Effect Different Multi-Walled Carbon Nanotubes MWCNTs Type on Mechanical Properties of Epoxy Resin Nanocomposites

Khalid R. Al-Rawi[1], Adawiya j. Hedar[2] , Olfat A.Mahmood[3]
[1]Department of physics, collage of women science,University of Baghdad.

[2]Nanotechnology and Advanced Materials Research center,University of Technology

[3]Department of physics, collage of women science,University of Baghdad.

ABSTRACT

Different type of Multi-walled carbon nanotubes (MWCNTs) long and short were used as reinforcement in epoxy resin with weight percentages (0.1,0.2,0.5,1.0,1.5,2.0,2.5,3.0,3.5,4.0,4.5,&5) wt.% respectively . The use direct mixing processes to prepared (Epoxy / MWCNTs) nanocomposites . Ultrasonic mixing process was used to disperse the nanotubes into the epoxy resin system. The results show improves the mechanical properties with increase percentage weight less than 2% . and then decreased with further increasing the MWCNTs content.The result show the long carbon nano tube have mechanical properties better than the short carbon nanotube.
Keywords: Multi walled carbon nanotubes (MWCNTs), Epoxy, Nanocomposites.

1.INTRODUCTION

Epoxy resin has weak mechanical properties, the relatively weak mechanical properties of epoxy resin have pre-vented its application in the components that demand high mechanical strength and stability.[1] It is well known that the physical properties of cured epoxy resins depend on their structure, the curing extent, and the curing time and temperature. For this reason, it is necessary to know and to understand the relationship between the network structure and the final properties of the material, in order to obtain resins suitable for high performance applica tions.[2]Individual carbon nanotubes (CNT) exhibit extraordinary mechanical and electronic properties.[3] Recently, carbon nanotube (CNT) as the filler in polymer matrix has attracted considerable interest due to its unique mechanical, thermal, and electrical properties. Due to their high aspect ratio and huge surface area, CNTs have strong tendency to agglomerate, which leads to inhomogeneous dispersion in the polymer matrix. Various techniques for dispersing CNTs have been extensively investigated and compared .[4] Multi- walled carbon nanotubes(MWCNTs) have unique atomic structure, very high a spectratio and extra ordinary mechanical properties ,making ideal reinforcing materials in nanocomposites.The recent researches have extensively focused on the epoxy resin -based carbon nanotube nanocomposites because of there applications in the electronics, a aeronautics and a astronautics industries. Some early research results just showed barely stronger or weaker carbon nanotube-epoxy nanocomposites than the net epoxy.[5,6]Such low reinforcing ability of the nanotubes in epoxy nanocomposites ,which is sometimes observed ,can be explained by a number of reasons . One is lack of interfacial adhesion, which is critical for load transfer in composites .Indeed carbon nanotube surfaces are atomically smooth, which may limit the transfer of load from matrix to nanotubes reinforcement(2.3).Another reason is the poor dispersion of nanotubes in the polymer matrix, which is also significant for the fabrication of reinforced nanocomposites.[7] In fact the dispersion of the CNTs in the matrix system is further main problem to be solved for nanotube-reinforced polymers .Nano-scaled particles exhibit an enormous surface area ($1000m^2$/g and more),which is several orders of magnitude larger than the surface of conventional fillers . This surface area acts as an interface for stress transfer, but is also responsible for the strong tendency of the CNTs to form agglomerations. An efficient exploitation of the CNT properties in polymers is therefore related to their homogenous dispersions in the matrix or an exfoliation of the agglomeration and a good wetting with the polymer. Various dispersions' methods (stirring, extrusion , sonication ,etc) for the distributions' of CNTs in polymers have been used . A common technique used in order to disperse CNTs is the sonications technique.[8,9] A pulsed ultrasound exfoliates agglomerates and disperses CNTs in the matrix effectively. To take full advantage of these unique mechanical properties , optimization of nanotube-polymer interface properties such as wet ability and adhesion is required . Carefully comparing these controversial results indicates that the stiffness of epoxy matrix seems to be a key point. In summary materials properties of epoxy are supposed to be improved by addition of carbon nanotubes . The focus on improving the mechanical properties led two common key issues to be investigated : (1) dispersion and (2) interfacial adhesion.[10] Ayatollahi et al.[11] investigated the effect that changing carbon nanotube aspect ratio had on electrical and mechanical properties of epoxy composites. Instead of changing the length, however, Ayatollahi et al. focussed on varying the diameter of the nanotubes. The results

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indicated that the best response in electrical conductivity and fracture toughness were observed in samples containing higher aspect ratio carbon nanotubes.

2. EXPERIMENTAL

Materials and Sample Preparation

The raw materials used to prepare the samples are; Epoxy as a matrix (Nitofill, EPLV with Nitofill EPLV hardener from Fosroc Company). The mixing ratio for resin and hardener is 3:1 and gelling time 40 minute at 35 ^{o}C, mixed viscosity 1.0 Poise at 35 ^{o}C; and the nanoparticle fillers are MWCNTs manufactured by NANOSHEL LLC USA.

Table (1) Materials properties

Materials	Length (μm)	Diameter (nm)	Surface area (m^2/g)	Purity %
Epoxy (EPLV)	--------	----------	-------------	-------
MWCNT LONG	~50	5-15	233	>97
MWCNT SHORT	3-8	20-30	90-350	99.8

Hand layup technique has been used to prepare specimens which is cost effective and easy process of manufacturing . Amount of epoxy was mixed with Different weight percentage of MWCNTS (1,0.2,0.5,1.0,1.5,2.0,2.5,3.0,3.5,4.0,4.5,&5wt.%) use direct mixing process by added MWCNT to epoxy resin .The mixture was sonicated for (30 minute)by ultrasonic path and then being stirred for (2 hr) to disperse the MWCNTs in polymer matrix . Using vacuum system (10^{-2} bar) to remove the bubble before molding After that the hardener added to mixture and stirred for (4 minute) .The mixture casting in a glass molds. The samples left for 72 hours before pulling out and leftin the vacuum chamber for (14) days before any test to get better curing conditions.

3. CHARACTERIZATION OF MWCNTs AND THEIR EPOXY COMPOSITES TESTING

3.1 Bending test

Three point bending test has been used to investeged the mechanism of crack propagation . Instron 1122 was used and the cross head speed were fixed (1mm/min) . load -deflection curves were obtained for different samples. The support span (distance between the supports) was depending on the specimen at the middle of support span for rectangular sample under a load in a three –point setup:

$$F.S = \frac{3PL}{2bd^2} \dots\dots\dots\dots (1)$$

$$E_b = \frac{ML^3}{4bd^3} \dots\dots\dots\dots (2)$$

F. S :- Flexural strength (N/mm^2)
E_B:-Flexural modulus (Mpa).
P:-The applied load at the highest point of (load- deflection) curve (N).
L:- The span length (cm) .
b:- The width of test specimens (cm).
d:- The thickness of test specimens (cm) , M:- The line of curve load –deflection

3.2 Micro-hardness test

The Vickers hardness (Hv) of the composites was measured by a micro- hardness testing machine (TH714 Time Group Inc). Each specimen was measured three times at random locations respectively .the average value of tests was recorded as the hardness value. (0.098N) force and (15 seconds) of dwell time were used in indentation.

3.3 Impact test

In order to evaluate the dynamic toughness, the Charpy impact value measured using a standard impact fracture energy measurement MODEL(XJJ series) ,ASTM standard(6110) , Laryee ,China Origin. The Charpy impact value is expressed by a following equation.

$$E = M \times \left[(\cos\beta - \cos\alpha) - (\cos\acute{\alpha} - \cos\alpha) \times \frac{\alpha+\beta}{\alpha+\alpha} \right] \dots (3)$$

$$G_s = \frac{E}{A} \dots\dots\dots\dots\dots (4)$$

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E: Impact fracture energy (KJ).

G_s: Impact strength (KJ/m^2)

M: Impact force torque (N .m).

β: pendulum raising angle after samples breaking .

α: actual measurement value of pendulum rising angle.

Ȑ:Avoding angle .

A: Area of the sample (m^2).

4. RESULTS AND DISCUSSION

FTIR spectra can provide further evidence for the variation in the thermal and mechanical properties of the different samples in terms of their molecular structures   . The FTIR spectra of the neat epoxy resin and MWCNT/epoxy nanocomposites are presented in Figure 1 and 2 .The FTIR spectrum of commercial epoxy resin is given in Figure.3 The bands at 2966.52 cm $^{-1}$,914.26 cm^{-1} and 1246.02 cm^{-1} , 830 cm^{-1} denote epoxy group . In Fig.4 , the FTIR of (epoxy /MWCNTs) shows the peak   (2966.52 cm $^{-1}$)   remove and   the peak area of the band at 914.26 cm^{-1} was increased it streaching to 937.40 cm^{-1} and decreased in the peak of -OH groups . It can be noted that the less intense peak at 3506.59 cm^{-1} to 3417.86 cm^{-1} . The good dispersion of MWCNT is a result of interaction between between epoxy resin and MWCNTs lead to occurs the change in peaks of FTIR epoxy groups.
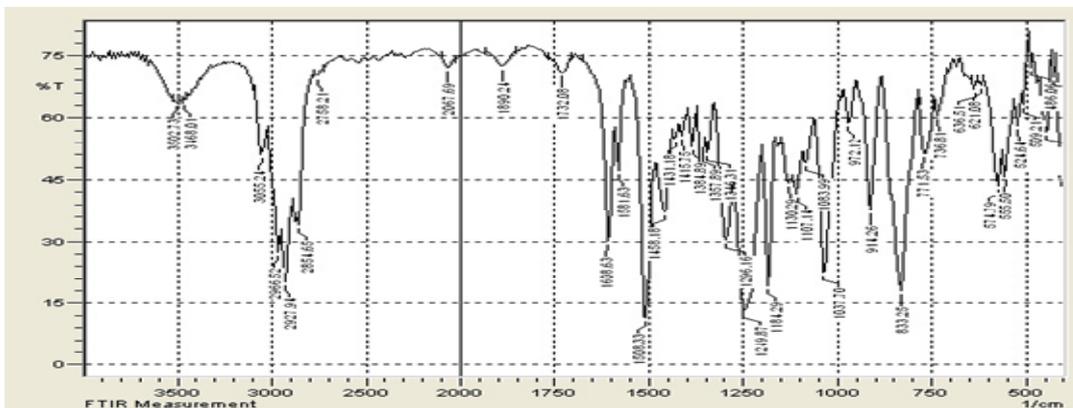
**Figure .1** FTIR spectrum of epoxy resin
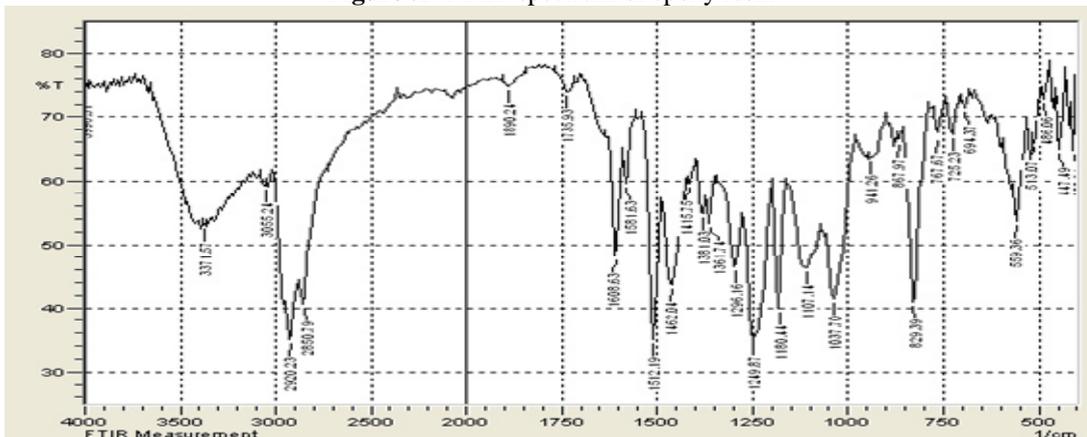
Figure.2 FTIR spectrum of epoxy /MWCNTs nanocomposites

Flexural modulus of pure resin   was found to be(75Mpa) . Figure.3 and Figure.4 shows the Flexural strength and Flexural modulus of epoxy nanocomposites   increase with various addition ratios of MWCNTs . It showed that the content of LMWCNTs (0.1 - 2 wt.%) enhance and increase the flexural strength from (75Mpa for epoxy resin to 151.7 Mpa) or this percentage lead to increase flexural strength 100% compare with the pure epoxy, and Flexural modulus can be improved from (1.4 Gpa) to the maximal value (2.34 Gpa). Creating attractive polar forces, and Van der-Waals bonding between chains and nanotubes this lead to increase constraint between tubes/epoxy chains and epoxy chains itself, complicated epoxy chains which approached one another, will reducing free volume space. This effect of MWCNTs leads epoxy chains to bear extra loading. After 2 % wt of MWCNTs flexural strength begin to decrease from maximum enhancement. Increasing the weight percentage of MWCNTs lead to increasing the constraint between epoxy chains, which decreases the length of epoxy chains lead to decreasing flexural strength, while flexural strength depends highly on chains length and free volume space (reducing free volume space indicate the increase in immobility of epoxy chains of matrix system), Flexural strength after the 2% wt. percentage of MWCNT still higher than that of epoxy resin

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because of Van der -Waals bonds which is weak bond but with huge numbers. .[12] When the content of MWCNTs increased further, flexural properties of nanocomposites decreased. The less efficient improvement at higher content is attributed to an increasing amount of observed agglomerates that cannot effectively transfer outside force. The local agglomerate formed stress point leads to the generation of fracture source, which decrease the mechanical properties .[13]. Figure.5 shows that in both type of MWCNTs the hardness was increased with increase MWCNTs content untile 1.5% after this percentage the values of hardness being to decrease .Increased hardness values due to an over lap and stacking , which reduced the movement of polymer molecules ,which lead to increase the resistance of material to scratch ,cut, and becoming more resistance to plastic deformation . Hardness of material depended on the type of forces that bind between atoms in the material.[14] The strong linkages at the interface Phases Nano carbon and epoxy result to increase the coherence of the mixture . The values of charpy impact strength for the net epoxy and Epoxy/MWCNTs Nanocompositesare shown in Figure.6 .Based on values ,the impact strength of the neat epoxy increased with increasing different type of LMWCNTs content,reaching to (1.5 wt.%). Which was attributed to the good dispersions of the nanotubes in the matrix and good reaction between the epoxy and the grafted nanotubes. Investigation of fracture surface in nanocomposites revealed that narrower crack-tips underneath the advancing cracks were more efficiently bridged by the nanotubes in epoxy/LMWCNTs resulting in an increased resistance aginst crack propagation more than the SMWCNTs .[15] The reduce of impact strength when increase the MWCNTs concentration lead to agglomerate.[16]
. All reduction in flexural modulus , flexural strength , hardness , and impact strength value of MWCNT/epoxy nanocomposites due to formation of agglomerates of nanotubes for both type inside polymer matrix that reduced reinforcing effects of the CNTs by acting as flaws in the resin .

5. CONCLUSIONS

1- The Flexural strength increase two times compare with the value of pure epoxy at 2% LMWCNTs concentration
2- Increase of the impact strength and hardness values with increase MWCNTs concentration.
3- All results shows that LMWCNTs enhance mechanical properties better than SMWCNTs.
4- For both MWCNTs type we observes decrease in flexural strength ,flexural modulus ,impact strength and hardness of nanocomposites materials when we increase the MWCNTs concentration above 2% that due to occur MWCNTs agglomerate in the nanocomposites structure .
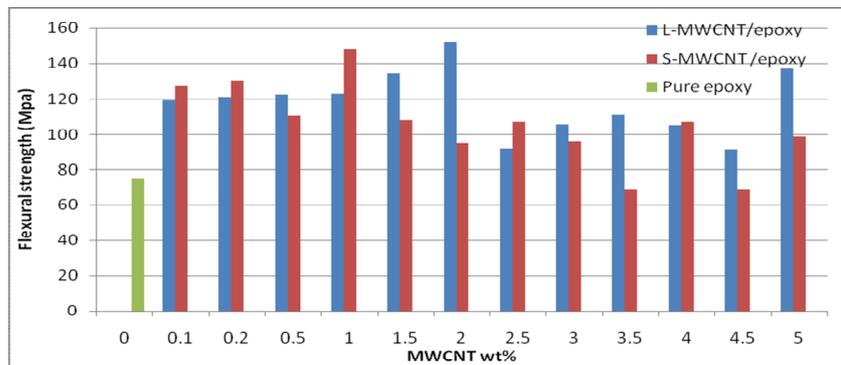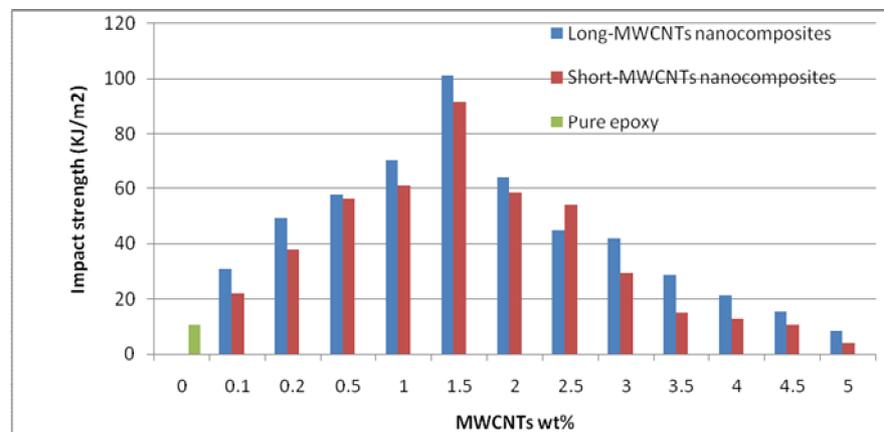

Figure .3. Flexural strength vs MWCNT wt% of Epoxy/ MWCNTs composites.
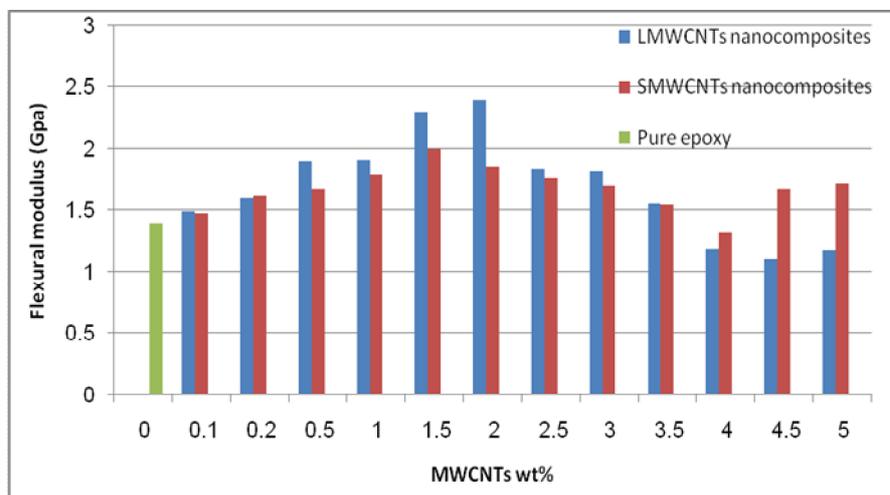
Figure.4. Flexural modulus vs MWCNT wt% of Epoxy/MWCNTs composites.
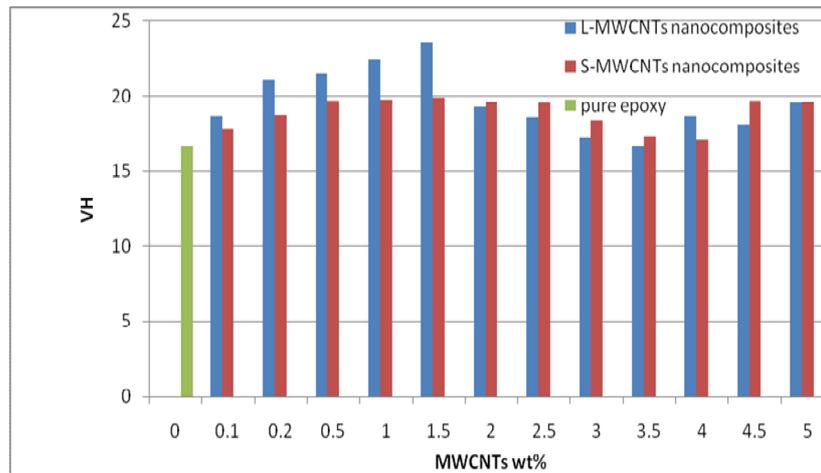
Figure.5. Vickers micro hardness vs MWCNT wt% of Epoxy/ MWCNTs composites.

Figure.6. Charpy Impact vs MWCNT wt% of Epoxy/ MWCNTs composites.

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