites filled with Graphene (GA)

Sr. No.	Specimens	Composition
1	PE	Plane Control beam
2	C1	Epoxy + 0.1wt% Graphene
3	C2	Epoxy + 0.2wt% Graphene
4	C3	Epoxy + 0.3wt% Graphene
5	C4	Epoxy + 0.4wt% Graphene

3.6.2 Dispersion of nanoparticles

Sonication was used for appropriate dispersion of nanoparticles in epoxy matrix. In order to cure the specimen nanoparticle integrated epoxy L-12-part A was mixed with part hardener K-6-part B at a stochiometric ratio of 90:10. Hand lay-up process followed by compression molding technique was employed to cast the specimens. The Specimens were positioned inside the compression mold, for 5 hours. For compression moulding process, a pressure of 150 kN and a temperature of 130°C was maintained to obtain cured graphene-epoxy composites. The cured laminated composites had thickness of about 3mm for tensile test and 6mm for flexural test.

A graphical diagram of the fabrication procedure adopted involves a hand-layup technique for particulate filled epoxy composites is shown in Fig 2.
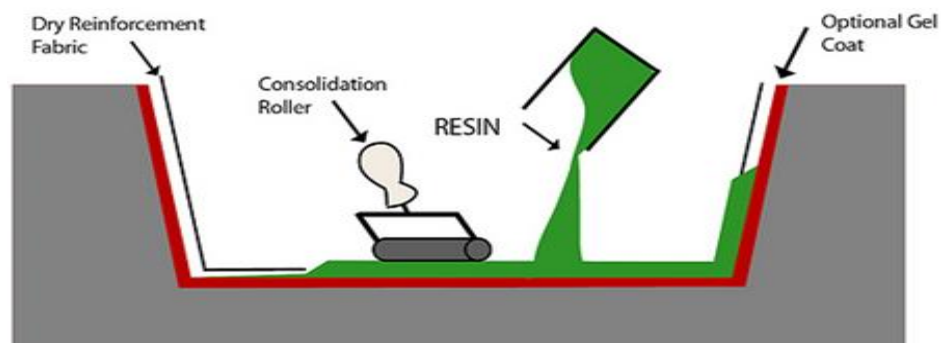
Fig. 3.4 Particulate filled epoxy composite fabrication by hand lay-up process

3.7 Instruments used for Experiment

3.7.1 Four Digit Weighing Machine

This Machine is used to measure the Nano-material. Also, this machine is used, in order to get the accurate results during the testing. The measuring pan of an analytical balance is inside a transparent enclosure with doors, so that dust does not collect and so any air currents in the room do not affect the balance's operation.

Fig. 3.5 Four Digit Weighing Machine

Table 3.6 – Specifications of Four Digit Weighing Machine

Capacity	0-200gm
Readability	0.0001 g
Repeatability	± 2 mg
Linearity	± 1 mg
Pan Size	Ø80 mm
Output Interface	RS232
Windshield Size	19 * 19 * 24 cm
Net Weight	7.2 kg
Overall Size	22 * 38 * 35 cm
Dimensions	49.5 * 33 * 49 cm
Power Supply	110 V/AC

3.7.2 Magnetic Stirrer

Magnetic stirrers or laboratory stirrers exist in multiple variations, from the simple single-place stirrer for daily use in the laboratory to the high-end reaction block for application in chemical laboratories. Magnetic stirrers are the ideal tool for laboratories, research and quality control. Magnetic stirrers are especially suitable for continuous use under most demanding conditions. Magnetic stirrers exist in different versions such as that is single-place stirrers, multiple-place stirrers, devices with or without continuous regulation of speed, with or without heating plate or as a combined high-end edition.

Fig 3.6 Magnetic Stirrer

Table 3.7 - Specifications of Magnetic Stirrer

Stirring places	1
Max. stirring capacity	2
Rotational sp. range	1250
Temperature range	20 to 300 C
Accuracy	<100 °C / ±1 °C >100 °C / ±3 °C
Power supply	220 V / 50 Hz
Operating conditions	+15 to +50 °C / <80 % RH
Dimensions	230 x 180 x 120 mm
Weight	2.2 Kg

3.7.3 Effect of ultrasonic energy (Ultra Sonicator)

Ultrasonication is a familiar physical technique to disperse nanoparticles into base fluids. Line voltage is converted to mechanical vibrations by ultrasonicator. Pressure waves are transferred in the medium by these mechanical vibrations. This phenomenon known as cavitation increases temperature of the liquid. Although the energy released by these bubbles lasts for a fraction of a second but cumulative effect of these bubbles causes extremely high levels of energy that causes distribution of the NPs [164]. The dispersion of NPs was conducted using bath sonicators and probe sonicator respectively as shown in Fig 3 and 4 respectively.

Fig. 3.7 Ultrasonic equipment used for dispersion of CFs and NPs

Table 3.8 Specification of Ultrasonic Equipment

| Properties	Inferences
Frequency	30 ± 3 KHz
Electric Supply	230V AC 50Hz Single Phase
Tank Size	225mm * 125mm *60mm
Tank Capacity	1.5 litre
Ultrasonic Power	50 Watt
Heater Power	120 Watt
Company	Rivotek
Cost	Rs. 18,300

3.7.4 Mould

The mould is used to prepare the casting of composite material. This mould is made up of Cast Iron. The dimensions of mould are 230mm * 160mm * 6mm. This mould is covered with the Aluminum Foil in order to get a perfect casting and surface finish and shouldn't get stuck with the mould.
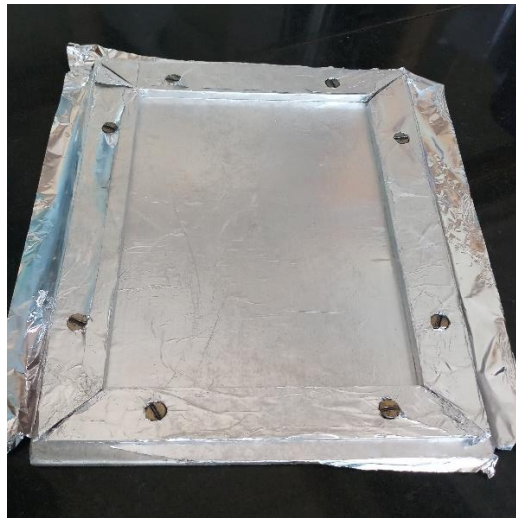

Fig. 3.8 Mould used for casting

### 3.8 ASTM Standard for Various Tests

It is an International Standards Organization that develops and publishes voluntary consensus technical standards for a wide range of materials, products, systems and services. Standards are developed within committees. ASTM International has no role in requiring or enforcing compliance with its standards, however, may become mandatory when referenced by an external contract, corporation or government. This ASTM standard is designed for Tensile testing and Flexure Testing. The Dimensions of various shapes like Dog bone Shape, three-point loading, etc are given in the fig. below.
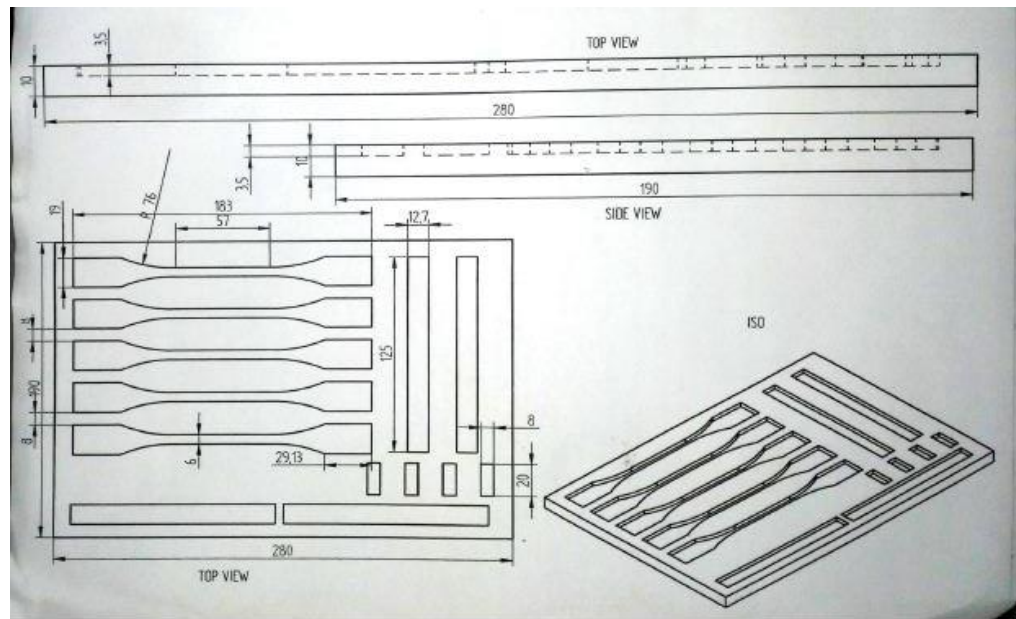
Fig.3.9 ASTM standardized shapes with dimensions for various testing
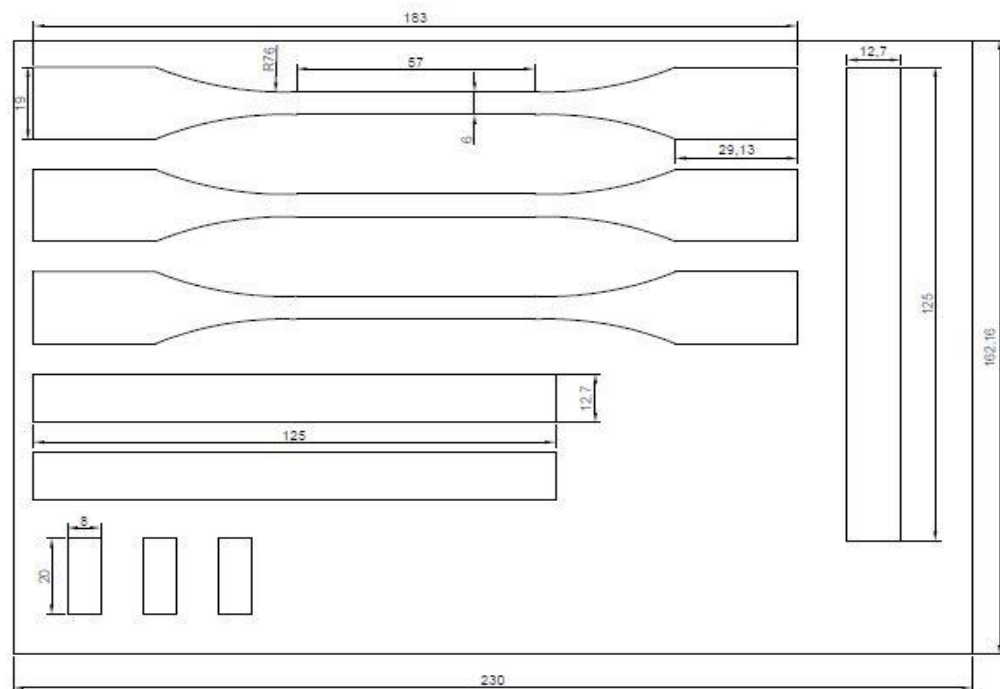
Fig.3.10 CAD Drawing Image
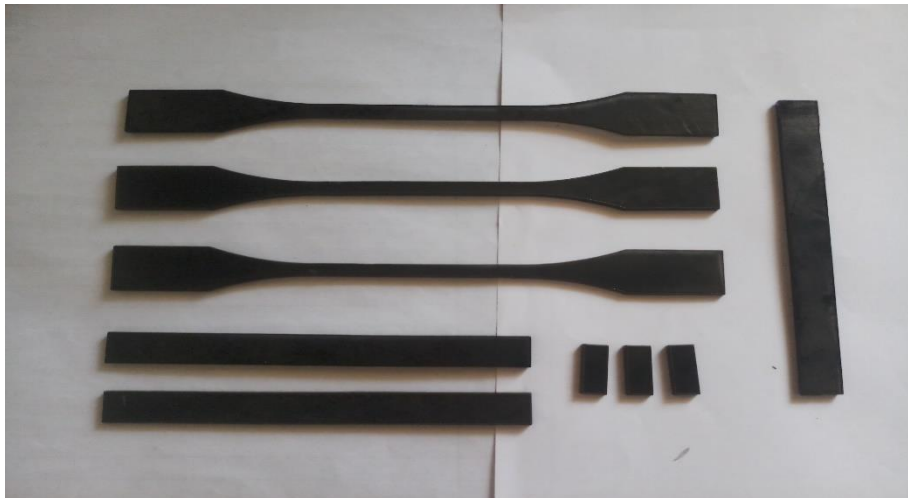
Fig 3.11. 0.1 % Graphene in polymer

Fig 3.12. 0.2% Graphene in polymer

Fig 3.13 0.3% Graphene in polymer

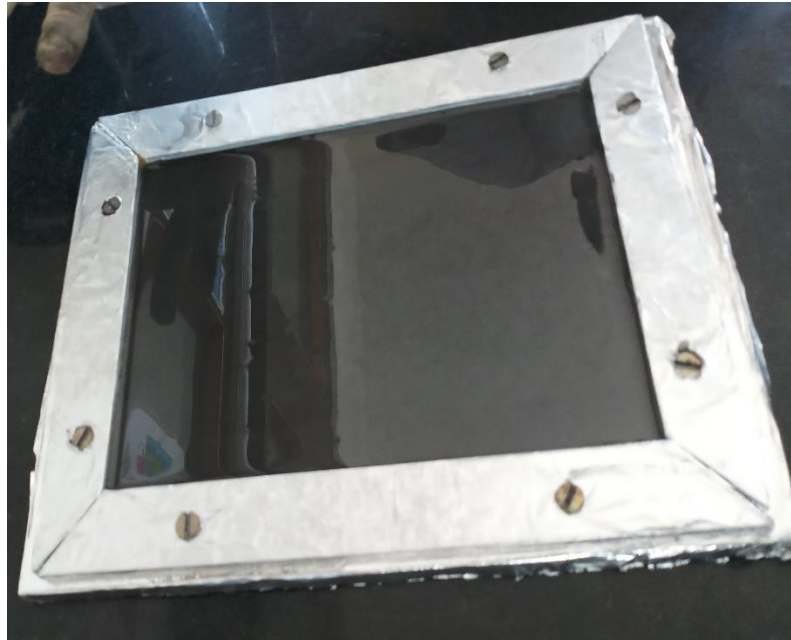**Fig.3.14** 0.4 % Graphene in polymer

Fig.3.15 Nano Composite kept in Ultra Sonicator

Fig.3.16 Covering the Mould with Aluminium Foil

Fig.3.17 Nano-Composite poured into the mould.

3.9 Testing of Specimen

3.9.1 Flexural Testing Machine

The UTC-5533 200 KN Capacity Manual Flexure Testing Machine is designed to perform reliable flexure tests. Being a low-cost alternative, UTC-5533 testing machine combines precision and simplicity with the unique design of the manual power pack. The UTEST range of flexural machines have the accuracy of Class 1 starting from 2% of the full capacity. UTC-5533 flexure testing machine consists of a heavy duty welded frame, manual hydraulic power pack and data acquisition system LPI.

Procedure for Flexure Testing -

1) Measure the width and thickness of the specimen.

2) Mark on the location where the load will be applied under three point bending. Note the length of between support.

3) Place the sample carefully on the stage of three point bending fixture of a machine.

4) Make sure that the loading point is placed on the marked location.

5) Carry out the bend test.

Fig.3.18 Flexure Test Machine UTC- 5533

Table 3.9– Specification of Flexure Test Machine UTC- 5533

Capacity	200 KN
Class 1 Range	4-200 KN
Resolution	1/65.000
Ram Travel	100mm
Max. Vertical Clearance	425mm (without accessories)
Max. Horizontal Clearance	650mm
Max. Clearance between Lower Rollers	900mm
Frame	UTC-56000
Power Pack	UTC-4810

Overall Dimension	**Frame**	1000 * 950 * 1130 mm
	Power Pack	300 * 250 * 500 mm
Weight (Approx.)	**Frame**	225 kg
	Power Pack	50 kg

3.9.2 Tensile Testing Machine

For testing of strength in tension, compression, bending, shearing, rapture and etc. It can be used for various raw materials, semi-finished and finished products which are essential to manufacturers performing quality control and academic organizations for studying the physical properties of raw materials.

Procedure For Tensile Testing –

1) Install the specimen in the testing machine (bottom connection first; take care not to unscrew the top bracket)

2) Mount the extensometer on the specimen and set it to zero (attach the support wire from the extensometer to the top of the tester frame support).

3) Set the Range Indicator. Set the machine load indicator and the limit pointer to zero.

4) Press the START button.

5) Apply load to the specimen at a very slow rate and record the load.

6) At the yield point, remove the extensometer. From this point, record the elongation.

7) At the ultimate strength, stop recording the elongation strain. Continue loading specimen to fracture (as a safety precaution stay away from the loading area).

8) Remove the fractured specimen from the machine.

Fig 3.19 – Tensile Testing Machine

Table 3.10 – Specification of Tensile Testing Machine

Model		GT-7001-Ls 20
Capacity		200 kN
Speed (without loading)		0.01~90mm/min
Display Method		PC based
Load Range		(full scales at the same amplification)
Resolution		1/200000
Accuracy		±1%
Driving Method		Servo Hydraulic Control System
Tension	Max. Space	460mm
	Grips for Rod(Dia.)	Ø10~ Ø40 mm
	Grips for Plate	40 * 60 mm
Dimension (W * D* H)	Main Unit	107 * 73 * 194 cm
	Control Unit	119 * 69 * 145 cm
Weight (Approx.)	Main Unit	1270 kg
	Control Unit	255 kg

3.10 CALCULATION

a) Calculation for plain epoxy polymer

i) Dimensions of Mould = 230mm * 160mm * 3 mm

ii) Volume of Mould = 230 * 160 * 3

$$= 110400 \text{ mm}^3$$

$$= 1.104 * 10^{-4} \text{ m}^3$$

iii) Density of Epoxy = 1162 kg/m^3

iv) Mass = Density * Volume

$$= 1162 * 1.104 * 10^{-4}$$

$$= 0.1282 \text{ kg}$$

$$= 128.2 \text{ gms}$$

Considering losses in the beaker, the total amount of Epoxy Resin and Hardener =

130 gm

Proportion of Epoxy to Hardener is 9: 1.

Epoxy = 117 gm

Hardener = 13 gm

b) Calculations for Polymer with 0.1% Nano-material

Total Mass of Epoxy and Hardener = 130 gm

Epoxy – 117 gm

Hardener – 13 gm

0.1 % Nanomaterial is calculated in proportion to the mass of Epoxy.

Mass of Nano-Material = (0.1/100) * Mass of Epoxy

$$= (0.1/100) * 117$$

$$= 0.117 \text{ gm}$$

Mass of Epoxy = Previous Mass of Epoxy – Mass of Nano-Material

$$= 117 – 0.117$$

$$= 116.883 \text{ gm}$$

For 0.1% Nano-Material

Epoxy = 116.883 gm

Nano-Material = 0.117 gm

Hardener = 13 gm

c) Calculations for Polymer with 0.2% Nano-material

Total Mass of Epoxy and Hardener = 130 gm

Epoxy – 117 gm

Hardener – 13 gm

0.2 % Nanomaterial is calculated in proportion to the mass of Epoxy.

Mass of Nano-Material = (0.2/100) * Mass of Epoxy

$$= (0.2/100) * 117$$

$$= 0.234 \text{ gm}$$

Mass of Epoxy = Previous Mass of Epoxy – Mass of Nano-Material

$$= 117 – 0.234$$

$$= 116.766 \text{ gm}$$

For 0.2% Nano-Material

Epoxy = 116.766 gm

Nano-Material = 0.234 gm

Hardener = 13 gm.

d) Calculations for Polymer with 0.3% Nano-material

Total Mass of Epoxy and Hardener = 130 gm

Epoxy – 117 gm

Hardener – 13 gm

0.3 % Nanomaterial is calculated in proportion to the mass of Epoxy.

Mass of Nano-Material = (0.3/100) * Mass of Epoxy

$$= (0.3/100) * 117$$

$$= 0.351 \text{ gm}$$

Mass of Epoxy = Previous Mass of Epoxy – Mass of Nano-Material

$$= 117 – 0.351$$

$$= 116.649 \text{ gm}$$

For 0.3% Nano-Material

Epoxy = 116.649 gm

Nano-Material = 0.351 gm

Hardener = 13 gm

e) Calculations for Polymer with 0.4% Nano-material

Total Mass of Epoxy and Hardener = 130 gm

Epoxy – 117 gm

Hardener – 13 gm

0.4 % Nanomaterial is calculated in proportion to the mass of Epoxy.

Mass of Nano-Material = (0.4/100) * Mass of Epoxy

$$= (0.4/100) * 117$$

$$= 0.468 \text{ gm}$$

Mass of Epoxy = Previous Mass of Epoxy – Mass of Nano-Material

$$= 117 – 0.468$$

$$= 116.532 \text{ gm}$$

For 0.4% Nano-Material

Epoxy = 116.532 gm

Nano-Material = 0.468 gm

Hardener = 13 gm

Table 3.11 - Experimentation on Nano-Materials with Various Compositions

Sr. No.	Mass of Epoxy (gm)	Mass of Nano-Material (gm)	Mass of Hardener (gm)	Total Mass (gm)
1	116.883	0.1 % = 0.117	13	130
2	116.766	0.2 % = 0.234	13	130
3	116.649	0.3 % = 0.351	13	130
4	116.532	0.4 % = 0.468	13	130
5	116.415	0.5 % = 0.585	13	130
6	116.298	0.6% = 0.702	13	130

4. RESULTS AND DISCUSSIONS

4.1 Mechanical Characterization

This section highlights on the flexural and tensile tests conducted on six replicas of each composition of the modified beams as per ASTM standards.

4.1.1 Three-point test

This test includes determination of optimum percentage of graphene dosage for reinforcing in epoxy beams based on three-point loading tests. This test ensures optimum percentage of graphene by wt. % of epoxy required to reinforce the plain epoxy beams, which gives highest structural efficiency in terms of load bearing capacity. Hence the flexural behavior of graphene reinforced epoxy beams is investigated. Modified beams were subjected to flexural test (Three-point loading) to determine their mechanical properties such as strength-deflection criteria etc. The outcome of the modified beams was compared with the unmodified beams to check the efficacy of the reinforcement for structural applications. The specimen characteristics utilized for flexural test as per ASTM(D2344) is shown in Table 11. Descriptions of the test specimen used are presented in Table 12. The load v/s deflection of the specimen is shown in Fig 15.

From the Fig. 15 it follows that as the percentage of GA in polymer matrix increased the strength increased proportionally till 0.3 wt.% of fillers in the epoxy matrix. The reason for this observed trend could be attributed to graphene platelet like structure that relive stress deposition by providing more surface area for stress distribution in the holding matrix. Beyond 0.3 wt.% of filler in the holding matrix declining trends in strength aspects were observed and this could be due to shrinkage of the polymer matrix at specific sites inhibiting the polymer chain linkage.

Table 4.1 : Specimen Characteristics utilized for Flexural test as per ASTM (D2344)

1.	Size of the Specimens	40mm x 12mm x 6 mm
2.	Epoxy resin	L-12
3.	Percentage of Graphene (GA)	0.1, 0.2, 0.3, 0.4% by weight of epoxy resins.

Table 4.2 : Result of three-point test

Sr. No.	Specimen	Ultimate load (kN)	Max. Deflection (mm)	Max. Flexural stress (N/mm^2)
1	PE (Plain epoxy)-PE	2.7	1.1	375
2	PE+ 0.1wt.% of Graphene-C1	0.6625	3.65	93
3	PE+ 0.2wt.% of Graphene-C2	0.57	5.63	80
4	PE+ 0.3wt.% of Graphene-C3	0.475	6.41	66
5	PE+ 0.4wt.% of Graphene-C4	0.77	5.69	108
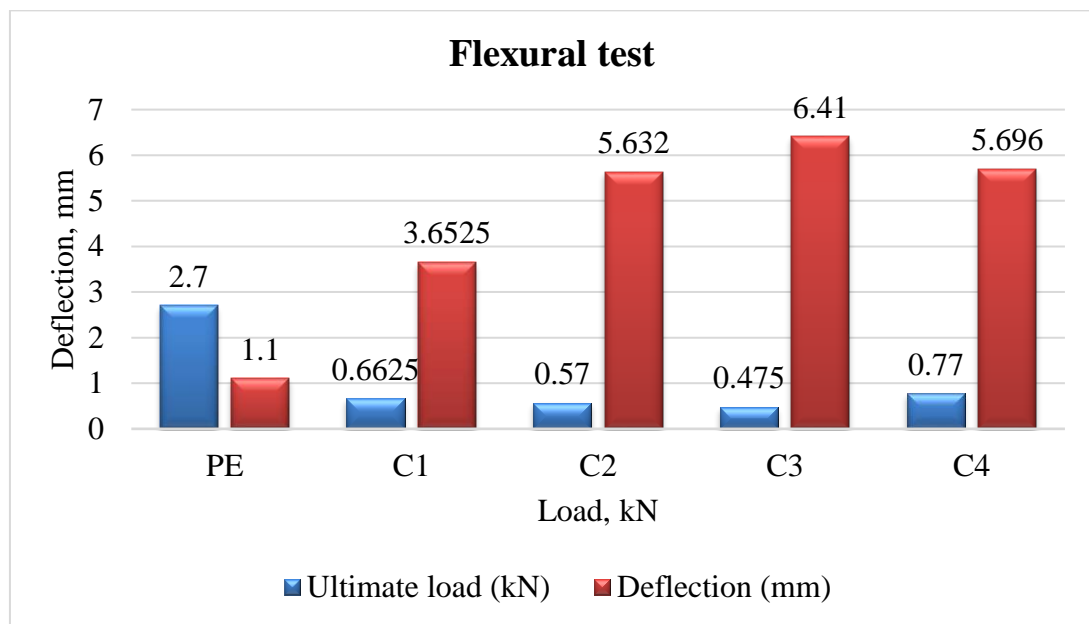
**Fig. 4.1** Load v/s Deflection of three-point test

4.1.2 Tensile test

This test includes determination of optimum percentage of graphene for reinforcing epoxy beams that will end up with enhanced ductility of the modified beams. This characterization correlates the optimum percentage of graphene by wt.% of epoxy that gives highest structural efficiency in terms of load bearing capacity. Hence the tensile behavior of graphene reinforced epoxy beams is investigated. Modified beams were studied under tensile loads to investigate their mechanical properties such as deflection, strength criteria etc. Tensile results of the modified beams were weighed against unmodified beams to identify the changes incurred for reinforcement. The outcomes obtained were then compared with unmodified beams. The specimen characteristics utilized for tensile test is shown in Table 13. The results of the test specimen used are presented in Table 14. The load v/s deflection of the specimen is shown in Fig. 16.

Table 4.3: Specimen Characteristics for Tensile test as per ASTM (D3039)

1.	Size of the Specimens	250mm x 25mm x 2.5 mm
2.	Epoxy resins	L-12
3.	Percentage of Graphene (GA)	0.1, 0.2, 0.3, 0.4% by weight of Epoxy resin.

Table 4.4: Result of tensile test

SR. No	Composition	Ultimate load (kN)	Ultimate Tensile strength (N/mm²)	Displacement (mm)
1	PE	1.56	24.5	1.6
2	PE + 0.1 wt.% of Graphene	3.47	24.00	4.5
3	PE + 0.2 wt.% of Graphene	4.91	22.25	4.9
4	PE + 0.3 wt.% of Graphene	5.94	67.50	6.3
5	PE + 0.4 wt.% of Graphene	4.75	21.22	2.1
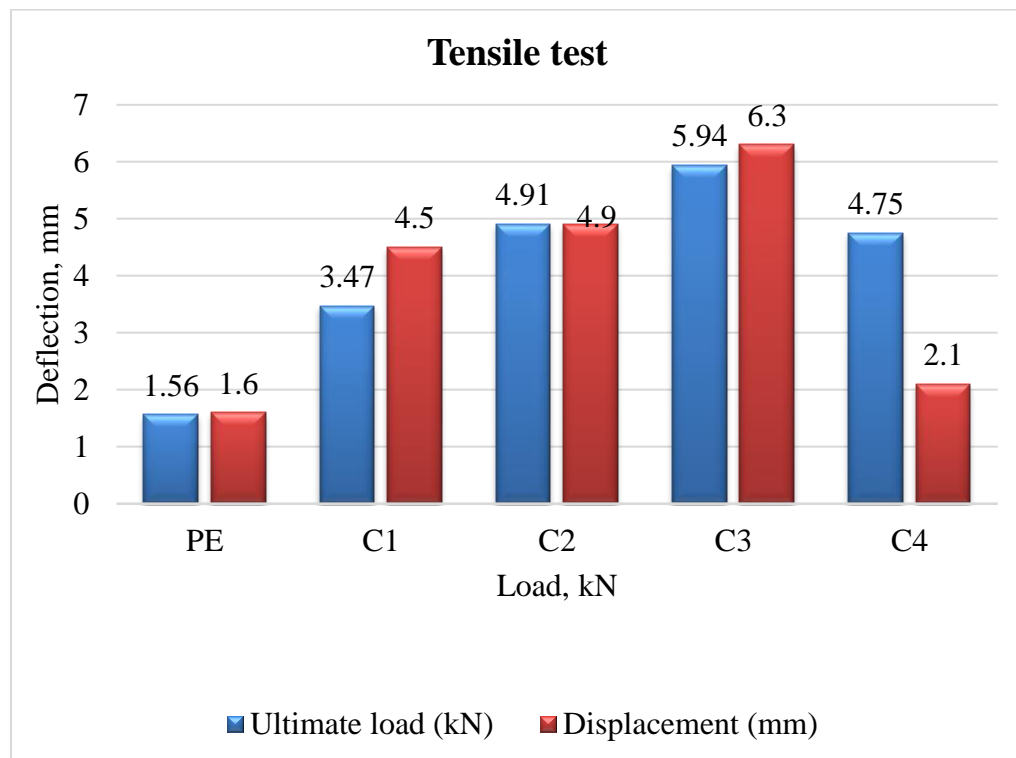
Fig. 4.2 Load v/s Deflection of tensile test

5. CONCLUSION AND FUTURE SCOPE

5.1 CONCLUSION

- We have successfully studied and prepared Nano-Particle Reinforced Polymer by using Epoxy Resin and Graphene. During the preparation, we undergone through the standard procedure for the dispersion of NPs in polymer-based matrix and standard procedure for the preparation of the Nano-composite specimens.

- After the preparation of Nano-Composite specimens, the following mechanical tests were performed.

  i) Flexural Test

  The percentage of GA in polymer matrix increased the strength increased proportionally till 0.3 wt.% of fillers in the epoxy matrix.

  ii) Tensile Test

  The percentage of GA in polymer matrix decreased the strength increased proportionally till 0.2 wt.% of fillers in the epoxy matrix. It suddenly increases for the 0.3% wt. of fillers in the epoxy matrix and again decreases further.

- According to the comparison of the results, it is concluded that, the polymer with 0.3% wt. of Graphene filler in the epoxy matrix is reliable for the structural applications.

- As per the above problem definition and objective, this material can be used for various applications like drone, turbine blades and other structural applications.

5.2 FUTURE SCOPE

The study can be extended with help of improvement in mechanical properties and with refers to this we can use for structural applications. We can use this material for structural applications in automobiles, drone materials, marine application, and defense related application. As the weight of material is less and strength is high we can use this for applications where light weight material is necessary.

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