Enhancement of Mechanical Properties in Glass Fiber Epoxy Composites Using Nanomaterials

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ISLAMABAD

AUGUST, 2015

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2011-NUST-MS PhD-Mech-23
(MS68)

A thesis submitted in partial fulfillment of the requirements for the degree of

MS Mechanical Engineering

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Declaration

I certify that this research work titled "*Enhancement of Mechanical Properties in Glass Fiber Epoxy Composites Using Nanomaterials"* is my own work. The work has not been presented elsewhere for assessment. The material that has been used from other sources has been properly acknowledged / referred.

Signature of Student

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This thesis has been read by an English expert and is free of typing, syntax, semantic, grammatical and spelling mistakes. Thesis is also according to the format given by the university.

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Copyright Statement

Acknowledgements

Countless words of praise and thanks to Allah Almighty with whom Blessings and Mercy the research exploration eventually in the form of thesis was accomplished. I would like to express the deepest gratitude to my advisor Dr. Rizwan Saeed Choudhry for his kind support, patience, magnificent knowledge and expert guidance in my research. I would also like to thank my guidance and evaluation committee (GEC) members Dr. Aamer Ahmed Baqai, Dr. Hasan Aftab Saeed & Dr. Laraib Alam Khan for their kind guidance and support. I would like to acknowledge my friend Mr. Laeeq Ur Rahman for his help and moral support. I would like to thank CEME (College of Electrical & Mechanical Engineering)-NUST (National University of Sciences and Technology) where I was enrolled to the Master studies program and where I found an appropriate platform for accomplishing the academic requirements and research.

To my family

Especially My Dearest Mother & My honorable late Father

Qazi Muhammad Khan

Who always cordially supported me in the educational domain

To see me at appropriate intellectual level in life

Abstract

Increasing the delamination resistance of composites without degrading the in-plane properties is one of key challenges faced by composite manufacturers and researchers. Adding nano-materials in the resin have been shown to have positive effect on delamination resistance. Aligning these fibers in through thickness direction within the matrix is expected to result in further improvement. Thus, in this study a novel technique, which used a combination of sonication and magnetic field application across the molding set up, was adapted to align the multiwall carbon nanotubes (CNTs) through the thickness direction. Resin infusion technique is selected for this purpose. In order to get optimized and valid results, design of experiments (DOE) with Taguchi Approach was utilized with two control factors and with three levels each. Two composite specimen groups were fabricated using vacuum-assisted resin infusion (VARI) molding; one with using magnetic field and another without the magnetic field, were prepared. The mechanical properties, including interlaminar shear strength (ILSS), flexural strength, flexural elastic modulus (E) were measured and compared. The experimental results indicate improvement in the interlaminar/interfacial properties of the magnetized composites with low weight percentage CNT loading when compared to the samples with the same CNTs weight percentages. Increased ILSS results in decreasing delamination, an increase in the out of plane loading capacity and bending resistance.

Key Words: *CNTs, DOE, VARI, ILSS, Magnetic field*

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CHAPTER 1: INTRODUCTION

1.1 Background and Motivation

From advanced requirements point of view, today's world is shifting towards lighter, stronger and stiffer structures which require development of fibre-reinforced composites with advanced materials. Fibre reinforced composites are with good characteristics of high strength, better stiffness to weight ratio, fatigue strength , better corrosion resistance that is why these are widely used in aerospace, automobile, marine, defense and bio engineering.

As a matter of fact, fiber reinforced polymer–matrix composites extensively used with success in a number of industries but their interlaminar shear strength (ILSS) and delamination resistance have been faced as limiting design characteristic [1]. The reason is that conventional fabrication lack in producing reinforcing fibers across the thickness direction to counter transverse loading [2]. Brittle nature of resin matrix and weak fiber/matrix or fabric-matrix interface in composite laminates principally may cause delamination failures [3]. Different methods/techniques have been tried to improve ILSS and flexural properties. Amongst the more effective techniques are to weave fibers in the thickness direction or using Z-pins to connect the laminae [4], toughening the matrix resin, and placing an interleaf, in the interplay regions of the laminate. These techniques have effectively served the purpose, but in some cases, the degraded in-plane direction properties have been reported [5]. But still, these techniques have appeared as labor intensive that can greatly increase cost of the composites by requiring additional manufacturing processes [6].

As carbon nanotubes have marvelous mechanical properties and high aspect ratio, these aspects have proven their use as reinforcing agents in composites [7]. Researchers have used nanoparticles and carbon nanotubes (CNT) to improve a number of properties [7-26] like ILSS, fatigue strength, flexural strength, interlaminar fracture toughness, tensile strength and stiffness critical stress intensity factor K_{IC} etc. ILSS is an important design parameter and it uncovers the interlaminar property of composite laminates. As in transverse loading, composite usually fails on the fiber/matrix interface, therefore, improving the interface adhesion has been an important goal of composite researchers. Zhihang fan et al. [1] introduced MWNT with mechanical stirring, ultrasonication and acid oxidation techniques in VARTM and achieved enhancement in ILSS up to 33%. F.L. Shan et al. [7] used epoxy resin E20 for spraying on the fabric surface to anchor MWCNTs thereby and finally found an increase of 24% in average propagation G_{IC}, 11% in propagation G_{IIC} and 12% in ILSS. These enhancements were told because of good distribution of CNTs and the fiber/ matrix interfacial adhesion. Hsio KT et al. [8] made use of MWCNTs in epoxy and came up with enhanced ILSS results. Zhou et al. [9] added 2 wt% carbon nanofibres into glass fiber-epoxy composites and improved ILSS by 22.3%. Gojny et al. [10] improved ILSS by 20% after adding 0.3 wt% DWCNTs into fiber reinforced epoxy composites. Wichmann et al. [11] used DWNT and came up with 20% enhancement in ILSS. Zhu et al. [12] got 35% enhancement in ILSS. They coated SWNTs on the glass fibers first and then filled the mold with vinyl ester. Veedu et al. [13] first grew MWNTs on the SiC woven fabrics then fabricated hybrid composites. They achieved remarkable improvement in delamination resistance and interlaminar fracture toughness. In 2014 M.S. Senthil Kumar et al. [14] made use of nanomaterials and CNTs with various combinations of matrix materials and noted improvements in ILSS 20% for DWCNTs, 25% for HNT (Hallosite Nano Tubes) and 40% in fracture toughness for silane treated CNTs. M.M. Rahman et al. [15] achieved 49% increase in ILSS by enhancing cross link density with 0.3wt.% amino-functionalized MWCNTs loading in epoxy system. Shusheng Chen et al. [16] improved ILSS and coefficient of thermal expansion of PEI-functionalized CFF/EP composites by 34% and 16.8% respectively. This enhancement was attributed to the enhanced bonding strength due to increase in interfacial cross linking density. Sun Lili et al. [17] succeeded in increasing ILSS by 8.16% with the use of surface treated MWCNTs. A. Godara et al. [18] investigated Interfacial shear strength (IFSS) and found gain over 90% when used CNTs in fibre sizing. V.C.S. Chandrasekaran et al. [20] in 2010 increased ILSS by 21.3% after using flow flooding chamber (FFC) method. They suggested that the fabrication process and MWCNT distribution could be the key reasons for ILSS enhancement. Chandrasekaran et al. [21] in 2011 investigated ILL enhancement in glass fiber epoxy composites and found 41% ILSS increase for MWCNTs and 61% for functionalized MWCNTs. Florian H et al. [22] got significant increase in ILSS by 20% by only loading 0.3 wt% DWCNT-NH2. Jiang Zhu et al. [23] coated nanotubes (0.015 wt%) in the mid plane ply on several types of nanotubes and a 45% increase in ILSS was observed when compared to the control composite. J.M. Wernik et al. [24] obtained 25% increase at loading MWCNT with 1.0 wt% concentration in the epoxy adhesive. Bai-Chen Wang et al. [26] gained improvements in flexural strength and ILSS by 32.2% and 32.9%, respectively with only 0.025wt% CNTs loading. According to them, these enhancements could be ascribed to the transferability of loads from matrix to fibers due to enhanced matrix properties of the resulting composites.

Flexural strength plays a vital role in resisting bending loading. Researchers and composite engineers have successfully used CNTs to improve this property [26-35]. Bai-Chen Wang et al. [26] observed 32.9% increase in flexural strength after loading 0.025wt% CNTs. S. Zainuddin et al. [27] used acetone as dispersant medium for MWCNTs combined with sonication and effectively distributed CNTs in the epoxy. Optimum enhancement was observed in 0.3 wt.% samples in flexural strength and modulus with an increase of 42.2% and 20.4% respectively. CNTs dispersion and sonication play a profound role in mechanical performance [28, 29] of the CNTs hybrid composites. Liao YH et al. [28] increased storage modulus by 50.8% after dispersing 0.5 wt.% nanotubes in SC-15 epoxy resin. They utilized tip sonication and used acetone as solvent. Peng-Cheng Ma et al. [30] made use of sonication, stirring and calendering. Best results were reported for calendering for flexural modulus and strength up to 6.41% & 3.71% respectively. Nicholas T. Kamar et al. [31] strategically injected 0.25 wt% GnPs into the interfacial regions of the glass fabric/epoxy composite and increased flexural strength and G_{1C} up to 29% and 25% respectively. Yuanxin Zhou et al. [32] used SC-15 epoxy resin and carbon nanofibers with ultrasonic liquid processor of high intensity and obtained a homogeneous mixture. Both the tensile and flexural strengths improved by 11% and 22.3%, respectively. Improved fatigue strength was also observed. M.M. Rahman et al. [33] investigated properties of amino functionalized MWCNTs by using e-glass in epoxy composites through a combination of dispersions and found that flexural strength and flexural modulus improved by 38% and 22% with 0.3 wt.% loading. Daniel C. Davis et al. [34] strategically incorporated amino functionalized SWCNTs in carbon fabric/epoxy – matrix interfaces and found improvements in the tensile strength, stiffness and resistance fatigue damage. M. B. A. Salam et al. [35] dispersed two types MWCNTs in epoxy resin system both at elevated temperature and room temperature. It was observed that at high temperature cure, samples with 0.2 wt% MWCNTs loading exhibited improvement in flexural strength and flexural modulus with 41.45% and 48% respectively. It was concluded that elevated temperature results in better dispersion due to decresing viscosity of the polymer.

While designing the composites, delamination is faced as big challenge for the designers. Matrix transverse cracking in a brittle manner, fiber-matrix interface de-bonding have been reported [36]. Elisa Borowski et al. [36] improved fracture toughness in the FRP with the addition of MWCNTs. 25% increase in the mode-I interlaminar fracture toughness was observed with 0.5 wt% MWCNTs loading. Vahid Mirjalili et al. [37] experimentally investigated the effect of MWNT as a toughening agent in laminated composites. Mode-I fracture toughness by 48% ,Mode-II fracture toughness by 143% and the stress intensity factor (K_{IC}) by 17% were improved. These enhancements were attributed to MWNT pull out from the matrix, crack deviation and crack pinning. Christopher S et al. [38] showed that small amounts of loading of MWCNTs in to the glass–fiber composites and noticed reduction in cyclic delamination crack propagation rates significantly. Such behavior was attributed to the use of CNTs that increased interlaminar fracture resistance. Kim, H et al. [39] investigated fracture toughness enhancement due to improved adhesion between CNTs and carbon fibers. Composites with CNTs exhibited the G_{IC} to be 51% higher than those of the baseline samples. Enhanced adhesion (due to CNTs) between the matrix and fibers toughened the interface, and this led to an enhancement in G_{IC}. H. Silva et al. [40] concluded that with incorporating about 0.5% of CNTs into the matrix, mixed-mode fracture toughness improves significantly. An increases of about 25% was achieved in critical strain energy release rate (G_C)

Yokozeki et al. [41] performed investigation about the damage accumulation behaviors in carbon fiber nanocomposite laminates subjected to tension. It was found that if cup-stacked carbon nanotubes (CSCNT) are dispersed between mats, the composites can experience delay in the context of matrix cracking. Chuanguo Ma et al. [42] studied the effects of CNT loading and magnetic field on fracture toughness (K_{IC}), glass transition temperature in epoxy resin. Significant enhancement of 51% in K_{IC} was achieved by composites with 3 wt.% aligned CNTs transverse to crack growth. M. Battistella et al. [43] manufactured epoxy composites with fumed silica nanoparticles and found a 54% increase in K_{IC} compared to the neat epoxy which indicates a strong effect on crack propagation resistance.

Sohel Rana et al. [44] studied vapour-grown carbon nanofibres (CNFs) effect on properties after dispersing these via sonication and stirring in carbon fabric reinforced epoxy composites. A dispersion of 0.5% carbon nanofibres in the matrix enhanced Young’s modulus by 37% and tensile strength by 18%. Sunny S et al. [45] investigated three-dimensional reinforcement of the

CNTs that were aligned radially and grown on the surface of fibers in a woven cloth. Bearing stiffness, critical strength and ultimate strength were improved by 19%, 9% and 5% respectively. Daniel C et al. [46] studied the effect of fluorine functionalized CNTs loading into carbon/epoxy composites on tension, compression and cyclic loading. Consequently, improvements in tensile strength up to 18%, in stiffness up to 24% and resistance to failure were observed. R.J. Allen et al. [47] produced VACNT composite samples and with 2.0 vol% CNT loading enhanced flexural storage modulus by 21% and tensile modulus by 17%.

Philip D. Bradford et al. [48] produced CNT composites with an increased volume fraction with CNTs aligned and millimeter long. Tensile testing showed tensile strengths up to 400 MPa. Muhammad M. Rahman et al. [49] studied polyol and uniformly dispersed amino-functionalized MWCNTs (in 0.3 wt.% loading) on the thermo-mechanical behavior of the three-phase toughened epoxy composites. Introduction of polyol in the resin system decreased the thermo-mechanical properties. However, incorporating amino-functionalized MWCNTs (in 0.3 wt.% loading and uniformly dispersed) into the polyol-toughened epoxy composites improved the properties back due to improved crosslink interactions among the resin, polyol and NH_2-MWCNTs. For the same weight percentage improvements of 14%, 16% and 7%, were observed in storage modulus, loss modulus and glass transition temperature respectively for the epoxy system and in the polyol/epoxy composite the same properties were enhanced by 12%, 13% and 8%, respectively. A.N. Upadhyay et al. [50] investigated the effect of CNT additive on $Se_{85}Te_{10}Ag_5$ glassy composite for the electrical, thermo-physical properties and mechanical behavior. Thermal conductivity increased by 59%, micro hardness increased by 68.73% and electrical conductivity was noted for its rapid change by several orders of magnitude from10^{-12} (S/cm) to 10^{-6} (S/cm) for the glassy composites with 3% and 5% CNT inclusions. Such enhancements were attributed to the percolation network. Smrutisikha Bal et al. [51] investigated mechanical and electrical properties of epoxy nanocomposites. They studiedtheir behavor under room temperature and refrigerated curing conditions using carbon nanofibers up to 1 wt.%. Refrigerated samples showed significant increase in flexural modulus and hardness as nanofibers could not aggregate during cure condition. Refrigerated nanocomposites, with 1% CNF, increased bending modulus of the substrate up to 98%. In case of refrigerated samples, for low weight percentage use of CNFs, better trend was observed in electrical conductivity in the direction of nano fibres.

Some researchers have used magnetic fields for aligning CNTs in the matrix as well. Yanjie Su et al. [52] successfully demonstrated the magnetic field influence on the synthesis of SWCNTs. These were synthesized with the magnetic field as per the selected diameters and distributions when in the arc plasma, it was applied perpendicular to the electric field. Purity and orientation of SWCNTs, their separation into two different regions with different diameter distributions were controlled by the magnetic field. It was noticed that modifying direction of the magnetic field improved CNTs diameter selection efficiency as well. B.K. Jang, Y. Sakka [53] investigated the effect of the shape and size of CNTs on their alignment in a magnetic field. They applied magnetic field of 12T to investigate the alignment response of MWCNTs in the suspension. Good alignment of the CNTs was obtained and it was noticed that it was depending on the direction of applied field. Erin Camponeschi et al. [54] used magnetic field and explored the effect of alignment of CNTs on the mechanical properties of the nanocomposite. Properties of the composites were found better than composites that remained unexposed to a magnetic field. S.G. Prolongo et al. [55] studied the properties of epoxy composites exposed to weak magnetic field of 0.3 T. CNTs alignment was performed in specific device designed to hold permanent magnets for the field application. The thermo-mechanical and tensile properties of magnetized composites were higher than neat epoxy resin. Anshu Sharma et al. [56] studied the properties of aligned CNT/Poly Carbonate nanocomposites fabricated under magnetic field of 1200 Gauss. 0.1wt.% of SWCNTs and MWCNTs were homogeneously dispersed throughout the matrix by ultrasonic agitation. They succeeded in aligning SWNT and MWNT under the effect of magnetic field. Dynamic mechanical analysis measurements confirmed the improved mechanical strength of the aligned nano composites. Donglu Shi et al. [57] applied 5T magnetic field on the MWCNT (coated with Ni/CoO) that were dispersed in the liquid. It was confirmed that nanotubes were responsive to magnetic field and aligned in the direction of the applied field. Resultantly, tensile strength was observed with strong anisotropy in the MWCNT–polystyrene composites.

The discussion so far indicates that if carbon nanotubes-epoxy suspension is infiltrated and distributed within layers, and made to infuse in the thickness direction, the CNTs in the suspension might orient in the thickness direction, and this exercise may result in improving the ILSS of fiber reinforced composites. [58].

Additionally, as CNTs are influenced by a magnetic field, therefore a novel technique which combine the techniques of sonication, mechanical stirring and magnetic field application across the glass fiber fabrics during VARI was deemed to get the MWCNTs aligned in the thickness direction resulting in increased values of ILSS and flexural strength without weakening the in-plane properties.

In order to materialize the above mentioned idea, glass fiber fabric/epoxy composites were fabricated by dispersing some amount of MWCNT in the epoxy using acetone as dispersant. The MWCNT/epoxy mixture was infused in the glass fiber fabrics with vacuum assisted resin infusion (VARI) molding and double vacuum assisted resin infusion (DVARI) moulding as shown in Fig 4.5

The ILSS and flexural properties of the composite were measured. Interlaminar shear strength was characterized by the short beam shear test and flexural strength and moduli were determined by three point bending test. Two composite specimen groups (both loaded with MWCNT at 0.5, 1.0 and 1.5 wt %.) were fabricated; one with a magnetic field and the other without it. All the three properties were compared with reference to the control samples and un magnetized composites.

1.2 Aims and Objectives

The aim of current research is to fabricate glass fiber epoxy composite with enhanced mechanical properties (ILSS, flexural strength etc) by strategically infusing the MWCNTs/epoxy suspension into stationary glass fiber fabrics using VARI technique and further aligning CNTs in the thickness direction with the application of magnetic field. The substantial objective leading to achieve the stated aim is described as follows:

- Literature review in developing an up-to-date and comprehensive understanding of the available experimental techniques for property enhancing with carbon nanotubes in glass fiber reinforced epoxy composite

- To study and observe influence of magnetic field on CNTs aligning at relatively higher magnetic fields

1.3 Methodology

First of all Design of Experiment (DOE) by Taguchi method [59] was performed in order to find out number of experimental trials required for the said purpose. For reference sake, using VARI method, one glass fabric/epoxy laminate was fabricated and tested for ILSS, flexural strengths and SEM images. Then 03 laminates of carbon nanotubes glass fabric/epoxy composites were fabricated (loading MWCNTs with 0.5wt%, 1.0wt% and 1.5wt% respectively) and tested for ILSS, flexural strengths and SEM images. Then 09 laminates of carbon nanotubes glass fabric/epoxy composites were fabricated (loading MWCNTs with 0.5wt%, 1.0wt% and 1.5wt% and applying 03 magnetic field levels on to each wt% level during curing) and finally tested for ILSS, flexural strengths and SEM images. Results of the laminates fabricated without magnetic fields were compared with those fabricated under magnetic fields with reference to the simple glass fabric/epoxy laminate. On the basis of improved properties, VARI with magnetic field was validated as properties enhancing method. Overall methodology adopted for this study is shown in flowchart in Figure 1.1

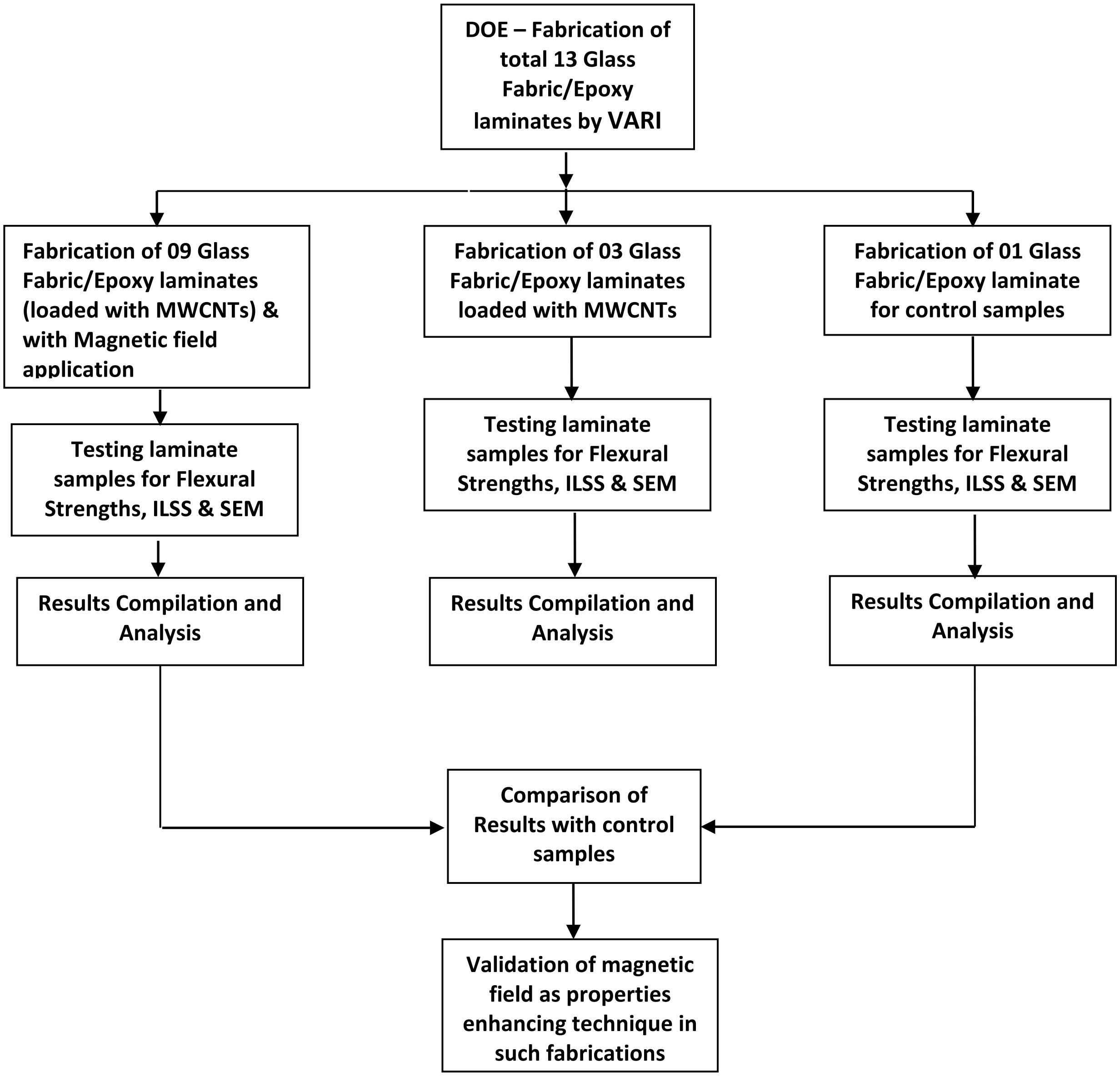


Fig 1.1: Methodology

1.4 Thesis Contribution

The present research will help in:

(i) Opening avenues for fabrication of CNTs/Glass fabric/Epoxy Composites with various magnetic fields for getting better out of plane properties.

(ii) Familiarization with DOE technique in experimental research concerns.

1.5 Thesis Organization

The thesis comprises of six chapters organized and summarized as follows:

Chapter 1: Introduction

Chapter 1 introduces back ground & motivation for the study, objective, methodology adopted for the research, contribution of the study in future and finally highlights organization of the whole research work.

Chapter 2: Property Enhancement Techniques – A Review

This chapter describes some of the methods that have been used and proven for properties improvement. Cases with magnetic field application for property enhancement are also discussed.

Chapter 3: Preparation for Fabrication Technique

In this chapter DOE is performed to determine how many experimental trials there shall be required for the research. It also tells about materials and fabrication processes selection along with magnetic field application design.

Chapter 4: Fabrication & Testing

This chapter tells about first fabrication detail and then testing that are performed on the samples for characterization of ILSS, flexural strength etc.

Chapter 5: Results & Discussions

In this chapter test results are compiled, compared and discussed on the part of improvements and discrepancies as well. For the future researchers, conclusions, limitations and future scope of research work is highlighted.

CHAPTER 2: PROPERTY ENHANCEMENT TECHNIQUES– A REVIEW

In the present study, glass fiber reinforced epoxy composites is deemed from transverse properties enhancement point of view when carbon nanotubes are strategically injected within interfaces of the composite laminates. In these composites, the ILSS appears as limiting design characteristic and these composites have weak (ILSS) along transverse loading. Carbon nanotubes can improve the transverse properties without weakening the in-plane mechanical properties [1]. Number of techniques have been used for improving the properties encompassing the in-plane and in-thickness properties, like interlaminar fracture toughness (G_{IC} and G_{IIC}), ILSS, tensile strength and flexural storage moduli [7] and all have been found with pleasant enhanced figures. Mechanical tests are performed on standard samples to experimentally determine the above mentioned properties. A number of techniques are employed by imaging on order of nano scale to observe closely the CNTs distribution and their preferable orientation. Magnetic field application has been found for aligning the CNTs thereby resulting in enhancement of properties [52, 53, 54, 55, 56]

Sequel to the above, found in the literature, glimpses of these approaches are presented in the following sections.

2.1 Literature Review

In this section, literature pertaining to the improvement of the mechanical properties of glass fibre epoxy composites fabricated with CNTs is reviewed in order to identify weaknesses or room for improvement in current approaches or to identify some novel methodology to come up with the enhanced transverse properties. To serve this purpose, a literature review about some of the experimental techniques that have been developed and used for the properties enhancement is being presented in the following sections.

2.1.1 Experimental Techniques used for Property Enhancement

Several experimental techniques have been used for dispersing and strategically injecting the CNTs in glass fibre epoxy composites and were found with the enhanced mechanical properties. Some techniques have used magnetic fields as well for CNTs alignment. Detailed overview is described as follows:

2.1.1.1 Use of Mechanical Stirring, Ultrasonication and Acid Oxidation

Zhihang fan et al. [1] measured the interlaminar shear strength of conventional glass fibre reinforced epoxy composites after enhancing it by injecting multi-walled carbon nanotube /epoxy suspensions into glass fibre mats in VARTM method of fabrication. High-speed mechanical stirring combined with ultrasonic agitation and acid oxidation were used for the suspension preparation. It was found that injection of MWCNTs caused enhancement in ILSS by 33%. MWNTs orientation across the thickness was deemed as to contribute to the increase in the said properties.

As depicted in Fig. 2.1A, fter having CNT-epoxy suspension distributed among layers, it might increase the ILSS of fibre reinforced composites. Also if this suspension is caused to flow across the thickness, the CNT present in suspension might take orientation in the thickness direction because of shear as depicted in Fig. 2.1B. They were of the opinion that if orientated CNT (between glass fibre layers in the thickness direction) effectively take load from the matrix, they could improve ILSS of the composite in an effective way [1].

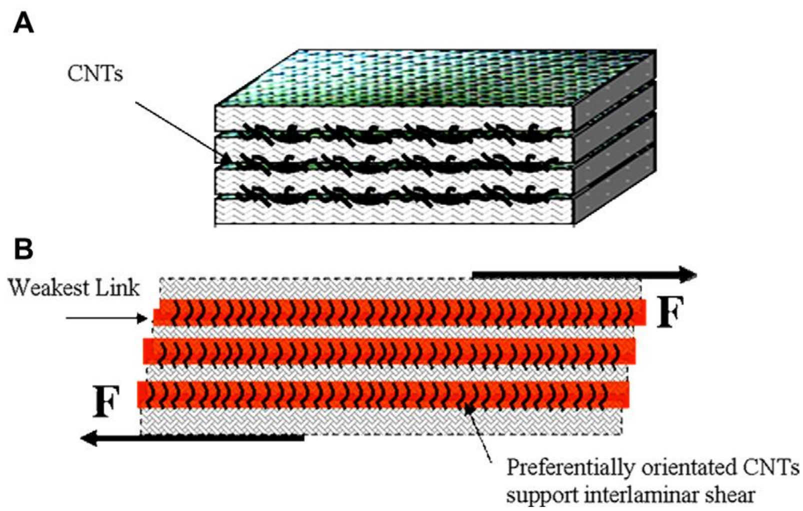


Fig. 2.1 Schematic showing CNTs between glass fiber layers which could increase the ILSS

2.1.1.2 Use of Developed Spray Process

F.L. Shan et al. [7] used vacuum-assisted resin infusion (VARI) molding method and prepared carbon fiber-reinforced epoxy composites, with multiwall carbon nanotubes (MWCNTs).

CNTs were deposited on the carbon fibre fabric with spraying epoxy resin E20 (with high viscosity) after spraying the CNTs. Actually E20 was used to fix CNTs on the fabric, to avoid flushing of the deposited CNTs by the infused resin during the infusion process. Then three type of composites were fabricated with different carbon fibre fabrics, like as-received, CNT-deposited with E20 and CNT-deposited without E20 and were subjected to study of the effects of CNTs on mechanical properties of the resulting composites.

They measured interlaminar fracture toughnesses, ILSS, tensile strength and flexural properties with enhanced figures.

Depositing CNTs with E20 increased the average propagation G_{IC} by 24%, the G_{IIC} by 11% and the ILSS by 12%, while preserving the in-plane properties. Depositing CNTs without E20 decreased the interlaminar fracture toughness. Differences in the CNTs distribution, fibre/matrix interfacial bonding and different spraying processing were regarded as played role in the properties enhancement.

2.1.1.3 A New Technique to Fabricate Nanocomposites with High Volume Fraction

Philip D. Bradford et al. [48] reported a method for the quick macroscopic CNT composites production resulting in high volume fraction. Millimetre long CNTs are well aligned produced by shear pressing which processes vertically aligned CNT arrays into dense preforms of aligned CNT. Preforms are then utilized in composites. Shear pressing is shown in Fig. 2.2.

SEM analysis confirmed CNTs alignment and TGA was used for CNT volume fraction to be 27% in the composites. Tensile tests reported good tensile strengths of 400 MPa.

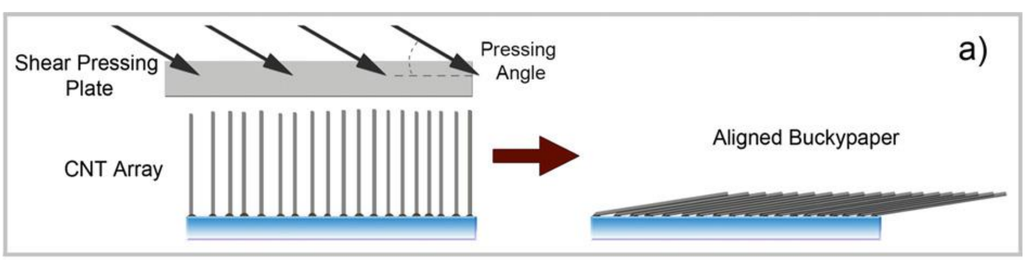


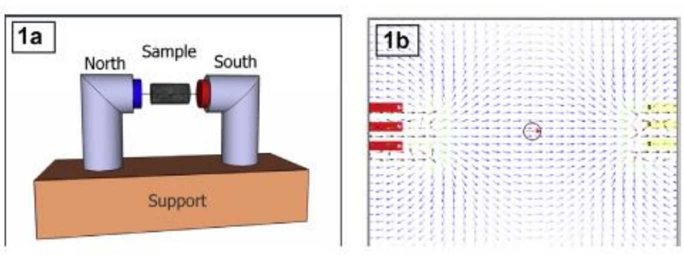
Fig.2.2: schematic showing CNTs array (before and after pressing)

2.1.1.4 Properties of CNTs–Polymer Composites with Magnetic Field Alignment

Erin Camponeschi et al. [54] performed experiments to explore the effect of alignment and orientation of CNTs. Epoxy polymer matrix was studied for CNTs alignment under a magnetic field for the per obtaining enhanced mechanical properties. It was shown that the changes in the the glass transition temperature, Young's modulus, elastic and viscous moduli, appeared as a function of the magnetic field strengths used for the CNTs aligning. Moreover, these enhancements were dependent on the resin system (epoxy) used. They showed that aligning CNTs with higher magnetic fields, gave superior results as compared to those composites that were not fabricated under a magnetic field.

2.1.1.5 Properties Enhancement with Use of Magnetic Field

S.G. Prolongo et al. [55] studied the properties of epoxy composites reinforced with magnetically aligned CNTs. A specific designed device was used for applying magnetic field of 0.3T. Permanent magnets were used as shown in Fig 2.3. CNTs were attached with magnetic nanoattachments (Fe_3O_4) and their bonding was confirmed by TEM. Composites were tested for thermo-mechanical as well as tensile properties and the results were higher for the magnetized composites than neat epoxy resin and were same for composites whether reinforced with pristine CNTs or magnetite–CNTs. An improved electrical conductivity was found in the direction of aligned CNTs.



(a) (b)

Fig. 2.3 (a) Application of magnetic field with the designed device (b) Spatial distribution of magnetic field lines

2.1.1.6 Effect of Magnetic Field and Multi-Wall Carbon Nanotubes Shape and Size on their Alignment

B.K. Jang , Y. Sakka [53] investigated the effect of shape and size of (CNTs) on their alignment under magnetism. A stable dispersion was observed when polyethylenimine (PEI) as a dispersant was added to CNT suspension.

To see the alignment response, CNT suspension was applied with a high magnetic field up to 12 T. CNTs were found with good alignment and it was noticed that it was depending on the field direction. Shape and size were also found influential on the part of degree of alignment. Thick, straight CNTs showed better alignment than the thin, curved CNTs.

2.1.1.7 Properties Enhancement in CNT/Polymer Nano Composites due to Magnetic Alignment

Anshu Sharma et al. [56] studied the properties of aligned CNT/Poly Carbonate nanocomposites fabricated under magnetic field of 1200 Gauss as shown in Fig 2.4.. 0.1wt% of SWCNTs and MWCNTs were homogeneously dispersed throughout the matrix by ultrasonic agitation. FTIR spectroscopy, Raman spectroscopy, electrical and mechanical measurements were performed to study the behavior of the nanocomposite. CNTs degree of alignment in polymer matrix was confirmed through Raman spectra. Mechanical strengths of the aligned CNT/polymer nano composites were characterized by dynamic mechanical analysis with enhanced figures.

Fig 2.4: Magnetic field application device for CNT/Poly Carbonate nanocomposites

2.1.2 Experimental Methods for Property Measurement

Different experimental methods/tests are used to characterize the behavior of composites. These are micro-tensile testing, mechanical testing like CST, SBS, DCB test, thermo-mechanical properties are determined by DMTA, thermo-gravimetric TGA is performed to obtain input material data for the micromechanics models (CNTs volume fractions etc).

2.1.2.1 Micro-Tensile Testing

Micro-tensile stage and load cell is used to perform the test on samples. Stage is attached to load cell and then samples are mounted into the stage with suitable gauge length. Tests are conducted with specific parameters like with 0.1 mm/min extension speed. Extension and load are recorded with a sample time of 500 ms until failure of the sample. When the test is over, sample dimensions and the collected data are utilized for calculating the tensile stress and strain. Stress–strain curves can be used for tensile young modulus calculation or from the tensile strength of the samples as well. It is better if three samples are tested (for better average results) for both the neat resin and the CNT composite [47].

2.1.2.2 Compression Shear Tests (CST)

CST is used to characterize a composite for ILSS [1]. As shown in Fig 2.5, in this this test, a two clamps set up is used and specimen is placed between the clamps in such a way that left clamp contacts the left half of the top surface, the right clamp contacts the right half of the bottom surface of the specimen in a symmetrical way. Vertical guides provide support to both sides of the specimen to constrain it from rotation or horizontal motion. The right clamp is fixed in position, and the left clamp generate direct interlaminar shear during its downward movement along the central plane of the specimen until failure. The failure load is divided by the area of the failure plane to determine ILSS of the specimen. This method provides a true ILSS.

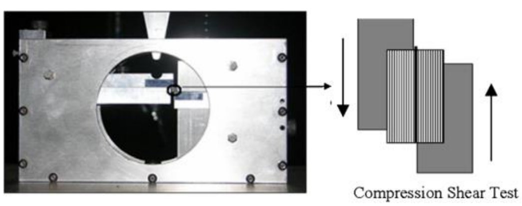


Fig 2.5: compression shear test (CST)

2.1.2.3 Short Beam Shear (SBS)

SBS method is used to measure the apparent ILSS of the composite through bending [1].
Interlaminar shear is generated indirectly to characterize the interlaminar shear strength of unidirectional, fiber-reinforced composites as per ASTM D 2344 [60]. As shown in Fig.2.6 in this test two cylindrical supports are used on which the specimen is placed in a balanced way. A cylindrical head is used for down motion at a specified speed and applies a force at the center of the span length and generates transverse load up to first failure. The failure load is then used to calculate the apparent ILSS of the composite.

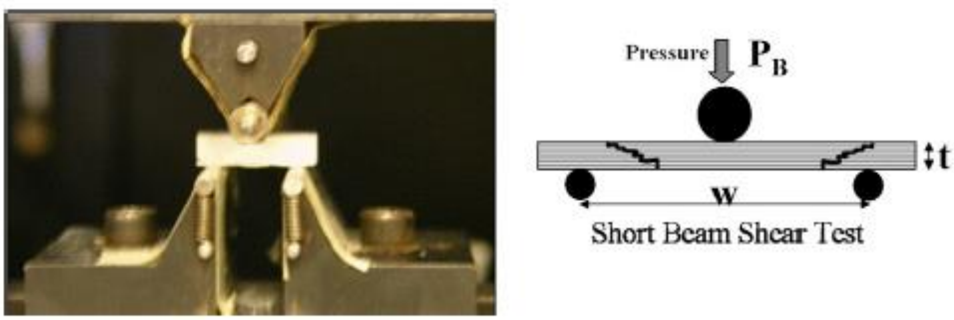


Fig 2.6: Short Beam Shear Test

2.1.2.4 DMTA

DMTA (Dynamic Mechanical Thermal Analysis) is performed in the single cantilever bending mode. Both fully cured neat epoxy resin and CNT composite samples are tested in the test. For sample specific analysis, the dimensions of samples are accurately recorded [47].
Measurements are conducted using a sinusoidal load with suitable displacement amplitude and frequency over a dynamic temperature profile at a heating rate of usually 3.0°C/min or as per requirement. The storage modulus (E′), loss modulus (E″) and tan delta are measured as functions of the sample temperature.

2.1.2.5 DCB Test

Double cantilever beam test is conducted according to ASTM standard D 5528, for interlaminar fracture toughness (G_{Ic}) measurement [7] illustrated in Fig. 2.7. Dimensions of specimens are

25 mm by 150 mm with PTFE film 65 mm in length inserted at the load application side. Load is applied through loading blocks, attached to both sides of the sample. The test is performed at a displacement rate of 1 mm/min on a universal testing machine. Crack extension is recorded for every millimeter of crack growth for stable growth and crack propagation. Interlaminar fracture toughness for mode-I is determined as per the following equation :

$$G_{IC} = \frac{3P\delta}{2b(a+|\Delta|)} \tag{2.1}$$

Where b is the width of specimen, a is the delamination length, P is the load, δ is the load point displacement, Δ is the horizontal axis intercept from a-$C^{1/3}$ curve. The compliance, C, is the ratio of the load point displacement to the applied load, δ/P

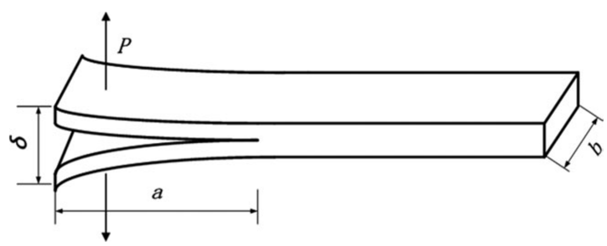


Fig 2.7: DCB test

2.1.2.6 Thermo Gravimetric Analysis (TGA)

TGA can be performed as per requirements on as-grown CNT forests, fully cured neat epoxy samples and fully cured CNT composite samples etc. In this method, samples are ramped to 700 C°- 850 C° at 10 C°/min rate and under a nitrogen flow of 50 ml/min to accurately measure the CNT mass present in the sample of composite. Using the recorded mass percentage values and calculated sample densities, average volume of CNT in the composite is calculated. The volume fraction of CNT is then calculated by following equation:

$$f_f = 1 - \frac{(1-m_f)\rho_C}{\rho_m} \tag{2.2}$$

Densities of composite(ρ_c) and neat resin (ρ_m) samples are measured by accurately measuring and weighing the samples and m_f is the CNT mass fraction calculated by TGA results (also can be calculate from the difference between neat resin and composite samples) [47].

2.1.2.7 Flexural Testing (Three Point Bend Test)

Flexural properties (flexural strength and modulus) are determined by 3-point bending test with specific speed, specimen dimensions and span length according to ASTM D 790 – 96a (Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials) [61]

2.1.3 Techniques for CNTs Alignment Confirmation / Property Characterization

Techniques like SEM, TEM, X-ray micro tomography, Raman spectroscopy etc. are concerned with getting insight at microscopic level (such as degree of CNTs alignment, dispersion, interaction between the CNTs and matrix etc).

2.1.3.1 Scanning Electron Microscopy (SEM)

The distribution pattern and alignment of as grown, CNT forest samples are examined at various magnifications using scanning electron microscope [47]. This is widely used for fractography to confirm ductile and brittle nature of failures of the samples.

2.1.3.2 Transmission Electron Microscopy (TEM)

The morphology of nano particles like bond of magnetite–CNTs in composite, CNTs dispersion in suspension etc is determined by transmission electron microscopy (TEM) [1].

2.1.3.3 Raman Spectroscopy

To investigate the interaction between the CNTs and matrix, to measure the degree of alignment of CNTs induced by any suitable mean can be ascertained through Raman spectroscopy [56].

CHAPTER 3: PREPARATION FOR FABRICATION TECHNIQUE

3.1 Fabrication Technique Selection

In this section reason for the fabrication methodology is described as follows:

- (Zhihang fan et al. 2008) Introduced MWNT with mechanical stirring, ultrasonication and acid oxidation into the VARTM technique and achieved increase in ILSS up to 33% [1]

- (F.L. Shan et al. 2013) Used MWCNTs with Resin E20 spraying on carbon fiber fabrics and then used VARI method for composite fabrication and achieved increase in GIc by 24%, in GIIc by 11% and in ILSS by 12% [48]

- Magnetic field application has shown properties enhancement mentioned in chapters 1 and 2 [52,53,54, 55, 56]

Therefore, this research will focus on fabricating the MWCNT/Glass fabric/Epoxy laminates with the combination of ultrasonic agitation, mechanical stirring using Vacuum Assisted Resin Infusion (VARI) method along with magnetic field application.

3.2 Magnetic Field Application Design

In order to apply a perpendicular magnetic field on the VARI set up, a solenoid(shown in fig 3.1) was designed and manufactured with the inner diameter of 8 inches to cater for the laminate dimensions minimum of (150x150) mm. Detail of the solenoid design and magnetic field calculations is given as follows:

Governing equation of the solenoid magnetic field is [62]

$$B = \frac{\mu_0 NI}{L} \qquad (3.1)$$

Where μ_0 is permeability of the free space, and $\mu_0 = 4\pi(10)^{-7}$ Hm^{-1}

I: current in the coil wire (Amperes)

N: number of turns of coil

L: length of the solenoid coil (m)

B: magnetic flux density (Tesla)

Anshu Sharma et al. [56] studied the effect of inclusions of CNTs in polycarbonate matrix with applying a magnetic field of 1200 gauss (0.12T) and they had magnetically aligned CNT/polymer nanocomposites with enhanced mechanical strength. This magnitude of magnetic field was selected for designing the magnet without core first as upon requirement; magnetic flux density could be enhanced with keeping mild steel in solenoid as core material. Length of the solenoid was assumed as 75mm. Winding wire was selected so that could withstand current up to 4.5A. With the above mentioned values, equation (3) gives N (number of turns) to be as follows:

$$N = \frac{BL}{\mu_0 I} \quad (3.2)$$

$= 0.12\text{ T }(0.075\text{m}) / 4\pi(10)^{-7}\text{ Hm}^{-1}(4.5\text{A})$

$= 1591$ turns OR 1600 turns

If solenoid is used with MS as core material with relative permeability μ_r = 2000

Then permeability μ can be expressed as [62]:

$$\mu = \mu_0 . \mu_r = 8000\pi(10)^{-7}\text{ Hm}^{-1} \quad (3.3)$$

Equation (3.2) then becomes as:

$$B = \frac{\mu N I}{L} \quad (3.4)$$

Fig 3.1: Solenoid

B.K. Jang et al. [53] obtained well aligned MWCNTs dispersed in a suspension with the use of higher magnetic field of 12 Tesla. Relatively a strong magnetic field is required to get the CNTs aligned for a specific purpose. Therefore taking 12 Tesla as reference, for the current research, three magnetic levels 8T, 12T and 16T were decided to investigate their effects on the properties. Equation (3.4) gives the corresponding currents of 0.15A, 0.224A and 0.3A to produce magnetic fields of 8T, 12T and 16T respectively. For this purpose a variable voltage regulator (step down transformer) shown in fig 3.2 was used with a multimeter to obtain the above mentioned currents on its output and as input to the solenoid. Correspondence tabulated in table 3.1 was established among voltage, current, and magnetic field values.

Fig 3.2: Variable Voltage Regulator

Table-3.1: Voltage, current and magnetic field correspondence for MS core solenoid

Parameters	Level 1	Level 2	Level 3	Unit
Voltage	52	79	105	Volt
Current	0.15	0.224	0.3	Ampere
Magnetic Field(B)	8	12	16	Tesla

During VARI moulding, CNTs were used with (0.5, 1.0 and 1.5) wt %ages which correspond to (1.03, 2.07 and 3.08) grams respectively. Higher magnetic field proves better in overcoming the resistance of viscous media as well faced by CNTs.

3.3 Design of Experiment (DOE) By Taguchi Method

Introduced by Dr. Taguchi, Taguchi method is a powerful technique used for problem solutions. This is widely utilized by industrials engineers and scientists for improving process performance and productivity [59]. He devised a statistical design of experiments (SDOE or DOE) which enables to design and manufacture products with high quality and low cost. This approach primarily focuses on elimination of the causes of poor quality and on making product performance insensitive to variation. He made use of the designs of orthogonal array (OA) like L_8, L_{16}, L_{18} and so on. Factors chosen for the experiment are assigned to the OA after having calculated degree of freedom of the experiment.

3.3.1 Application of DOE for the Current Research

DOE technique has been applied to determine how many experimental trials were required (here number of laminates) in achieving the enhanced properties while taking into account the control factors affecting the objective of the research (enhancing the ILSS, flexural strength of the glass/epoxy composite with MWCNTs). In laminate fabrication, weight of CNTs and magnetic fields were deemed as having vital effect on the resulting properties. Following is the step by step detail of the DOE application to this study.

Thesis Topic: Enhancement of mechanical properties in glass fibre epoxy composites using MWCNTs

Problem Statement: Glass fibre epoxy composites have weak interlaminar shear strength (ILSS) for loads applied along the thickness direction.

Objective: to enhance ILSS with the use of MWCNTs and magnetic field.

Control Factors: weight of CNTs and magnetic fields affect the stated objective and could be controlled during the experimentation conditions, therefore deemed as control factors and with three levels each. CNTs were used with (0.5, 1.0 and 1.5) wt %ages (discussed in Chapter 4) which correspond to (1.03, 2.07 and 3.10) grams respectively

Table-3.2: Control factors with defined levels

S.No.	Control Factors	Labels	Level 1	Level 2	Level 3	Unit
1.	Magnetic Field	A	8	12	16	Tesla
2.	CNTs Weight	B	1.04	2.07	3.10	gram

Noise factors: following are the possible noise factors which could affect the objective:

- Re agglomeration of CNTs
- Non uniform magnetic field

Interaction Factors: as the effect of one control factor (CNTs weight) would not be the same at different levels of the other factor (magnetic field), hence an interaction exists between magnetic field and CNTs weight, considered as follows:

AxB = AB

Total DOF of the experiment: DOF of a control factor is less by one unit from the number of levels associated to it. Following is DOF of the experiment:

(DOF of control factors) + ∑ (DOF associated with control factor)(number of interactions with that control factor)

= (2x2) + 2(1)

= 4+2

=6

Design of Orthogonal Array (OA):

Criterion: Number of experimental trials > Total DOF

L9>6

Hence L9 can be selected as OA for the current case.

Following is the outcome of Minitab for DOE by Taguchi.

Taguchi Orthogonal Array Design

L9 (3^2), Factors: 2, Runs: 9

Table-3.3: Total number of experimental trials

Trials	Magnetic field(Tesla)	CNTs Wt. (gram)
1.	8	1.04
2.	8	2.07
3.	8	3.10
4.	12	1.04
5.	12	2.07
6.	12	3.10
7.	16	1.04
8.	16	2.07
9.	16	3.10

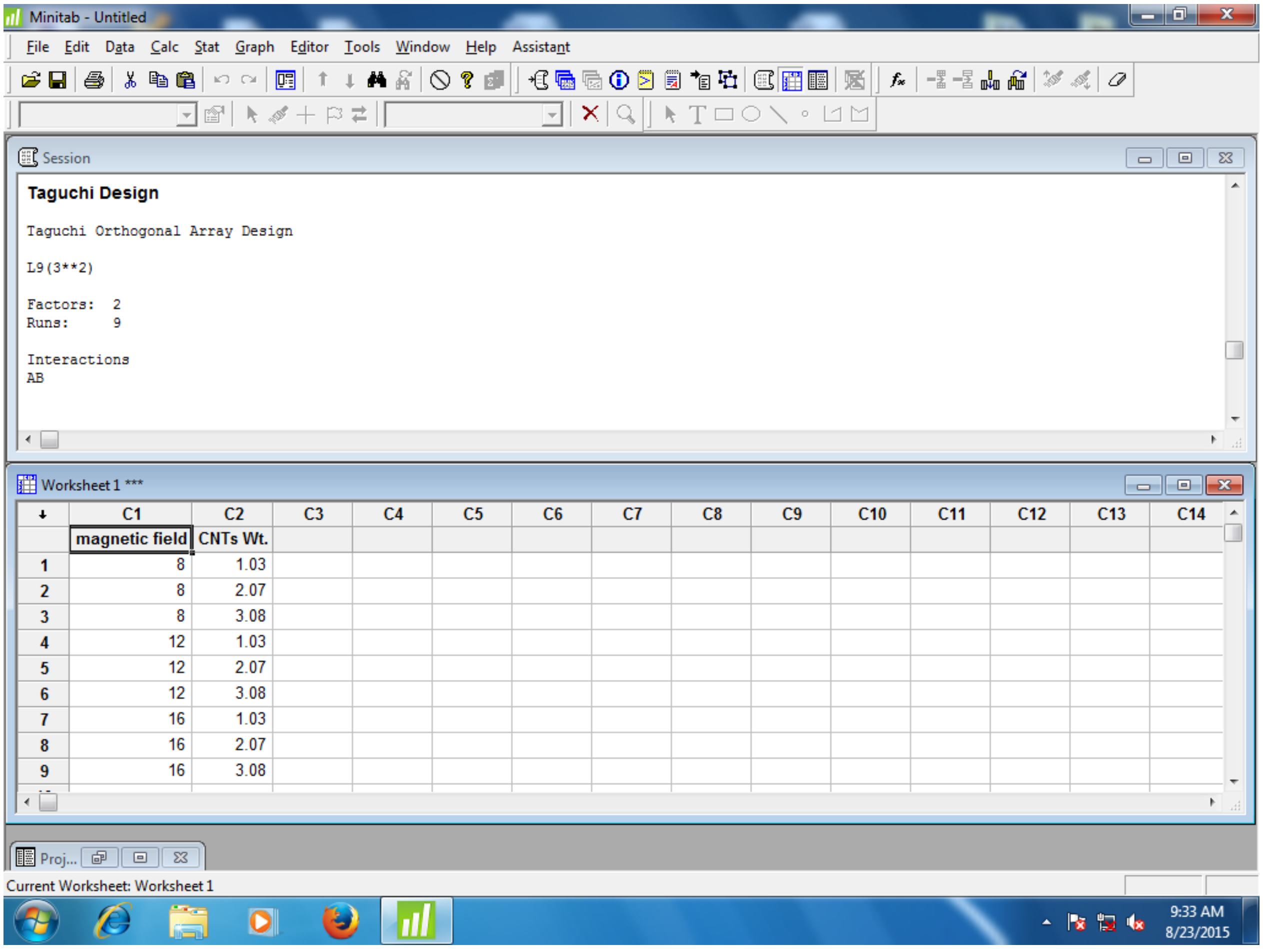


Fig: 3.3 Snap of the screen of Minitab showing number of experimental trials with combinations of CNTs weights and magnetic fields

CHAPTER 4: FABRICATION AND TESTING

4.1 Fabrication Scheme

In this section fabrication scheme is described as follows:

1. Simple glass fabric/epoxy laminate fabrication quantity 01
2. CNTs/glass fabric/epoxy laminates fabrication quantity 03 (with 0.5, 1.0 and 1.5 CNTs weight %ages respectively)
3. CNTs/glass fabric/epoxy laminates fabrication with magnetic field quantity 09 (each three with 0.5, 1.0 and 1.5 CNTs weight %ages and magnetic field levels respectively) as shown in table 3.3

4.2 Fabrication Materials

Fabrication materials used are mentioned as follows:

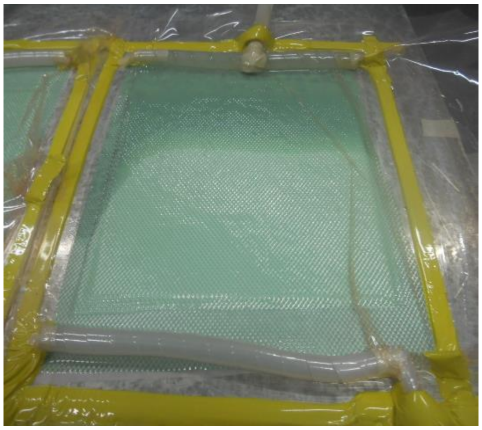
1. MWCNTs (OD: 50 – 80 nm), Length: 15 – 20 µm, BET Nitrogen surface area: 55-75 m^2/g , density: $120kg/m^3$, volume resistivity: $(2 – 5)x10^{-4}$ Ω/cm)
2. Resin LY 5052(gel time 45 min at room temperature, curing time 08 hrs)
3. Hardener: HY 5052
4. Glass Fabric No. 7725 plain weave (08 layers 150 x 150 mm each)
5. Acetone

4.3 Fabrication Processes

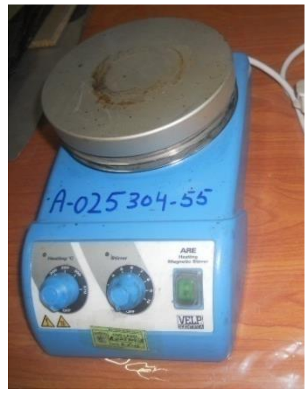
Fabrication processes of the required laminates have been depicted as follows:

4.3.1 Glass fabric/ Epoxy laminate Fabrication quantity 01

This was fabricated as a control sample. Hardener HY 5052 was added in pre-defined quantity (57gram) in 150 gram Resin LY 5052. Mixture was stirred in magnetic stirrer for a while till the two constituents properly mixed up. Then mixture was infused under vacuum of 760mm of Hg into the mold through vacuum assisted resin infusion method at room temperature as shown in the figure 4.1. VARI mould with vacuum bagging, with 8 glass fabrics (150x150mm) stacked together, with distribution media placed on the top, breather cloth; release film placed above the fabric stacks was used. A vacuum pump was used to create the necessary pressure gradient to get the suspension infiltrated into the glass fabrics. After letting it cured, laminate was demolded and subjected for post curing at 100^{0}C for 04 hours [20]. A schematic of process flow is shown in fig 4.2



(a)



(b)

(c)

(d)

Figure 4.1 (a) Vacuum Assisted Resin Infusion (VARI) with single vacuum bagging (b) Magnetic stirrer (c) Vacuum Pump Reservoir (d) Bath Sonicator

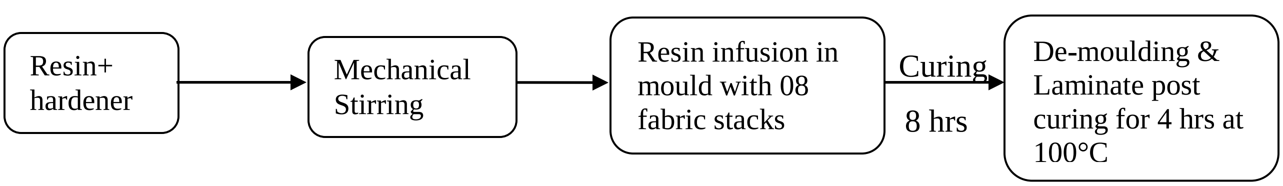


Figure 4.2 Resin Infusion Process of Glass fabric/ Epoxy laminate

4.3.2 MWCNTs /Glass fabric/ Epoxy laminate quantity 03

Some researchers have found use of acetone or ethanol as dispersant without or in combination with sonication that effectively distribute the CNTs in epoxy matrices [27, 28]. These three laminates were fabricated with adding MWCNTs in 0.5wt%, 1.0wt% and 1.5wt% respectively. In each case, MWCNTs were added to a pre-determined amount (50gram) acetone and agitated in bath sonicator for 1 hour. Then epoxy resin (150gram) was added to it and again agitated for 1 hour. Finally the mixture was placed at a magnetic stirrer at 80ºC and stirred for 4 hour to fully evaporate the acetone. This method was adopted for the CNTs uniform dispersion and distribution in epoxy [1]. Before infusion, hardener was added to the mixture, stirred and infused into the mould as previously. For concentrations more than 0.5 wt.% CNTs, VARI with double vacuum bagging was used [1]. In double vacuum bagging, after arrival of the suspension at the exit vent, the infusion and vacuum was stopped in the inner bag and applied in the outer bag for about 30 minutes [1] to relax the fabric stacks compression and to allow flow the mixture via capillary action up to lower layer. Then vacuum was re applied only in the inner bag to compress the stacks and squeeze out the excessive mixture. After the suspension had been cured, vacuum was discontinued and the hybrid MWCNT/glass/epoxy composite laminate was de-moulded. After demolding, laminates were subjected for post curing at 100^{0}C for 04 hours [20]. A schematic of process flow is shown in fig 4.3

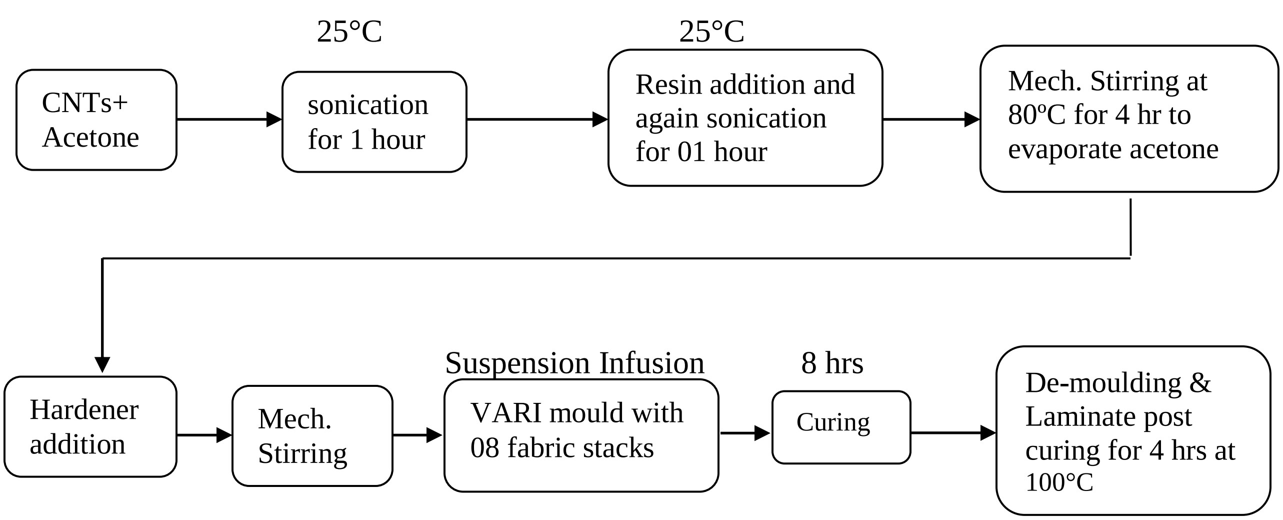


Figure 4.3 Resin Infusion Process of MWCNTs /Glass fabric/ Epoxy laminate

4.3.3 MWCNTs /Glass fabric/ Epoxy laminate quantity 09 with magnetic field

These laminates were fabricated with the magnetic field applied upon the set up for at least 2.5 hours (enough more than the resin jell time). Field was applied via solenoid for 8T, 12T and 16T as per combinations worked out by DOE as shown in table 3.3. The rest fabrication method is exactly similar as described in 4.3.2. A schematic of process flow is shown in fig 4.4. Schematic of the magnetic field applied on the set and its picture is shown in fig 4.5 (a) and (b) respectively.

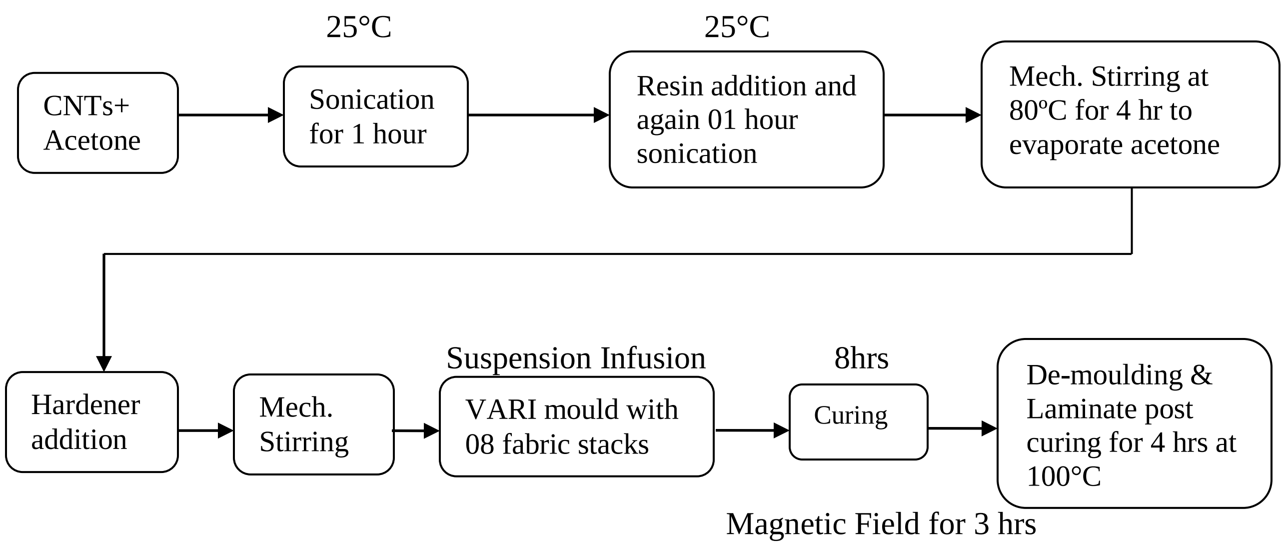
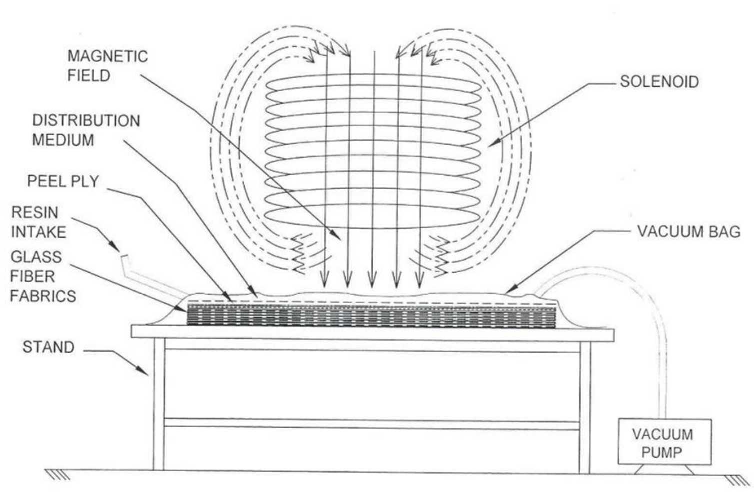
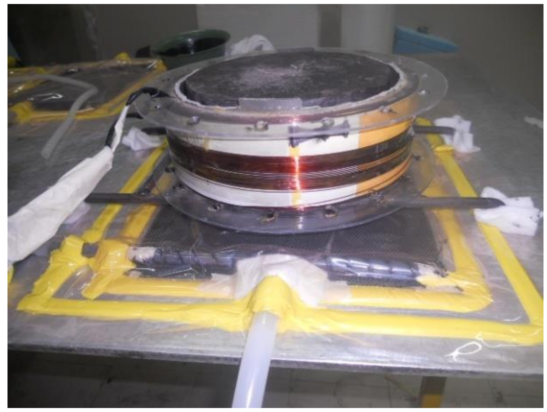


Figure 4.4 Resin Infusion Process of MWCNTs /Glass fabric/ Epoxy laminate with magnetic field application



(a)



(b) (c)

Fig 4.5: (a) Schematic of MWCNT/glass/epoxy composite being processed by VARI method and magnetic field application to align CNTs in the thickness direction (b) & (c) Snap Shot of the actual set up (VARI with double vacuum bagging with magnetic field applied on it)

4.3.4 Detail of Constituents of the Mixture

For each laminate, 08 glass fabrics each of size 150 x150 mm were used in VARI mould. If weights of resin and hardener are supposed as "X = 150gram" and "Y = 38%X" gram respectively, then the rest detail of the weights of the constituents defined in terms of X and Y is tabulated in table 4.1.

Table-4.1: Constituents of the Mixture

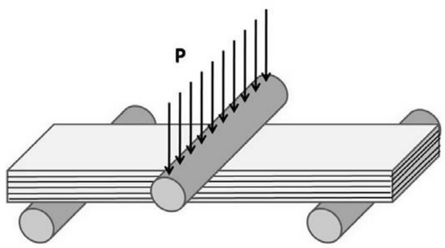
S.No.	**Constituents**	**%age**	**Weight(grams)**
1.	Resin LY 5052	-	150
2.	Acetone	33%X	50
3.	Hardener	38%X	57
4.	MWCNTs	0.5%(X+Y)	1.03
		1.0%(X+Y)	2.07
		1.5%(X+Y)	3.08

4.4 Samples Testing

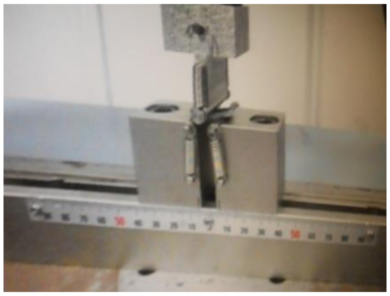
Samples were tested for flexural strength and interlaminar shear strength (ILSS). The interlaminar shear strength (ILSS) of the composite was measured using the short beam shear (SBS) method as per ASTM D 2344 [60]. Flexural strength was characterized by three point bending test as per ASTM D 790-96a [61].

4.4.1 Short Beam Shear (SBS)

SBS method is used to measure the apparent ILSS of the composite through bending [1]. Interlaminar shear is generated indirectly to characterize the interlaminar shear strength of unidirectional, fiber-reinforced composites [60]. As shown in Fig. 4.6, six samples per laminate with dimensions of 20 x 10 mm and with span length of 10mm were tested with universal testing machine (UTM). Cross head movement was kept as 1.3mm/min.



(a)

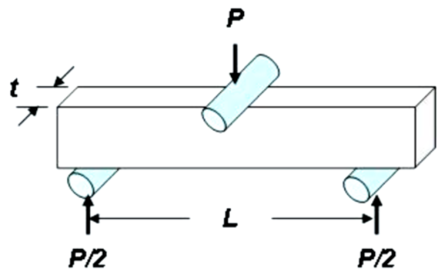


(b)

Fig 4.6: (a) Schematic of Short Beam Shear Test (b) Sample testing for SBS in UTM.

4.4.2 Flexural Test by Three Point Bend test

Flexural strength is measured by three point bending test according to American Society for Testing and Materials (ASTM-D 790-96a) [61]. Six samples per laminate with dimension 80 x 25 mm and with span length of 50mm were tested with universal testing machine. Cross head speed was kept as 2.8mm/min. Fig 4.7 shows the test schematic and a sample during testing phase.



(a) (b)

Fig 4.7: (a) Schematic of 3 Point Bend Test (b) Sample testing for flexure with UTM.

CHAPTER 5: RESULTS AND DISCUSSIONS

5.1 Test Results and Discussions

Test results for the tested samples for ILSS, flexural strengths and flexural moduli have been presented for the control (glass fiber fabric epoxy composite), CNTs loaded (unmagnetized and magnetized) glass fiber fabric epoxy composites tabulated in Table 5.1. Percent increase or decrease in the properties has been described by taking the control composite "A" as reference. As evident from the Table 5.1, only two composites (D and EM3) have shown improvement in flexural strength and flexural modulus. However, when magnetized samples are compared to the unmagnetized ones with their respective weight percentages, an increase is found for flexural strength, flexural modulus and ILSS for 0.5wt% magnetized samples. This trend of increase first almost becomes equal (for average values of the magnetized composites) for 1.0 wt.% composites and as weight percentage of CNTs increases up to 1.5%, a decrease is observed in all the three properties of magnetized composites compared to the unmagnetized composites.

Section 5.1.1 presents detail for the ILSS of CNTs loaded unmagnetized and magnetized glass fiber fabric epoxy composites first in comparison to the control composite "A" and then compares and explains results of the CNTs loaded magnetized composites with the unmagnetized composites. Section 5.1.2 describes flexural strengths and flexural moduli of CNTs loaded unmagnetized and magnetized glass fiber fabric epoxy composites both in comparison to the control composite and the unmagnetized composites.

Table 5.1: Mechanical properties of MWCNT/glass fabric/epoxy nanocomposites

Samples	Flexural Strength (MPa)	C.V	% change	Flexural Modulus (GPa)	C.V	% change	ILSS (MPa)	C.V	% change
A	406.651	0.040	-	20.869	0.018	-	34.051	0.057	-
B	292.113	0.050	-28.166	16.923	0.043	-18.910	18.234	0.182	-46.449
C	371.436	0.074	-8.6597	19.973	0.044	-4.2951	30.611	0.091	-10.104
D	407.05	0.046	+0.0979	22.07	0.034	+5.750	29.74	0.060	-12.662
EM1	259.506	0.145	-36.184	17.495	0.036	-16.169	17.939	0.146	-47.315
EM2	289.296	0.200	-28.858	18.705	0.288	-10.372	19.955	0.149	-41.395
EM3	392.930	0.045	-3.3741	21.331	0.036	+2.2124	31.429	0.047	-7.6996
FM1	316.044	0.051	-22.281	17.563	0.033	-15.843	22.887	0.075	-32.787
FM2	381.908	0.051	-6.0846	19.768	0.018	-5.2802	31.442	0.043	-7.6631
FM3	233.126	0.132	-42.671	17.090	0.014	-18.111	16.712	0.086	-50.921
GM1	358.689	0.055	-11.794	18.447	0.033	-11.606	26.928	0.048	-20.917
GM2	336.44	0.024	-17.265	18.103	0.040	-13.255	25.545	0.053	-24.979
GM3	383.743	0.057	-5.6334	19.298	0.025	-7.5291	27.799	0.071	-18.361

Where

A: glass fabric/epoxy composite

B: 0.5% MWCNT/ glass fabric/ epoxy composite

C: 1.0% MWCNT/ glass fabric/ epoxy composite

D: 1.5% MWCNT/ glass fabric/ epoxy composite

EM1, EM2, EM3: 0.5% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively

FM1, FM2, FM3: 1.0% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively

GM1, GM2, GM3: 1.5% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively

5.1.1 Interlaminar Shear Strength (ILSS)

As evident from fig 5.1 and table 5.1, behavior of decrease in ILSS has been exhibited by both the magnetized and un magnetized composites when compared to the control composite. Bai-Chen Wang et al. [26] observed 32.9% increase in flexural strength with only 0.025wt% CNTs loading and beyond to this limit, flexural and ILSS properties began to degrade. An explanation to this trend was mentioned due to reagglomeration of CNTs at higher loading and tendency to entangle there by reducing impregnation of the fabrics. Reagglomeration of CNTs can cause decrease in fiber/matrix interfacial strength [17, 24, 63]. However, when magnetized composites are compared to the un magnetized for ILSS, an increase is observed for 0.5wt% as the magnetic field increases from 8T to 16T. This behavior could be seen in table 5.2 and fig 5.2. This increase could be attributed to the effect of CNTs alignment with magnetic field for higher levels (12T and 16T) [54] and loading with low weight percentage [30].

Interaction of CNTs inclusions with polymer chains restricts movement of polymer chains. As alignment occurs, polymer chains come across more oriented and aligned CNTs and their movement becomes difficult that result in the increase of the stiffness properties [56].

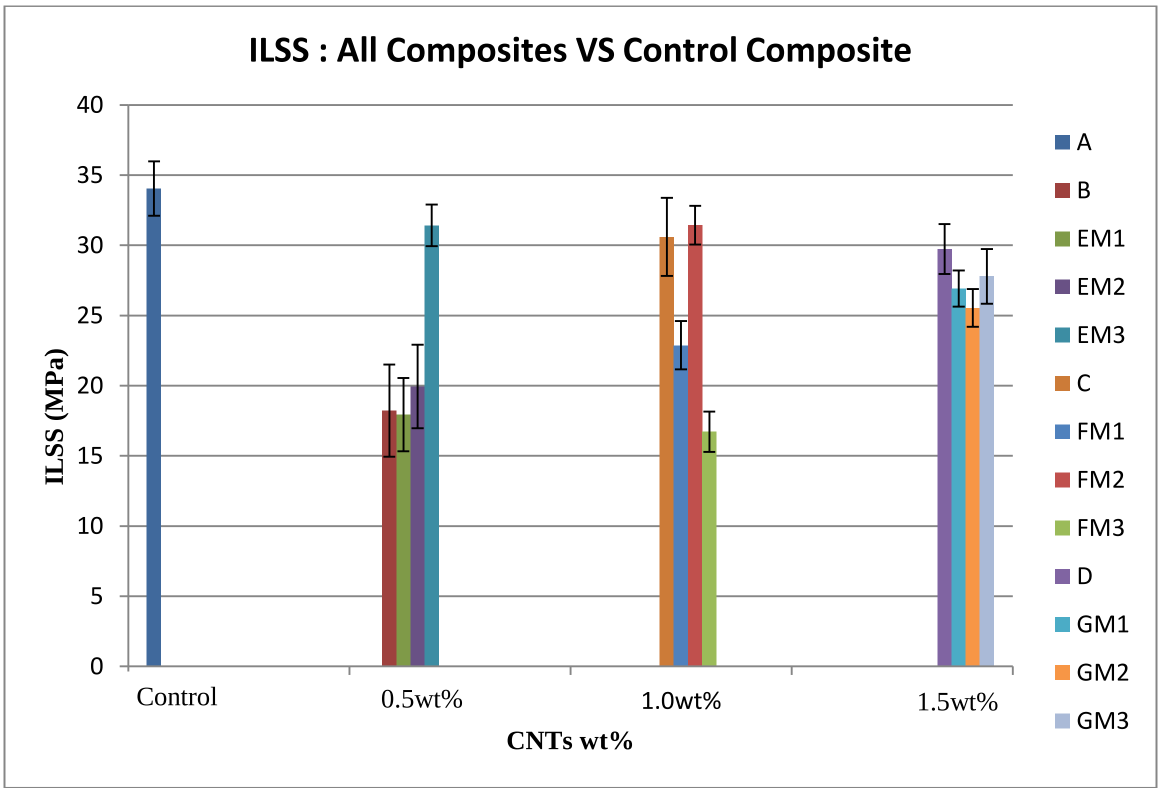


Fig 5.1 Comparison of ILSS of all composites with control composite A (0 wt%)

Where

A: glass fabric/epoxy composite

B: 0.5% MWCNT/ glass fabric/ epoxy composite

C: 1.0% MWCNT/ glass fabric/ epoxy composite, D: 1.5% MWCNT/ glass fabric/ epoxy composite

EM1, EM2, EM3: 0.5% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively

FM1, FM2, FM3: 1.0% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively

GM1, GM2, GM3: 1.5% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively

Table 5.2- Mechanical properties of MWCNT of Magnetized and Unmagnetized 0.5% MWCNT/ Glass fabric/ Epoxy Composites

Samples	Flexural Strength (MPa)	C.V	% change	Flexural modulus (GPa)	C.V	% change	ILSS (MPa)	C.V	% change
B	292.1133	0.051	-	16.9233	0.044	-	18.2347	0.182	-
EM1	259.5067	0.145	-11.162	17.4955	0.036	+3.381	17.9399	0.146	- 1.616
EM2	289.2967	0.200	- 0.964	18.7052	0.288	+10.53	19.9558	0.149	+9.438
EM3	392.9308	0.045	+34.51	21.3316	0.036	+26.05	31.4298	0.047	+72.36

Where

B: 0.5% MWCNT/ glass fabric/ epoxy composite

EM1, EM2, EM3: 0.5% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively

As percentage of CNTs increases from 0.5% to 1.5%, at higher concentrations of CNTs, there occurs reagglomeration which cause hindrance in the homogenous dispersion in epoxy and finally results in large agglomerates or “spot structures”. Such agglomerates cause decrease in ILSS of the nanocomposites [17, 24, 26] that can be seen in tables 5.3 and table 5.4. But here at 1.5wt%, the unmagnetized composite exhibited better than the magnetized samples at the same concentration. At higher CNTs concentrations, under the influence of magnetic field, a decreased degree of alignment has been reported because of the neighboring CNTs causing hindrance for their alignment [30].

Table 5.3-Mechanical properties of MWCNT of Magnetized and Unmagnetized 1.0% MWCNT/ Glass fabric/ Epoxy Composites

Samples	Flexural Strength (MPa)	C.V	% change	Flexural modulus (GPa)	C.V	% change	ILSS (MPa)	C.V	% change
C	371.43	0.074	-	19.973	0.0444	-	30.611	0.091	
FM1	316.04	0.051	-14.91	17.563	0.0332	-12.06	22.887	0.075	-25.23
FM2	381.91	0.052	+2.819	19.768	0.0188	-1.029	31.442	0.044	+2.715
FM3	233.12	0.132	-37.23	17.090	0.0144	-14.43	16.712	0.086	-45.40

Where

C: 1.0% MWCNT/ glass fabric/ epoxy composite

FM1, FM2, FM3: 1.0% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively

Table 5.4 - Mechanical properties of MWCNT of magnetized and un magnetized 1.5% MWCNT/ Glass fabric/ Epoxy Composites

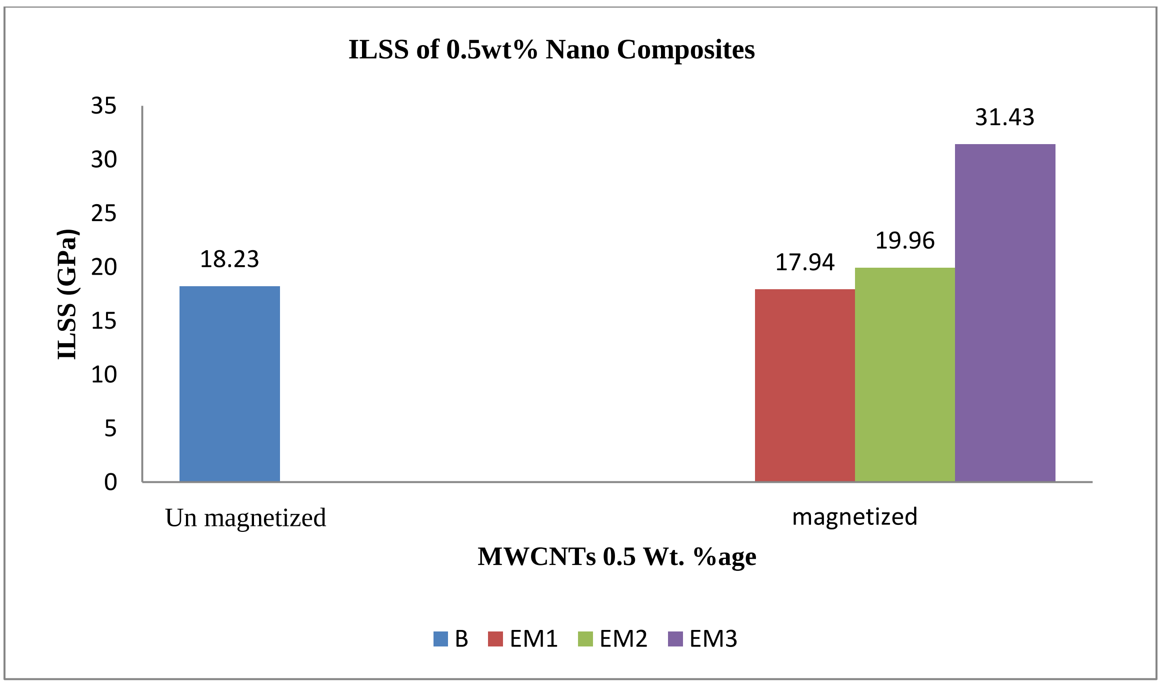
Samples	Flexural Strength (MPa)	C.V	% change	Flexural modulus (GPa)	C.V	% change	ILSS (MPa)	C.V	% change
D	407.05	0.046	-	22.07	0.034	-	29.74	0.060	-
GM1	358.69	0.055	-11.88	18.45	0.033	-16.41	26.93	0.048	-9.452
GM2	336.44	0.024	-17.35	18.103	0.040	-17.97	25.54	0.053	-14.103
GM3	383.74	0.057	-5.72	19.298	0.025	-12.55	27.799	0.071	-6.526

Where

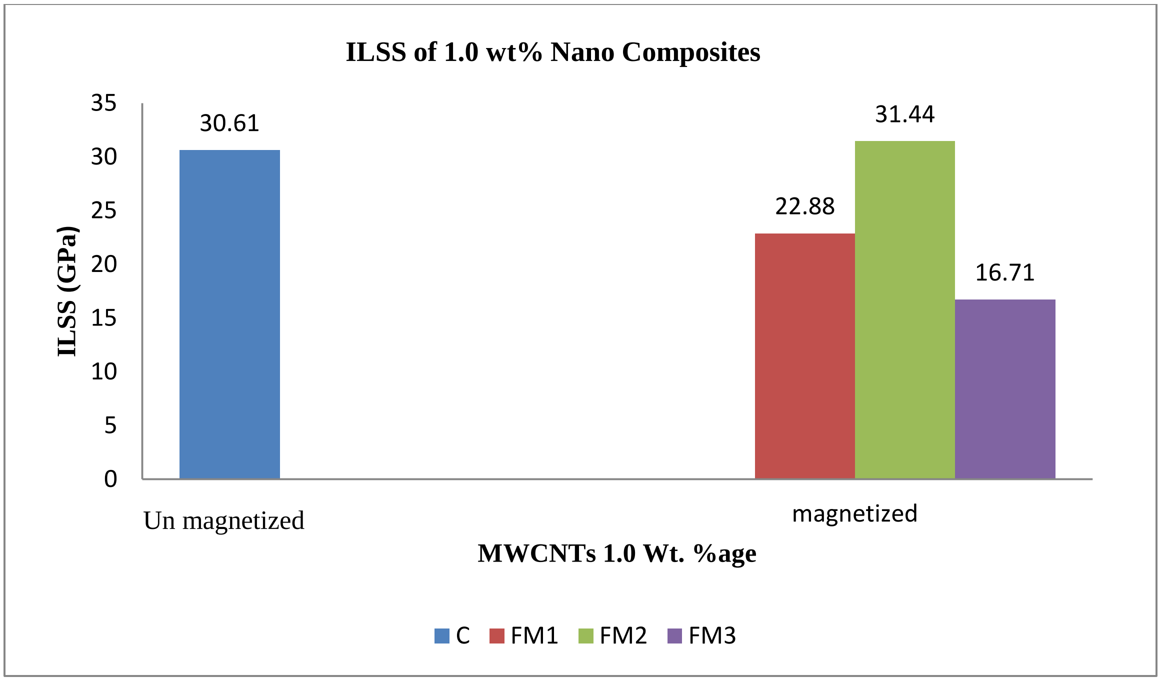
D: 1.5% MWCNT/ glass fabric/ epoxy composite

GM1, GM2, GM3: 1.5% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively

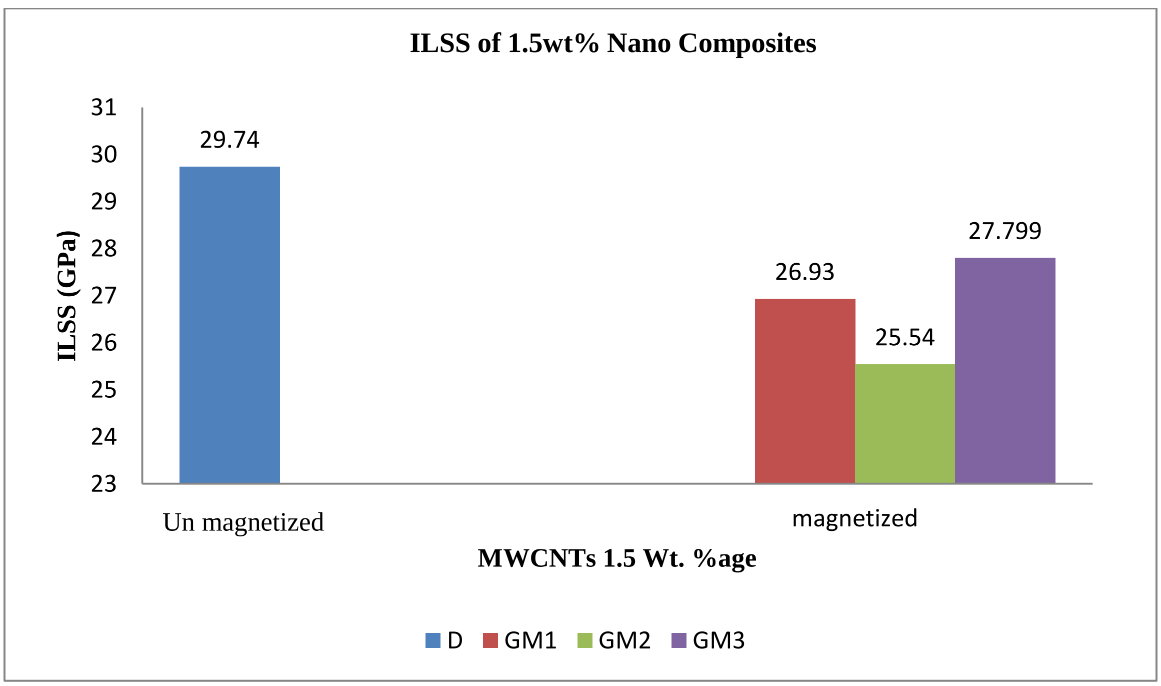
Contrary to the above stated reason, behavior of resulting in lower ILSS and flexural properties can be explained by slight negative effect of the applied magnetic field as well. Chuanguo Ma et al. [42] argued that aligned CNTs do not form 3D network easier than randomly oriented CNTs. With this they become less efficient in constraining movements of the macromolecular chains of epoxyIn such situation, interlaminar slippage could be understood under higher concentrations and higher magnetic fields. This slippage would have resulted in decreased ILSS values of the higher concentration magnetized composites. Such samples underwent relatively through higher failure displacements under lower loading applications. Average failure results of the composite "FM3" (1.0 wt%) specimens indicate failure at higher displacement (0.45mm at 484.22N) compared to "C" with lower displacement (0.37mm at 720N) respectively as shown in fig 5.3.



(a)

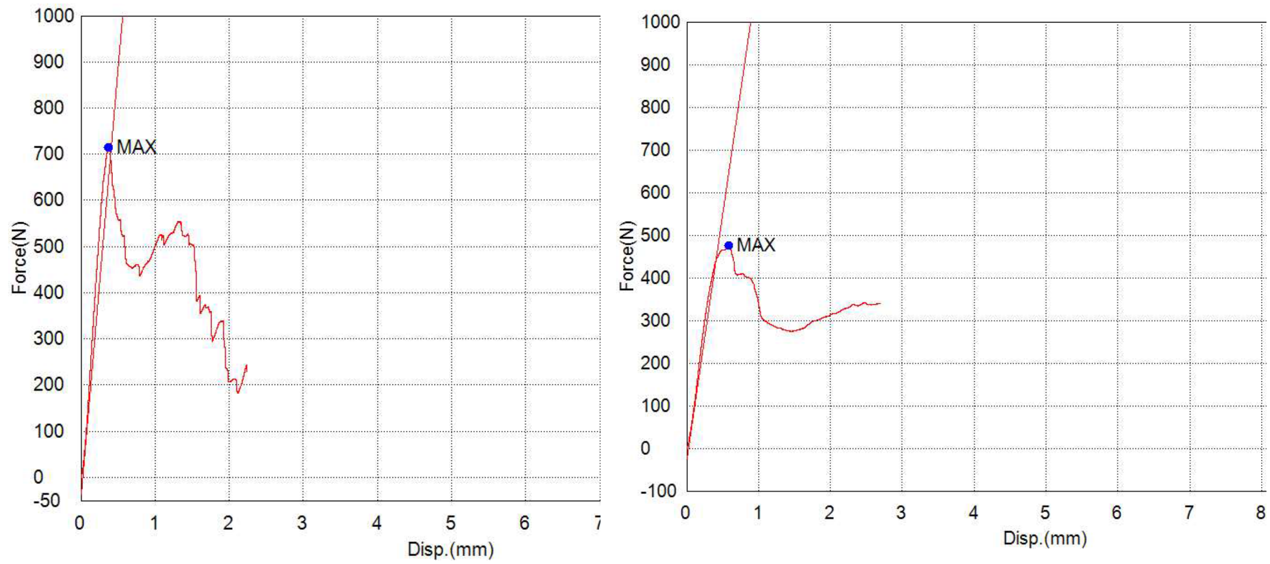


(b)



(c)

Fig 5.2 Comparison of ILSS of magnetized with un magnetized composites (a) 0.5wt% CNTs composites (b) 1.0wt% CNTs composites (c) 1.5wt% CNTs composites.



(a) (b)

Fig 5.3 load-displacement diagrams of (a) un-magnetized 1.0 wt% CNTs composite “C” (b) magnetized 1.0wt% CNTs composite “FM3”

5.1.2 Flexural Strength and Flexural Moduli

For 0.5wt%, 1.0wt% and 1.5wt% CNT/glass fabric/epoxy composites, results have been shown in table 5.1. Fig 5.4 and fig 5.5 show results for flexural strengths and fig 5.6 and 5.7 explains the same for flexural moduli of the composites. Composite D has shown a marginal enhancement of 0.098% in flexural strength and 5.75% in modulus as compared to the control composite “A”. Composite EM3 has shown enhancement of 2.21% only in flexural modulus .The rest all composites have shown decrease in flexural strength and moduli. Bai-Chen Wang et al. [26] reported good results for flexural strength and ILSS up to 0.025wt% CNTs loading and beyond to this limit, flexural and ILSS properties began to degrade due to re agglomeration of CNTs. Some researchers have obtained good enhancement in flexure strength up to 0.25wt % loading and as loading was increased, a fall in flexure properties was observed [31]. At higher weight loading of CNTs, sonication plays a vital role in good dispersion and finally in achieving better mechanical properties [28]. Decrease in the flexural properties could be because of CNTs non uniform dispersion at higher concentrations of CNTs loading. Reagglomeration has been reported for causing decrease in mechanical properties [17, 24, 26]. However, an increase in flexural properties is found at 0.5wt% level in magnetized samples compared to unmagnetized composite as shown in table 5.2, figure 5.5 and 5.7 respectively and a clear trend towards rise in flexural properties (flexural strength and flexural moduli) is noticeable with the rise in magnetic field from 12T to 16T. For higher concentrations (1.0wt% and 1.5wt% CNTS loading), magnetized composites have shown decrease in flexural properties as indicated in table 5.3, table 5.4, fig 5.5 and fig 5.7. At higher concentration of CNTs, there could be two possibilities for lower flexural properties, first tendency of reagglomeration and second a slight negative effect of the applied magnetic field indicated by Chuanguo Ma and the co-researchers [42]. In this context of comparison, where with same higher concentrations (1.0wt% and 1.5wt% CNTS loading), magnetized composites exhibited lower flexural properties and ILSS than the unmagnetized ones and hence the reason of re-agglomeration can be eliminated (although re-agglomeration would exist in CNTs loaded composites as these have resulted in almost lower values for all the three properties in comparison with control composite A). We are only left with the magnetic alignment of CNTs being responsible for some tendency of preventing CNTs to form a good 3D network in the epoxy [42]. Due to this effect, CNTs become less efficient in constraining the movements of the macromolecular chains of epoxy. Hence interlaminar slippage could easily occur under higher CNTs concentrations and higher magnetic fields. This slippage might result in decreased flexural properties and ILSS values of the higher concentration magnetized composites meaning that such samples would relatively show higher failure strains under lower loading applications.

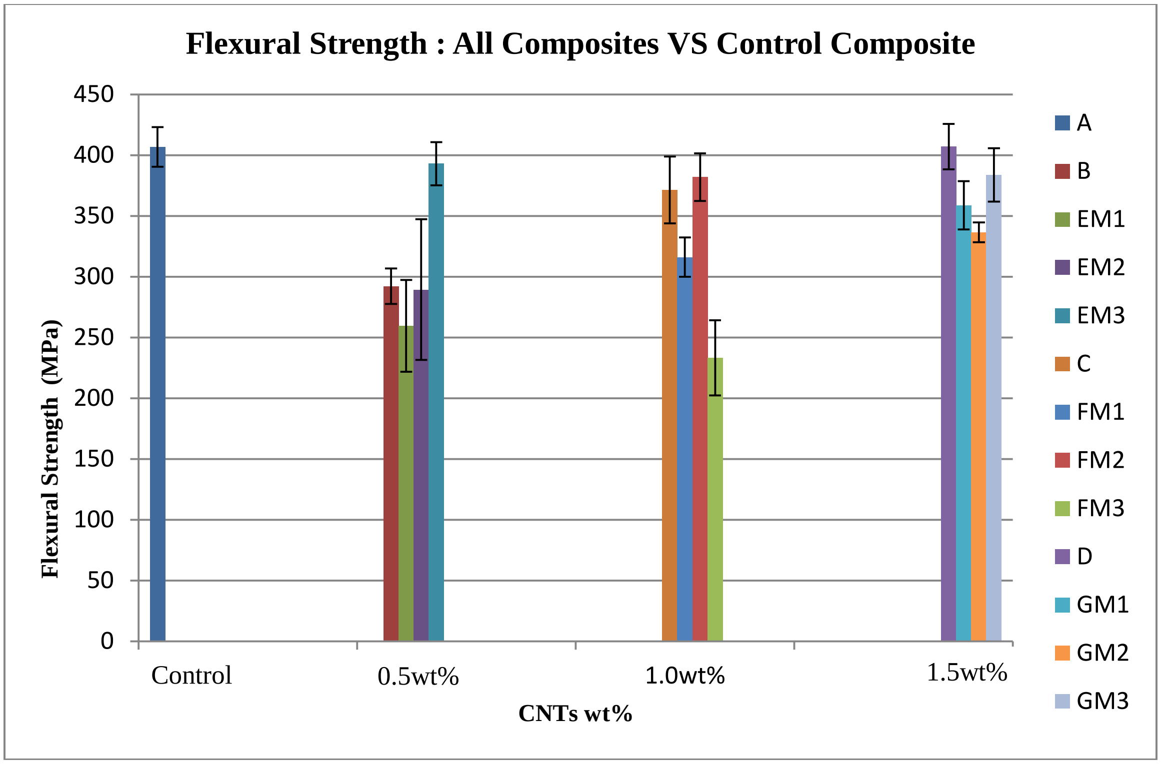


Fig 5.4 Comparison of Flexural strengths of all composites with control composite A (0 wt%)

Where

A: glass fabric/epoxy composite

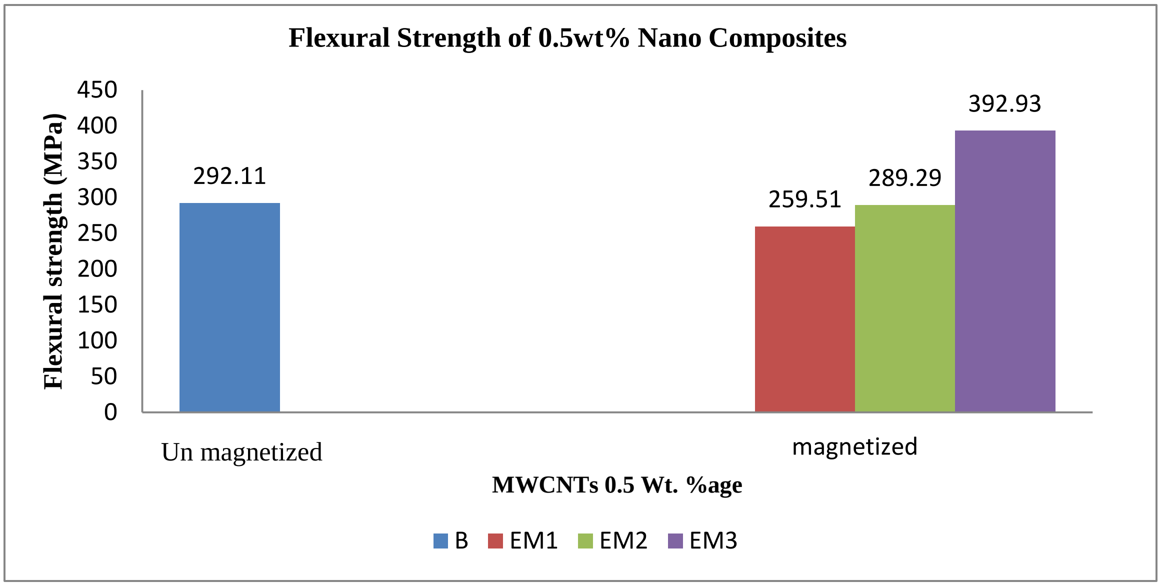
B: 0.5% MWCNT/ glass fabric/ epoxy composite

C: 1.0% MWCNT/ glass fabric/ epoxy composite, D: 1.5% MWCNT/ glass fabric/ epoxy composite

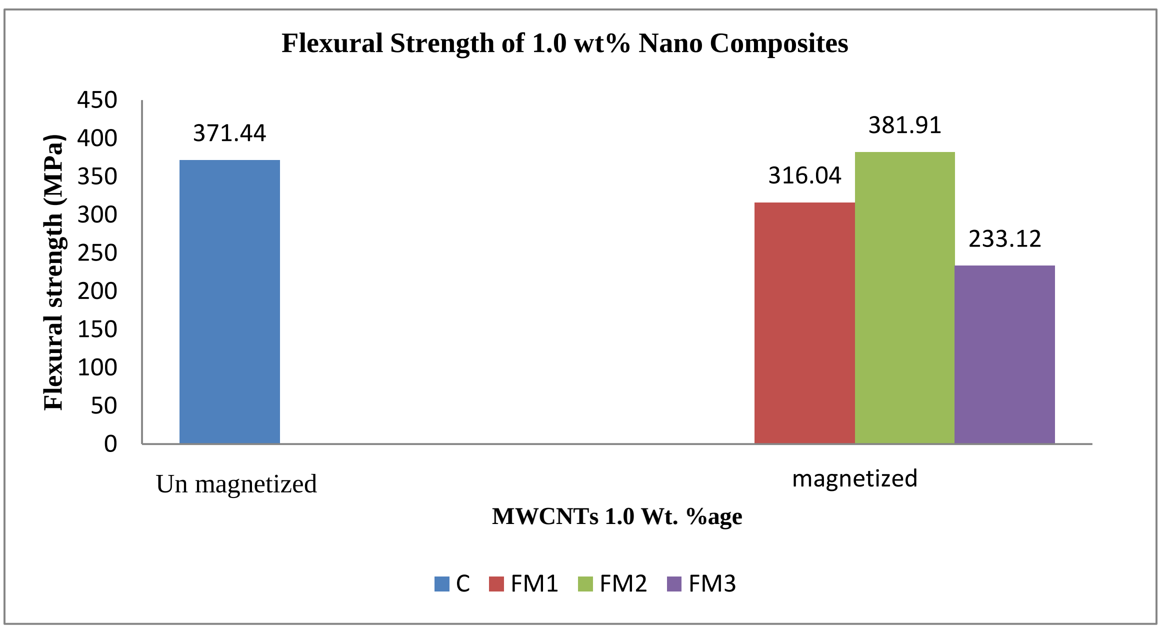
EM1, EM2, EM3: 0.5% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively

FM1, FM2, FM3: 1.0% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively

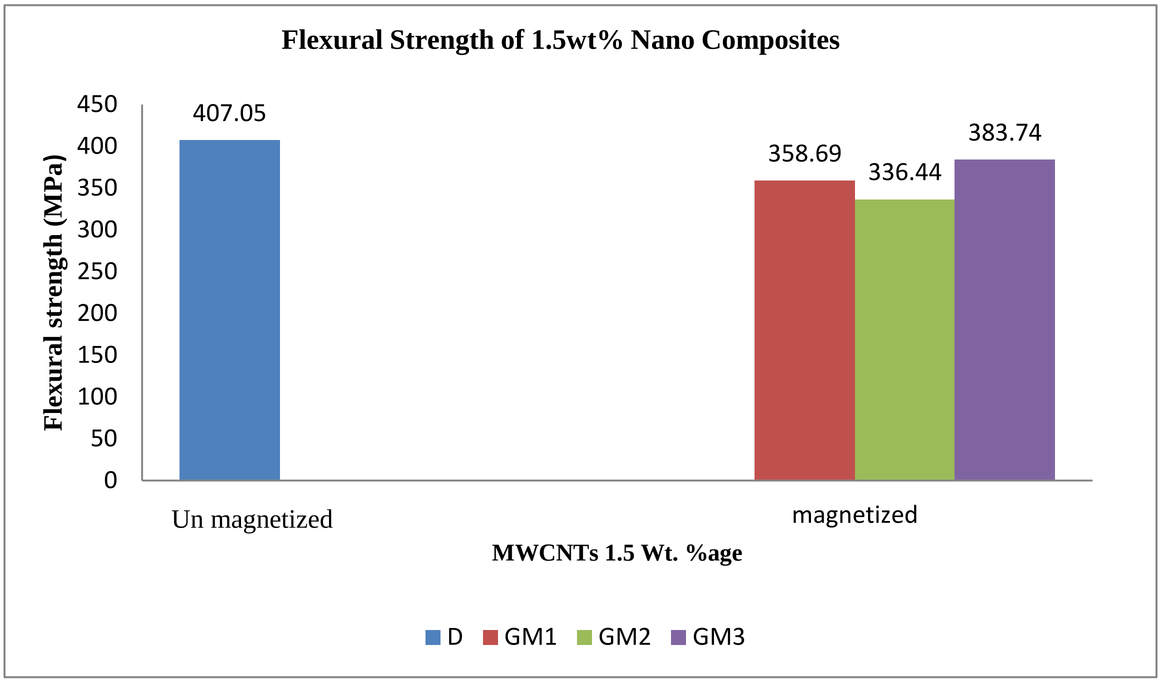
GM1, GM2, GM3: 1.5% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively


Flexural Strength of 0.5wt% Nano Composites
Flexural strength (MPa)
450
400
350
300
250
200
150
100
50
0
292.11
259.51
289.29
392.93
Un magnetized
magnetized
MWCNTs 0.5 Wt. %age
B
EM1
EM2
EM3

(a)


Flexural Strength of 1.0 wt% Nano Composites
Flexural strength (MPa)
450
400
350
300
250
200
150
100
50
0
371.44
316.04
381.91
233.12
Un magnetized
magnetized
MWCNTs 1.0 Wt. %age
C
FM1
FM2
FM3

(b)



(c)

Fig 5.5 Comparison of Flexural strengths of magnetized with un magnetized composites (a) 0.5wt% CNTs composites (b) 1.0wt% CNTs composites (c) 1.5wt% CNTs composites.

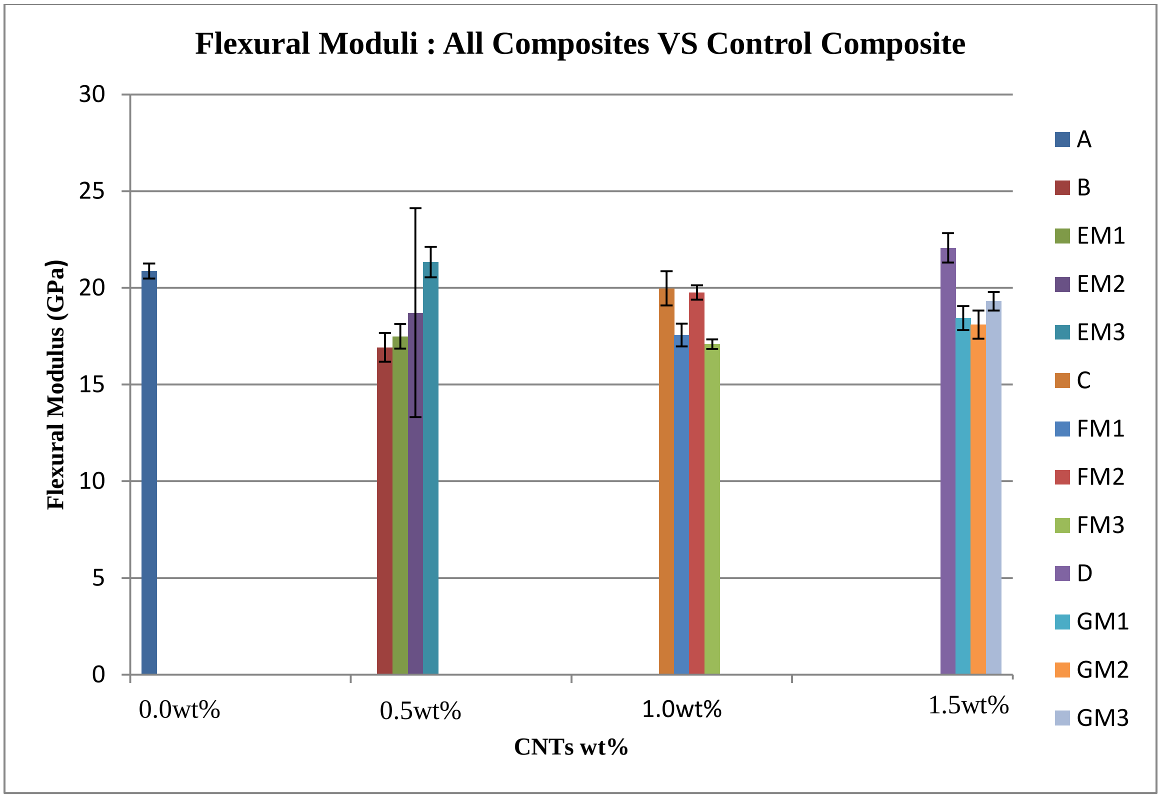


Fig 5.6 Comparison of flexural moduli of ALL composites with control composite A(0 wt%)

Where

A: glass fabric/epoxy composite

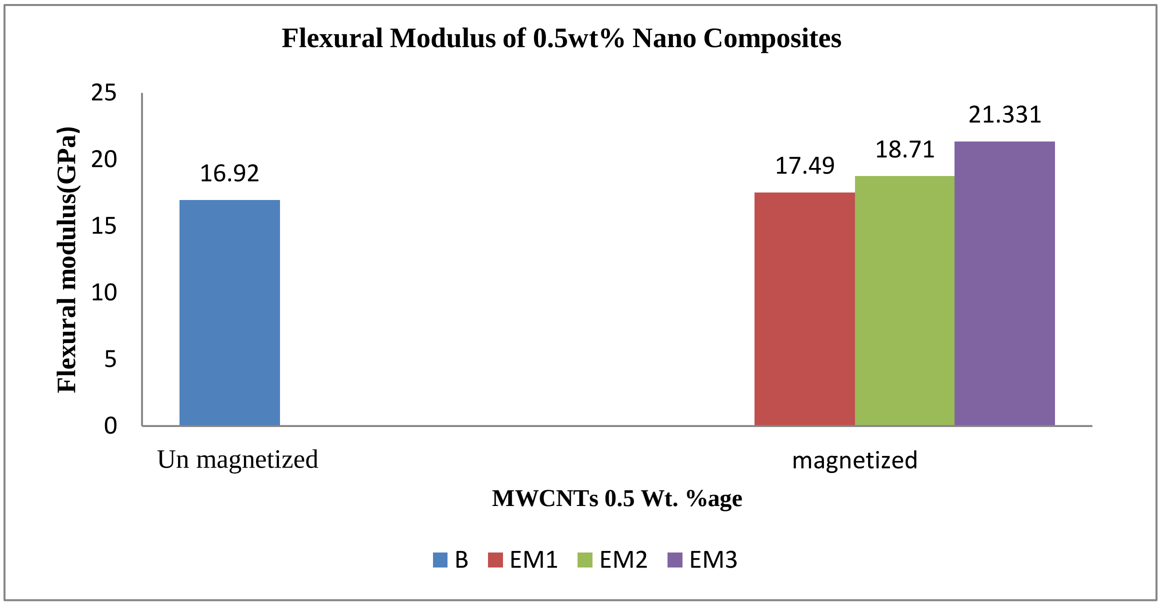
B: 0.5% MWCNT/ glass fabric/ epoxy composite

C: 1.0% MWCNT/ glass fabric/ epoxy composite, D: 1.5% MWCNT/ glass fabric/ epoxy composite

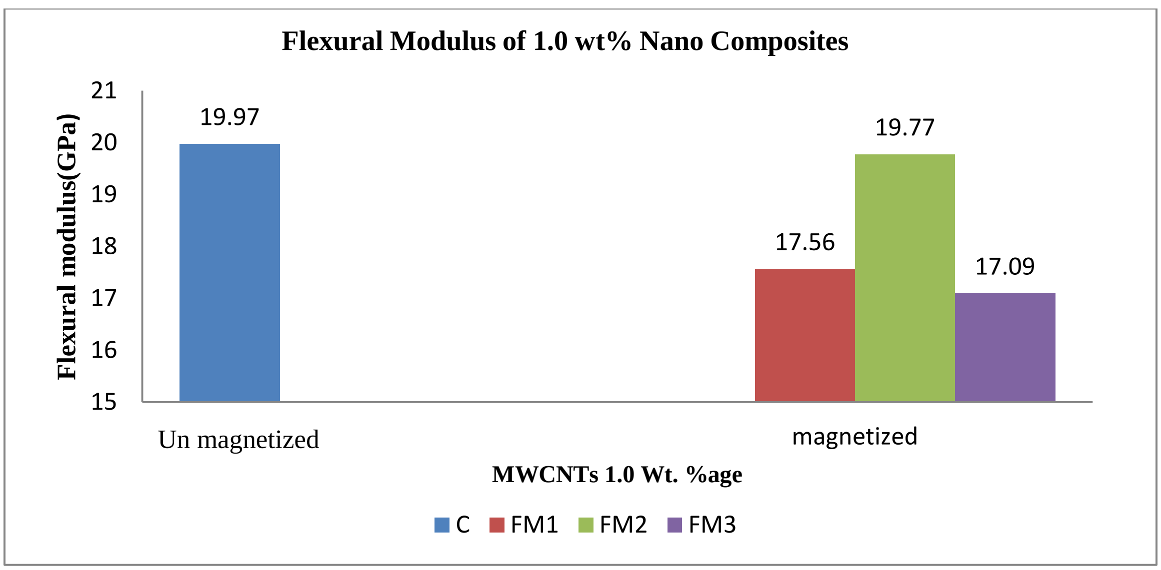
EM1, EM2, EM3: 0.5% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively

FM1, FM2, FM3: 1.0% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively

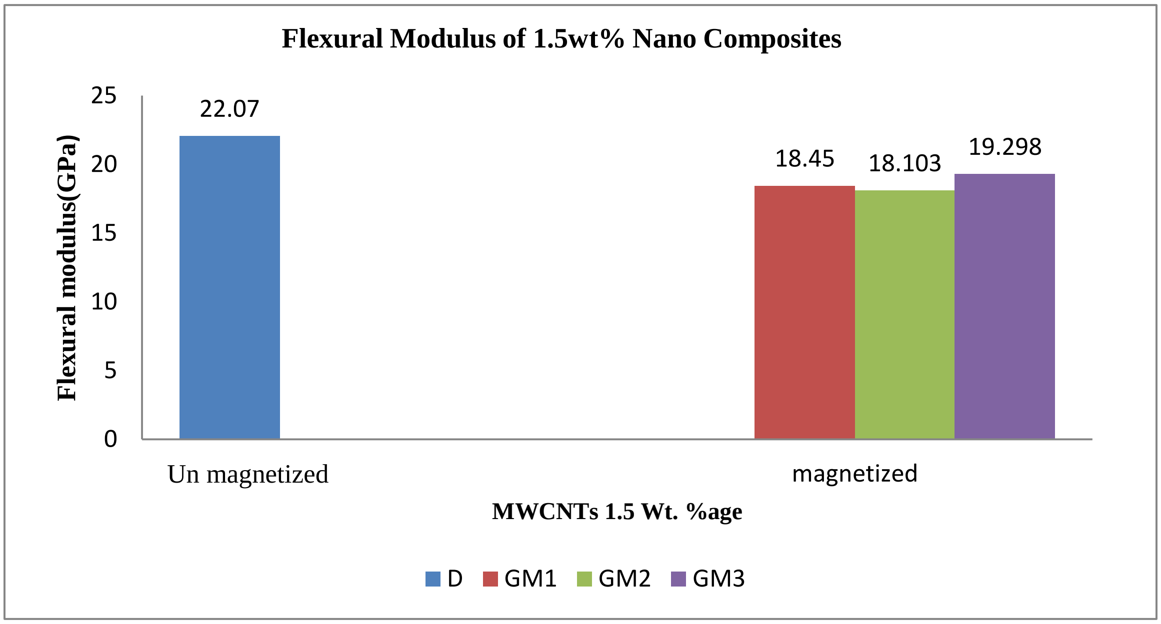
GM1, GM2, GM3: 1.5% MWCNT/glass fabric/epoxy composites fabricated under magnetic fields of 8T, 12T and 16T respectively


Flexural Modulus of 0.5wt% Nano Composites
Flexural modulus(GPa)
25
20
15
10
5
0
16.92
17.49
18.71
21.331
Un magnetized
magnetized
MWCNTs 0.5 Wt. %age
B
EM1
EM2
EM3

(a)


Flexural Modulus of 1.0 wt% Nano Composites
Flexural modulus(GPa)
21
20
19
18
17
16
15
19.97
17.56
19.77
17.09
Un magnetized
magnetized
MWCNTs 1.0 Wt. %age
C
FM1
FM2
FM3

(b)



(c)

Fig 5.7 Comparison of Flexural moduli of magnetized with un magnetized composites (a) 0.5wt% CNTs composites (b) 1.0wt% CNTs composites (c) 1.5wt% CNTs composites.

5.2 Conclusions

From the literature survey of glass fiber/epoxy composites, it was found that these composites show decreased delamination resistance towards transverse loading [1] and further weak fiber/fabric-matrix interface and brittle nature of resin matrix in composite laminates principally may cause delamination failures [3]. It was found that CNTs have shown enhancement of properties when strategically injected into the composites. Magnetic field application on the fabrication set up has shown properties improvement as well.

As MWCNTs could be easily produced and it was thought if these are infused into the interplay regions of the glass fabrics through vacuum assisted resin infusion (VARI) process and further be aligned with magnetic field, these might result in enhanced ILSS and flexural properties.

From the experimental results of the research work, it was observed that when MWCNTs were used in 0.5wt % in glass fabric epoxy composite, an increase was noticed for ILSS and flexural properties under the effect of magnetic field application. With this CNTs concentration, an increasing trend in the above mentioned properties was noticed with the increasing magnetic fields from 8T to 16T. However, at higher CNTs concentrations (1.0wt % and 1.5wt %), decrease in the ILSS and flexural properties was observed. This tendency can be due to the decreased degree of alignment occurring at higher CNTs concentrations [30] or can be explained by a contrary argument argued by Chuanguo Ma and the co-researchers [42]. According to them, increased magnetic alignment of CNTs in between the layers can be regarded responsible for some tendency of preventing CNTs to form a good 3D network in the epoxy(as compared to the randomly oriented CNTs) and in the absence of such networks, polymer chains can easily move resulting in the decrease in properties.

5.3 Limitations

- Pristine MWCNTs require powerful magnetic field to get them aligned in the field direction [53] until these are functionalized with nano attachments like Fe_3O_4
- MWCNTs show less susceptibility towards magnetic field as compared to SWCNTs

5.4 Future Scope of Work

The same work can be re explored for further research in the context of focusing under modern property characterization techniques like Raman spectroscopy, SEM, TEM to look into the actual causes of negative effects of strong magnetic alignment of CNTs in the thickness direction occurring at higher concentrations and higher magnetic fields.

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