Effect of MWCNTs on static, dynamic, and wear performance of Vacuum Assisted Resin Infusion Molding (VARIM) processed glass fabric/epoxy polymer composites

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Abstract: In the present work, MWCNTs were used in wt.% of 0.075, 0.15, and 0.3 in epoxy resin, and then glass fabric/epoxy polymer (GF/EP) composites were fabricated using VARIM setup. The mechanical and wear properties of GF/EP composites were compared with and without the addition of MWCNTs. Maximum improvement in tensile and flexural performance were seen in GF/EP composites at 0.15 weight percent MWCNT addition when compared to a neat GF/EP one, based on static testing such as tensile and flexural tests. High-cycle fatigue test results show a relatively higher cycle count with 0.15 wt.% MWCNTs addition in the GF/EP composite compared to the GF/EP without the addition of MWCNTs. The wear performance also improved with a lower friction coefficient as well as a lower track depth and width for MWCNTs with added GF/EP than plain GF/EP composite. Therefore, in addition to decreasing surface softening and improving the wear performance of glass fabric epoxy composites, MWCNTs (0.15 weight percent) increased the interfacial adhesion at the fiber/matrix interface. This study establishes an optimum ratio of 0.15 wt.% of MWCNTs addition in glass fabric/epoxy polymer composites for enhancement in their mechanical and wear performance.

Keywords: Glass fabric; Epoxy, Multiwalled carbon nanotubes (MWCNTs); Tensile; fatigue; wear.

1. INTRODUCTION

Glass fabric-epoxy (GF/EP) composites have wide applications in aerospace, wind turbine blades, and other industrial products due to their excellent mechanical performance and lower weight (Shivamurthy *et al.*, 2020; Ding *et al.*, 2018; Chen *et al.*, 2014). Despite this, failures of GF/EP composites are reported in harsh environments (Tomasz *et al.*, 2012; Huang *et al.*, 2023; Bai *et al.*, 2014). Poor tribological performance of GF/EP composites is another major concern that limits their applications (Barrena *et al.*, 2014; Upadhyay *et al.*, 2019). The mechanical and wear performance of GF/EP composites can be improved by the addition of carbon nanotubes (MWCNTs). MWCNTs exhibit excellent mechanical properties, such as higher tensile and bending strengths, as well as improved anti-friction and anti-wear properties (Rathore *et al.*, 2016). Some of the previous works reported the addition of MWCNTs to glass-fiber epoxy composites. The interfacial shear strength of glass fiber/epoxy composites modified

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with carbon nanotubes were investigated (Godara *et al.*, 2010). The mechanical properties of glass fiber/carbon nanotubes/epoxy hybrid composites were studied. Out of different weight percentages of MWCNTs, such as 0.1, 0.5, and 1 wt.% epoxy composites, the highest improvement in mechanical properties such as tensile strength, Youngs modulus, and toughness were observed with 0.5 wt.% MWCNTs (Dehrooyeh *et al.*, 2021). The effect of MoO_3/carbon nanotubes on the friction and wear characteristics of glass fabric epoxy composites under dry sliding conditions was studied (Yuxin *et al.*, 2020). The results depicted improved wear performance of MoO_3-CNT hybrid material/GF/EP polymer by around four times compared to GF/EP composites. The flexural behavior of glass/epoxy and MWCNT/glass/epoxy (0.3 wt.% of MWCNT) at different in-service environment temperatures were assessed (Prusthy *et al.*, 2015). The open-hole tensile strength of unidirectional glass fiber-reinforced plastic (UD-GFRP) laminates relative to MW-CNT-infused UD-GFRP composites (Pothnis *et al.*, 2021). The presence of MW-CNTs increased the tensile strength by approximately 27% compared to neat UD-GFRP. Several other works reported improvement in the properties of composites such as silicon rubber nanocomposites with the addition of nanoparticles (Kumar *et al.*, 2021; Verma *et al.*, 2018; Park *et al.*, 2022).

Previous works reported the effects of MWCNTs addition on the mechanical properties of glass fiber epoxy polymer composites and the wear performance of carbon nanotube coatings on glass fabric epoxy composites. No work has been reported on the effect of MWCNTs addition on the static (tensile, flexural), dynamic (high cycle fatigue), and wear performance of glass fabric-reinforced epoxy polymer composites and to evaluate an optimum ratio of MWCNTs addition in these composites for enhancement of the aforementioned properties. The study would be useful to researchers to estimate the optimum ratio of MWCNT addition in GF/EP composites for an improvement in their mechanical and wear performance.

2. MATERIAL AND METHODS

2.1. Materials

Multi-walled carbon nanotube/glass fabric/epoxy composite laminates and plain glass fabric/epoxy composite laminates were prepared using glass fabric (Type: uni-directional plain-woven, thickness: 0.33-0.41 mm, obtained from Bhor Chemicals & Plastics Pvt. Ltd., India) as the structural reinforcement. A mixture of epoxy resin (Epotec YD-585) and hardener (TH7257E) supplied by Aditya Birla Chemicals Pvt. Ltd. was used as the matrix. The MWCNTs (diameter: 10-20 nm, length: 3-8 μm, purity: >99%, average interlayer distance: 0.34 μm) supplied by Nanoshel, US, were used as the nano reinforcement.

2.2. Composites Fabrication

The MWCNTs/epoxy resin/glass fabric composites were fabricated in three stages. The MWCNTs were mixed with epoxy resin in two stages. An accurately weighed MWCNTs were mixed with epoxy resin by high-shear mixing using a shear homogenizer (Make: Daihan, Model: HG-15A). at 20000 rpm for 10 minutes, followed by sonication at 20 KHz on pulse mode (2s on, 5s off) for 20 minutes using a probe sonicator (Make: Stericox, Model: DH-92-IIDN). Further, hardener was mixed in epoxy resin (mixing ratio 100:32), followed by degassing under vacuum to remove the air bubbles. After degassing, glass fabric/epoxy (GF/EP) and glass fabric/epoxy/MWCNTs (GF/EP/MWCNTs) laminates were fabricated using the vacuum-assisted resin infusion molding (VARIM) process. A stack of 8 layers of unidirectional glass fabric was laid on the mold and then sealed with vacuum bagging film, as shown in Fig. 1(a). The vacuum was then applied to infuse the epoxy resin mixture into the glass fabric laid up via spiral tubing. The VARIM process is comprised of mainly two stages. The first stage is infusion, where an epoxy hardener mixture is sucked under a vacuum into the glass fabric layers, where vacuum pressure is maintained within the range of 650 mm of Hg to 670 mm of Hg using a vacuum pump. The laminates were then cured at 80˚C for 5 hours. The GF/EP and GF/EP/MWCNTs laminates prepared by the VARIM process have a size of 300 mm x 300 mm and a thickness in the range of 2.42 mm to 2.65 mm. Three batches of matrices comprised of MWCNT with weights of 0.075, 0.15, and 0.3 were prepared for GF/EP/MWCNTs composites. Samples were designated as GF/EP for neat glass fabric epoxy composite and GF/EP+0.075MWCNTs, GF/EP+0.15MWCNTs, and GF/EP+0.3MWCNTs for wt.% of 0.075, 0.15, and 0.3 MWCNTs in epoxy resin.

2.3. Composites characterization

The in-plane tensile properties like ultimate tensile strength, Young's modulus, and percentage elongation at break for GF/EP and GF/EP/MWCNTs composites were as per the ASTM D3039 standard. The flexural properties of these composite laminates were evaluated under the ASTM D7264 standard. The sample dimensions for tensile and flexural tests were as per ASTM D3039 and ASTM D7264 standards, respectively, and tests were performed using a universal testing machine (Make: Walter + Bai; Model: LFV-100 kN). At least, three samples were tested for each batch to ascertain the repeatability of the performance of these composites. Field emission scanning electron microscopy (FE-SEM) (Make: JEOL, Model: JSM 7900F) was used for fractography analysis of the fractured samples obtained after the tensile test and micrograph analysis of polished GF/EP and GF/EP/MWCNTs composites after VARIM fabrication. Gold sputtering was done on composite sample surfaces to get good SEM images of fractured samples. High-cycle fatigue (HCF) tests were performed at different stress levels for GF/EP and GF/EP/MWCNTs composites under stress-controlled constant-amplitude axial fatigue loading conditions following ASTM standard D3479. The load ratio was 0.1, and stress (MPa) vs. number of cycles to failure (nf) (S-N) curves were plotted for GF/EP composites with and without MWCNTs. The fatigue run-out condition was 1000000 cycles for both with and without MWCNTs added to GF/EP composites. Friction and wear tests for GF/EP and different weight percentages of GF/EP/MWCNTs composites were performed using a ball-on-disc tribometer (Make: Ducom Instruments) under dry sliding conditions at room temperature (25 ± 1 ˚C) as per ASTM standard G133. Disc-shaped composite samples were rotated against a stainless-steel ball (AISI 316) of 6 mm diameter with a normal load varying from 10 N to 30 N in steps of 10 N and a sliding speed of 0.1 m/s for a test duration of 60 min (3600 seconds). Similar test parameters were used in previous work on tribological studies on epoxy-carbon nanofiber composites (Chanda *et al.*, 2019). Before testing, all samples were polished to 1200 grit and cleaned with acetone to remove surface impurities. Friction force was measured with a load cell in the ball holder, and weight loss measurements were also done using an electronic balance. Worn surfaces of steel ball and composite samples were further investigated under optical microscopy (Make: Olympus, Model: DSX510) and FESEM analysis to depict the possible wear mechanisms.

3. RESULTS

3.1. Mechanical performance of GF/EP and GF/EP/CNT composites

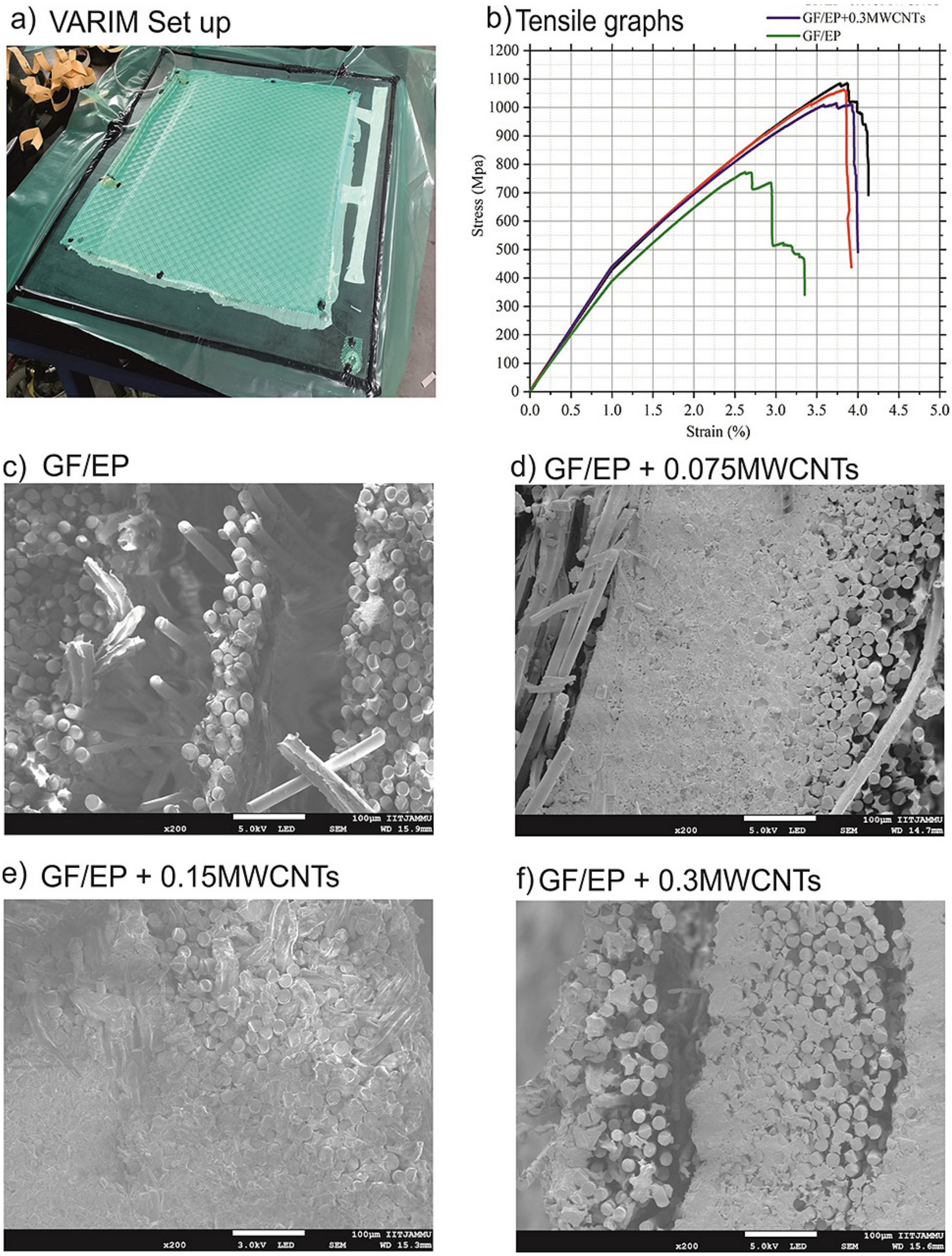
The mechanical performance of GF/EP/MWCNTs composites with different weights of MWCNTs and neat GF/EP composites was evaluated via static tests, which included tensile and flexural tests besides high-cycle fatigue tests. The average values of important properties such as tensile strength, Youngs modulus, elongation at break (%) for the tensile test, flexural stress, flexural strain, and flexural modulus for the flexural test are listed in Table 1. With the addition of MWCNTs in the GF/EP composite, an increase in tensile strength was noticed. MWCNTs with weight percentages of 0.075, 0.15, and 0.3 exhibited improvements in tensile strength of approximately 37.69%, 40.54%, and 31.47%, respectively, compared to GF/EP composites without MWCNTs (Fig. 1b). A similar trend was observed in the flexural test, where maximum improvement in flexural stress was noted for GF/EP+0.15MWCNTs by around 55% compared to GF/EP (Fig. 2a). Youngs modulus and flexural modulus also increased slightly with the addition of MWCNTs in the GF/EP composite. The fractography results, as shown in (Figs. 1c-f), showed the fractured sample morphology of GF/EP composites with and without MWCNTs addition after the tensile test. For the GF/EP composite, glass fibers were detached, whereas glass fiber detachment was not evident for MWCNTs added to the GF/EP composite. Another important observation was the rise in elongation at break (%) in the tensile test and flexural strain with the addition of MWCNTs. The highest increase in elongation at break and flexural strain was observed with 0.15 wt.% MWCNTs relative to a neat GF/EP composite. Also, the least rise in tensile strength, flexural stress, elongation at break, and flexural strain among different wt.% MWCNTs-added composites were noted for the GF/EP/MWCNTs composite with 0.3 wt.% MWCNTs. The tensile and flexural properties of the GF/EP/MWCNTs composite with 0.075 wt.% MWCNTs were increased to a certain extent at 0.15 wt.% MWCNTs and then decreased considerably at 0.3 wt.% MWCNTs relative to the GF/EP composite without MWCNTs. HCF tests were conducted on GF/EP and GF/EP+0.15MWCNTs at the same stress levels, and S-N curves were graphically represented with an exponential fit as shown in Figs 2b,c. Maximum tensile and flexural strengths were observed with 0.15 wt.% MWCNTs, GF/EP+0.15MWCNTs was chosen for HCF comparison with the GF/EP composite. The MWCNTs added to the GF/EP composite possessed a higher cycle count at different stress levels relative to the GF/EP composite. Both with and without MWCNTs added, GF/EP composites withstand the fatigue runout condition at lower stress levels. SEM micrographs (Figs. 3a,b) for GF/EP and GF/EP+0.15MWCNTs composites showed enhanced interfacial adhesion for GF/EP composites with MWCNTs addition which restricted interfacial defects and debonding at fiber/matrix interface (Figs. 3a,b). Similar observations were reported on interface enhancement of glass fiber fabric/epoxy composites by modifying fibers with functionalized MWCNTs (Zeng *et al.*, 2019).

S. No.	**Type of composite**	**Tensile Strength (MPa)**	**Youngs Modulus (GPa)**	**Elongation at break (%)**	**Flexural stress (MPa)**	**Flexural strain**	**Flexural Modulus (GPa)**
1	GF/EP	754 ± 10	40 ± 2	2.63	535 ± 6	0.0231	24.88 ± 1
2	GF/EP+0.075MWCNTs	1045 ± 6	44 ± 1	3.84	791 ± 3	0.0312	27.21 ± 1
3	GF/EP+0.15MWCNTs	1082 ± 3	43 ± 1	3.87	831 ± 4	0.0317	27.93 ± 2
4	GF/EP+0.3MWCNTs	969 ± 8	45 ± 2	3.73	735 ± 8	0.0253	26.81 ± 1

Table 1. Average values for tensile and flexural test parameters for GF/EP and GF/EP/MWCNTs composites.

3.2. Wear performance of GF/EP and GF/EP/MWCNTs composites

The friction tests were conducted, and friction coefficient versus time duration graphs were plotted at different loading conditions of 10N, 20N, and 30N for GF/EP and GF/EP+0.15MWCNTs composites, as shown in Figs.4(a-c). The friction coefficient for composites reached a stable level after a certain time, as shown in the figures. The friction coefficient increased with the rise in loading for composites. At 10N load, no significant difference in stable friction coefficient was noted between GF/EP and GF/EP+0.15MWCNTs composites. However, at 20N and 30N loads, considerable variations were observed between composites, as the GF/EP+0.15MWCNTs composite possessed a substantially lower friction coefficient relative to the GF/EP one. The wear track generated after the ball-on-disc test on these composites Fig. 5(a-d) show ball morphology after wear testing for composites at 10N load, and Figs. 6(a-d) show the same at 30N loading conditions for GF/EP composites with and without CNTs. The wear track depth in 2D and 3D views for composites is shown in Figs 5(b-f) at 10N load and in Figs 6(b,c,e, f) at 30N load. At 10N load, track width was lower for GF/EP+0.15MWCNTs than GF/EP, and not much significant change in ball morphology was observed. The wear track depth was 33 µm for GF/EP+0.15MWCNTs and 89 µm for GF/EP. A similar trend was noted related to wear track depth at 30N load, where 154 µm for GF/EP and 127 µm for GF/EP+0.15MWCNTs. The particles of the GF/EP composite were evident in their larger content on the ball surface compared to the GF/EP+0.15MWCNTs one. So, MWCNTs-added GF/EP composites have improved wear performance relative to GF/EP composites. SEM micrographs (Figs. 7a-b) taken at wear track at 30N load for both GF/EP and GF/EP+0.15MWCNTs composites showed considerable variations in wear track mechanisms. In both composites, cracks were evident in the epoxy layer which showed there was softening at the surface due to frictional heating. For GF/EP composite, epoxy layer was broken besides debonding of the epoxy with glass fibers was evident whereas no such characteristic was noticed for GF/EP+0.15MWCNTs composite.


a) VARIM Set up

b) Tensile graphs

c) GF/EP

d) GF/EP + 0.075MWCNTs

e) GF/EP + 0.15MWCNTs

f) GF/EP + 0.3MWCNTs

Figure 1. (a) VARIM setup; (b) Tensile stress vs. strain graphs; (c) to (f) Fractographs of fractured samples after tensile test (c) GF/EP; (d) GF/EP+0.075MWCNTs; (e) GF/EP+0.15MWCNTs; (f) GF/EP+0.3MWCNTs.

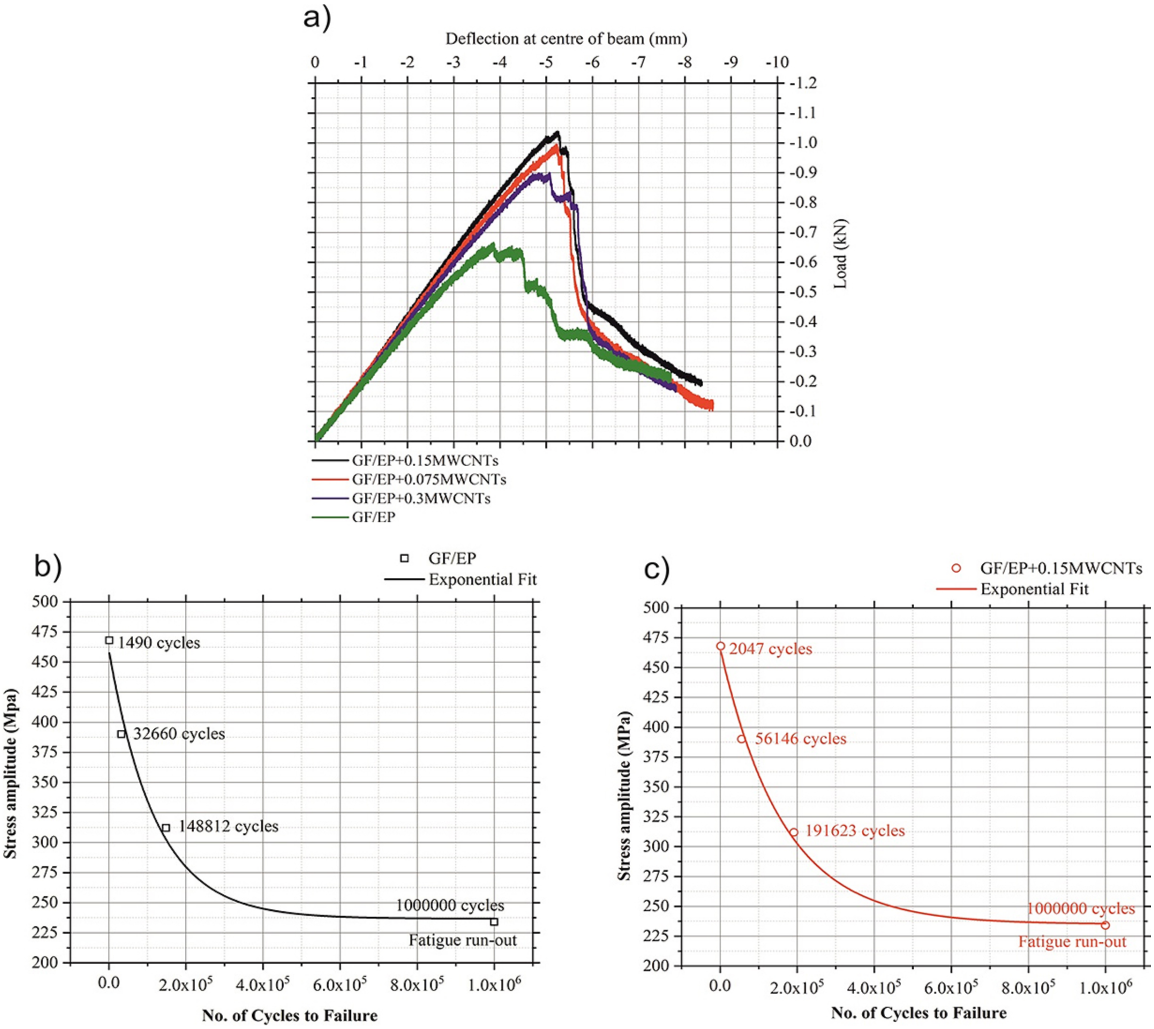


Figure 2. (a) Deflection at center of beam (mm) vs. load (kN) plots for three-point bend samples; (b) S-N curve for GF/EP composite; (c) S-N curve for GF/EP+0.15MWCNTs.

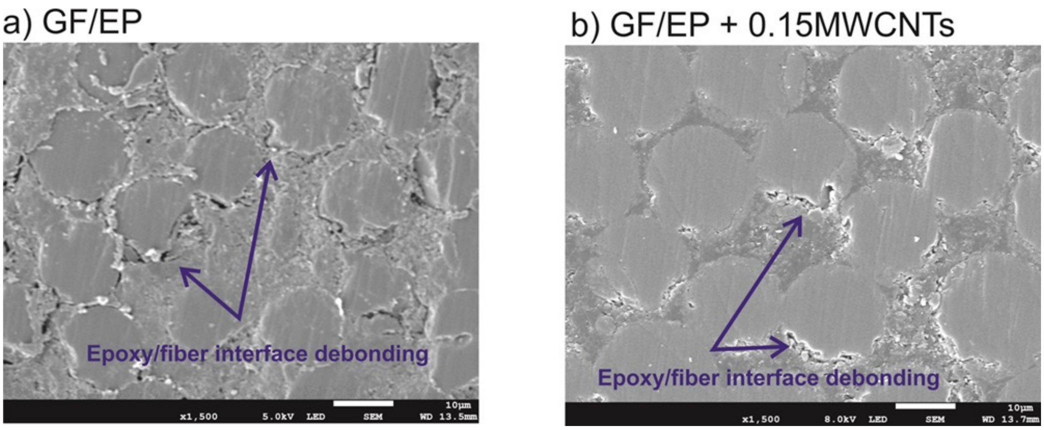


Figure 3. SEM micrographs for fiber/matrix interface of (a) GF/EP composite; (b) GF/EP+0.15MWCNTs composite.

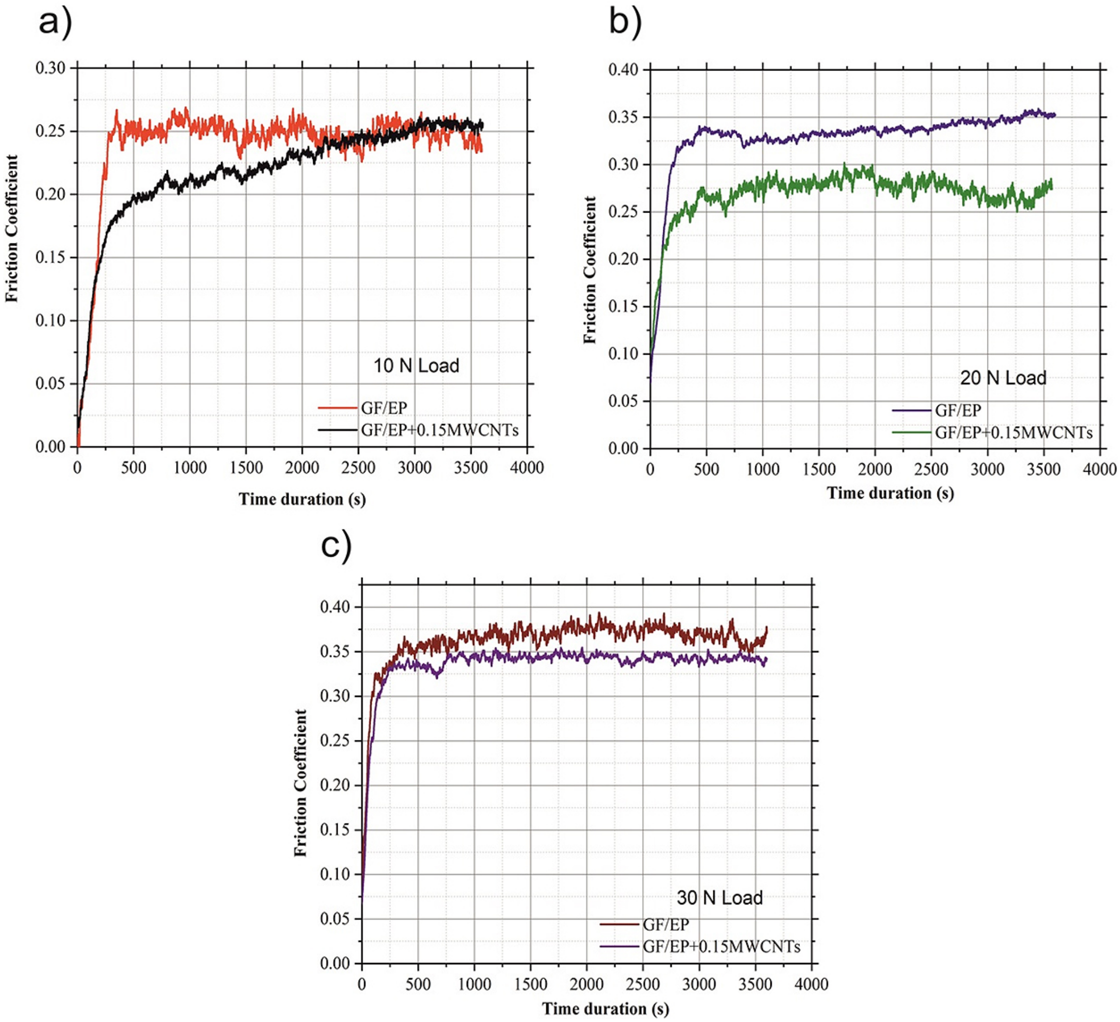


Figure 4. Friction coefficient vs. Time duration (s) plots at different loading conditions for GF/EP and GF/EP+0.15MWCNTs composites: (a) 10N; (b) 20N; (c) 30N.

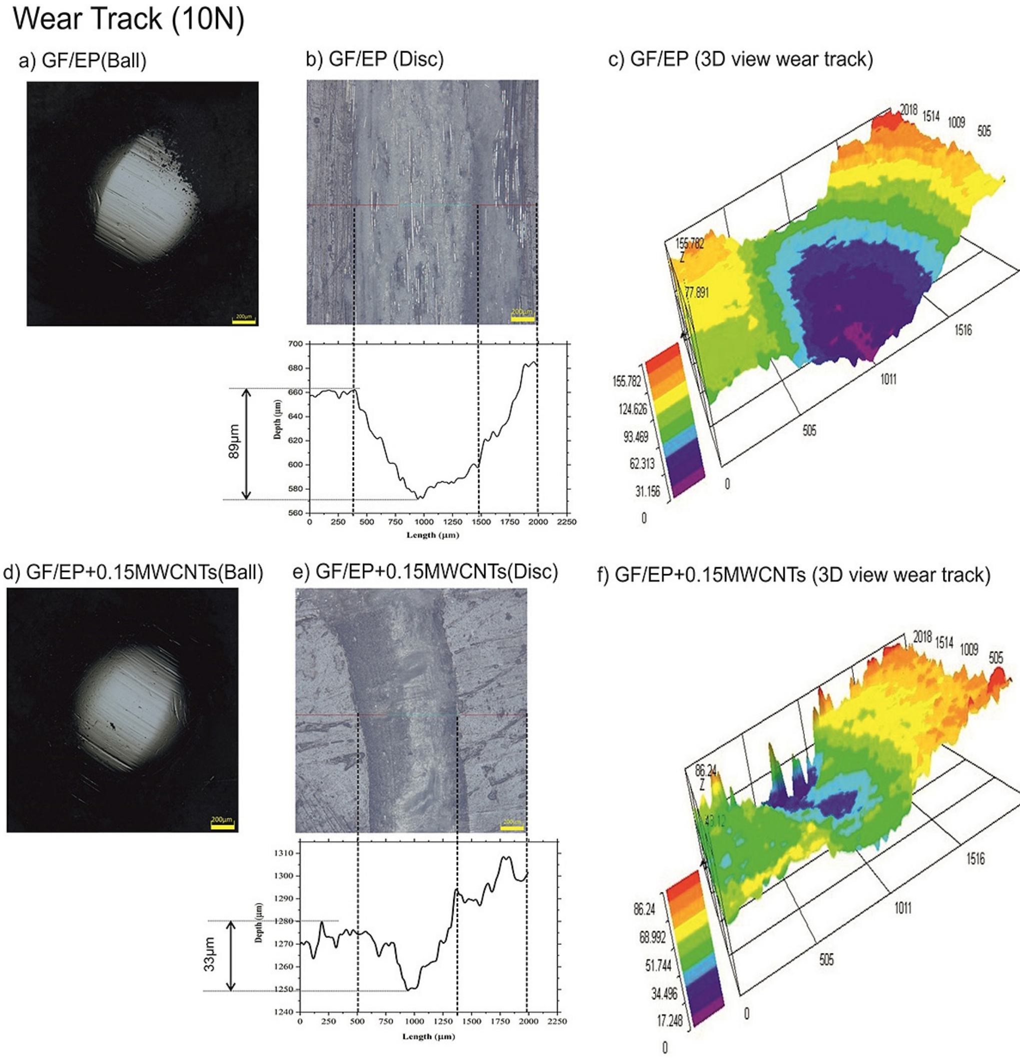


Figure 5. Wear track results for GF/EP and GF/EP+0.15CNT at 10N load: (a) & (d) Ball morphology; (b) & (e) 2D view of wear tracks; (c) & (f) 3D view of wear tracks.

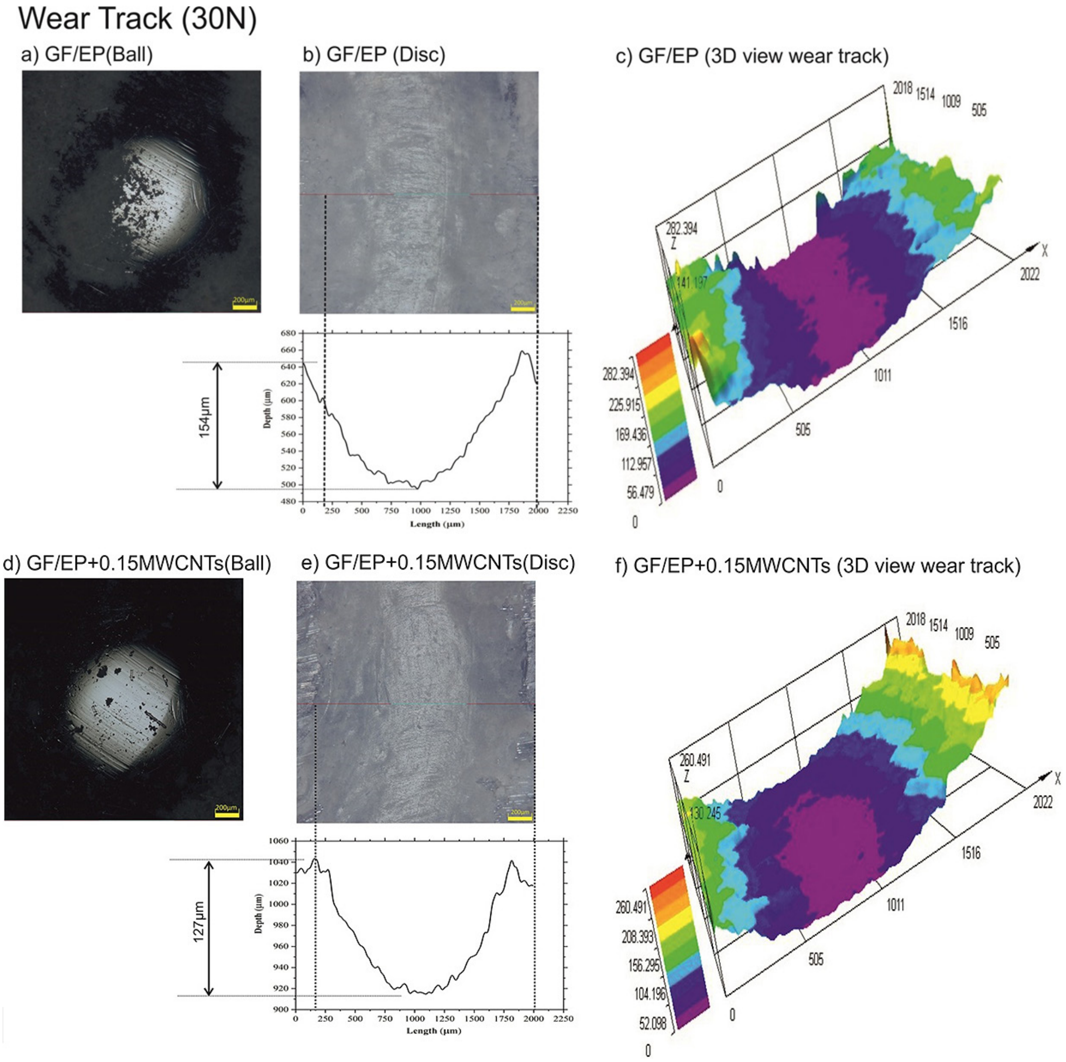


Figure 6. Wear track results for GF/EP and GF/EP+0.15CNT at 30N load: (a) & (d) Ball morphology; (b) & (e) 2D view of wear tracks; (c) & (f) 3D view of wear tracks.

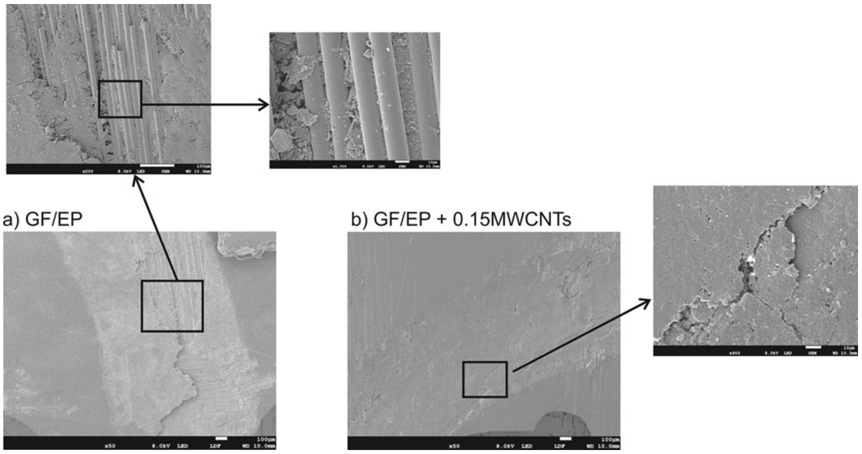


Figure. 7. SEM micrographs taken at the wear track of (a) GF/EP composite; (b) GF/EP+0.15MWCNTs composite at 30N Load.

4. DISCUSSION

The improvement in tensile and flexural properties of MWCNTs with added GF/EP composites was attributed to enhanced interfacial interactions, which further resulted in effective load transfer from matrix to CNT and CNT to fabric. Previous work showed that MWCNTs proved to be beneficial in enhancing the tensile and flexural strengths of MWCNTs added to glass fiber-reinforced polymers (GFRP) (Kappan *et al.*, 2019). As CNTs are high-strain energy-induced materials, uniform CNT dispersion in epoxy resin improves rigidity and hardness through interfacial interaction. The addition of MWCNTs to GFRP results in increased interfacial adhesion, which leads to the enhancement of both flexural and interlaminar shear strengths (Singh *et al.*, 2019). Fractography results showed relatively lower glass fiber detachment for GF/EP/MWCNTs composites compared to GF/EP composites. The homogenized dispersion of MWCNTs creates more filler surfaces for bonding with the epoxy, which enhances the mechanical properties (Weiking *et al.*, 2014). The optimum wt.% of 0.15 wt.% MWCNTs were identified as weight% with maximum mechanical properties among different MWCNTs weight% added GF/EP composites. There was a decline in the mechanical properties beyond 0.15 wt.%, which was evident for the GF/EP+0.3MWCNTs composite. An increase in MWCNTs content beyond 0.1 wt.% in epoxy results in agglomeration due to MWCNTs large specific surface area, and these aggregated fillers act as stress concentrators that are detrimental for mechanical performance (Cui *et al.*, 2013). Higher percentages of CNT addition in GFRP tend to degrade the fatigue performance of GFRP due to the formation of agglomerates (Burrego *et al.*, 2014). The increase in high cycle fatigue of GF/EP+0.15CNT relative to GF/EP was attributed to the formation of nanoscale damage zones owing to carbon nanotubes. Lower MWCNT volume fraction addition to the matrix tends to increase significantly high cycle fatigue performance owing to energy absorption from the fracture of nanotubes bridging across nanoscale cracks and from nanotube pull-out from the matrix that results in an improvement in fatigue life (Grimmer *et al.*, 2009). Wear performance improvement due to MWCNTs addition in GF/EP composite was attributed to MWCNTs self-lubrication characteristic. CNTs as additives can effectively reduce the friction coefficient of materials and improve their anti-friction and anti-wear properties (Yuxin *et al.*, 2020). Combining epoxy resin with MWCNTs is an effective method to enhance wear resistance and lower its friction coefficient (Cui *et al.*, 2013).

5. CONCLUSION

The major challenges faced during MWCNTs addition in glass fabric/epoxy polymer composites were improper dispersion and irregular alignment of MWCNTs in the polymer matrix. Besides this, agglomeration of MWCNTs was another major issue noticed at higher MWCNTs weight percent addition during VARIM fabrication of MWCNTs added glass fabric/epoxy polymer composites.

The tensile and flexural properties increased with the addition of MWCNTs in the GF/EP composite due to enhanced interfacial adhesion that leads to effective load transfer from epoxy matrix to CNT to glass fabric.

The optimum ratio in wt.% for MWCNTs addition was identified as 0.15 wt.% CNT in glass fabric epoxy composite for its improved mechanical properties. The maximum improvement observed at 0.15 wt.% addition was approximately 40% and 55% for tensile and flexural strengths, respectively. Beyond 0.15 wt.% CNT, tensile and flexural properties declined at 0.3 wt.% CNTs added to the GF/EP composite, which was still higher than the neat GF/EP composite. This decline in mechanical properties was due to the agglomeration of MWCNTs fillers.

High cycle fatigue results showed a significant rise in cycle count for 0.15 wt.% added GF/EP composite relative to neat GF/EP composite. The friction and wear performance were improved with the addition of 0.15 wt.% CNT in the GF/EP composite.

The MWCNTs incorporation in glass fabric/epoxy polymer composites enhanced the interfacial adhesion at the fiber/matrix interface thereby reducing the interface defects which resulted in improvement in static and dynamic mechanical properties of these composites. Also, MWCNTs addition prevents softening of the glass fabric/epoxy polymer composite surface in frictional heating conditions that affect the wear performance of these composites. Thus, improvement in mechanical and wear properties of MWCNTs addition in glass fabric/epoxy polymer composites explore opportunities for applications in wind turbine blade fabrication and the automobile sector.

Declaration of competing interest

The authors declare that they have no known competing interests that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available from the corresponding author on valid request.

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