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INTERNATIONAL JOURNAL OF RESEARCH IN AERONAUTICAL AND MECHANICAL ENGINEERING
WWW.IJRAME.COM
ISSN (ONLINE): 2321-3051

Vol.7 Issue 8,
August 2019
Pg: -13-23

EXPERIMENTAL INVESTIGATION ON FATIGUE BEHAVIOR OF GLASS FIBER REINFORCED COMPOSITE WITH NANO PARTICLES

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ABSTRACT: ***In this paper presents the glass fiber reinforced polymers (GFRP) have become a preferable material for reinforcing or strengthening reinforced concrete structures due to their corrosion resistance, high strength to weight ratio, and relatively low cost compared with carbon fiber reinforced polymers (CFRP). However, the limited fatigue life of GFRP hinders their use in infrastructure applications. For instance, the low fatigue life of GFRP caused design codes to impose stringent stress limits on GFRP that rendered their use non-economic under significant cyclic loads in bridges. In this paper, we demonstrate that the fatigue life of GFRP can be significantly improved by an order of magnitude by incorporating Multi-Wall Carbon Nanotubes (MWCNTs) during GFRP fabrication. GFRP coupons were fabricated and tested under static tension and cyclic tension with mean fatigue stress equal to 40% of the GFRP tensile strength. Microstructural investigations using scanning electron microscopy (SEM) and Fourier Transform Infrared (FTIR) spectroscopy were used for further investigation of the effect of MWCNTs on the GFRP composite. The experimental results show the 0.5 wt% and the 1.0 wt% MWCNTs were able to improve the fatigue life of GFRP by 114.3% and 98.6%, respectively, compared with neat GFRP.***

1. INTRODUCTION

The development of composite materials and related design and manufacturing technologies is one of the most important advances in the history of materials. Composites are multifunctional materials having unprecedented mechanical and physical properties that can be be tailored to meet the requirements of a particular application. Many composites also exhibit great resistance to high-temperature corrosion and oxidation and wear. These unique characteristics provide the mechanical engineer with design opportunities not possible with conventional monolithic materials. Composites technology also makes possible the use of an entire class of solid materials, ceramics, in applications for which monolithic versions are unsuited because of their great strength scatter and poor resistance to mechanical and thermal shock. Further, many manufacturing processes for composites are well adapted to the

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ISSN (ONLINE): 2321-3051

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fabrication of large, complex structures, which allows consolidation of parts, reducing manufacturing costs.

Polymer composite materials have generated wide interest in various engineering fields, particularly in aerospace applications. Research is underway worldwide to develop newer composites with varied combinations of fibers and fillers so as to make them useable under different operational conditions. The improved performance of polymer composites in engineering applications by the addition of filler materials has shown a great promise and so has become a subject of considerable interest. Ceramic filled polymer composites have been the subject of extensive research in last two decades [1]. Various kinds of polymers and polymer matrix composites reinforced with ceramic particles have a wide range of industrial applications such as heaters, electrodes [2], composites with thermal durability at high temperature [3] etc. These engineering composites are desired due to their low density, high corrosion resistance, ease of fabrication, and low cost [4-6].Hard particulate fillers consisting of ceramic or metal particles and fiber fillers made of glass are being used these days to dramatically improve the wear resistance even up to three orders of magnitude [7]. The inclusion of inorganic fillers into polymers for commercial applications is primarily aimed at the cost reduction and stiffness improvement [8, 9]. Along with fiber-reinforced composites, the composites made with particulate fillers have been found to perform well in many real operational conditions. It is reported by Bonner [10] that with the inclusion of micro-sized particulates into polymers, a high filler content (typically greater than 20 vol. %) is generally required to bring the above stated positive effects into play. But at the same time, this may also have detrimental effects on some important properties of the matrix polymers.

2. MATERIALS AND METHODS

This chapter describes the details of processing of the composites and the experimental procedures followed for their characterization and tribological evaluation. The raw materials used in this work are: E-glass fiber, Carbon Nano Tubes, Epoxy resin.

2.1. Processing of the Composites

2.1.1. Specimen preparation

E-glass fibers (360 roving taken from Saint Gobian) are reinforced with Epoxy LY 556 resin, chemically belonging to the ‘epoxide’ family is used as the matrix material. Its common name is Bisphenol A Diglycidyl Ether. The low temperature curing epoxy resin (Araldite-LY 556) and corresponding hardener (HY951) are mixed in a ratio of 10:1 by weight as recommended. The epoxy resin and the hardener are supplied by Ciba Geigy India Ltd.

E-glass fiber and epoxy resin has modulus of 72.5 GPA and 3.42GPa respectively and possess density of 2590 kg/m3 and 1100kg/m3 respectively. The filler material MWCNTs/Graphene) are provided by NICE Ltd India sieved to obtain particle size in the range

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Pg: -13-23

70-90 µm. Composites of three different compositions such as 30wt%, 40wt% and 50wt% glass fiber are made and the filler content (weight fraction of TiO2 in the composite) is kept at 10% for all the samples The castings are put under load for about 24 hours for proper curing at room temperature. Specimens of suitable dimension are cut using a diamond cutter for physical characterization of specimens before testing as shown in Fig.2.1 and universal hydraulic fatigue testing machine as shown in Fig.2.2.

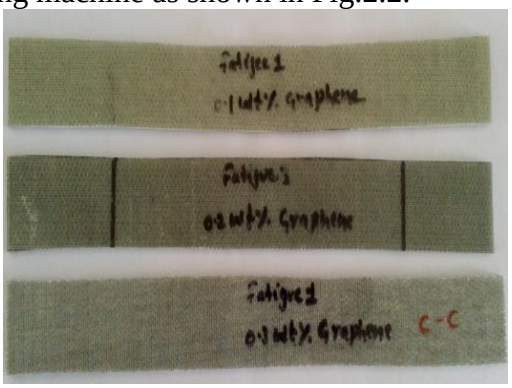


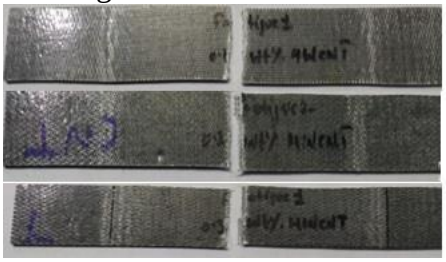
Fig.2.1 Composite materials

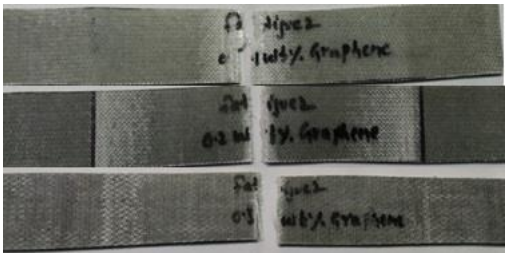
Fig.2.2 Universal Hydraulic Fatigue Testing machine

3. RESULTS AND DISCUSSION

3.1 Tensile Strength

GFRP composite coupons were tested under static tension loads up to failure. The tensile strength for the individual specimen is presented in Table 1. Median stress-strain curves for the GFRP materials with different MWCNTs content are shown in Fig.2.3 (a), (b) &(c). It can be observed from Fig. 2.3a that incorporating MWCNTs had no effect on the tension modulus of elasticity of GFRP composites at low strain level. This can be attributed to the fact that the stiffness of glass fibers dominates the elastic response of GFRP for being much higher stiffness compared with the epoxy matrix. However, at high strain levels it was observed that GFRP composites incorporating MWCNTs are stiffer than the neat ones. The increase of stiffness associated with the increase in strain is attributed to the effect of fiber straightening during test. In a vacuum assisted hand layup fabrication technique, the fibers would not be fully stretched during the fabrication of the GFRP composites process.





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(a) (b)

(c)

Fig.2.3 After testing of sampled specimens

The fiber stretching. Thus, more stiffening was observed in the case of GFRP incorporating 0.2wt% and 0.2 wt% MWCNTs/Graphene-epoxy Nano composites than that observed in neat GFRP composites. On the other hand, the mean tensile strength for the five coupons in each GFRP composite group was found to be 703 ± 55 MPa, 904 ± 107 MPa, and 714 ± 117 MPa for the GFRP composites incorporating 0 wt. % (neat), 0.2 wt. % MWCNTs, 0.2 wt. % Graphene and wt. % MWCNTs/Graphene respectively. The results show the tensile strength of the GFRP composites increased by 28% for 0.2 wt. % MWCNTs-epoxy Nano composite matrix. However, very limited increase of only 2% was observed for 0.2wt. % MWCNTs/Graphene -epoxy Nano composite matrix. A bar chart for comparison is presented in Fig. 2.3(c).

Table 1. Tensile strength (MPa) for individual Glass Fiber Reinforced Polymer (GFRP) coupons.

Specimen number	Neat	0.2wt% MWCNTs	0.2wt% Graphene	0.2wt% MWCNTs/Graphene
1	690	891	785.5	680
2	783	763	679	595
3	729	1058	983.5	909
4	657	931	815	699
5	650	876	782	688
Mean	703	904	809	714

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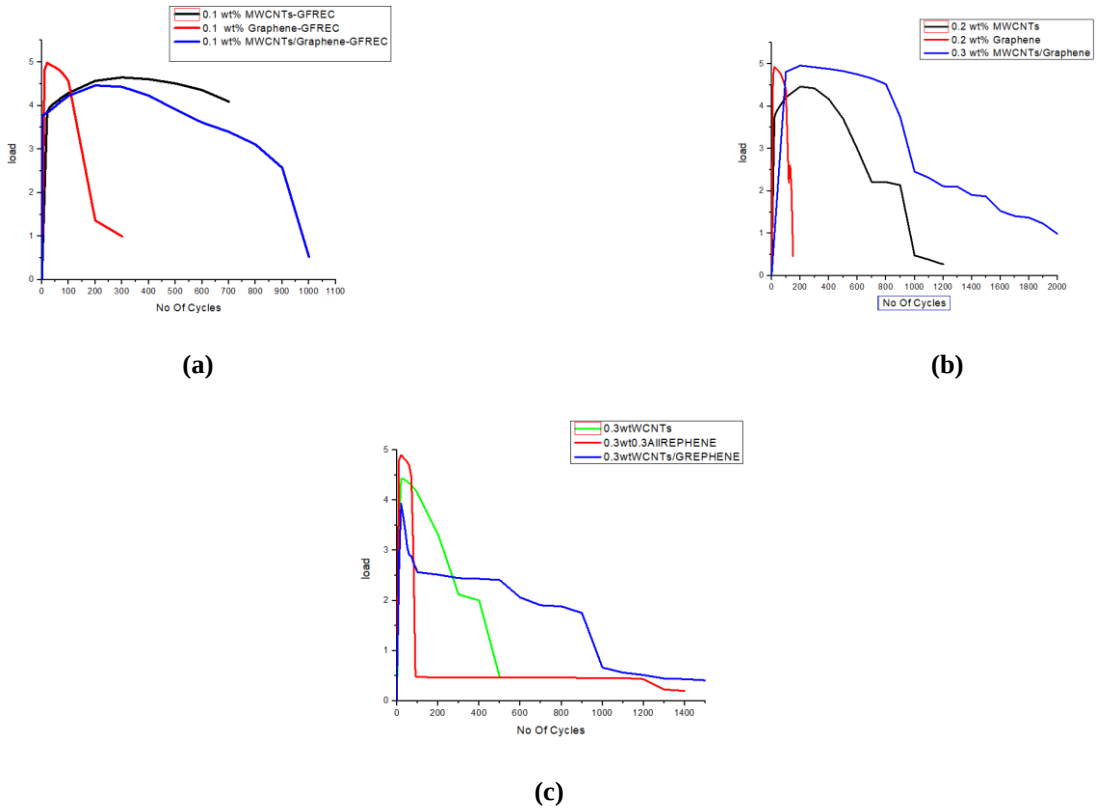

Fig 2.4 (**a**) Example median load-no.of cycles curves for the neat, (a)0.1-0.3%, MWCNTs,(b) 0.1-0.3%, Graphene and (c) 0.1-0.3% MWCNTs/Graphene under static tension load

Statistical analysis using student *t*-test with 95% level of confidence shows that the increase in the tensile strength of the GFRP composites incorporating 0.2 wt% MWCNTs was statistically significant. On the other hand, the difference in the tensile strength between the neat GFRP and GFRP coupons incorporating 0.1 wt% MWCNTs was statistically insignificant. The failure modes of GFRP composites incorporating different MWCNTs content are shown in Fig 2.5 It is obvious that incorporating MWCNTs in epoxy matrix alters the failure mode of the GFRP composites. A typical broom-like failure of the GFRP composites was observed in the coupons fabricated using neat epoxy due to the weak Interfacial bond between glass fibers and the epoxy matrix as reported by others [11–12]. However, the figure shows that incorporating 0.2 wt.% and 0.2 wt% MWCNTs/Graphene significantly limited the broom-like failure of the GFRP composites giving a sign of enhancing the cohesive strength of the epoxy Nano composite matrix.

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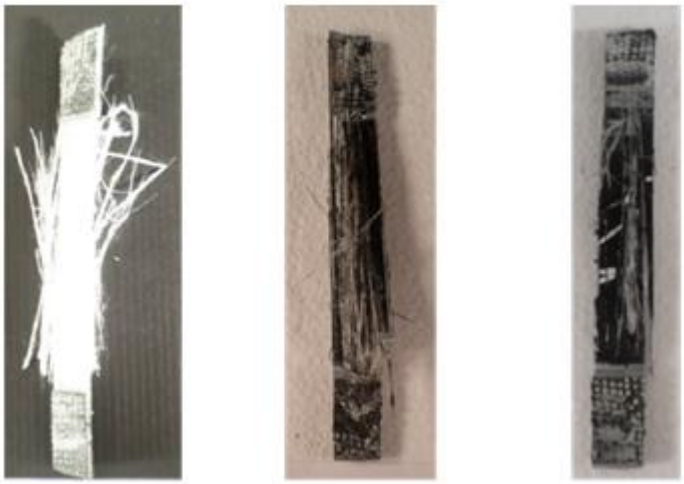

(a) (b) (c)

Fig.2.5. Failure modes of the GFRP composites including (a) 0% (b) 0.2wt% MWCNTs and (c) 0.2wt% Graphene

Moreover, the strain at failure for the GFRP composites incorporating 0 wt% (neat), 0.2 MWCNTs, 0.2 wt% Graphene and 0.2wt% MWCNTs/Graphene was found to be 11.3%, 12.8%, and 10.7% respectively. The results show that the ductility was increased by 13% for the GFRP composites incorporating 0.2 wt% MWCNTs-epoxy Nano composites and decreased by 5% for the GFRP composites incorporating 0.3 wt% MWCNTs-epoxy Nano composites. Statistical analysis using student *t*-test with 95% level of confidence shows that difference in the ductility for the GFRP composites incorporating MWCNTs-epoxy Nano composites were statistically significant. It is apparent from Fig 2.5, that incorporating 0.2 wt% MWCNTs, 0.2 wt% Graphene ,in epoxy was able to significantly increase the mean fatigue life of GFRP from 87.51 ± 5.6 k (neat) to 1.09 ± 0.62 M (0.2 wt% MWCNTs) and 0.95 ± 0.27 M (0.2 wt% MWCNTs/Graphene) representing an increase of 114.3% and 98.6%, respectively, as shown in Figure 6. The improvement of fatigue life of GFRP is exceptional, reaching one order of magnitude. Although the variability of the fatigue life of GFRP coupons was relatively high, the minimum fatigue life achieved by incorporating MWCNTs in the GFRP composites was 530 k cycles. Significant scatter in fatigue life of composites was reported by other researchers and is attributed to heterogeneous nature, higher sensitivity to batch variability, and complex failure modes of composite materials [13]. This represents a minimum fatigue life improvement of 500%. Table 2 shows the fatigue life of individual GFRP composite coupons. Statistical student *t*-test was conducted and showed the increase of the fatigue life for both the 0.2 wt% MWCNTs and 0.2 wt% Graphene to be statistically significant with a 95% level of confidence. However, the

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WWW.IJRAME.COM
ISSN (ONLINE): 2321-3051

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Pg: -13-23

difference between the 0.3% and the 0.3% MWCNTs/Graphene was statistically not significant.

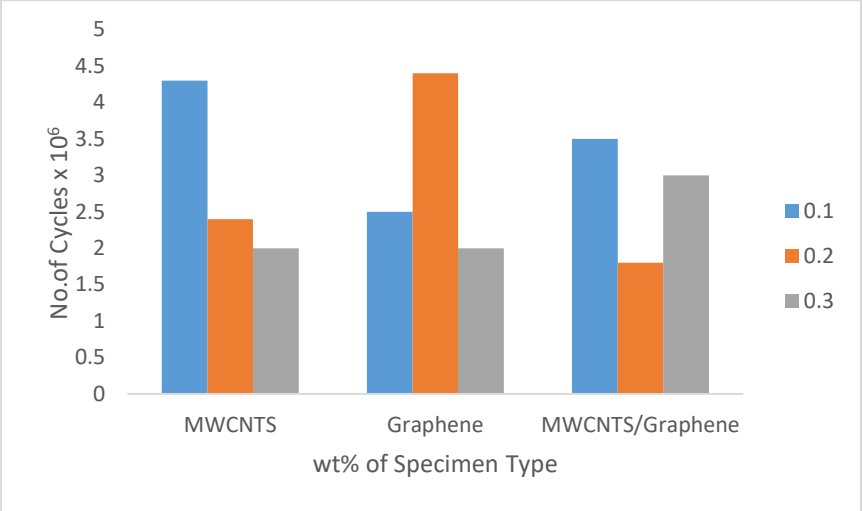

Fig.2 6. Number of cycles in million until failure for the neat, 0.1-0.3wt% MWCNTs, 0.1-0.3wt% Graphene and 0.1-0.3wt% MWCNTs/Graphene. % presented represents the improvement of the number of cycles to failure of GFRP composites compared to reference (neat).

Table 2. Fatigue life (Number of cycles until failure) for individual GFRP coupons.

	Fatigue life (number of cycles until failure)			
Specimen Number	**Neat**	**0.2wt% MWCNTs**	**0.2wt% Graphene**	**0.2wt% MWCNTs/Graphene**
1	85,138	531,006	584,472	637,398
2	92,111	2,082,378	1,392,607.5	702,837
3	79,543	585,078	855,039	1,125,000
4	87,472	1,094,855	1,170,592.5	1,246,330
5	93,301	1,144,095	1,093,047.5	1,042,000
Mean	87,513	1,087,482	1,019,097.5	950,713

The above observations on both the static and cyclic tensile strength of GFRP incorporating MWCNTs can be explained by our hypothesis that COOH functionalized MWCNTs react chemically with the epoxy and produce a new MWCNTs-epoxy Nano composite that has a higher bond (adhesion) strength with glass fibers than neat epoxy and a higher shear (cohesive) strength of the MWCNTs-epoxy Nano composite compared with neat epoxy. The improvement of the tensile strength of GFRP by 28% of GFRP incorporating 0.2wt%MWCNTs is attributed to the

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ISSN (ONLINE): 2321-3051

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enhancement in the adhesion/bond strength between the fibers and the matrix. It is well established that tension failure of GFRP is initiated by adhesion failure of glass fibers and epoxy [14]. The inability of the 0.3wt% MWCNTs to improve the GFRP tensile strength can be explained by the significant effect of increasing MWCNTs content on the viscosity of epoxy, which in turn resulted in an observable increase in epoxy viscosity. While this viscosity increase might do little to affect the chemical reaction between epoxy and MWCNTs within the Nano composite, as it takes place during the sonication and mechanical stirring processes, it would significantly hinder the ability of epoxy to impregnate the glass fibers cloth during the fabrication process. It is possible that the use of 1.0 wt% MWCNTs improves the cohesion strength of the matrix but does not improve the adhesion strength of the matrix with glass fibers.

3.2 Microstructure Characterization

Fig.3 (a)-(j) shows the dispersion of the 0.1-0.3wt% MWCNTs, 0.1-0.3wt% Graphene and 0.1-0.3wt% MWCNTs/Graphene in the epoxy Nano composites. It is apparent from Fig 3(a)-(c) shows that the 0.1-0.3wt% MWCNTs are well dispersed as single individual nanotubes and not bundled together. This good dispersion enables the suggested chemical reaction to take place and also allows MWCNTs to work as fibers bridging the Nano and micro cracks and therefore delaying fatigue crack propagation in the epoxy matrix. A close look at the FTIR spectrum where both peaks appear is shown in Figure 3(d)-(f). It becomes obvious from Figure 3(f) that these peaks are all missing in the neat epoxy. The strong appearance of the two peaks with epoxy 0.2wt% MWCNTs compared with epoxy incorporating 0.2 wt% Graphene is attributed to the higher quantity of the COOH in the former compared with the latter. We also note in Figure 3(g)-(i) shows that other classical peaks of epoxy at 3350 cm^{-1} appearing in all three materials with and without MWCNTs/Graphene. The above FTIR observations confirm our hypothesis that the addition of COOH functionalized MWCNTs to amine-based epoxy resulted in chemical reaction and the formation of a new epoxy-MWCNTs Nano composite with improved mechanical properties, including adhesion with glass fibers and cohesion strength of GFRP matrix. Such chemical reaction between the COOH functionalized MWCNTs and epoxy were reported to improve the dispersion of the MWCNTs in the epoxy and enhance the mechanical properties of the MWCNTs-epoxy Nano composites significantly [15-16].

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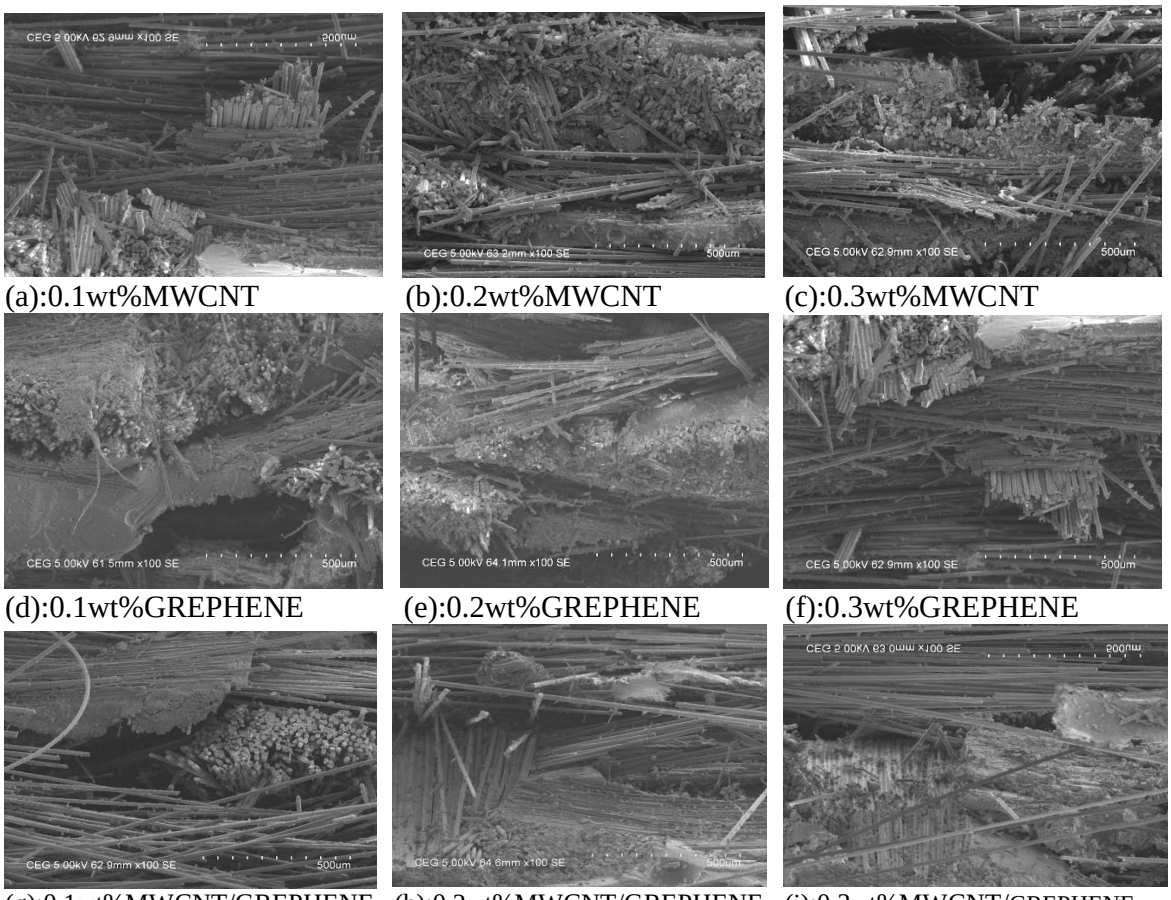

(a):0.1wt%MWCNT (b):0.2wt%MWCNT (c):0.3wt%MWCNT

(d):0.1wt%GREPHENE (e):0.2wt%GREPHENE (f):0.3wt%GREPHENE

(g):0.1wt%MWCNT/GREPHENE (h):0.2wt%MWCNT/GREPHENE (i):0.3wt%MWCNT/GREPHENE

Figure 3. SEM images showing dispersion of the **(a)-(c):** 0.1-0.3 wt% MWCNTs; (**d)-(f)** 0.1-0.3 wt% Graphene and **(g)-(i)**: 0.1-0.3 wt% MWCNTs/Graphene in epoxy.

Our investigation showed that MWCNTs-epoxy Nano composite could significantly improve the fatigue life of GFRP composites such that fatigue design limitations can be eliminated. While the above investigation has limitations represented by (1) the method of GFRP fabrication which would typically introduce relatively high variability; (2) the limited number of layers in fabricating GFRP; and (3) the relatively high mean stress of cyclic loading applied, the above study still sheds light on the potential of using MWCNTs in improving the fatigue performance of GFRP. The improvement in the fatigue life was remarkable reaching one order of magnitude allowing GFRP to pass the one million-cycle mark compared with GFRP incorporating neat epoxy that did not reach 100,000 cycles. The ability of MWCNTs to improve the tensile strength and the fatigue life of GFRP in a tension-tension fatigue test proves the well dispersion of MWCNTs along the GFRP length and across the thickness. This is attributed to the fact that fatigue failure under tension is dominated by a

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single critical crack [17]. The ability to improve the tensile strength and fatigue life of GFRP means MWCNTs limited crack propagation of that critical crack within the whole coupon. This further proves the well dispersion of MWCNTs within the whole coupon. Furthermore, microstructural investigations proved the good dispersion of MWCNTs in the epoxy matrix and provided evidence of chemical reaction of the functionalized MWCNTs with epoxy. Moreover, with the percolation threshold for MWCNTs of 0.2 wt%, the dispersion and damage evolution can be assessed using electrical measurements as presented by the authors elsewhere [18]. In general, it is evident that GFRP without the current fatigue limitations can be produced using MWCNTs. The fact that MWCNTs are added at a very low quantity (0.2 wt% of the weight of epoxy) makes insignificant addition to GFRP manufacturing cost.

4. CONCLUSIONS

Incorporating 0.2 wt% MWCNTs in epoxy improved the tensile strength and fatigue life of the GFRP coupons by 28% and 1143%, respectively. Tensile strength and fatigue life of GFRP composites incorporating 1.0 wt% MWCNTs were improved by 2% and 986%, respectively. Statistical analysis confirmed that the improvements in fatigue life using 0.2 wt% and 0.2 wt% MWCNTs and Graphene were significant. The improvement of tensile strength and fatigue life of GFRP composites incorporating MWNCTs was associated with absence of the classical broom-like failure mode.

We hypothesize that the significant improvement in tensile strength and fatigue life of GFRP incorporating COOH functionalized MWCNTs is attributed to the chemical reaction between the COOH group and epoxy forming a new epoxy-MWCNTs Nano composite with improved adhesion strength with glass fibers and cohesion strength within the epoxy matrix. However, increasing MWCNTs content increases epoxy viscosity, which would hinder epoxy impregnation during GFRP composite fabrication and thus affects epoxy bond with glass fibers. The non-significant effect of 1.0 wt% on tensile strength of GFRP compared with 0.2 wt% of MWCNTs is attributed to the fact that the tensile strength of GFRP is significantly governed by adhesion of epoxy and glass fibers. The ability of both 0.2 wt% and 0.2 wt% MWCNTs and Graphene contents to improve fatigue life of GFRP is attributed to the fact that fatigue of GFRP is more governed by matrix cohesion that controls fatigue crack propagation and is significantly improved with the addition of MWCNTs. As GFRP incorporating MWCNTs remarkably passed the one million-cycle mark at a relatively high mean stress, it is concluded that a new GFRP without fatigue limitations for infrastructure applications can be produced using MWCNTs. More work is needed to investigate the effect of MWCNTs on the fatigue strength of GFRP by examining fatigue life at different stress levels. Moreover, additional CNTs contents between 0.2 wt% and 0.3 wt% need to be investigated to achieve the optimum CNTs content.

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