Tensile behavior of MWCNT enhanced glass fiber reinforced polymeric composites at various crosshead speeds

K K Mahato*, D K Rathore, R K Prusty, K Dutta, B C Ray

Composite Materials Group, Metallurgical and Materials Engineering Department, National Institute of Technology, Rourkela-769008, India

*Email: kishorepce@gmail.com

Abstract. Fiber reinforced polymeric (FRP) composite materials are subjected to different range of crosshead speeds during their in-service life. The work has been focused to investigate the effect of carbon nanotube (CNT) addition in glass fiber reinforced polymer (GFRP) composite on tensile behavior. The Control GFRP composites and CNT modified composites were tested at different crosshead speeds viz. 1, 10, 100 mm/min. CNT modified matrix was processed with epoxy as a matrix materials and multi-walled carbon nanotube (MWCNT) as a filler with different MWCNT content (i.e. 0.1, 0.3 and 0.5 wt. %). Increase in the CNT content upto 0.3% the tensile strength increasing for all the crosshead speeds as compared to the control GFRP composite. The tensile strength are dependent on the CNT content in GFRP composite. It has been observed that addition of 0.1% CNT and 0.3% CNT enhanced the tensile strength by 6.11% and 9.28% respectively than control GFRP composite. The tensile modulus is found to be mostly unaffected on an optimum CNT content in the GFRP composite. The tensile strength of control GFRP and all CNT modified GFRP composites were found to be crosshead speed sensitive and increased with increasing crosshead speeds in the aforesaid loadings. However, slight decrease in tensile modulus was observed with addition of CNT due to agglomeration of the CNT in the polymer matrix composites. The DSC analysis was also carried out to understand the effect of the CNT content on the glass transition temperature (Tg) of GFRP composites. Different failure patterns of GFRP composite tested at 1, 10, and 100 mm/min crosshead speeds were identified.

1. Introduction

In this present era, the demand and usage of FRP composites was increasing in rapid manner. The applications of these composites are not limited to a field, but it is widely spread over to various arena. The field of applications includes automotive, sporting goods, aerospace, structural, cryogenic vessels, solar panels and oil and gas pipelines. Incorporation of nanofillers into the polymer matrix has been an active area of research around the world. Reproduction of the distinct behaviors of nanofillers in the polymer matrix is one of the crucial objective to achieve better mechanical, electrical and/or thermal properties. The usage of CNT as nanofillers is due to its strength (about 22 GPa [1]) and modulus ( about 1 TPa [2]). Various researchers have studied on the mechanical performance of GFRP composites and incorporation of different nanofillers [3–5] at various environments and loading conditions [6–9]. There is a dearth in the literature of tensile behavior of CNT embedded into polymer matrix with crosshead speed variations.
The current experimental study deals with the incorporation of multi-walled carbon nanotubes (CNTs) in epoxy resin that caused improvement in tensile strength over conventional GFRP. In the present study, epoxy of GFRP composite was modified with 0.1 wt. %, 0.3 wt. % and 0.5 wt. % of CNT and laminates were fabricated. Also, the variation in the loading rate with CNT content is evaluated.

2. Materials and Methods

2.1 Testing Materials

The current experimental investigation consists of fabrication of GFRP composite and Diglycidyl Ether of Bisphenol A (DGEBA) based epoxy resin was used as matrix and triethylene tetra amine (TETA) was used as hardener. The woven fabric E-glass fibers used in the experimental program was supplied by Saint Gobin industries. Epoxy resin and hardener were obtained from Atul Industries Ltd, Gujarat having the trade name of Lapox L-12 and K-6 respectively. The epoxy to hardener weight ratio was taken as 10:1 as per the supplier’s recommended standard.

2.2 Preparation of GFRP and CNT enhanced GFRP laminates

The GFRP composite laminated was fabricated with 10 layers of woven fabric E-glass fibers having 60:40 weight fraction of glass fibers and epoxy respectively. The fabrication was done using hand layup method. To ensure appropriate distribution of CNTs in the polymer matrix is the crucial issue to get better mechanical properties. Therefore, the CNTs and polymer suspension were thoroughly mixed and stirred with the help of magnetic stirrer and ultra-sonicator. The control GFRP and CNT-GFRP laminates were cured in hot press compression moulding machine at 60°C temperature for 20 minutes at a pressure of 5 kgf/cm². As per the ASTM D3039 standard the samples were cut using diamond cutter. After that the samples were post cured in an oven at 140°C for 6 hours [10].

2.3 Testing Methods of GFRP composite

2.3.1 Tensile Test

The test was performed with tension fixture in Instron 8862 Universal testing machine (UTM) shown in figure 1(b) in accordance to the ASTM D3039 standard samples to evaluate the tensile properties. The samples were tested at room temperature with different loading rates viz. 1, 10, 100, 500 and 1000 mm/min.

2.3.2 Scanning Electron Microscopy (SEM) Analysis.

| Property                        | Epoxy  | Woven Fabric E- Glass Fibers |
|--------------------------------|--------|-----------------------------|
| Density (g/cm)                 | 1.162  | 2.58                        |
| Tensile Strength (GPa)         | 0.11   | 3.4                         |
| Tensile modulus (GPa)          | 4.1    | 72.3                        |
| Strain at failure (%)          | 4.6    | 4.8                         |
| Areal weight of fabric (g/m²)  | -      | 360                         |

Table 1. Mechanical Properties of E-glass fibers and epoxy
The fractured surfaces of the tested specimens were further analysed using scanning electron microscope (SEM) for determining the various dominating modes failure mechanisms using JEOL-JSM 6480 LVSEM at 20KV.

3. Results and discussion

3.1 Effect of tensile behavior of MWCNT enhanced glass fiber reinforced polymeric composite at various crosshead speeds

The tensile stress-strain curves for control GFRP, 0.1wt% CNT, 0.3wt% CNT and 0.5wt% CNT at different loading rates is shown in figure 1. It is observed that with increase in the CNT content upto 0.3% the tensile strength increasing (figure 1) for all the loading rates as compared to the control GFRP composite. Further, addition of CNTs causes decrease in the strength value and it may be attributed to the agglomeration of CNTs in the polymer matrix, where due to poor stress transfer across the fiber/matrix interface.

![Stress–strain curves for GFRP composites at various MWCNT contents at room temperature with different loading rate](image)

**Figure 1.** Stress–strain curves for GFRP composites at various MWCNT contents at room temperature with different loading rate (a) 1 mm/min, (b) 10 mm/min and (c) 100 mm/min.

The tensile properties i.e. tensile strength and tensile modulus obtained from figure 1 were plotted against various CNT content for GFRP composite in figure 2 as revealed below. It is evident from figure 2 that the tensile strength and tensile modulus are strongly dependent on the CNT content for GFRP composite. Incorporation of 0.1% CNT and 0.3% CNT resulted in enhancement in tensile properties at room temperature than control GFRP composite as revealed from figure 2(a). It has been observed that addition of 0.1% CNT and 0.3% CNT enhanced the tensile strength by 6.11% and
9.28% respectively than control GFRP composite. The reason can be attributed to very high specific surface area of CNTs, which in-turn exhibits very high interfacial area in the final composite. Hence the stress transfer ability across the interface is easily facilitated, as a result higher strength in case of CNT/GFRP composite is observed than control GFRP composite at room temperature environment.

![Figure 2](image)

**Figure 2.** Variation in (a) Tensile strength and (b) Tensile modulus with CNT content in GFRP composite at various crosshead speeds.

But in the meantime drop in tensile modulus is observed with addition of CNT and the reason may be attributed to agglomeration of the CNT in the polymer matrix. Agglomeration causes poor wettability in the fiber/matrix interface resulting in weaker adhesion in the interfacial bonding between the CNT enhanced fiber/matrix composite. It is observed from figure 2 (a) that with increase in CNT content upto 0.3 wt% with loading rate, the tensile strength of the GFRP and CNT/GFRP composite is increasing. Generally, thermal stresses induces micro-cracks in the polymer matrix and/or, at the fiber/matrix interface may possibly grow without blunting at a stable state. Some of these microcracks turn up potential cracks at low loading rates and causes substantial reduction in tensile stress of the composite system. But, at the higher loading rates, the time availability to propagate the microcracks is very less. The modulus value represented in figure 2 (b) shows no significant variation in the tensile modulus for control GFRP and CNT-GFRP composites at all the crossheads speeds. In the meantime slight drop in tensile modulus is observed with addition of CNT due to agglomeration of the CNT in the polymer matrix. But, with respect to loading rate increase in modulus value was observed in case of 10 mm/min crosshead speeds at all the CNT content. This may be attributed to proper adhesion of fiber/matrix/CNT in the composite and propagation of cracks at 10mm/min is neither very fast nor very slow constitutes proper stress transfer from matrix to the fiber.

### 3.2 Different failure patterns of GFRP composite

Figure 3 shows the bulk pictures of GFRP composites with different failure pattern tested at (a) 1mm/min, (b) 10 mm/min (c) 100 mm/min loading rates. At lower loading rates the initiation of crack generates at the middle portion of the specimens. But at higher loading rates multiple numbers of cracks are generating throughout the samples i.e. at middle portions as well as at the tab portions.
Figure 3. Figure: Different failure patterns of GFRP composite and CNT embedded GFRP composite tested at (a) 1mm/min, (b) 10 mm/min (c) 100 mm/min crosshead speeds.

3.3 Temperature modulated differential Scanning Calorimetry (TMDSC) Measurement

The glass transition temperature ($T_g$) of the matrix phase of the composites were analyzed using TMDSC. Figure 4 indicates the TMDSC curves for GFRP composite at RT, 0.1% CNT, 0.3% CNT and 0.5% CNT-GFRP content. The control GFRP composites indicates $T_g$ about 95.32 °C, whereas the CNT-GFRP at 0.1, 0.3 and 0.5 wt. % of CNT exhibits 114.23 °C, 113.55 °C and 112.91 °C respectively. This indicates addition of CNT into GFRP composites significantly enhances the $T_g$. This may be attributed to the proper distribution of CNTs in the matrix resulting rise in the $T_g$.

Figure 4. Temperature modulated differential scanning calorimetry (TMDSC) plot for GFRP composites at RT, 0.1% CNT, 0.3% CNT and 0.5% CNT

3.4 Fractography analysis

After, the tensile testing post failure analysis were done using nano nova FESEM (field emission scanning electron microscope) to examine the dominant mode of failures in the composites. Figure 5(a) and 5(b) indicates SEM images of 0.1% CNT/GFRP and 0.3% CNT/GFRP composites
respectively. It is evident from figure 5(a) that dispersion of MWCNTs in epoxy matrix was better in case of 0.1% CNT/GFRP composite as compared to 0.5% CNT/GFRP composites [figure 5(b)]. Good dispersion of MWCNTs imparts proper stress transfer from the polymer matrix to fiber. But, when the MWCNTs particles start to agglomerate at a particular region in the polymer matrix, the stress transfer across the composites is not uniform and hence drop in tensile modulus was observed with increasing percentage of MWCNTs in the composites.

**Figure 5.** FESEM images of (a) 0.1% CNT/GFRP and (b) 0.5% CNT/GFRP composites.

4. Conclusions

The present investigation comprises on the tensile response of the CNT addition with GFRP composites and further tested at various crosshead speeds includes the following conclusions

- Increase in the CNT content up to 0.3% the tensile strength increasing for all the crosshead speeds as compared to the control GFRP composite.
- Addition of 0.1% CNT and 0.3% CNT enhanced the tensile strength by 6.11% and 9.28% respectively than control GFRP composite.
- Decrease in tensile modulus was observed with addition of CNT due to agglomeration of the CNT in the polymer matrix composites.
- The glass transition temperature (Tg) of CNT embedded composites are found to be more as compared control GFRP.

Acknowledgement

The authors are heartily expresses their deep gratitude to NIT Rourkela for providing the necessary infrastructural and equipment facility.

References

[1] Li F, Cheng H M, Bai S, Su G and Dresselhaus M S 2000 Tensile strength of single-walled carbon nanotubes directly measured from their macroscopic ropes Appl. Phys. Lett. 77 3161–3

[2] Salvetat J-P, Briggs G A D, Bonard J-M, Bacsa R R, Kulik A J, Stöckli T, Burnham N A and Forró L 1999 Elastic and Shear Moduli of Single-Walled Carbon Nanotube Ropes Phys. Rev. Lett. 82 944–7
[3] Nayak R K, Mahato K K and Ray B C 2016 Water absorption behavior, mechanical and thermal properties of nano TiO2 enhanced glass fiber reinforced polymer composites *Compos. Part Appl. Sci. Manuf.* **90** 736–47

[4] Nayak R K, Mahato K K, Routara B C and Ray B C 2016 Evaluation of mechanical properties of Al2O3 and TiO2 nano filled enhanced glass fiber reinforced polymer composites *J. Appl. Polym. Sci.* **133** n/a–n/a

[5] Kathi J, Rhee K-Y and Lee J H 2009 Effect of chemical functionalization of multi-walled carbon nanotubes with 3-aminopropyltriethoxysilane on mechanical and morphological properties of epoxy nanocomposites *Compos. Part Appl. Sci. Manuf.* **40** 800–9

[6] Mahato K K, Biswal M, Rathore D K, Prusty R K, Dutta K and Ray B C 2016 Effect of loading rate on tensile properties and failure behavior of glass fiber/epoxy composite *IOP Conf. Ser. Mater. Sci. Eng.* **115** 12017

[7] Mahato K K, Rathore D K, Dutta K and Ray B C 2017 Effect of loading rates of severely thermal-shocked glass fiber/epoxy composites *Compos. Commun.* **3** 7–10

[8] Ray B C 2006 Loading Rate Sensitivity of Glass Fiber–Epoxy Composite at Ambient and Sub-ambient Temperatures *J. Reinf. Plast. Compos.* **25** 329–33

[9] Ray B C 2004 Effects of crosshead velocity and sub-zero temperature on mechanical behaviour of hygrothermally conditioned glass fiber reinforced epoxy composites *Mater. Sci. Eng. A* **379** 39–44

[10] Kumar D S, Shukla M J, Mahato K K, Rathore D K, Prusty R K and Ray B C 2015 Effect of post-curing on thermal and mechanical behavior of GFRP composites *IOP Conf. Ser. Mater. Sci. Eng.* **75** 12012