



Research Article

Enhancement of out-of-plane mechanical properties of carbon fiber reinforced epoxy resin composite by incorporating the multi-walled carbon nanotubes

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Abstract

In this study, epoxy hybrid nanocomposites reinforced by carbon fibers (CFs) were fabricated by a filament winding. To improve out-of-plane (transverse) mechanical properties, 0.5 and 1.0 Wt.% multi-walled carbon nanotubes (MWCNTs) were embedded into epoxy/CF composites. The MWCNTs were well dispersed into the epoxy resin without using any additives. The transverse mechanical properties of epoxy/MWCNT/CF hybrid nanocomposites were evaluated by the tensile test in the vertical direction to the CFs (90° tensile) and flexural tests. The fracture surfaces of composites were studied by scanning electron microscopy (SEM). The SEM observations showed that the bridging of the MWCNTs is one of the mechanisms of transverse mechanical properties enhancement in the epoxy/MWCNT/CF composites. The results of the 90° tensile test proved that the tensile strength and elongation at break of nanocomposite with 1.0 Wt.% MWCNTs improved up to 53% and 50% in comparison with epoxy/CF laminate composite, respectively. Furthermore, the flexural strength, secant modulus, and elongation of epoxy/1.0 Wt.% MWCNT/CF hybrid nanocomposite increased 15%, 7%, and 9% compared to epoxy/CF laminate composite, respectively.

Keywords Epoxy · Carbon fibers · MWCNT · Nanocomposite · Transverse properties

Abbreviations

CFs	Carbon Fibers
MWCNTs	Multi-Walled Carbon Nano-Tubes
SEM	Scanning Electron Microscopy
90° UTS	90° Ultimate Tensile Strength
FRP	Fiber Reinforced Polymer
GFs	Glass Fibers
CFRP	Carbon Fiber Reinforced Polymer
DGEBA	DiGlycidyl Ether of Bisphenol A
CNTs	Carbon NanoTubes
CNTBPs	Carbon NanoTube BuckyPapers
RFI	Resin Film Infusion
CAI	Compression-After-Impact
DMA	Dynamic Mechanical Analysis

LVI	Low Velocity Impact
90° tensile test	Tensile test in the vertical direction to the CFs
E	Modulus
UTS	Ultimate Tensile Strength
UD90	Samples of 90° tensile test
SSA	Specific Surface Area

1 Introduction

In fiber reinforced polymer (FRP) composites, the fiber with excellent mechanical, physical, and thermal properties such as CF, glass fiber (GF), and aramid fiber is dispersed

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into the polymer matrix. These composites have unique properties (such as lightweight, high performance, higher stiffness, and so on) and are widely used in different industries [1–3]. Recently, researchers have been investigated the industrial functionalities of FRP composites. Aamir et al. [4] reviewed aerospace applications of carbon fiber reinforced polymer (CFRP) composites. Hassan et al. [5] focused on marine applications of epoxy/GF composite that Kevlar fiber stitched in the z-direction of samples.

Epoxy, as thermoset polymers, have been characterized by great chemical, thermal, and mechanical resistance, long dimensional stability, and good adhesiveness. These properties have made them be widely utilized in FRP composites as a matrix [6]. Diglycidyl ether of bisphenol A (DGEBA) is a thermoset epoxy with great mechanical, dynamical, and thermal properties, chemical resistance, and good fiber impregnation characteristics in comparison with the other polymers which has been widely used in CFRP composites [7].

In recent years, CF reinforced epoxy resin composites have attracted much attention due to their excellent properties when compared to the polymer matrix alone or relative conventional composites [8]. The epoxy/CF composites possess excellent properties, such as high stiffness and strength, corrosion resistance, easy processing, and facile large-scale production, which have been widely utilized in aerospace and railway [9, 10]. Great in-plane specific mechanical strength in the fiber direction also is important for CF-reinforced composites which facilitate carrying loads in the embedded CF direction. Although mechanical properties of composite laminates in the fiber direction are strong and these properties are insignificant in the vertical direction. Furthermore, the out-of-plane properties of epoxy/CF composites are matrix-dependent [11]. Therefore, out-of-plane properties are weaker than in-plane properties in CFRP due to the lower mechanical resistance of matrixes as compared to CFs [12]. This weakness may be problems that can cause matrix cracking and thereby ply delamination when out-of-plane loads apply [13].

The carbon nanotubes (CNTs) have been widely employed to augment properties of CFRP composites due to their great multi-functional properties, such as strong interfacial interactions and excellent stress transferring [14, 15]. The in-plane and out-of-plane properties can be improved by embedding CFs and nano-fillers, respectively [9]. Cheng et al. [16] studied the effect of carbon nanotube buckypapers (CNTBP) on the flexural properties of laminated FRP and quoted that CNTBP increases flexural properties. Avil et al. [17] studied the effect of the CNTs on the damping behavior of epoxy and CFRP composite. Yourdkhani et al. [18] were manufactured CFRP composite by resin film infusion (RFI) process. They investigated the effect of the dispersion of CNTs

on compression-after-impact (CAI) strength and electrical conductivity of samples. Han et al. [19] compared the effects of halloysite nanotubes, CNTs, and silicon carbide whiskers on the mechanical properties of epoxy/CF composites. Islam et al. [20] were added surface-modified MWCNTs and nanoclay to epoxy reinforced by CF composites. They investigated mechanical properties of composites using Dynamic Mechanical Analysis (DMA), Low Velocity Impact (LVI), and flexural tests [20]. In the literature, there is still a limited number of studies conducted for the epoxy matrix composites reinforced with CFs and MWCNTs which are evaluated the 90° tensile test properties.

To improve mechanical properties, the appropriate dispersion of CNTs in hybrid nanocomposite matrices is a critical issue. The CNTs are prone to be easily agglomerated, due to the bearing strong Van der Waals forces [21]. Some investigators have studied the methods for uniformly dispersing CNTs into FRP composites [13, 22–25]. Specific techniques have to be developed to grow CNTs on the surface of CFs [26]. The spray coating method was found to be effective in depositing CNTs onto the epoxy/CF prepregs [27]. Islam et al. [20] also used a mechanical mixer, ultra-sonic, and three roll mixers to disperse embedded the MWCNTs into the epoxy matrix.

The objectives of this paper are (i) to well-dispersed MWCNTs into epoxy resin using the high-power probe-ultrasonic and mechanical mixer method without any solvent addition, (ii) to investigate the effectiveness of MWCNTs incorporation for transverse mechanical properties of epoxy/CF laminate composite, (iii) to compare the effects of various weight percent of MWCNTs on the transverse mechanical properties of the composites, (iv) to perform the tensile test in the vertical direction to the CFs (90° tensile test) and three-point bending tests for evaluation of out-of-plane mechanical properties, and finally (v) to comprehensive microstructure characterization of composites.

2 Materials and methods

2.1 Materials

In this study, DGEBA type epoxy resin (2,2-Bis(4-glycidylphenoxy)propane Epoxide A) from MERCK (CAS Number: 1675-54-3), MWCNT (multiple rolled layers of graphene) from US Research Nanomaterials, Inc. (Code: US4306), hardener (methyl tetrahydrophthalic anhydride) from Chemwill (CAS Number: 19438-64-3), accelerator (1-Methylimidazole) from MERCK (CAS Number: 616-47-7), and CF (hexagonal aromatic rings of carbon atoms) from CSTsales (UNSPSC code: 11,162,100) were used as the primary materials. The epoxy resin system consisted of DGEBA, hardener, and accelerator. The resulting epoxy

system is an anhydride and hot-cure polymer. This system showed high potential for manufacturing the composites with filament winding, pultrusion, and pressure molding methods. The ultimate tensile strength (UTS) and modulus (E) of CFs are 4.9 and 230 GPa, respectively [28]. In this study, CFs with sizing was purchased to enhance wettability and structural properties. The outer diameter of the un-functionalized MWCNTs is 10–20 nm (Fig. 1). The true density and purity of MWCNTs are 2.1 g/cm³ and greater than 95%, respectively. The theoretical UTS and E of CNTs are 130 GPa and 1 TPa, respectively [29].

2.2 Sample preparation and testing

2.2.1 Dispersion method and sample preparation

Using ultrasonic and high-speed shearing is a simple method to improve the dispersion of nano-fillers in an epoxy resin matrix [30]. As can be seen in Fig. 2a, the MWCNTs were initially added to the hardener with relatively low viscosity (50–100 mPa s) at 25 °C, which allows for ultra-sonicating CNTs without solvent. The MWCNTs were sonicated in the hardener by probe-ultrasonic for 30 min under 200 W power at room temperature. Then, the DGEBA epoxy resin was added to the ultra-sonicated hardener/MWCNT. To facilitate the appropriate mixing of hardener/MWCNT in the epoxy with high viscosity, a mechanical mixer was used at 1500 rpm for 30 min at 75 °C. Next, the accelerator was added to the hardener/MWCNT/epoxy mixture. A mechanical mixer with 800 rpm was used to stir

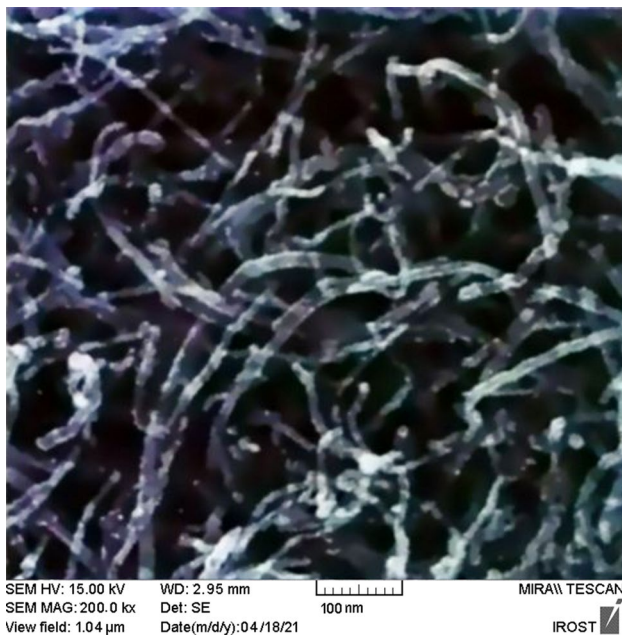


Fig. 1 SEM image of MWCNTs

hardener/MWCNT/epoxy/accelerator mixture for 10 min at 40 °C. Finally, the mixture was vacuumed to remove trapped air. The vacuum level was increased step by step up to 10⁻² mbar. To prevent the epoxy from boiling, the vacuum pump was stopped before boiling began.

The DGEBA epoxy resin as a matrix was mixed with hardener and accelerator in the ratio of 100:70:2.5 Wt.%, respectively. Components were thoroughly mixed to ensure homogeneity. The unidirectional composite laminates with 60 Wt.% CFs and 0, 0.5, and 1.0 Wt.% MWCNTs were fabricated by filament winding machine (Fig. 2b). Then, composites were cured at 2 h/70 °C + 3 h/130 °C.

2.2.2 90° tensile test

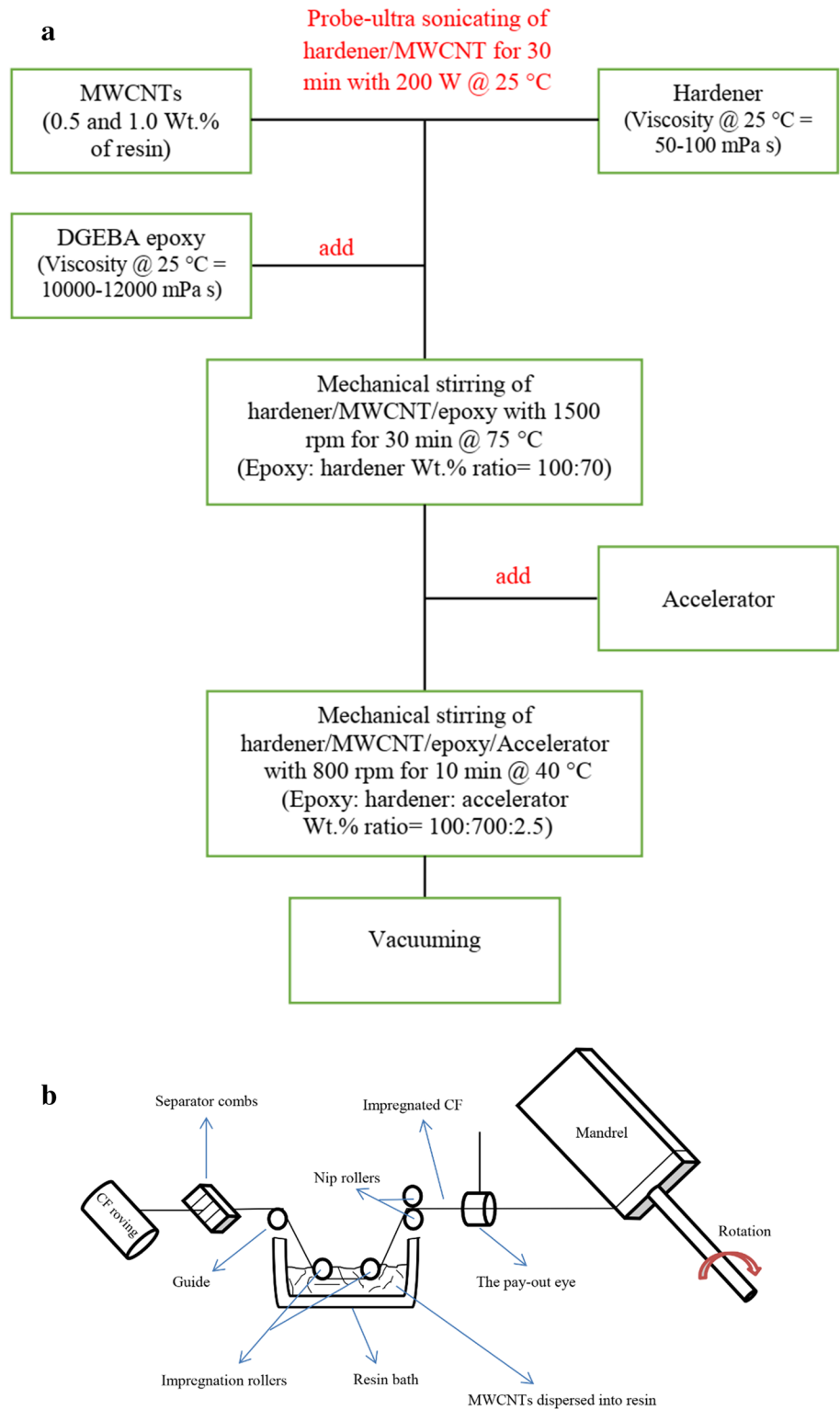
Samples of 90° tensile test (UD90) with dimensions of 2.5 × 25 × 175 mm³ were cut by waterjet from a twelve-ply plate in the vertical direction to the CFs according to ASTM D 3039 [31]. To effectively apply force to the samples during the tensile test, 25 × 25 × 1.5 mm³ Aluminum tabs with 90° bevel angle were bonded to the specimens (Fig. 3).

Tensile experiments of samples were performed by a universal testing machine at a cross-head speed of 2.0 mm/min according to the standard ASTM D 3039 [31]. The transverse mechanical properties (i.e., 90° UTS, E, and elongation at break) were evaluated. At least, five samples were tested for each composite to achieve reasonable results.

2.2.3 Flexural test

The flexural properties of epoxy/CF composites with and without MWCNTs were evaluated by a three-point bending test according to the ASTM D 790 [32]. The flexural strength, secant modulus, and elongation at break were measured by a three-point bending test. Samples were cut by waterjet from a twelve-ply plate (as shown in Fig. 3a) with dimensions of 2.5 × 15 × 250 mm³ in the parallel direction to the CFs. Figure 4 exhibits a schematic of the three-point bending test and test sample. The loading nose and supports were designed by cylindrical surfaces. The radius of the loading nose and supports were chosen to avoid excessive indentation and stress concentration directly under the loading nose which causes unacceptable failure. To prevent contact of the sample with the sides of the nose, the arc of the loading nose in contact with the sample was sufficiently large. The epoxy/CF composite with a high-strength was designed to the span-to-depth ratio of 60:1 to occur failure in the outer surface of fibers. This span-to-depth ratio was caused to eliminate the shear effect.

Fig. 2 Schematic of (a) MWCNTs dispersion into epoxy resin and (b) filament winding process for fabricating composites

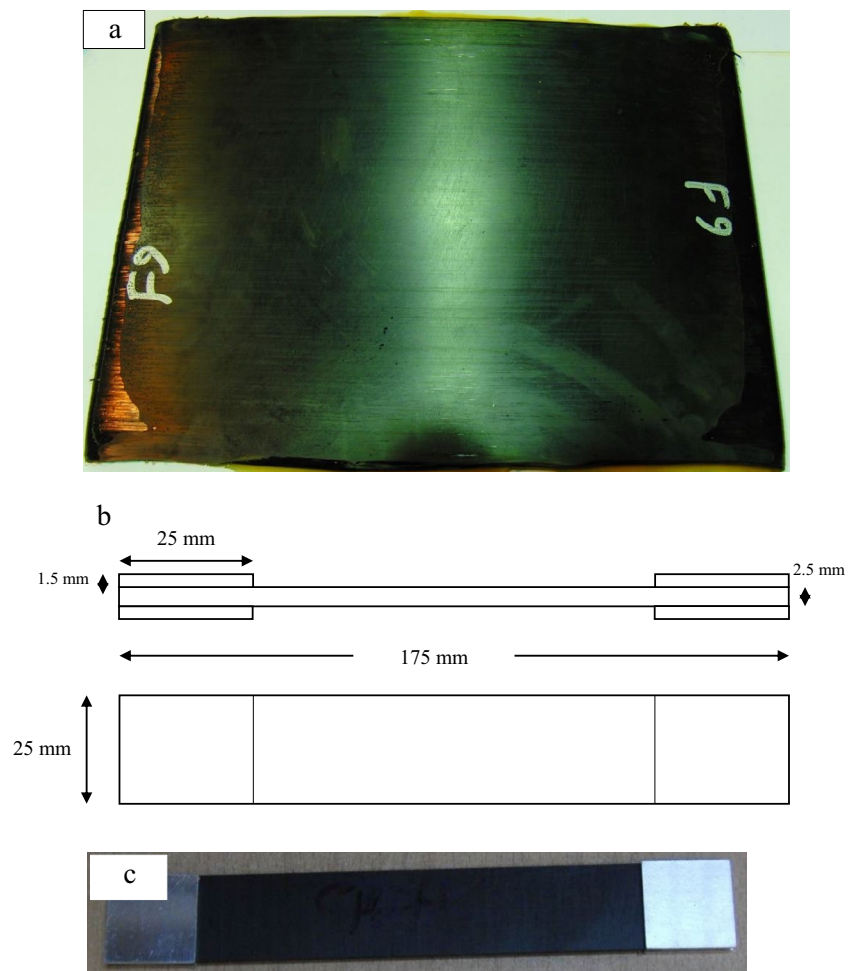


According to the ASTM D 790, the speed of the cross-head motion of the three-point bending test machine was 15.0 mm/min can be calculated by Eq. 1:

$$R = ZL^2/6d \tag{1}$$

where R (mm/min) is the rate of crosshead motion, L (mm) is the support span, d (mm) is the thickness of the sample,

Fig. 3 The details of sample preparation for 90° tensile test **(a)** Twelve-ply plate of composite, **(b)** schematic of UD90 sample with dimensions, and **(c)** UD90 tensile test specimen



and Z (min^{-1}) is the rate of straining of the outer fiber and shall be equal to 0.01 min^{-1} .

The flexural stress of the sample which is loaded at the midpoint and supported at two points was calculated by using Eq. 2:

$$\sigma_f = 3PL/2bd^2 \quad (2)$$

where σ_f (MPa), P (N), and b (mm) are the flexural stress, load, and width of the sample, respectively.

The flexural strain is the nominal fractional change in the length of an element of the outer surface of the sample at midspan, where the maximum strain occurs. It was calculated for any deflection by Eq. 3:

$$\varepsilon_f = 6Dd/L^2 \quad (3)$$

where ε_f (mm/mm) is the strain in the outer surface and D (mm) is the maximum deflection of the center of the test sample.

The slope of the straight line is the secant modulus when it joins the selected point and origin on the actual stress–strain curve. On the other hand, it is the ratio of

stress to corresponding strain at any selected point on the stress–strain curve [32].

3 Results

3.1 Microstructural study

The CF-reinforced epoxy laminate nanocomposite delaminates when subject to transverse or compressive load confirming the nature of laminated structure [33]. To solve this problem, it is necessary to add the MWCNTs in epoxy/CF laminate composite. The dispersion of the MWCNTs in epoxy-based composite was studied in Fig. 5, using SEM.

Incorporating CNTs with high specific surface area (SSA) into the matrix increased the internal energy of the system. The Van der waals forces among CNTs cause nanotube-nanotube adhesion. To minimize the SSA and internal energy, CNTs form aggregates. Ten to hundreds of individual CNTs make these agglomerations that are difficult to separate [34]. Embedding a high percentage of the MWCNTs into the epoxy-based composite increases

Fig. 4 (a) Schematic of the three-point bending test and (b) test sample

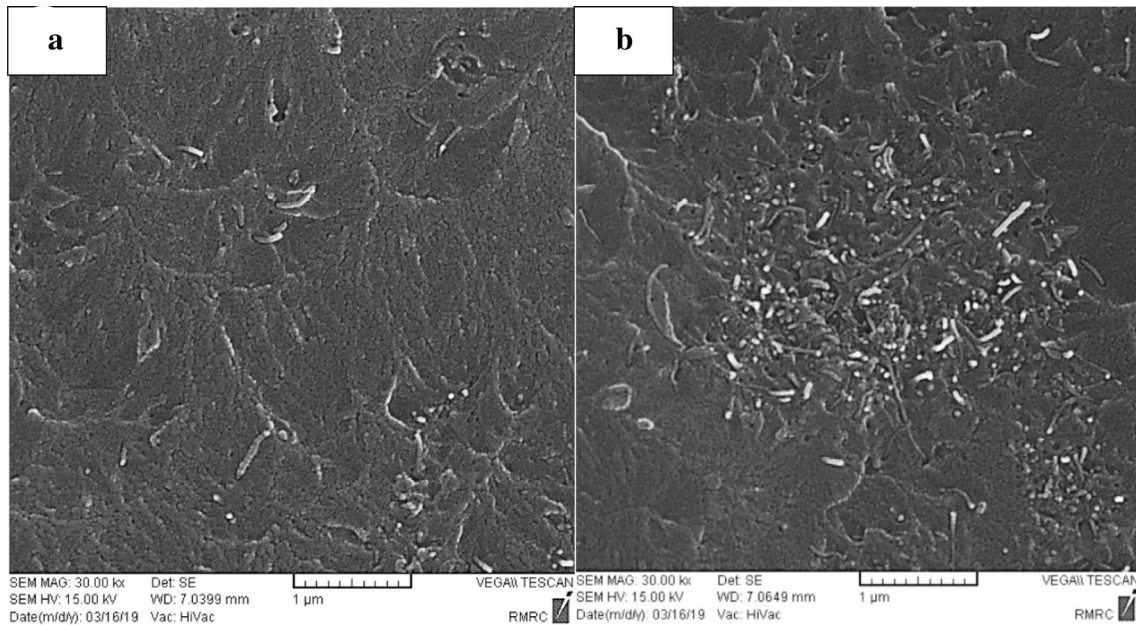
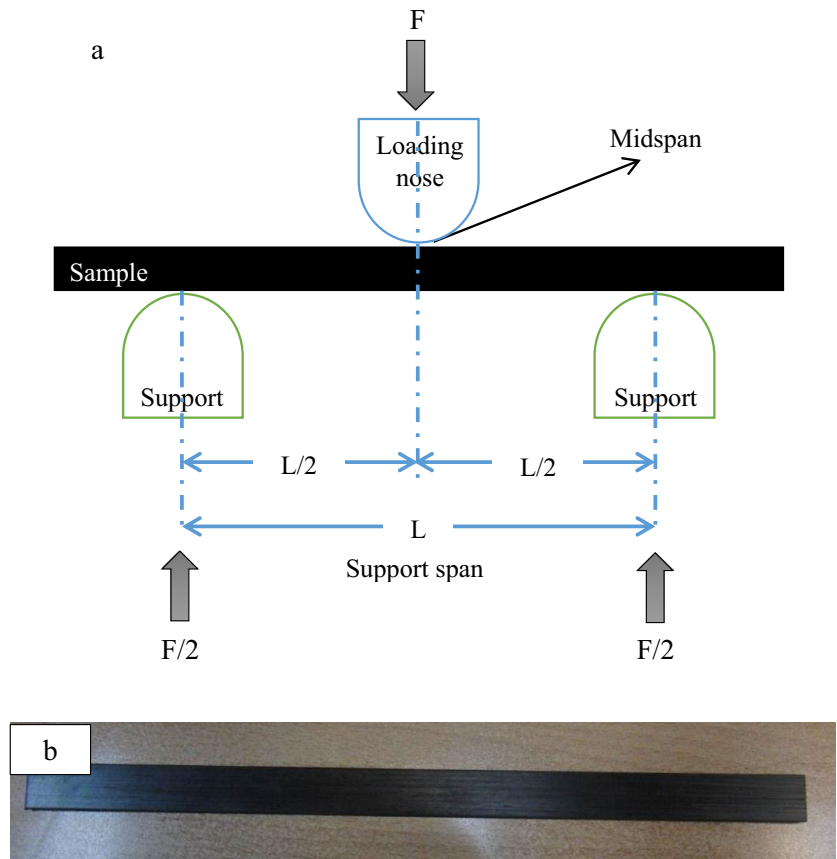


Fig. 5 The MWCNTs dispersion in nanocomposites (a) epoxy/0.5 Wt.% MWCNT and (b) epoxy/1.0 Wt.% MWCNT

the possibility of agglomeration. In nanocomposite with 1.0 Wt.% MWCNTs, the Van der Waals forces among

MWCNTs are higher than the other specimens, so the possibility of aggregation is high. However, Fig. 5b indicates

a homogenous dispersion of MWCNTs throughout the epoxy resin in epoxy/1.0 Wt.% MWCNT nanocomposite.

In this study, no additive was used for the MWCNTs distribution. The addition of additives may be helpful to achieve homogenous MWCNTs dispersion, but it may lead to the following problems:

- I. An extra step in the preparation process to remove additives before curing.
- II. Softening of the yielded composite due to low molecular weight [35].
- III. The reagglomeration of the MWCNTs may occur during the removal process [24].

3.2 Mechanical study

The transverse mechanical properties of epoxy/MWCNT/CF hybrid nanocomposites are evaluated by the 90° tensile test. Typical tensile stress–strain curves of epoxy/MWCNT/CF hybrid nanocomposites fabricated by 0, 0.5, and 1.0 Wt.% MWCNTs in the vertical direction to the CFs are illustrated in Fig. 6. It is seen from Fig. 6 that the 90° UTS and elongation increase as the MWCNTs loading increases. Also, the composites show pronounced linear behavior with large strains to failure.

The 90° UTS, E, and elongation at break of epoxy/CF composite and epoxy/MWCNT/CF hybrid nanocomposites with 0.5 and 1.0 Wt.% MWCNTs are presented in Table 1.

As can be seen in Table 1, the 90° UTS and elongation of composites continually enhance as the MWCNT content increases. It was seen that the epoxy/1.0 Wt.% MWCNT/CF sample showed higher 90° UTS and elongation than the other specimens. The 90° UTS and elongation of epoxy/CF composite were lower than the samples with MWCNTs. The average of the 90° UTS, E, and elongation of the composite without MWCNTs are 27.5 ± 1 MPa, 6.72 ± 0.17 GPa, and $0.34 \pm 0.01\%$, respectively. The composite containing

1.0 Wt.% MWCNTs shows a maximum of 90° UTS and elongation of 42 ± 1.41 MPa and $0.51 \pm 0.06\%$, respectively which are approximately 53% and 50% greater than that of epoxy/CF composite. Furthermore, the E of epoxy/1.0 Wt.% MWCNT/CF hybrid nanocomposite, which measures by the slope of the linear region in the stress–strain curve reaches approximately 6.8 GPa, which is about 2% more than that of the composite without the MWCNT incorporation. However, as shown in Table 1, composites had an E of the order of 6.45–7.17 GPa. In comparison with epoxy/CF composite, the E showed a very slight change. So, it should be noted that by adding 1.0 Wt.% MWCNTs the 90° UTS and elongation increased but E is constant.

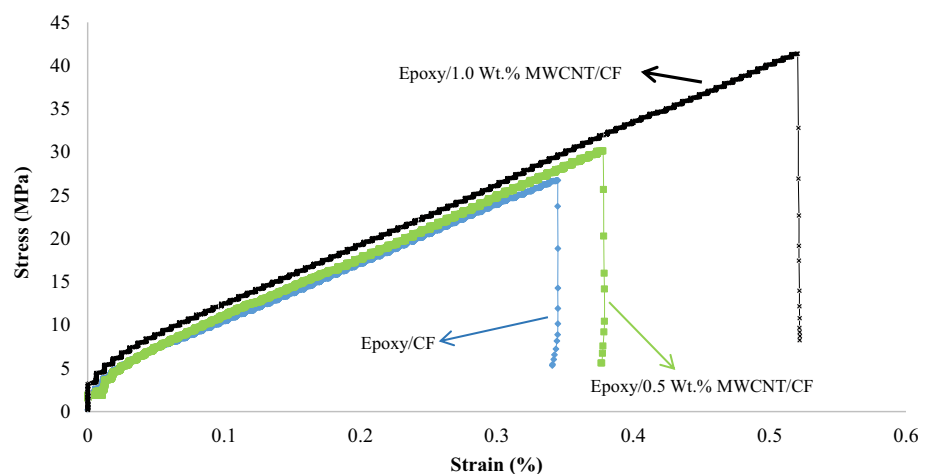
These results proved that the use of homogeneously dispersed MWCNTs improves the transverse mechanical properties of the fabricated composite as it could provide more the MWCNTs surfaces available to interact with the surrounding epoxy and increasing the strength of the epoxy resin matrix.

The fracture surface of the epoxy/CF laminate composite and epoxy/MWCNT/CF hybrid nanocomposite after the 90° tensile test showing the fracture regions are presented in Fig. 7. As shown in Fig. 7a, interfacial de-bonding in epoxy/CF composite observes with no epoxy adhere to embedded CFs. There is a poor connection between the epoxy and CFs resulting in weak interfacial bonding. In

Table 1 The transverse mechanical properties of samples

Sample	90° UTS (MPa)	E (GPa)	Elongation at break (%)
Epoxy/CF	27.5 ± 1.0	6.72 ± 0.17	0.34 ± 0.01
Epoxy/0.5 Wt.% MWCNT/CF	30.75 ± 4.34	6.45 ± 0.83	0.41 ± 0.05
Epoxy/1.0 Wt.% MWCNT/CF	42 ± 1.41	6.8 ± 0.08	0.51 ± 0.06

Fig. 6 The 90° tensile stress–strain curves of manufactured epoxy/MWCNT/CF hybrid nanocomposite with 0, 0.5, and 1.0 Wt.% MWCNTs



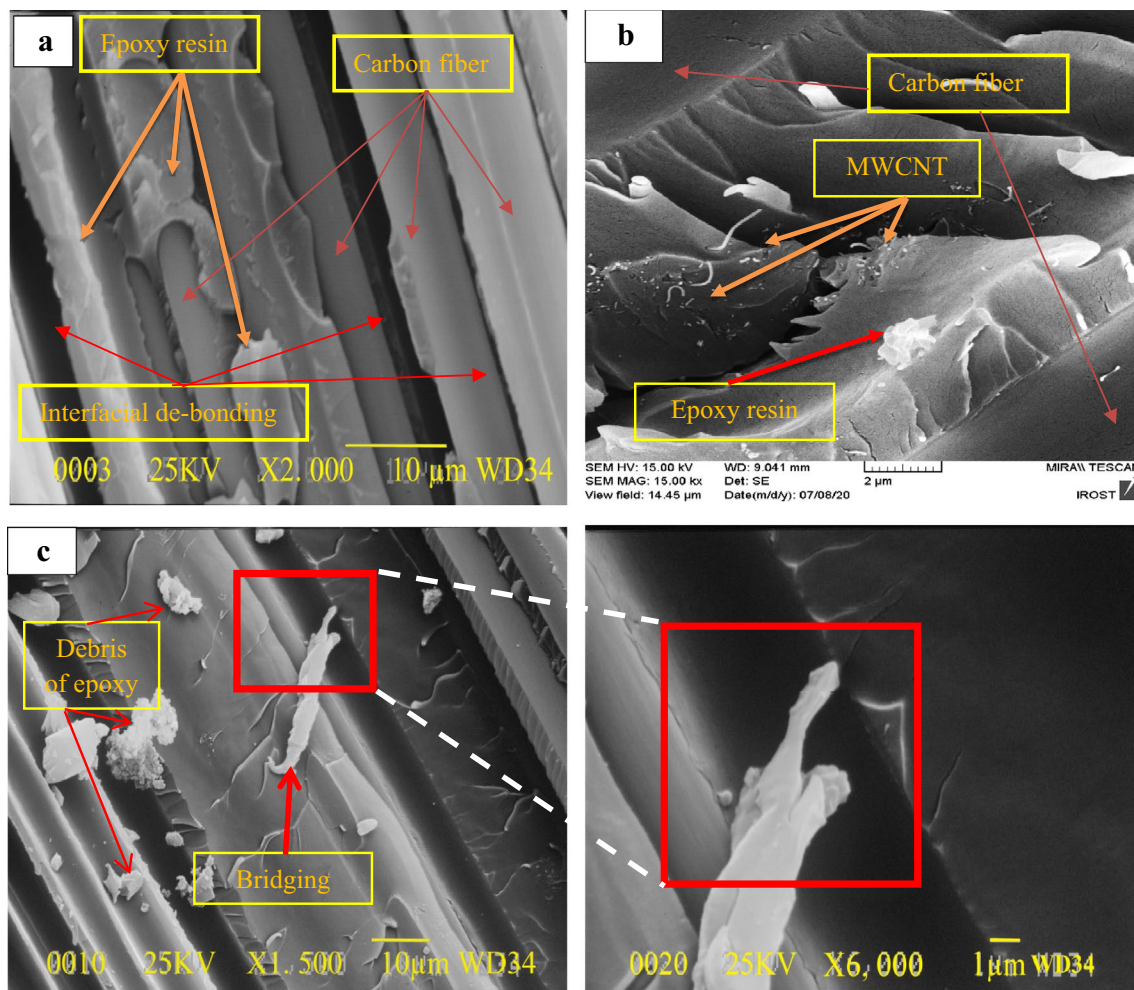


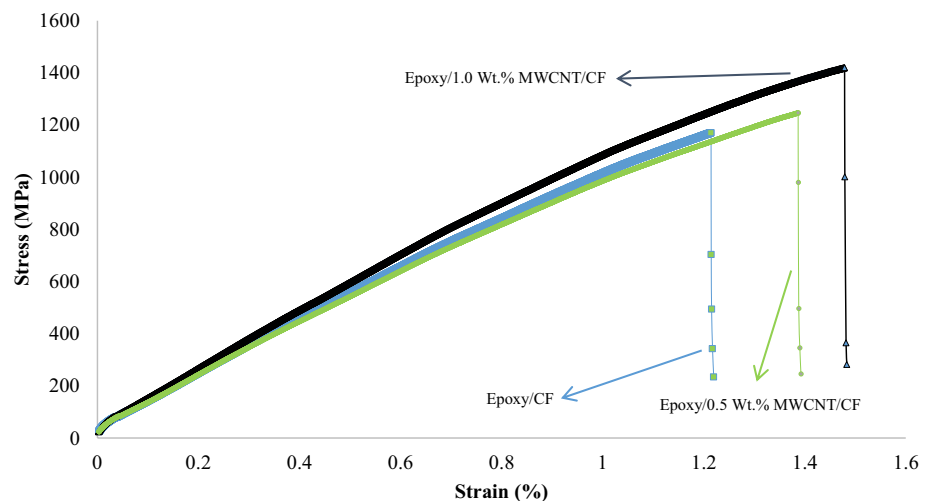
Fig. 7 SEM images of the fracture surfaces of (a) epoxy/CF laminate composite and (b, c) epoxy/MWCNT/CF hybrid nanocomposite

contrast to epoxy/CF composite, by the MWCNTs incorporation, the fracture surface of nanocomposite changes that epoxy resin adheres to the CFs, indicating the strong interfacial bonding (Fig. 7b). Figure 7c shows the debris of epoxy and MWCNT. The indicated MWCNT made a bridge as an interlock between the CFs. As can be seen, the shape of bridged MWCNT (rope and cylindrical shape) and debris of epoxy (shapeless with a porous surface) is completely different due to epoxy resin was cured on the surface of MWCNTs during the preparation process of the nanocomposite. The bridged MWCNTs increased load transfer and interlaminar strength result in improving mechanical properties.

The flexural properties of epoxy/CF composites and epoxy/MWCNT/CF hybrid nanocomposites fabricated by 0.5 and 1.0 Wt.% MWCNTs are measured by a three-point bending test. The flexural stress–strain curves of samples are shown in Fig. 8.

As can be observed in Fig. 8, flexural stress–strain curves of the composites illustrate the reinforcing effect of the MWCNTs and brittle fracture of the nanocomposites with almost no plastic deformation. The results of the three-point bending test are presented in Table 2.

According to Table 2, the flexural strength, secant modulus, and elongation at break of epoxy/CF composite are improved by the addition of the MWCNTs. As it is obvious in Table 2, the flexural strength values are 1374.02 ± 7.45 and 1251.26 ± 3.8 MPa for composites containing 1.0 and 0.5 Wt.% MWCNTs, respectively. These values are 15% and 5% higher than that of the flexural strength of epoxy/CF composite. The secant modulus and elongation of the nanocomposite with 1.0 Wt.% MWCNTs are 7% and 9% more than that of the composite without the MWCNTs. The interaction between the epoxy polymer matrix with the MWCNTs in the interlaminar and intra-laminar region of the laminated composites resulted in proportional enhanced mechanical properties of the samples. Tariq

Fig. 8 Flexural stress–strain curves of nanocomposites**Table 2** The flexural strength, secant modulus, and elongation at break of composites achieved from three-point bending test

Sample	Flexural strength (MPa)	Secant modulus (GPa)	Elongation at break (%)
Epoxy/CF	1191 ± 5.32	90.04 ± 2.09	1.3 ± 0.10
Epoxy/0.5 Wt.% MWCNT/CF	1251.26 ± 3.8	90.95 ± 1.95	1.36 ± 0.05
Epoxy/1.0 Wt.% MWCNT/CF	1374.02 ± 7.45	96.48 ± 2.6	1.42 ± 0.12

et al. [9] reported enhancement in flexural properties for epoxy/0.25 Wt.% MWCNT/carbon fabric composite. Sharma et al. [36] stated that the maximum improvement by 25% in flexural strength was achieved by incorporating 0.5 Wt.% amine-functionalized MWCNTs in CFRP composites.

Fracture surfaces of nanocomposites after the three-point bending test were observed by SEM (Fig. 9).

Figure 9a exhibits the fracture surface of an epoxy/CF laminate composite with low magnification, which showed a typical brittle fracture with island-like patterns. The pull-out of fiber, fiber breakage, and interfacial debonding was observed in the fracture surface of epoxy/CF composite (Fig. 9b). As can be seen in Fig. 9c, well dispersion of the MWCNTs into the epoxy resin results in the enhancement of flexural strength. The MWCNTs enable to make a strong mechanical link between the fracture surfaces due to their excellent mechanical properties as well as large specific surface area.

The difference between elongations which are measured by the tensile and flexural tests might be due to the strain rate of the test. The speed of cross-head motion of the flexural test 6.5 times more than the tensile test. These speeds were chosen according to related standards.

The improvement of dispersion and interfacial bonding of the MWCNTs in the epoxy resin enhances an effective stress transfer between epoxy and CFs. Incorporating 1.0

Wt.% MWCNTs compared to 0.5 Wt.% MWCNTs provide higher SSA in epoxy/CF composite. So, there is a more desirable interface for stress transfer in epoxy/1.0 Wt.% MWCNT/CF in comparison with epoxy/0.5 Wt.% MWCNT/CF hybrid nanocomposites.

The MWCNTs hold the epoxy resin matrix together by nano-stitches and enhance mechanical properties of the matrix, which lead to better transverse mechanical properties of epoxy/MWCNT/CF hybrid nanocomposite than that of epoxy/CF laminate composite. The composites containing 1.0 Wt.% MWCNTs that are homogeneously dispersed have two times more MWCNTs contents as epoxy/0.5 Wt.% MWCNT/CF composites. Hence, the mechanical properties of the matrix of the hybrid nanocomposite were further improved by 1.0 Wt.% MWCNTs.

The delamination and matrix cracking in the laminated composite may be due to the voids created in the interface of CF and the epoxy resin matrix. The MWCNTs improve interlaminar bonding between CF and epoxy and effective amount of voids reductions in the interface of reinforcement and matrix. Hence, an improving adhesion between CFs and epoxy plays an important role in stress transfer and causes the improvement of mechanical properties. The higher content of well-dispersed MWCNTs further enhanced interlaminar bonding and adhesion between CFs and epoxy. Therefore, interlaminar bonding and adhesion between CFs and matrix in epoxy/1.0 Wt.% MWCNT/

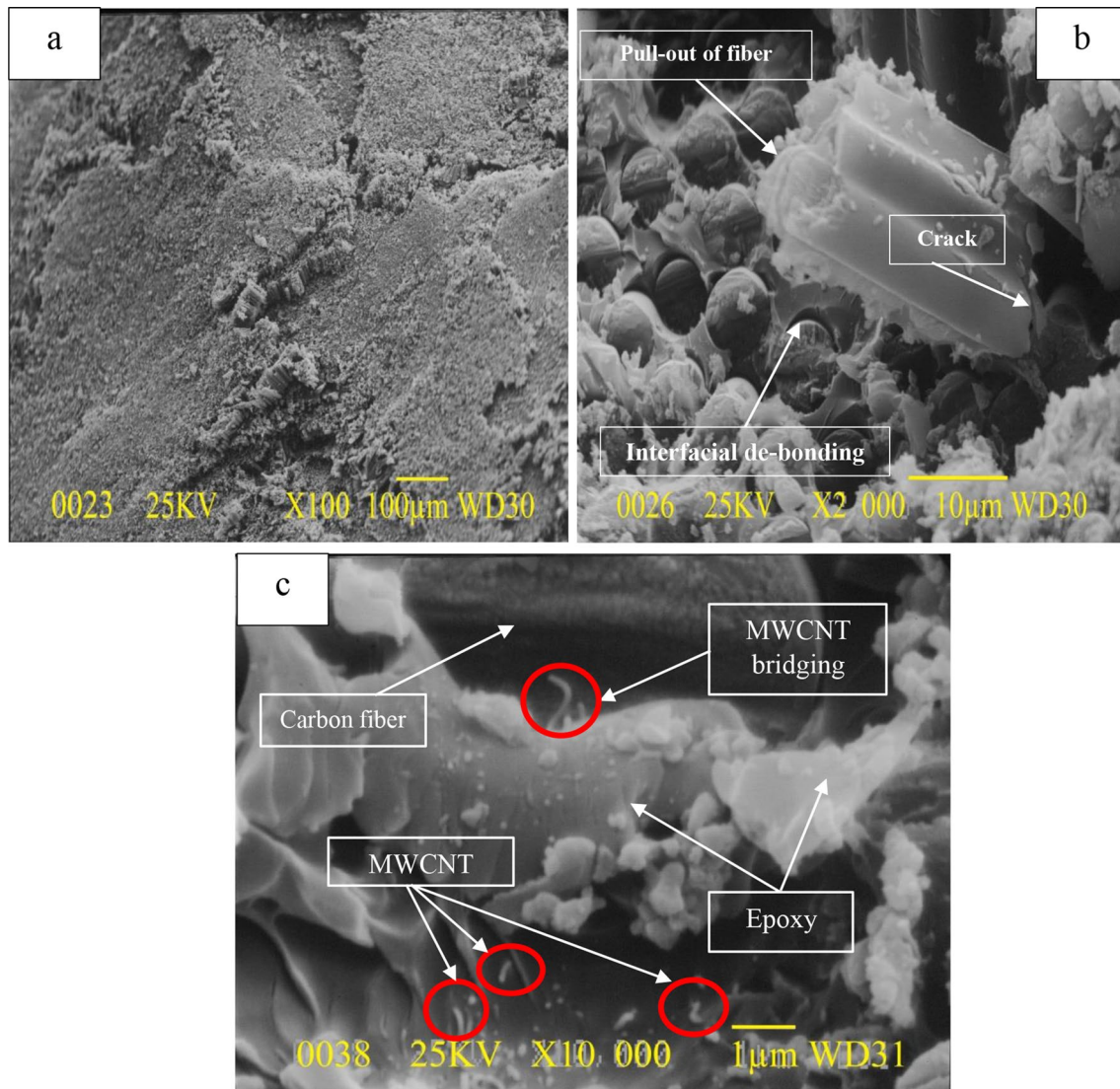


Fig. 9 SEM images of fracture surfaces of three-point bending samples (**a**, **b**) epoxy/CF composite and (**c**) epoxy/MWCNT/CF hybrid nanocomposites

CF composite is stronger than epoxy/0.5 Wt.% MWCNT/CF hybrid nanocomposite.

4 Conclusions

The epoxy/CF laminate composite and epoxy/MWCNT/CF hybrid nanocomposites with 0.5 and 1.0 Wt.% MWCNTs were fabricated by a filament winding machine. The flexural and 90° tensile properties of samples were evaluated by the three-point bending and 90° tensile tests, respectively. The results show that the out-of-plane properties of epoxy/CF composite were improved by incorporating the 1.0 Wt.% MWCNTs. The following conclusions were drawn:

1. The MWCNTs were homogeneously dispersed into the epoxy resin matrix using a high-power probe-ultrasonic and mechanical mixer without using any additives.
2. The 90° UTS and elongation at break of epoxy/1.0 Wt.% MWCNT/CF hybrid nanocomposite were approximately 53% and 50% greater than that of epoxy/CF composite, respectively.
3. In comparison with epoxy/CF composite, the flexural strength, secant modulus, and elongation of epoxy/1.0 Wt.% MWCNT/CF hybrid nanocomposite increased 15%, 7%, and 9%, respectively.

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Declarations

Conflicts of interest The authors declare that they have no conflict of interest.

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