The Effects of Adding MWCNTs on Fatigue properties of Epoxy and Composites Reinforced with Pre-Stressed Fiber

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https://doi.org/10.31972/iceit2024.030

Abstract:

The Failure due to fatigue is responsible of more than 85% materials failure, which occurs under ultimate strength. The fatigues properties of polymers can be improved by utilize reinforcement materials (fibers, particles, etc) to form composite materials with good properties. The improving in fatigue properties for composite can be done by subjecting the reinforcement fibers to preload and make them under prestressing that give extra stiffness to material. The Nanoparticles can be added to the resin to enhance the mechanical properties. In this research, the tensile prestress of (2.5, 5, 7.5, and 10MPa) was applied on fibers to make material under compression after curing. Nanoparticles of Multi Wall Carbon Nanotubes (MWCNTs) added to epoxy resin and composite (epoxy +carbon fiber) with various weight ratios (0.5, 1 and 2wt.%) . The fatigue test results showed the fatigue life for the epoxy enhanced by 350% at 1wt.% of MWCNTs, while it reach to 2850% for composite at 1wt.%MWCNTs and 10MPa of fibers prestressing . The amount of 2wt% MWCNTs will reduce the fatigue life for epoxy and composite due to agglomeration phenomena of MWCNTs inside the matrix and been as weakness zone inside the resin that may decrease the fatigue properties.

Keywords: Epoxy, Composite, Carbon Fiber, MWCNTs, Fatigue properties

1. Introduction

Polymeric composites (FRP) have good strength /weight ratio with high corrosion resistance and thermal insulation. The one FRP used in the mechanical application is carbon fiber reinforced polymer (CFRP) as it has high specifications like tensile properties, toughness, and corrosion resistance [1]. So, CFRP is used in many applications such as aircraft wings, automotive structures, blades of wind turbines, and other applications subject to load fluctuation. Fatigue is the failure that occurs for materials when subjected to repeated load that gradually decreases the working life of the material [2]. Fatigue failure happens for material when it can no longer withstand loads. Therefore, the CFRP fatigue properties improvement gained importance [3]. The pre-stressing of fibers is one of the useful method can be utilized to enhancing CFRP properties. The technique of fiber pre-stressing in composite during material solidification has grown in the last few years. It has enhanced the composite's mechanical properties without adding fibers, dimensions, mass, and extra costs. [4,5]. In addition to using the fiber pre-stressing technique to improve mechanical properties, the adding nanoparticles to the resin was also used to improve composite materials , and one of the nanoparticles types used were Multi-Wall Carbon Nanotubes (MWCNTs) that had superior mechanical strength, high elastic modulus, ability to absorbing shocks and so on[6]. **Nawras et al**., Studied experimentally the impact of using different fabric pretension on the fatigue properties of composite (Polyester + E-glass woven fiber) with fiber pre-stress levels up to 100MPa. The results showed the optimum pre-stress to fibers was 50MPa, and the fatigue life was extended by 43% [7]. **S.chen et al**. studied impacts of adding (CNTs) to composite of (epoxy resin + fibers of glass) on its mechanical properties. Results showed the improving in impact strength and interlaminar shear strength by a 189% and 28% respectively [8]. **Y.M. Jen and W. L. Ni,** studied experimentally the impact of adding the mixing of (GNPs + CNTs) on fatigue properties of composite. Results showed that the sample with a ratio of 1:9 GNPs/CNTs has clear improving in fatigue properties [9]. **S. Turaka et al.** studied impacts of using (CNTs + GNPs) between (0.1-0.3wt. %) to the composite of (epoxy resin/ fibers of glass) on fatigue properties. The results showed increasing in life of fatigue by 56% at MWCNT/GNPs of 0.2wt.%[10]. **M. Genedy et al.** concluded the impact of using 0.5 and 1wt. % of CNTs with the Glass Fiber Reinforced Plastics (GFRP). Results showed the improvement in fatigue life of GFRP may reach to 1143% [11]. **L. Böger et al.** conclude effects of using carbon nanotubes with composite of (epoxy resin+ fibers of glass).Results showed good effect of adding CNTs on increasing the fatigue life of composite [12]. **M. Shokreih et al**. studied the effect adding (0.1- 0.5wt. %) of GNPs to epoxy resin. The results showed increasing about 2700% in life of fatigues for composite material compared with control samples [13]. **L.Mahdi, and A.Alithari**, presented the effects of using GNPs to composite with fibers prestressing on the mechanical properties of composite (epoxy/Kevlar fibers). The results showed the enhancement in flexure properties and stiffness may reaches to 90% and 134% respectively. [14].Current research presents the possibility of improving fatigue life of epoxy and composite (epoxy resin/fibers of carbon) by adding MWCNTs and using fiber prestressing technique.

2. Used Materials and Methods of Experiment:

2.1 Materials**:** The samples of composite materials for testing made from epoxy resin and reinforced materials such as (carbon fibers and Nanoparticles of MWCNTs):

A- Epoxy Resin**:** The matrix material of composite made from epoxy has a trading name (Renksan-Renfloor HT 2000) with mixing ratio of resin to hardener of (2:1), with good specifications like (low creep, medium mechanical properties, and good transparency). Epoxy resin was used to produce a nanocomposite matrix system to develop a hybrid CF / Nanoparticle / Epoxy composite system; Table 1 shows the specification of resin matrix [15].

Table 1: Properties of Epoxy. [15]

Specification	**Bulk Density [g/cm³]**	**Compressive Strength [Pa]**	**Durability [N/m$^{3/2}$]**	**Tensile Property [Pa]**	**Flexural Property [Pa]**	**Elastic Property [Pa]**
Value	1.05	$70*10^6$	0.6	$27*10^6$	$63*10^6$	$2.8*10^9$

B- The reinforcement: The fibers of carbon can be used as stiffeners made by company of Sika in Italy with properties can be show in below. [16]

Table 2: Carbon Fibers Properties. [16]

Specification	**Bulk Density [g/cm³]**	**Tensile s Strength [Pa]**	**Elastic Modulus [Pa]**	**Appearance**
Value	1.8	$4000*10^6$	$230*10^9$	Black

C- MWCNTs: Multi-wall carbon Nanotubes are used as nano additives to epoxy to improve its properties. It made by CCVD and using concentrated acid chemistry with a purity of 90 % to purified it. It has an outer diameter of (20 – 40 nm), length of (10-30 μm) as shown in table 3 [17,18].

Table 3: Properties of MWCNTs. [18]

Specification	**Bulk Density [g/cm³]**	**Tensile Strength [Pa]**	**Modulus of Elasticity [Pa]**	**Surface Area [m²/g]**	**Fusion Temp. [°K]**
Value	2.1	$140000*10^6$	$1000*10^9$	70-250	4025

2.2. Preparation of Metallic Mold:

Method of hand layout are used to produce epoxy and composites samples from epoxy resin , reinforcement of carbon fiber and MWCNTs at weight fractions of (0.5, 1, and 2wt. %).The metallic mold (open type) that used to produce samples for test can be shown in figure1

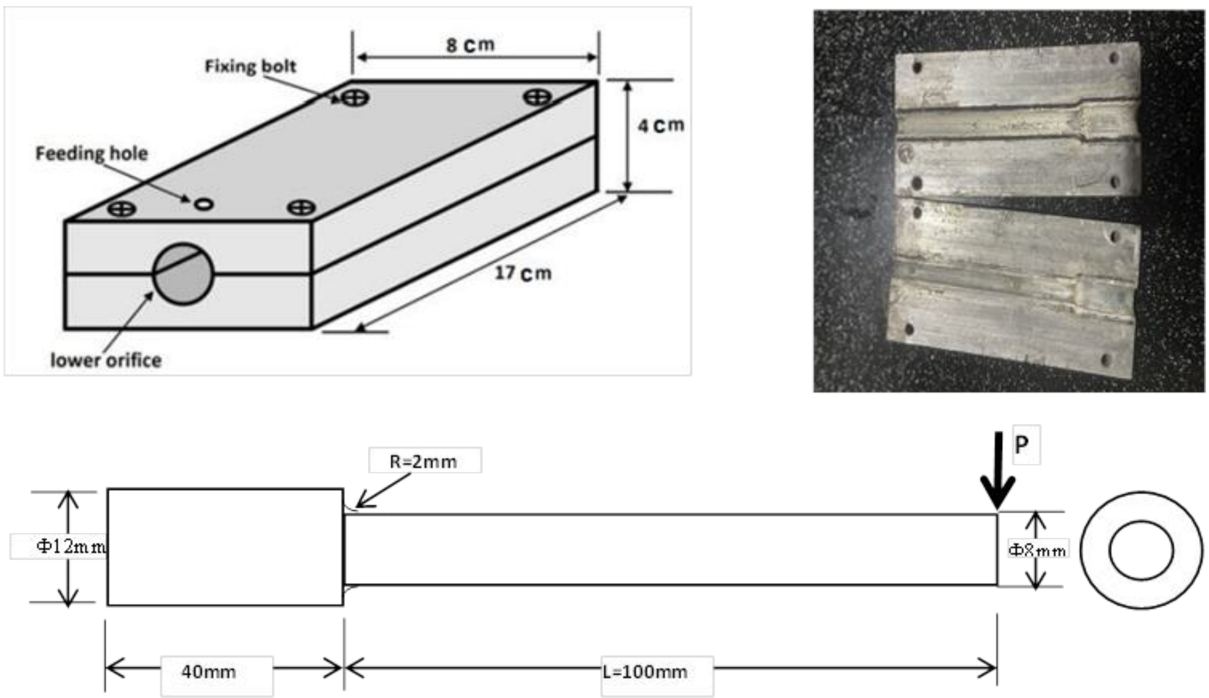


Figure.1.Metalic Mold and fatigue test sample with dimensions

2.3 Applying the Pre-stress to the Fibers:

The fiber expose to pre-determined tensile load (under elastic strength) at process of curing. After fully solidifies for composite, the tensile stress is removed, and the composite will be under compressive stress in the longitudinal direction of fibers to return to its original length. This compressive stress will give more material strength, decrease the probability of crack initiation, reduce flaws and cracks propagation rate, and ensure the alignment of fibers inside the matrix as sown in figure 2A, and metallic mold will be supported by a rig of wood in the vertical direction, and carbon fiber will be fixed from one end and subjected to tensile load by dead loads on the other end. The levels of fiber pre-stressing applied to the carbon fiber during the curing process are (2.5, 5, 7.5, and 10MPa) as shown in Figure 2B.

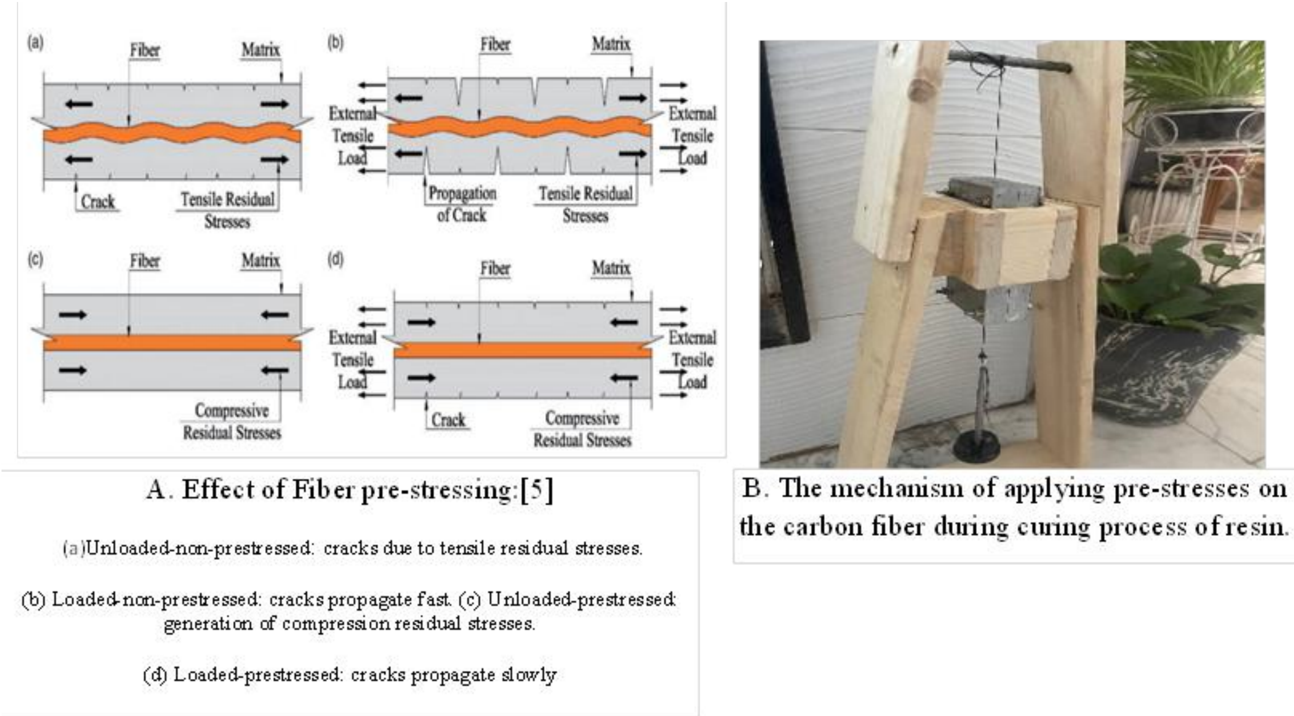


Figure 2: A- The fiber pre-stressing Effects
B- The applying pre-stressing on the fiber in composite during curing process

2.4 Method of MWCNTs Mixing with Resin of Epoxy: [19, 20]

The mixing of MWCNTs with epoxy resin by were done with three weight fractions (0.5, 1, and 2wt. %).The first step was MWCNTs with epoxy resin mechanically by using shear mixer of speed 2000 r.p.m for half hour then subjected to sonication of high-power (250W) by sonic dis-membrator type MTI for half hour. The power of sonication increased progressively till reaches to 250W. The ice jacket used to keep the temperature of mixture lower than 30°C to prevent temperature increasing in sonicating pot. Vacuum oven used at 40°C for half hour to reduce the ability of bubbles formation and then the hardener will added to the mixture by rate of one/two and then subjected to mechanical mixing for half hour. The mixture must be degasses under vacuum at 25 ± 5 °C for half hour to remove the bubbles, then using slowly injection method to supply the mixture to the metallic mold. The mathematical model of Halpin-Tsai is used to calculate the elastic modulus for samples of Nanocomposite (epoxy+MWCNTs) based on shape and physical properties of nanoparticles and matrix. [21]

$$E_C = \frac{1+2\frac{w}{t}\eta V_n}{1-\eta V_n} E_m \qquad (1)$$

Where: $\eta = \frac{(\frac{E_n}{E_m}-1)}{(\frac{E_n}{E_m}+2\frac{w}{t})}$, ($E_c$) is elastic parameters for composite, (E_m) is elastic parameters of matrix, (E_n) is elastic parameters for Nanoparticles, (V_n) is volume fraction for Nanomaterials, and (w/t) is factor of the shape

$$V_n = \frac{\frac{w_n}{\rho_n}}{\frac{w_c}{\rho_c}} \qquad (2)$$

Where: (w_n) is weight of Nanomaterial, (ρ_n) is density of Nanomaterial , (w_c) is weight of composite , and (ρ_c) is density of composite.

Table 4 show the samples that produced for fatigue test

Table 4: Fatigue test samples

No.	Specimen Group	Symbol	Average	pre-stress MPa
1	(A)Epoxy without Nanomaterials	A1-1,A1-2,A1-3	A1	no fiber
2		A2-1,A2-2,A2-3	A2	0
3		A3-1,A3-2,A3-3	A3	2.5
4		A4-1,A4-2,A4-3	A4	5
5		A5-1,A5-2,A5-3	A5	7.5
6		A6-1,A6-2,A6-3	A6	10
7	(B) with MWCNTs 0.5wt.%	B1-1,B1-2,B1-3	B1	no fiber
8		B2-1,B2-2,B2-3	B2	0
9		B3-1,B3-2,B3-3	B3	2.5
10		B4-1,B4-2,B4-3	B4	5
11		B5-1,B5-2,B5-3	B5	7.5
12		B6-1,B6-2,B6-3	B6	10
13	(C) with MWCNTs 1wt.%	C1-1,C1-2,C1-3	C1	no fiber
14		C2-1,C2-2,C2-3	C2	0
15		C3-1,C3-2,C3-3	C3	2.5
16		C4-1,C4-2,C4-3	C4	5
17		C5-1,C5-2,C5-3	C5	7.5
18		C6-1,C6-2,C6-3	C6	10
19	(D) with MWCNTs 2wt.%	D1-1,D1-2,D1-3	D1	no fiber
20		D2-1,D2-2,D2-3	D2	0
21		D3-1,D3-2,D3-3	D3	2.5
22		D4-1,D4-2,D4-3	D4	5
23		D5-1,D5-2,D5-3	D5	7.5
24		D6-1,D6-2,D6-3	D6	10

3. Test of Samples:

3.1 Fatigue Test

The standard specification of ASTM E467-21[22], were used to test the samples towards the fatigue by using flexure load with rotating type, and using the machine of Fatigue tester from GUNT in Germany as in Figure 6. The rotating samples subjected to flexure stress of 15, 20, 25, and 30MPa with stress ratio R= -1(fully reversed load), and record the number of cycles (fatigue life) for three samples and get the average value for each test.

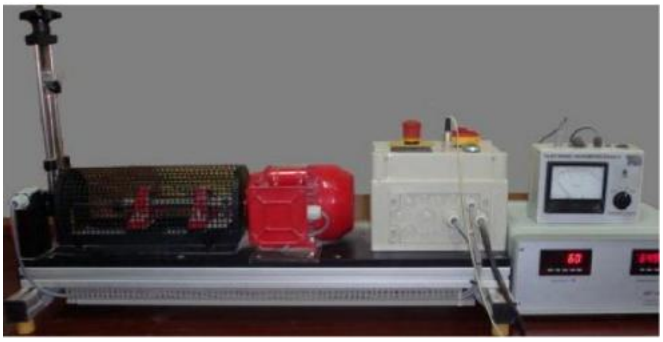


Figure.3. Fatigue tester Machine

Flexural stresses can determined by:

$$\sigma_b = \frac{M * y}{I} \quad (3)$$

Where: M=F* ℓ (4)

F----Applied flexure load (N)

ℓ----Samples active length (m)

y---- Radius of sample cross-section (m)

I-----Second moment of area (m^4)

$$I = \frac{\pi * d^4}{64} \quad (5)$$

d-----diameter of the sample (m)

3.2 SEM Test

The scanning electron microscope is used to get images of MWCNTs homogeneity inside the resin and how MWCNTs distribution effects on the performance of material especially on fatigue state.

4. The Results and Discussion:

From Figure 4 can show the improvement in fatigue life for Nanocomposite samples (Epoxy resin +MWCNTs) experimentally under various bending stress values. Also can be showed the impact of add different values of Nano carbon tubes to the matrix of epoxy and how fatigue properties can be improved. Comparison between epoxy and epoxy with MWCNTs can show the positive effect of Nanoparticles of Carbon tubes in increasing the life of fatigue for the epoxy due to the superior properties of Nano carbon tubes that work as stiffeners and reducing the probability of cracks creation and propagation into the epoxy resin. Higher improvement in fatigue life occurs when using 1wt.% of MWCNTs, which reaches 350% at 1wt.% of MWCNTs. The reduction in improving the ratio of fatigue life when using 2wt.% occur due to agglomeration phenomena of nanoparticles into the epoxy will decrease the required effect of MWCNTs and reduce the fatigue life that can be shown in figure 5 for epoxy and epoxy+ MWCNTs.

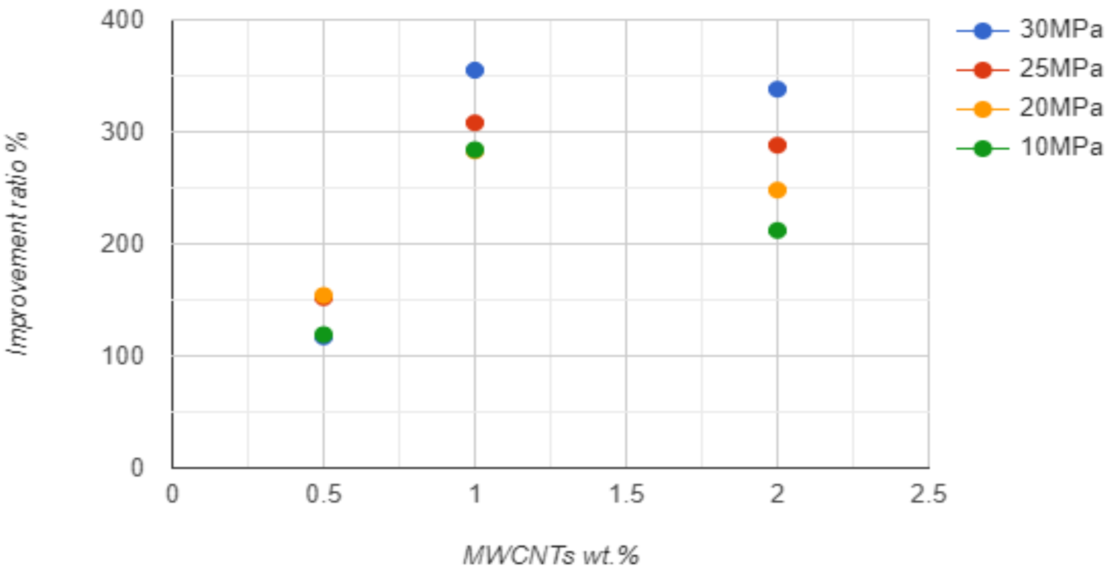


Figure.4.Fatigue life improvement for epoxy under different value of bending stress

Figure 5 shows improvement in fatigue life for samples of composites (epoxy resin +30% fibers of carbon + MWCNTs). The best result was a sample of 1wt.% MWCNTs, due to the beneficial effects of high properties nanoparticles in decreasing the cracks initiation and propagation inside the composite and work and cracks stoppers. The best improvement ratio was for fatigue life to reach 2500% at 1wt.% of MWCNTs , compared with pure epoxy and reduced to for sample of 2wt.% due to MWCNTs agglomeration inside the epoxy resin as in figure 6 .

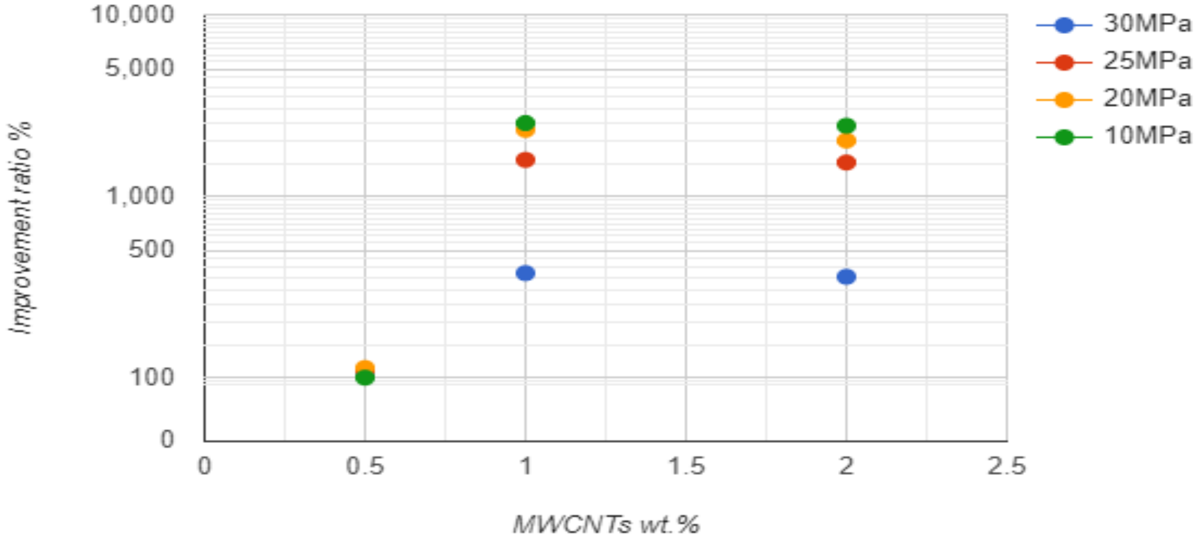
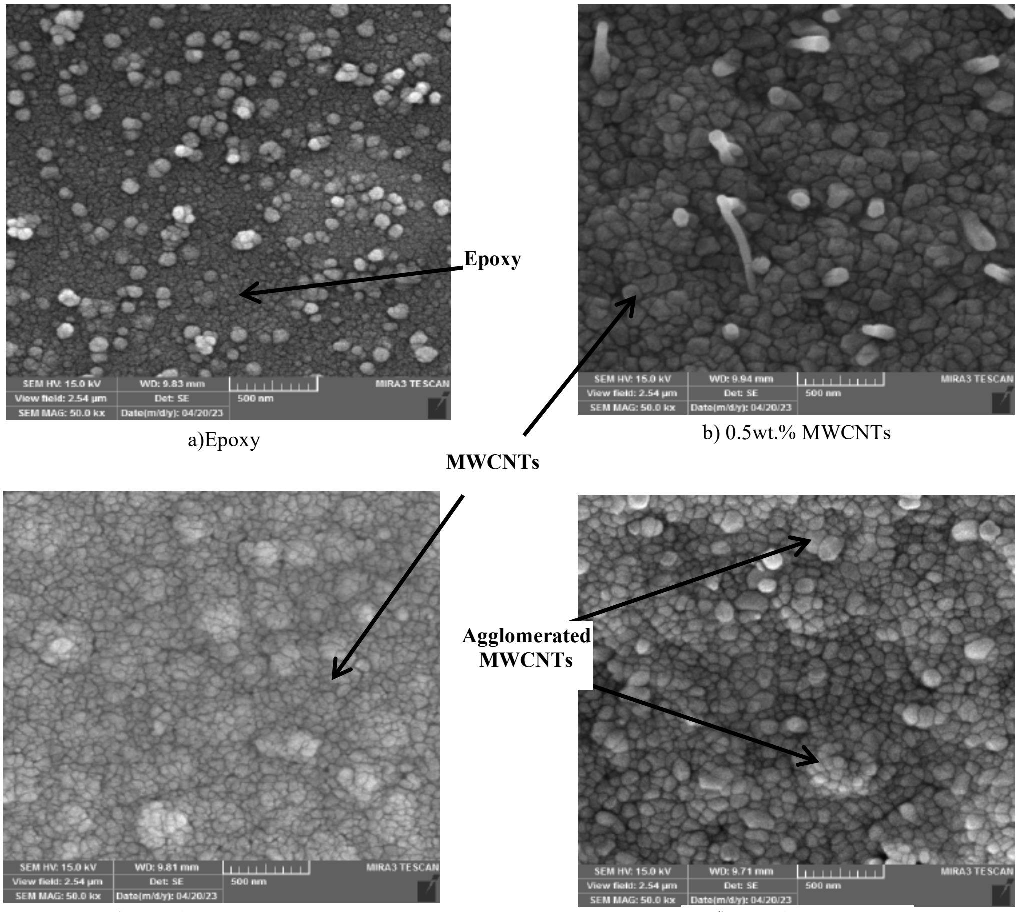


Figure.5. Fatigue life improvement for composite samples of (epoxy+30%Carbon Fibers) with different value of



a)Epoxy b) 0.5wt.% MWCNTs

c) 1wt.% MWCNTs d) 2wt.% MWCNTs

Figure.5.Images of SEM

a) Only resin. b) Resin with 0.5wt.% Nanomaterial. c) Resin with1wt.% Nanomaterial.
d) Resin with 2wt.% Nanomaterial

5. Conclusions:

Epoxy resin and composite material have low fatigue strength and fatigue life, so it needs some improvement in their properties to enhance it to withstand the circumstances of operation, increasing the life of work. This research showed that the effect of using Nanomaterials of Multi-Wall Carbon Nanotubes (MWCNTs) would increase fatigue life for epoxy by about 350% compared with net epoxy due to the super properties of MWCNTs that contribute to increasing strength, stiffness, shock resistance, and decrease the probability of micro-cracks creation and propagation into epoxy resin. The effect of fiber pre-stressing will improve the fatigue life with a good ratio by increasing the stiffness and strength of composite that enhance the fatigue life. Adding MWCNTs to composite subjected to fiber pre-stressing will increase the fatigue life to high percent depending on the ratio of MWCNTs addition and value of fiber pre-stressing. The max improvement in life of fatigue reach to 2850% for samples at 1wt.% MWCNTs and 10MPa fiber pre-stress, which change the material from low cycle fatigue (less than 10^5 cycles) type to high cycle fatigue (more than 10^5 cycles).Adding MWCNTs more than 1wt% (such as 2wt.%) will reduce the strength of composite as agglomerated nanomaterials inside the epoxy , works as stress zones for stress concentration , and act as barriers to load transfer between epoxy and fibers. These agglomerating zones acts as blemishes inside the resin matrix, and may causes decreasing in the material stiffness due to loosely bounded particles had no capacity to transfer loads from one particle to other.

Acknowledgement: The Authors want to express the thanks and gratitude for the help support offered by the faculties of labs of Nanotechnology and Advanced Materials Research Unit/ University of Kufa and Labs of Mechanical Engineering Department/the University of Kufa.

Conflicts of Interest: Authors declares there are no exist conflict of interest in this submission.

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