

Research Article

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Improving the fatigue life of composite by using multiwall carbon nanotubes

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Abstract: The fatigue life of polymer materials like epoxy can be improved by using stiffeners such as carbon fiber and/or adding multiwall carbon nanotubes (MWCNTs). This article studies the effect of adding MWCNTs with different ratios (0.5, 1, and 2 wt%) to epoxy and composite (epoxy + 30% carbon fibers). The experimental results of the fatigue test with fully reversed bending stress (with $R = -1$) showed a maximum increase of 788% in fatigue life when adding 1 wt% MWCNTs to epoxy, while the maximum improvement ratio reaches 2,500% when adding 1 wt% MWCNTs to composite. The best results of fatigue life improvement were observed for samples with MWCNTs of 1 wt%. The material will be transferred from low cycle fatigue (less than 10^5 cycles) to high cycle fatigue (more than 10^5 cycles) by adding 1 wt% of MWCNTs. At the same time, the ratio of MWCNTs of more than 1 wt% (such as 2 wt%) will decrease the fatigue life due to the agglomeration of nanotubes inside the resin and reduce the positive effect of it. These agglomeration points work as a barrier to load transfer and stress concentration points. The numerical model was built to simulate the fatigue test and compare the results with the experimental with a discrepancy value of 7.5%.

Keywords: epoxy, carbon fibers, CFRP, MWCNT, fatigue life, ANSYS

1 Introduction

Composites are utilized in various structures, including automotive, airplanes, and space crafts, because of their excellent characteristics of hardness-to-density and strength-

to-density ratios [1]. Owing to its light weight, high tensile strength, and superior corrosion and fatigue resistance, carbon fiber reinforced polymer has been widely used in the fields of aircraft, ships, and automobiles [2]. In this case, the used materials are subjected to cyclic loadings, triggering questions concerning their fatigue behavior [3]. In lightweight applications, a composite made of epoxy and carbon fibers has higher/better properties than usual materials such as aluminum, copper, and steel. Still, it is inferior to other materials for high-load applications. The chassis of a vehicle will experience various loading, including fatigue, lateral bending, and longitudinal torsion loading [4]. A fundamental problem in the composite material is fatigue load, which is a major concern. Since composite materials are anisotropic and heterogeneous, the fatigue behavior is more complicated, particularly in applications of structures with high loads. The composite materials exhibit a highly complex failure mechanism under fatigue loading. Fatigue failure in composite materials occurs for the following reasons: matrix cracking, fiber breakage, casting defects, and their interaction [5]. Tang et al. [6] studied the effect of multiwall carbon nanotubes (MWCNTs) on the thermal conductivity and mechanical properties of epoxy resin, like tensile strength, Young modulus, and impact toughness. The results showed increases in the thermal conductivity by about 61%, and mechanical properties improved with a range of 30–45% when adding about 0.5 wt% of MWCNTs to the pure epoxy resin. Wang et al. [7] presented the effect of adding carbon nanotubes (CNTs) to the composite of (epoxy + glass fibers) on the strength and toughness of the composite. The results showed improvement in the interlaminar shear strength and toughness with a 28 and 189% ratio when adding CNTs to epoxy glass fiber composite. Jen and Ni [8] experimentally studied the effect of adding CNT and graphene nanoplates on the fatigue properties of carbon fiber-reinforced epoxy materials. The results showed that the samples with a 9:1 MWCNT-to-graphene nanoparticle (GNP) ratio improved fatigue strength and life. Turaka et al. [9] concluded the effect of adding MWCNT and GNPs in the range of 0.1–0.3 wt% to the glass fiber/epoxy composite on the fatigue life of the composite. The results showed that the fatigue life increased by 56% when using MWCNT/GNPs of 0.2 wt%. Genedy et al. [10] presented the effect of adding the MWCN

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with a ratio of 0.5 and 1 wt% to the glass fiber-reinforced plastics (GFRPs). The results showed a maximum improvement of 1,143% in fatigue life of GFRP when using MWCNT of 0.5 wt%. Böger et al. [11] concluded the influence of adding CNTs to the glass fiber/epoxy hybrid composite. The results presented the positive effect of adding CNTs in increasing the fatigue life of the composite material to more than 50%. The properties of composites are greatly affected by the nature of bonding at the interface, the strength of the interface, and the mechanical load transfer from the surrounding matrix to nanotubes. Shokrieh et al. [12] presented the effect of experimentally adding nanographene platelets to the nanocomposite of the epoxy matrix with ratios of 0.1, 0.25, and 0.5 wt%. These results showed that the fatigue life on nanocomposite would increase about 27 times compared with neat epoxy. Mahdi and Alithari [13] investigated the effect of adding nanoparticle additives on the mechanical properties of epoxy/Kevlar fiber composite. The nanographene platelets were added in three different percentages of 0.5, 1, and 2 wt%. The results showed improvement in material stiffness reaching 134 and 90% for flexure strength. Islam et al. [14] presented a review of the effects of adding CNTs to composite and the agglomeration effects of CNTs inside the resin on reducing the properties of the composite. The present work studies the effects of adding MWCNTs to the epoxy and composite on the fatigue life and how that will enhance the composite's working life subjected to fully reversed loads and how to transfer the material from low fatigue life to high fatigue life.

2 Materials and methods

2.1 Materials

The materials used in preparing the composite samples for fatigue tests from resin and reinforcement materials (carbon fibers and/or MWCNTs) are summarized as follows:

2.1.1 Epoxy resin

Renksan-Renfloor HT 2000 is the epoxy resin used in this study, and it is used as a matrix for the composite material;

it has properties such as low viscosity, low creep, good mechanical resistance, transparent color, and mixing ratio with its hardener at 2 epoxy/1 hardener. The epoxy was used as the matrix system to develop a composite material. Table 1 shows the properties of this type of epoxy [13].

2.1.2 Fibers

Properties of carbon fiber used as reinforcement with trade name (SikaWrap®-300 C/60) made by Sika from Italy are shown in Table 2 [15].

2.1.3 MWCNTs

MWCNTs (shown in Figure 1) are used as nanoadditives to epoxy to improve its properties. The MWCNTs were made by combustion chemical vapor deposition and purified using concentrated acid chemistry with a purity of 90%, an outer diameter of 20–40 nm, and a length of 10–30 μm . Table 3 shows the properties of MWCNTs [16,17].

2.2 Mold preparation

The hand layout method was used to manufacture the composite samples from epoxy and composite with and without MWCNTs with different ratios of 0.5, 1, and 2 wt%.

Figure 2 shows that the open metallic mold was used to produce a fatigue test of rotating bending composite samples with dimensions shown in Figure 3.

2.2.1 Mixing process of MWCNTs with epoxy resin [18,19]

The MWCNTs are mixed with epoxy resin at three ratios, i.e., 0.5, 1, and 2 wt%.

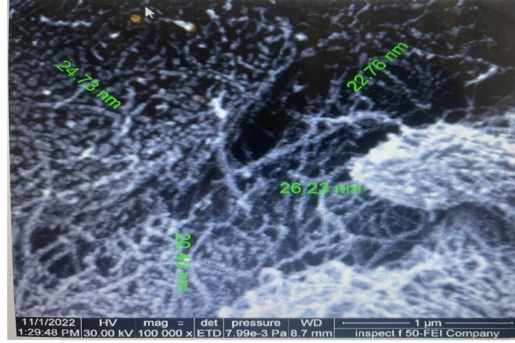
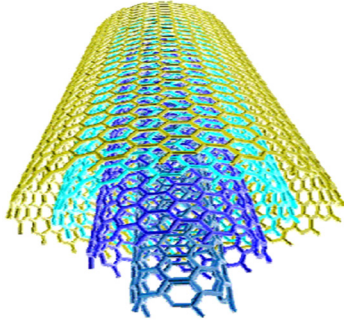
The epoxy was mixed with MWCNTs mechanically by a shear mixer with the speed of 2,000 rpm for 30 min and then sonicated with a high-power (250 W) sonic dismembrator type MTI for 30 min with a probe diameter of

Table 1: Properties of epoxy [13]

Property	Density (kg/m^3)	Viscosity (Poise) at 35°C	Compression strength (MPa)	Fracture toughness ($\text{MPa}\cdot\text{m}^{1/2}$)	Tensile strength (MPa)	Bending strength (MPa)	Modulus of elasticity (GPa)
Epoxy	1,050	1	70	0.6	27	63	2.8

Table 2: Carbon fiber properties [15]

Type	Density (kg/m ³)	Tensile strength (MPa)	Young's modulus (GPa)	Poisson's ratio	Color
Carbon fiber	1,800	4,000	230	0.2	Black

**Figure 1:** MWCNTs [17].**Table 3:** Properties of multiwall carbon nanotubes [16,17]

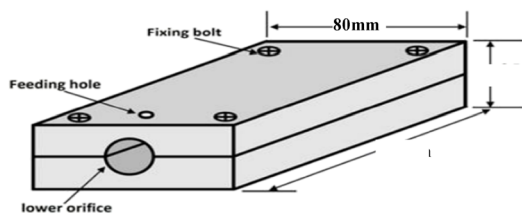
Type	Density (kg/m ³)	Tensile strength (GPa)	Young's modulus (GPa)	Specific surface area (m ² /g)	Melting temperature (°C)
MWCNTs	2,100	140	1,000	70–250	3,750

20 mm. The sonication power increased gradually until it reached 250 W, maintaining the mixture's temperature at 30°C using the ice jacket that worked as a heat sink to avoid overheating inside the sonication vessel. Then the mixture was placed inside a vacuum oven at 40°C for 30 min to remove the bubbles that may be found in the mixture and then poured into the mixture hardener with a ratio of 1:2 and mixed mechanically for 30 min. Before curing, the mixture was degassed under vacuum at $25 \pm 5^\circ\text{C}$ for 30 min, and then the mixture was poured into a metallic mold using the technique of slow injection by a feeding tube to completely produce fatigue test samples with a volume fraction of fibers of 30%, as shown in Figure 4.

The Halpin-Tsai mathematical model was used to determine the modulus of elasticity for nanocomposite samples depending on the geometry and orientation of reinforcement and the values of elastic modulus of matrix E_m and for nanomaterial E_n [20].

$$E_c = \frac{1 + 2\frac{w}{t}\eta V_n}{1 - \eta V_n} E_m, \quad (1)$$

where $\eta = \frac{\left(\frac{E_n}{E_m} - 1\right)}{\left(\frac{E_n}{E_m} + 2\frac{w}{t}\right)}$, E_c is the modulus of elasticity for composite, w/t is the shape factor, and V_n is the volume fraction of nanomaterials.

**Figure 2:** Metallic mold.

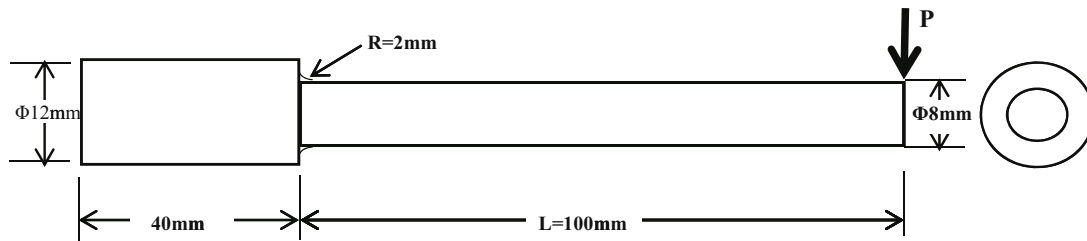


Figure 3: Dimensions of composite sample for fatigue test.

2.3 Samples test

2.3.1 Fatigue Test

The fatigue test (rotating bending) for composite specimens was done according to the standard specification of ASTM E467-21 [21], using a fatigue tester machine of GUNT from Germany, as shown in Figure 5, with applying different load values on the rotating samples to produce fully reversed bending stress of 15, 20, 25, and 30 MPa (chosen according to past researches) on cantilever rotating shaft with a stress ratio of $R = -1$, and to calculate the average value of a number of cycles for five testing samples for each test [22,23].

The bending stress can be calculated as follows [22,23]:

$$\sigma_b = \frac{M \times y}{I}, \quad (2)$$

where

$$M = F \times L, \quad (3)$$

where F is the applied bending load (N), L is the active length of the sample (m), y is the maximum distance of the surface from the center of cross-section (m), and I moment of inertia of sample (m^4).

$$I = \frac{\pi \times d^4}{64}, \quad (4)$$

where d is the diameter of the sample (m).



(a)



(b)

Figure 4: Fatigue tests samples: (a) without MWCNTs and (b) with MWCNTs.

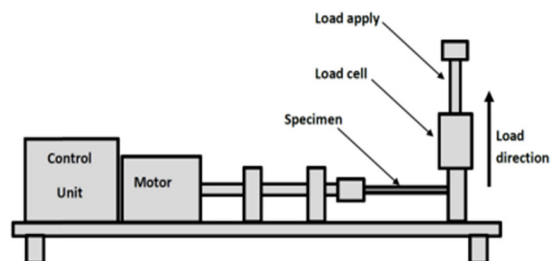
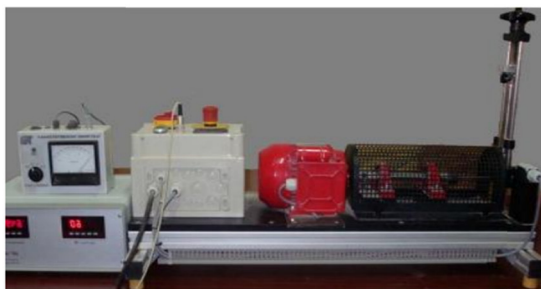


Figure 5: Parts of test machine.



Figure 6: Scanning electron microscopy.

2.3.2 Scanning electron microscope (SEM) test

The test composite samples were observed under SEM to obtain high-resolution images of MWCNTs distribution inside the resin and to know how that affects the properties of the composite especially fatigue life. Figure 6 show the SEM.

2.4 Numerical models

The numerical model for fatigue analysis was done using the finite element method using ANSYS Workbench R17.2 to simulate the fatigue test with 10860 elements of

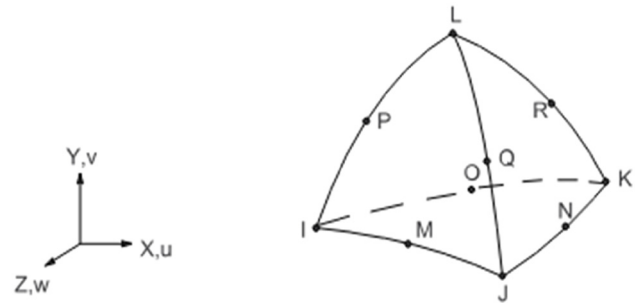


Figure 7: Tetrahedral element of 3D solid187 [13].

element type solid 187 (3D 10-node tetrahedral), as shown in Figure 7 [13].

Figure 8 shows the finite element meshed model for fatigue test for epoxy resin only, epoxy + MWCNTs with different weight ratios 0.5, 1, and 2 wt%, and composite (epoxy with carbon fiber of 30% volume fraction with and without MWCNTs). The sample is fixed in X , Y , and Z directions at the fixed end and freely rotates about the Z -axis with a bending load at the free end to produce fully reversed bending fatigue stress with $R = -1$ to set the same boundary conditions of experimental work. The numerical results are compared with experimental results.

3 Results and discussion

Figure 9 shows the S-N curve of the epoxy sample for different values of bending stresses for experimental and numerical analyses. This figure shows the experimental

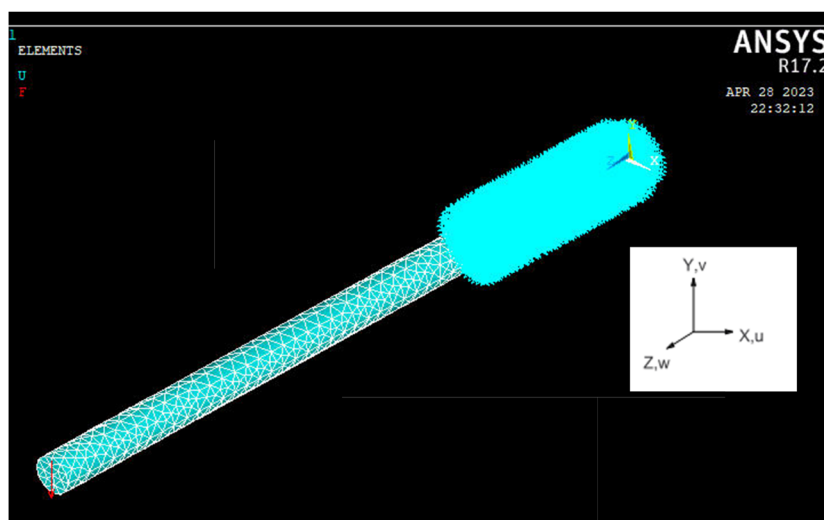


Figure 8: Numerical model of fatigue test sample.

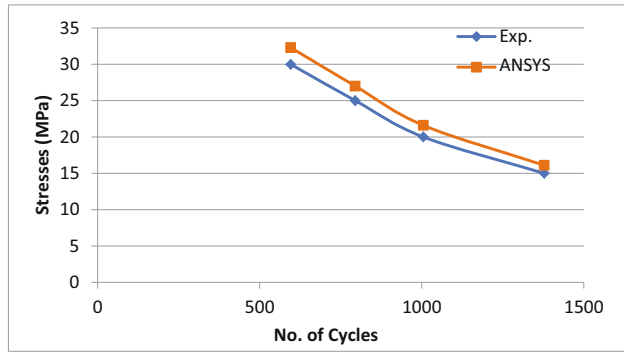


Figure 9: S–N curve for epoxy resin.

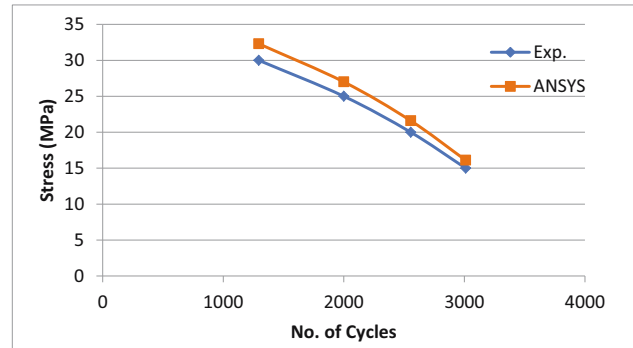


Figure 11: S–N curve for nanocomposite (epoxy + 0.5 wt% MWCNTs).

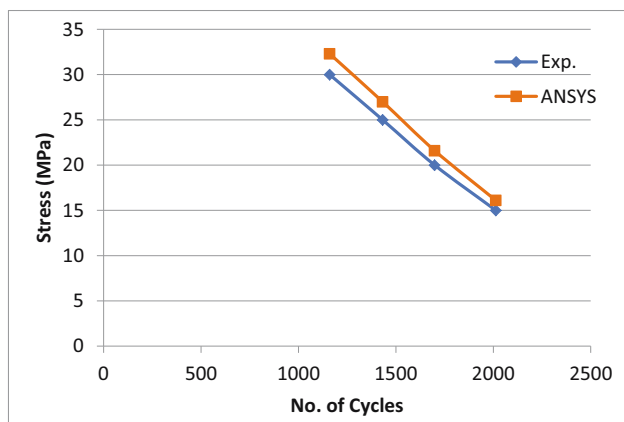


Figure 10: S–N curve for composite (epoxy + 30% carbon fiber).

low values of fatigue life for the epoxy resin that ranged between 1,378 cycles at bending stress of 15 MPa and 596 cycles at 30 MPa due to the high possibility of crack initiation and propagation in the epoxy resin with low toughness property that leads to increasing ability of fatigue failure with very low life cycles. The divergence between the experimental and numerical results was 7.5%.

Figure 10 shows the S–N curve for the composite sample of epoxy + 30% carbon fiber experimentally and numerically. This figure shows that fatigue life ranged between 2,013 cycles at a bending stress of 15 MPa and 1,160 cycles at a bending stress of 30 MPa, with an increasing ratio in fatigue life of 46% compared with epoxy only due to the positive effect of adding the carbon fiber that had high mechanical properties such as high strength and high Young modulus that improve the fatigue life of the composite.

Figure 11 shows the S–N curve for samples of (epoxy + 0.5 wt% MWCNTs) experimentally and numerically. This figure shows the effect of adding MWCNTs to the epoxy and how that affected fatigue life. The fatigue ranged between 3,011 cycles at bending stress of 15 MPa and 1,295

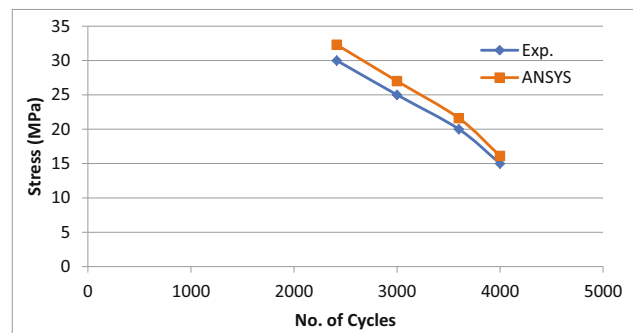


Figure 12: S–N curve for hybrid composite (epoxy + 30% carbon fiber + 0.5 wt% MWCNTs).

cycles at bending stress of 30 MPa, with an increasing ratio for fatigue life of 118% due to the positive effect of adding MWCNTs that had a high interface with epoxy and work as micro stiffeners that gave extra strength and stiffness to the composite, helped in stress transfer between nanomaterials, and reduced the ability to initiate the micro-cracks inside epoxy that may lead to starting of fatigue failure.

Figure 12 shows the S–N curve for composite samples of (epoxy + 30% carbon fiber + 0.5 wt% MWCNTs) experimentally and numerically. This figure shows the dual effect

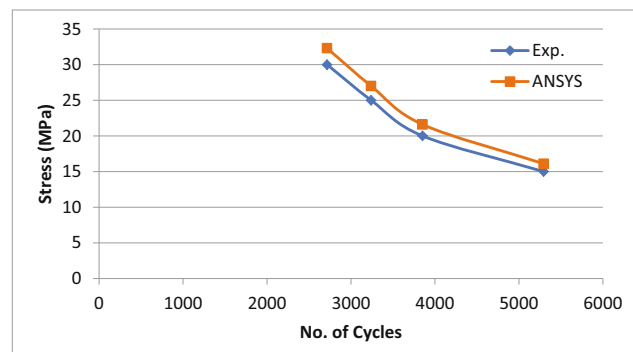


Figure 13: S–N curve for nanocomposite (epoxy + 1 wt% MWCNTs).

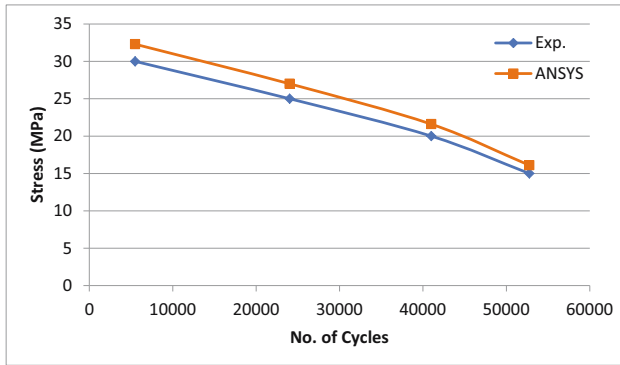


Figure 14: S–N curve for hybrid composite (epoxy + 30% carbon fiber + 1 wt% MWCNTs).

of adding carbon fiber and MWCNTs on the fatigue life of epoxy. The fatigue life ranged from 4,000 cycles at the stress of 15 MPa to 2,415 cycles at 30 MPa, thus increasing fatigue life by about 99% compared with composite samples. This improves fatigue life due to the effect of MWCNTs that have a good interface with epoxy and increase composite strength, stress transfer between nanomaterials, reduce the crack initiation ability, and increase fatigue life.

Figure 13 shows the S–N curve for samples of epoxy + 1 wt% MWCNTs experimentally and numerically. This figure shows the effect of adding 1 wt% of MWCNTs to the epoxy and how that will improve the fatigue life. The fatigue life ranged from 5,295 cycles at the stress of 15 MPa to 2,715 cycles at 30 MPa, with the increasing ratio in fatigue life by 788% due to MWCNTs increase in the strength and stiffness of epoxy, stress transfer between nanomaterials, and decrease in the ability of crack initiation and propagation that causes fatigue failure.

Figure 14 shows the S–N curve for composite samples of (epoxy + 30% carbon fiber + 1 wt% MWCNTs) experimentally and numerically. This figure shows the dual effect of adding carbon fiber and MWCNTs on the fatigue life of

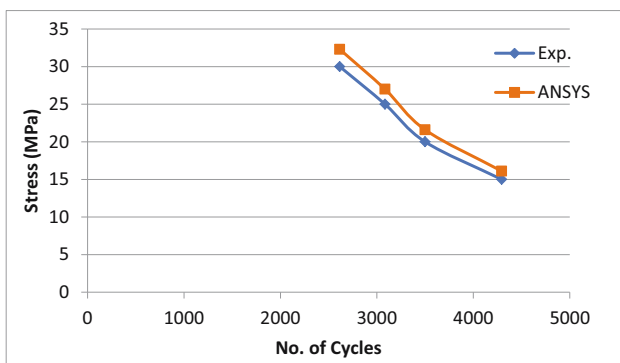


Figure 15: S–N curve for nanocomposite (epoxy + 2 wt% MWCNTs).

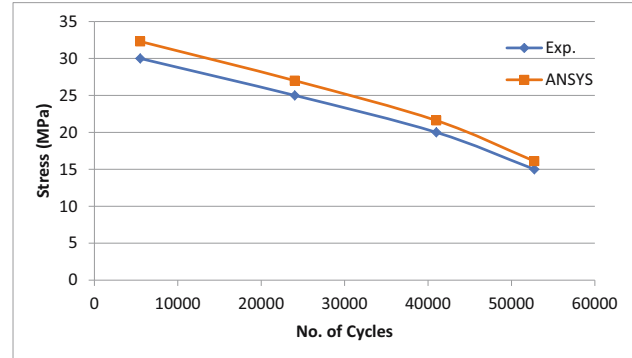


Figure 16: S–N curve for hybrid composite (epoxy + 30% carbon fiber + 0.5 wt% MWCNTs).

epoxy. The fatigue life ranged from 52,756 cycles at the stress of 15 MPa to 5,500 cycles at 30 MPa, thus increasing the fatigue life by about 2,500% compared with composite without nanomaterials. The material will be transferred from low cycle fatigue (LCF; less than 10^5 cycles) to high cycle fatigue (HCF; more than 10^5 cycles) by adding 1 wt% of MWCNTs. The improvement in fatigue life is due to the effect of MWCNTs that increase the strength and stiffness, help in stress transfer between nanomaterials, and reduce the ability of crack initiation and growth, leading to increasing fatigue life.

Figure 15 shows the S–N curve for samples of epoxy + 2 wt% MWCNTs experimentally and numerically. This figure shows the effect of adding 2 wt% of MWCNTs to the epoxy and how that will improve the fatigue life. The fatigue life ranged from 4,294 cycles at the stress of 15 MPa to 2,615 cycles at the stress of 30 MPa with an improving ratio in fatigue life of 620% due to the effect of MWCNTs that work as microstiffeners with epoxy to increase the strength, help in stress transfer between nanomaterials, and reduce the ability of crack initiation crack growth.

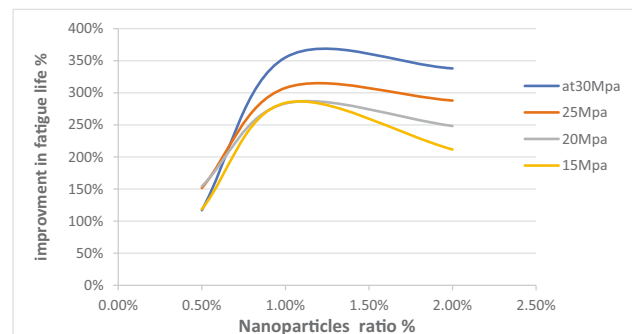


Figure 17: The improvement ratio of fatigue life of nanocomposite (epoxy + % MWCNTs).

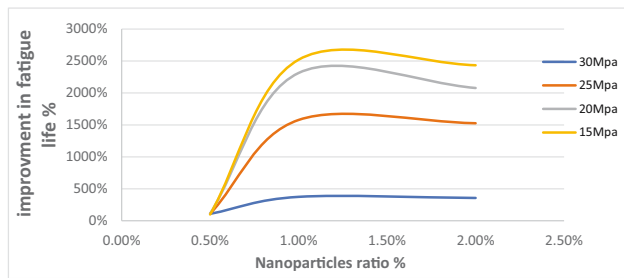


Figure 18: The improvement ratio of fatigue life of hybrid composite (epoxy + 30% carbon fiber + MWCNTs).

Figure 16 shows the S–N curve for composite samples of epoxy + 30% carbon fiber + 2 wt% MWCNTs experimentally and numerically. This figure shows the dual effect of adding carbon fiber and MWCNTs on the fatigue life of epoxy. The fatigue life ranged from 51,000 cycles at the stress of 15 MPa to 5,299 cycles at 30 MPa, increasing fatigue life by about 2,430% compared with composite without nanomaterials. The improvement in fatigue life is due to the effect of MWCNTs that reduces the crack initiation and growth ability, increasing the fatigue life.

Figure 17 shows the ratio of improvement in fatigue life for the epoxy when adding MWCNTs with different ratios of 0.5, 1, and 2 wt% experimentally. This figure shows the positive effect of adding MWCNTs and how they work as stiffeners for epoxy. It increases the strength and stiffness of the composite, reduces the ability of crack initiation and growth inside the epoxy, and enhances its fatigue life. The best-adding ratio was 1 wt%, giving the best fatigue life for epoxy.

Figure 18 shows the ratio of improvement in fatigue life for the composite (epoxy + 30% carbon fiber) when adding MWCNTs with different ratios (0.5, 1, and 2 wt%) experimentally. This figure shows the positive effect of adding MWCNTs and how they work as stiffeners for composite and reduce the ability of crack initiation and crack growths inside the epoxy that enhance epoxy's fatigue life. The best-adding ratio was 1 wt%, giving the best fatigue life for epoxy.

Figure 19 shows the SEM images for surfaces of epoxy without and with different amounts of MWCNTs. The properties of composites are greatly affected by the nature of bonding at the interface, the strength of the interface, and the mechanical load transfer from the surrounding matrix

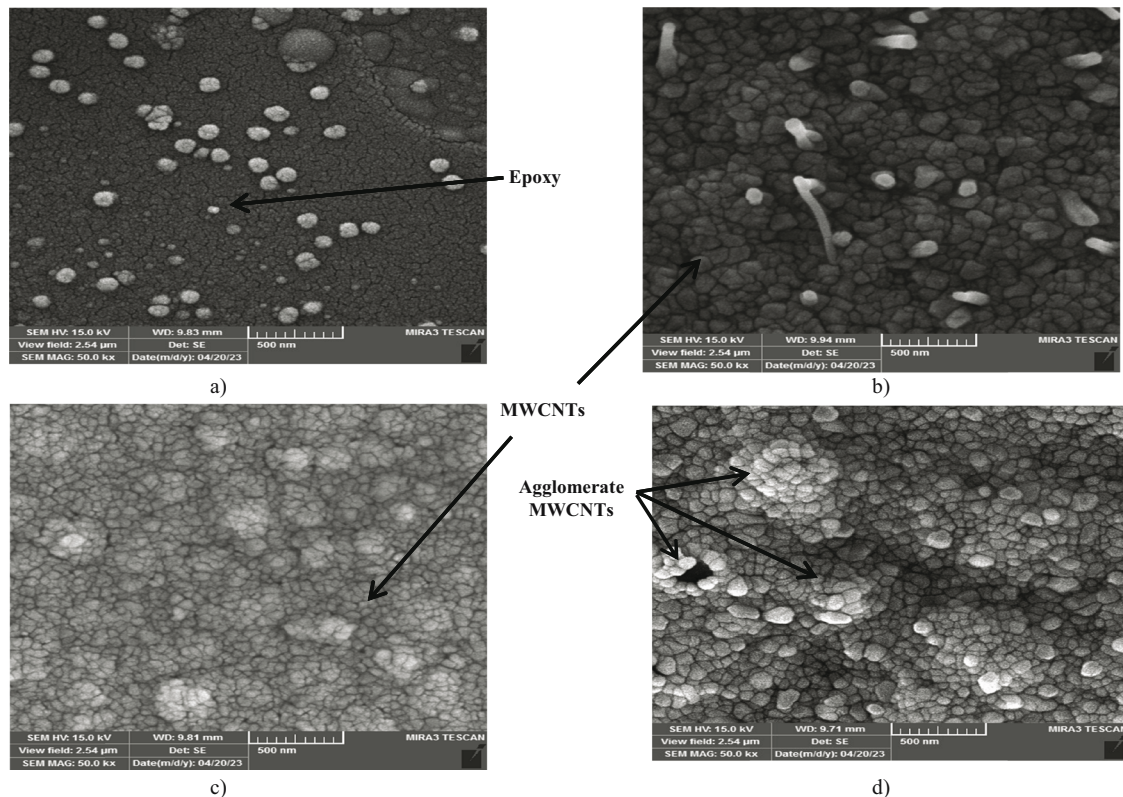


Figure 19: SEM images of epoxy and composite samples: (a) epoxy only, (b) epoxy + 0.5 wt% MWCNTs, (c) epoxy + 1 wt% MWCNTs, and (d) epoxy + 2 wt% MWCNTs.

to nanotubes. The best homogenization and distribution of MWCNTs in epoxy are shown for sample of 1 wt%. The figure shows also the agglomeration of nanomaterials when added 2 wt% of MWCNTs to the epoxy. The agglomeration points will work as stress concentration points and be as a barrier to stress transfer between nanomaterials inside the resin that decreases the strength of material.

4 Conclusions

The fatigue properties of epoxy and composite material can be improved by adding superior properties of MWCNTs that work as extra stiffeners to the materials and make a strong interface with matrix that increases the ability of stress transfer between nanotubes inside the resin. The properties of composites are greatly affected by the nature of bonding at the interface, the strength of the interface, and the mechanical load transfer from the surrounding matrix to nanotubes. The fatigue life of epoxy increased with a maximum ratio of 788% for 1 wt% of MWCNTs, while the improving ratio reached 2,500% for composite (epoxy + 30% carbon fiber) due to the presence of carbon fiber that works as stiffeners, and MWCNTs will work as extra stiffeners. The MWCNTs will decrease the cracks' ability to initiate and grow inside the composite due to the super strength property of MWCNTs, especially when using the ratio of 1 wt% and its very good improvement when compared with Turaka et al. [9], Genedy et al. [10], and Shokrieh et al. [12]. The unique findings of this work are transferring the material from LCF to HCF. The improvement ratio in fatigue life will start decrease to 2,430% when using 2 wt% of MWCNTs as nanomaterials start to agglomerate inside the resin. This agglomerated point works as stress concentration points and a barrier for stress transfer between nanotubes and that will reduce the positive effect of adding the MWCNTs. The numerical model simulates the experimental tests done with a divergence of 7.5% due to the effect of using numerical solution hypotheses that tried to approach the experimental conditions.

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Data availability statement: Most datasets generated and analyzed in this study are in this submitted manuscript.

The other datasets are available on reasonable request from the corresponding author with the attached information.

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