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# Effect of Carbon Nanotube on Thermo-Mechanical Properties of Glass Fiber/Epoxy Laminated Nanocomposites

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***Abstract-*** Nanocomposites has emerged, penetrated and conquered the market over the past years due to its wide range of applications. The processing of nanocomposites is one of the fastest growing areas in materials research due to the potential of significantly changing material properties even at low nanomaterial concentrations.

The thermo-mechanical behaviors were investigated experimentally for two systems of glass fiber reinforced epoxy composites: with carbon nanotubes (CNTs) and without CNTs. An experimental campaign which involves 4 layers laminates were conducted in this study. A manufacturing process using hand lay-up technique followed by hot pressing techniques was used to produce glass fiber-reinforced laminates with the epoxy matrix and with the CNT based epoxy matrix. The surface characterization and thermo-mechanical behavior of the glass fiber-epoxy system and glass fiber-CNT/epoxy system was characterized through Scanning Electron Microscopy (SEM) and Dynamic Mechanical Analysis (DMA).

***Index Terms***- Carbon nanotube, Nanocomposites, Glass fiber, DMA, SEM.

## I. Introduction

Composite materials [1] containing carbon fibers/glass fibers are widely used in applications ranging from aerospace to sports equipments. Carbon nanotube based nanocomposite is one of the fastest growing areas of materials research in developing new materials with new and improved properties [2, 3, 4]. The advantages of carbon nanotube based nanocomposites over conventional composites are lighter, high specific strength, high specific modulus, high chemical resistance, high electrical conductivity, etc. Yet, as the use of nanocomposite materials in modern applications is increasing, these materials are now extensively used in primary parts of aeronautical applications. The use of epoxy resin as matrix for the fiber-reinforced composites in the structural applications has been increased significantly [5]. As far as many studies have been achieved on nanocomposites [6, 7], only few investigations were conducted on truly multiscale composites including both fibers and nano reinforcements [8, 9, 10].

The aims of this study are (1) to fabricate glass fiber reinforced laminated nanocomposites using CNT-epoxy resin and (2) to compare the thermo-mechanical properties of glass fiber reinforced epoxy laminated composites and glass fiber reinforced CNT-epoxy laminated nanocomposites.

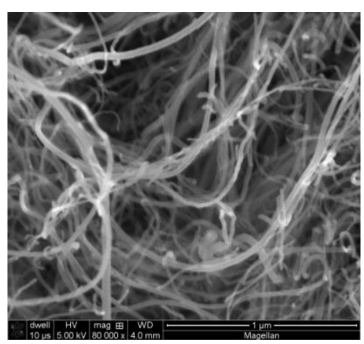
## II. EXPERIMENTAL

### *A. Material System*

The NH2 functionalized-multiwalled carbon nanotubes (MWCNTs) were supplied by Cheaptube. The manufacturer specified dimensions, diameter (d) ~10-20nm and length (l) ~5-10 µm, are confirmed by SEM observation (Fig 1(a)). According to the manufacturer, the MWNTs material has over 97% nanotube content with very small traces of metals as impurities. Unidirectional (UD) glass fiber has been used as reinforcing material in this study.

Unidirectional (UD) glass fibers from Pearl Industries, New Delhi., have been used as reinforcing material. The surface density of the fabric was 220 g/m2 and its thickness is 0.18 mm. Average diameter of the fibers is estimated to 20 µm (Fig 1(b)). Epoxy resin Araldite LY556 and the compatible hardener (HY917) used as the matrix were obtained from Ciba Specialty Chemicals.

(a)



(b)

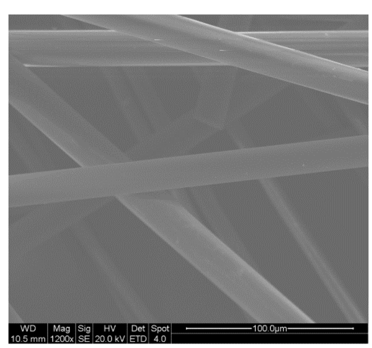


Fig 1. (a) SEM image of MWCNTs and (b) SEM image of glass fiber

*B. Preparation*

*Step 1: Dispersion*

In the first step, the MWCNTs were added into the epoxy in such a way that the final material was 1.0wt% of CNT by wt% in epoxy. Next, this mixture was ultrasonicated by using ultrasonicator as well as stirred using magnetic stirrer for 2 hr at room temperature. After this step, the required amount of hardener was added into the mixture (ratio of 10:9 by weight). It was thoroughly stirred for 15 minutes at room temperature to make a homogeneous mixture.

*Step 2: Curing Behavior*

The cure behavior of epoxy matrix and CNT modified epoxy are characterized by a differential scanning calorimeter (DSC). In the DSC plot, one exothermic peak at 1st run was appeared at 140oC due to the chemical reaction of epoxy and hardener, as shown in Fig 2. This value is found from DSC under nitrogen atmosphere with the heating rate of 5°C/min. The exothermic peak gives a measure of the heat liberated during chemical reaction between epoxy and hardener. In the second run, peak is not appeared, which indicate the completion of curing after 1st run.

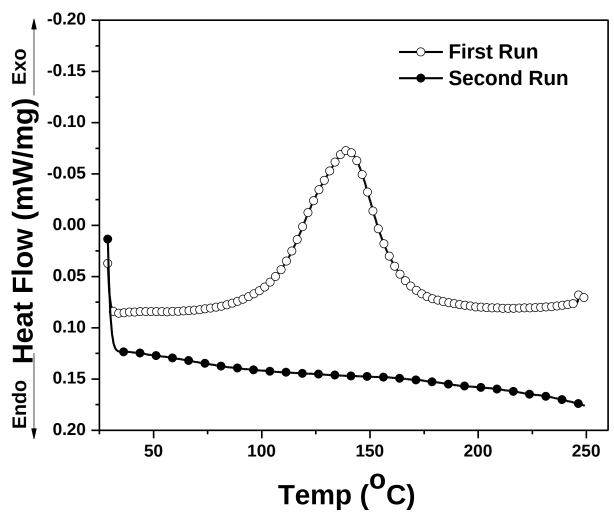
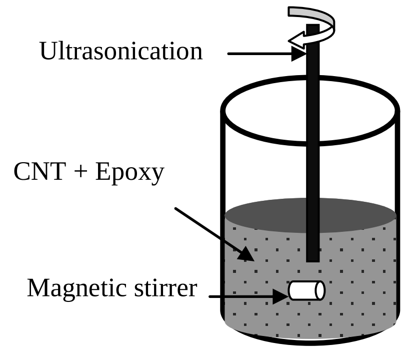


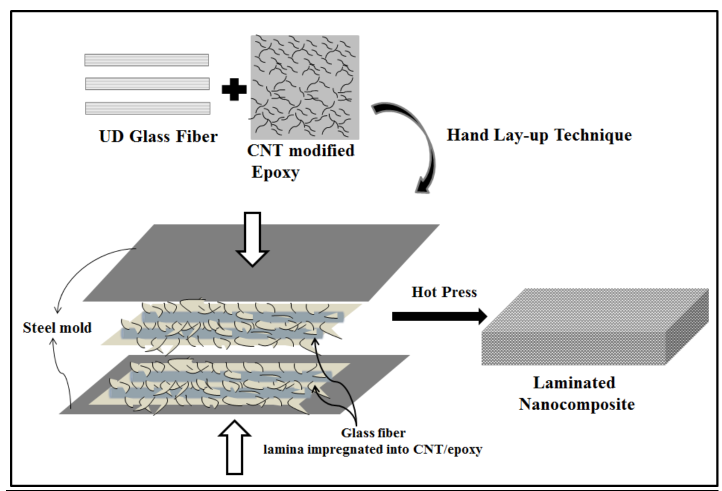

Fig 2.DSC analysis of epoxy resin

*Step 3: Lamination and Curing*

Fig 3 shows the schematic of the preparation of glass fiber reinforced CNT modified epoxy composite. Two different material configurations designated as $GF_E$ and $GF_{CE}$ were considered. Details of material configuration are shown in Table 1. Material $GF_E$ is made of epoxy resin and glass fiber, whereas, material $GF_{CE}$ is made of CNT-epoxy resin mixture and glass fiber with classical processing. The hand lay-up technique was used for the preparation of laminated composite. The epoxy resin and the curing agent were taken in the ratio of 10:9 by wt% in a beaker and mixed thoroughly until a clear homogeneous viscous liquid was obtained. Four layers of fabric were impregnated with epoxy resin and placed in a mold. The laminates were fabricated by using conventional compression molding at a constant pressure of 10 kg/cm2. The mold was kept under a pressure for 4 hrs at 80°C to obtain a cured laminate. The cured sample was removed from the mold and placed in an oven for 8 hrs at 160°C for post curing. Three specimens of each series were prepared.




Step-1




Step-2

Fig3.Schematic of the processing of glass fiber reinforced CNT modified epoxy composite.

| Designation | Materials |
|---|---|
| $GF_E$ | Glass fiber reinforced epoxy composite |
| $GF_{CE}$ | Glass fiber reinforced CNT-epoxy composite |

Table 1.The details of the materials configuration

*C. Characterization of carbon nanotubes and their composites*

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Scanning Electron Microscopy (SEM): The surface morphology of glass fiber, MWCNTs and fractured composite samples were investigated using SEM. The samples were coated with a thin layer of gold to reduce charging during analysis.

Dynamic Mechanical Analysis (DMA): The thermo-mechanical behavior of composite specimens was investigated using a dynamic mechanical analyzer (DMA Pyris Diamond) in the bending mode. Rectangular specimens (50 x 15 x 2mm) were subjected to load-controlled sinusoidal loading (peak load: 5N, frequency: 1Hz, span length: 50 mm) at a heating rate of 3°C/min in the temperature range from 25- 250°C.

Mechanical Test: To investigate the mechanical behavior, tensile tests were performed for all the materials using molded samples per the ASTM/D638-03 type I standard. Straight laminated samples (length: 200 mm, width: 20 mm) were cut from the plate using an automatic diamond saw. All tests were performed using an Instron 8252 universal testing machine with an extension rate of 1 mm/min.

## III. Results and Discussion

The storage modulus (E′) of $GF_E$ and $GF_{CE}$ are presented in Fig4. In this Figure, it has been observed that the $GF_E$ shows a lower storage modulus than $GF_{CE}$ over the entire range of temperatures i.e., 30 to 250°C. The storage modulus of $GF_{CE}$ material at 30°C is 24.4 GPa, whereas the E′ values at 30°C for $GF_E$ is 17.8GPa. Clearly at the room temperature the E′ value of $GF_{CE}$ sample is enhanced by more than 37% when compared to the $GF_E$ sample. The modulus is increased due to the high modulus of CNT. Besides this the improvement is also due to the good interaction between the CNT and glass fiber; CNT and matrix; and glass fiber and matrix.

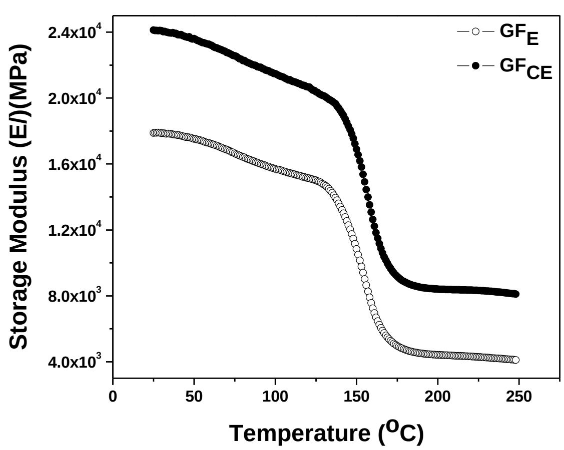



Fig4. Storage modulus of glass fiber epoxy laminated composites with and without MWCNTs

Fig5.presents the Tan δ curves of $GF_E$ and $GF_{CE}$ material. It has been observed that the Tan δ of the $GF_{CE}$ is decreased, when these are compared with the $GF_E$ material. From the Tan δ vs temperature curves, the values of glass transition temperature ($T_g$) of the $GF_{CE}$ is observed at 166.7, whereas the values of $T_g$ of the glass-epoxy is observed at 156°C. The $T_g$ are shifted to the higher temperatures in presence of CNTs, because, the movement of epoxy backbone is restricted by CNT and glass fiber. In Fig 5, the Tan δ values of $GF_E$ and $GF_{CE}$ material at the $T_g$ are 0.0708 and 0.0613, respectively. This lower Tan δ values for $GF_{CE}$ is attributed to the reduction of mobility of the epoxy chains near the glass fiber surface and carbon nanotubes.

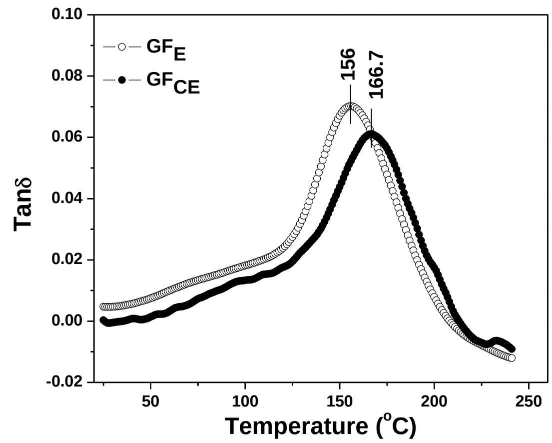



Fig 5. Tanδ of glass fiber epoxy laminated composites with and without MWCNTs

Fig5 shows the stress-strain responses from monotonic tensile tests conducted on the $GF_E$ and $GF_{CE}$ material. The elastic modulus of the composites was evaluated from the initial slope (0.1% strain) of the stress-strain curves of the specimens. The elastic modulus of $GF_{CE}$ sample is higher than that of $GF_E$ sample. The tensile modulus of the $GF_E$ was 14.6GPa. This value is increased by 30% to 19.2GPa for laminates containing 1.0 wt% MWCNTs. The modulus is increased due to the high modulus of MWCNT. In addition to this the improvement is also due to the good interaction between the CNT and matrix and glass fiber and matrix.
From the tensile test results as shown in Fig 6, we obtained similar conclusions despite modulus improvements were smaller than for the DMA results (see Table 2). The displacement levels up to 2.3 x 10-3 m was used in simple tensile test to evaluate Young's modulus, whereas displacement amplitude used in DMA test was 10-8m. It has been confirmed that this divergence is due to the large difference in displacement levels used in simple tensile test in comparison with the DMA test.

| Material | E/(GPa)(30oC) | Tanδ | Tg(oC) | E(tensile test)(GPa) |
|---|---|---|---|---|
| $GF_E$ | 17.8 | 0.0708 | 156 | 14.6 |
| $GF_{CE}$ | 24.4 | 0.0613 | 166.7 | 19.2 |

Table 2. Storage modulus at room temperature, peak value of Tanδ, glass transition temperatures and Young's modulus for the $GF_E$ and $GF_{CE}$ material.

The SEM of the fractured surfaces of the glass fiber reinforced CNT-epoxy composite has been done to investigate the CNT-epoxy interaction and the micro structural changes involved on the incorporation of the CNT into the epoxy matrix. From the micrograph of the fractured surface of the $GF_E$ specimen (Fig 7(a)), it has been observed that the fracture is ductile in nature. There is smoothness in the fractured surface with very little ruggedness.

The SEM micrographs of the $GF_{CE}$ composite reveal that CNTs have the good contact with the glass fiber and matrix, which shows a good interaction among CNTs/epoxy matrix (Fig7 (b)). With introducing CNTs content there is a remarkable difference in the surface morphology of the fractured surface. The smoothness of the fractured surface goes on decreasing in presence of CNTs suggesting a ductile to brittle transformation in the composite material

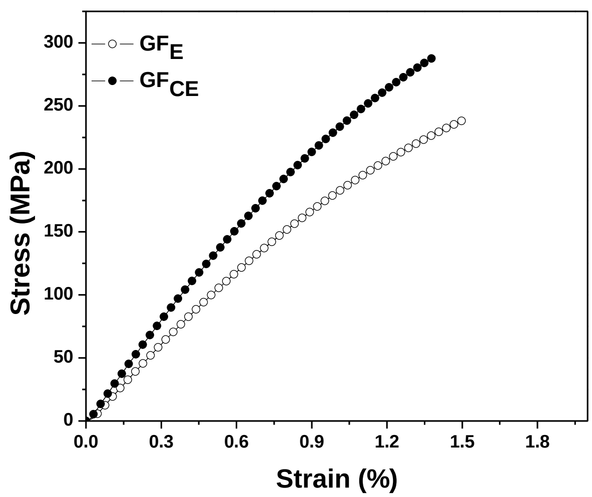
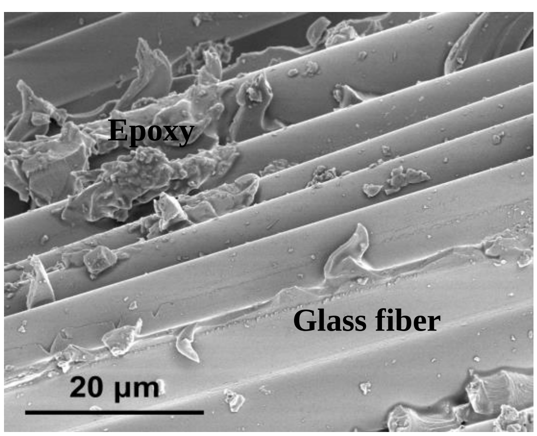



Fig. 6.Tensile stress-strain curves for glass fiber epoxy laminated composites with and without MWCNTs

(a)




(b)

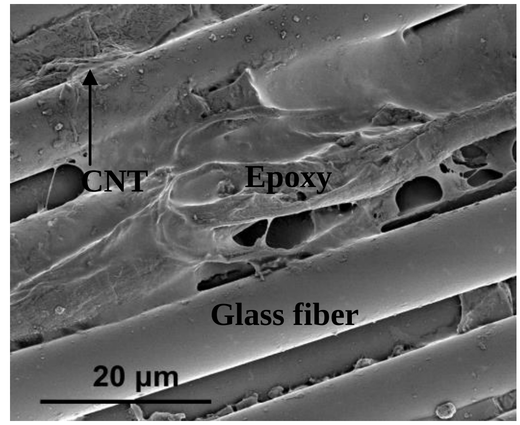



Fig 7. SEM micrographs of fractured samples (a) $GF_E$ and (b) $GF_{CE}$ material

## IV. CONCLUSION

The influence of CNTs in laminates has been studied using DMA and mechanical testing, etc. The dynamic properties of laminated composites reveal that the E′ value of $GF_{CE}$ sample is enhanced by more than 38% when compared to the $GF_E$ sample. The young's modulus and storage modulus of the $GF_{CE}$ sample is enhanced due to the presence of CNTs and CNTs-epoxy and CNTs-glass fiber interaction. The Tg for $GF_{CE}$ is shifted to the higher temperatures, because the movement of epoxy backbone is restricted by CNTs and glass fiber.

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