Thermo-physical and Mechanical Characterization of Epoxy/MWCNTs' Nanocomposites

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Abstract— In this study, we add MWCNTs to enhance the properties of the epoxy as a resin matrix for a nanocomposite material. Thermal properties are enhanced by improving the matrix properties. An investigation was performed to find the relation between the thermos-physical properties and the MWCNTs percentage in epoxy matrix. A various weight percentage of MWCNTs was dispersed in epoxy matrix to be examined these were (0.1, 0.25, 0.5, 1.0) %. The samples were prepared with the sonication technique for about an hour and cured in an open mold in autoclave at 80°C for about four hours and made into (6x6) mm square with (1.0) mm thickness. The thermal conductivity (k) was obtained by measuring the thermal diffusivity (α) and thermal effusivity (e) using the photoacoustic (PA) technique. The composites exhibit about (180) % improvement in k at (1.0) wt. %. A micromechanical models were evaluated to predict through-thickness thermal conductivity of the manufactured sample, and then compared with the experimental results. A Finite Element Model (FEM) was developed to reveal heat transport mechanisms of the resultant nanocomposites. The nanocomposite design for finite element analysis (FEA) provided close predictions and performed better than the micromechanical models.

Key words— Thermal Conductivity, Carbon Nano-tubes, Composite, thermal properties, Micro-mechanical model.

1. INTRODUCTION:

The composite materials are widely used in the manufacturing of the automotive, airplanes and aerospace vehicles. The polymer matrix composite (PMC) has a low thermal conductivity especially in the cross-sectional direction, through thickness, but characterized by high mechanical properties and lightweight. Therefore, improving through thickness thermal conductivity leads to use of PMCs in more applications [1, 2]. The matrix constituents in PMCs, such as epoxy resin, have a low thermal conductivity up to 2 W/mK [3], it is also have good characteristics as stiffness, strength, dimension stability, chemical resistance and considerable adhesion property to the embedded fibers. There are various methods to improve the epoxy resin thermal properties as using the high conductive fillers or reinforcing fibers. The filler materials modifying the epoxy thermal properties are nanoparticles, nanotubes, clay and nanofibers to produce high performance composites [4, 5]. These Nano fillers produces a nanocomposite with an enhanced modulus of rigidity, strength and heat deflection but decreases the fracture toughness of the epoxy [4, 6].

Liao Y et al. in 2004, study the enhancement of epoxy resin thermal conductivity in the direction through thickness using multiwall carbon nanotubes (MWCNTs). It is a multi-function Nano-reinforcement for polymer matrices because of the high strength (about < 100 times than steel) and modulus (about 1000 GPa), high thermal conductivity (twice as high as diamond), excellent electrical capacity (< 1000 times than copper), and thermal stability (2800°C in vacuum) [7]. Different polymer/CNT nanocomposites have been produced by incorporating CNTs into various polymer matrices, such as polyamides, polyimides, epoxy, polyurethane and polypropylene [8, 9]

Y. X. Zhou et al in 2008 [10], evaluate the electrical, thermal and mechanical properties of multi-walled carbon nanotubes (MWCNTs) reinforced epoxy.

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Firstly, 0.1, 0.2, 0.3, and 0.4 wt. % of (MWCNTs) were infused into epoxy. Electric conductivity, dynamic mechanical analysis (DMA), three point bending tests and fracture tests were performed. Experimental results show significant improvement in electric conductivity, the resistivity decreased from (1014 Ω·m) of neat epoxy to (10 Ω·m) for (0.4%) Epoxy/MWCNT and increase of permittivity with increasing of MWCNTs percentage. The results of DMA revealed that adding MWCNTs to epoxy produce (90%) enhancement in storage modulus and (17°C) increase in Tg. Mechanical test results that the modulus increased with higher MWCNTs percentages, but the (0.3%) MWCNTs infusion system showed the maximum strength and fracture toughness enhancement. The increase of MWCNTs percentage leads to poor dispersion and then less strength and fracture toughness.

Michael Zimmer et al in 2012 [11], exploring by modeling and experimentally the effect of nanoparticles filler concentration, aluminum nanoparticles and carbon nanotubes, on through thickness conductivity of (epoxy/nanoparticle) and (fiber reinforced/nanoparticle) multiscale composites study. The nanocomposite design for finite element analysis (FEA) provided close predictions and performed better than the micromechanical models. On the multiscale composite system, predictions were concluded to be dependent upon FEM design where the interactions between nanoparticles and fibers are critical to determine the through-thickness conductivity.

Measurement of thermal parameters such as thermal diffusivity (α), thermal effusivity (e) and thermal conductivity (k) of epoxy/MWCNTs nanocomposite are very essential for their applications, particularly in the devices fabrication. (α) m2s−1 is a significant thermo-physical parameter which measures how effectively phonons carry heat through the sample. Whereas the measurement of the heat exchange rate or the thermal impedance for heat exchange of a given material is essentially determined by (e) Ws1/2m−2K−1, (e) is a relevant thermo-physical parameter for surface heating or cooling as well as in quenching processes. These quantities are defined by $\alpha = {}^{k}/_{\rho c}$ and $e = \sqrt{k\rho c}$.

Knowledge of the thermal conductivity (k) of epoxy/MWCNTs composite, aids in the selection of candidate for engineering applications such as computer parts and automobile engines. Photoacoustic technique (PA) is a photo-thermal detection technique; that is proved a powerful tool to study the optical and thermal properties of such materials without particular sample treatment in a nondestructive manner [12, 13]

2. EXPERIMENTAL WORK:

2.1. Epoxy Nanocomposites materials and preparation.

The Epoxy nanocomposites samples in this research were made using biresin two parts matrix; part A CR82 (resin) and part B CR80-6 (hardener). Both are made by German company called “Sika Deutschland GmbH”. A Multiwall Carbon nanotubes (MWCNTs) were purchased from the Egyptian Petroleum Research Institute (EPRI), this MWCNT’s were produced by a high-yield catalytic process based on chemical vapor deposition (CVD) with an outer mean diameter of (8-10 nm) and inner mean diameter of (4nm) and length from (5-10 μm). The MWCNTs purity was greater than 90%. The MWCNTs dispersed in the epoxy with a variable weight fraction (0.1, 0.25, 0.5, and 1%) to investigate the thermal properties enhancements.

Each sample was manufactured by dispersing the MWCNTs in epoxy using ultrasound waves (sonication) in a constant temperature as in Figure 1-a, and then a magnate stirring to ensure a good dispersion condition. In the dispersion process a Sonics (vibra-cellTM) was used with (50%) amplitude and temperature not more than 80°C for about 45 minutes. Afterward a magnetic stirrer named “hotplate stirring” device model “SB162-3” was used in (500 RPM) for 10 minutes as in Figure 1-b, then the sonication process was repeated for more 15 minutes with the same conditions. The samples were cured in an Aluminum mold according to the dimensions as per ASTM D-3039 Figure 1-c, the mold and the curing processes were performed in the Aircraft Factory/Arab

Manufacturing Organization, and it was designed to hold five tensile test specimens and a thermal conductivity specimen. The Epoxy/MWCNT specimens will entered the autoclave Figure 1-d with a protectioin from the internal air stream, this object was held in the autoclave at 80°C for four hours with a temperature increasing and decreasing rate 5°C/min.

2.2. Micro-Mechanical Models.

The micromechanical models are widely used for through thickness thermal conductivity predictions of composites [11]. This research uses each model to predict the thermal conductivity of the various concentrations of (MWCNTs) samples based on their constituent materials properties. The prediction results were compared to the measured value of the samples to evaluate the effectiveness of each model.

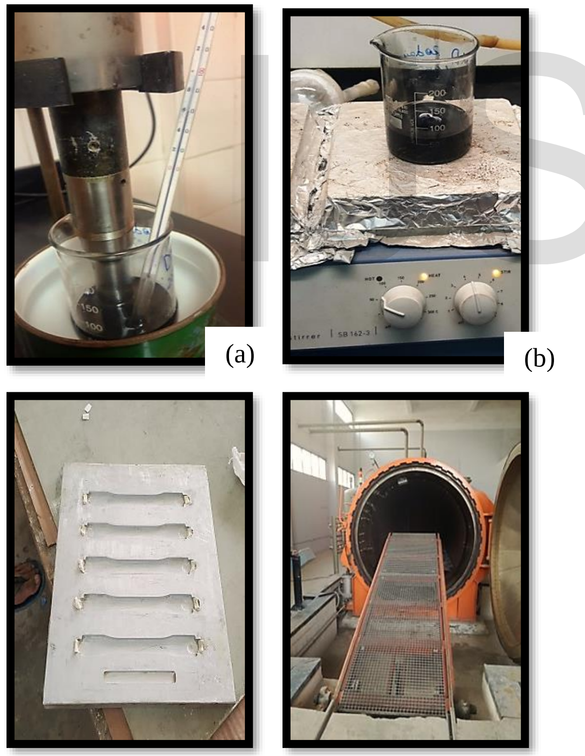


Figure 1- a) sonication probe, b) magnetic stirrer, c) curing mold, d) autoclave.

2.3. Finite Element Model.

A COMSOL Multi-physics 5.2a software package was used to simulate functional model of the nanocomposite samples, the created COMSOL unit was represented based on the epoxy matrix filled with the mentioned MWCNTs percentages. The unit dimension is 5μm x 5μm and the MWCNTs were presented with its real dimensions and was distributed randomly.

These models were built in time dependent methodology where the applied heat flux to the lower edge of the functional unit is 0.5 W/m2. The parameter conditions were set as a constant heat flux applied against an exposure period 180s; the initial temperature selected is the room temperature.

The insulated surfaces prevented convection and maintained a constant temperature gradient. For the predetermined heat flux, the temperature value at the end of the exposure time was derived. The thermal conductivity was calculated as in section 3.3.

$$\alpha = f_c L^2 \;\; {m^2}/{s} \qquad \text{Equation 1,}$$

2.4. Nanocomposite Epoxy/ MWCNTs thermal measurements.

The experiment work was executed in the Faculty of Engineering/ Banha University. In the physics lab there is a photoacoustic (PA) apparatus for measuring the thermal conductivity of the solid materials. This technique of measurements was carried out by a gas microphone detection technique. Tl t beam produced by ht source was mecnanically modulated by an optical chopper (SR540), and focused onto the sample which was mounted carefully inside the cell (MTEC Model 300). The sound wave generated from the sample can be subsequently detected as an acoustic signal by a highly sensitive microphone fixed inside the cell. The PA signal was then amplified by a low noise preamplifier and further processed using a lock in amplifier (Stanford Research System, Model SR830 DSP). A personal computer was interfaced to the system for automatic data acquisition and analysis [14].

3. RESULTS AND DISCUSSIONS:

3.1. Nanocomposite Epoxy/ MWCNTs Results.

The analytical model depends on the compression between the micromechanical model equations and the predicted values of the epoxy nanocomposite with different MWCNTs percentages. In Figure 2, it shows the different behavior of the various equations of nanocomposite thermal conductivity calculated according to MWCNTs percentage, from this figure we may explain that "Lewis-Nielsen" methods shows an unconventional behavior as a result it is preferred to disregard this method. The other techniques (Halpin-Tsai, springer-Tsai and Rayleigh) show increase in the thermal conductivity by increase in the percentages of MWCNTs until (0.5%) afterword, the thermal conductivity decreases neglecting the MWCNTs percentage. At last, role of mixture (ROM) shows slight variation of thermal conductivity, seems to be constant with neat specimen.

All the analytical techniques considering the same initial conditions to compare the variation of thermal conductivity by increasing MWCNTs in the epoxy matrix. The thermal conductivity of the neat epoxy is about (0.12) W/m.K while MWCNTs thermal conductivity is (3000) W/m K. The weight fraction of the nanocomposite varies for MWCNTs percentages (0.1, 0.25, 0.5 and 1) % wt. considering part of the epoxy weight.

TABLE 1

THERMAL CONDUCTIVITY CALCULATION

	0	0.1	0.25	0.5	1
ROM	0.12	0.1201	0.1203	0.1206	0.1212
HALPIN-TSAI	0.12	0.1467	0.2000	0.3600	NA
SPRINGER-TSAI	0.12	0.0842	0.0757	0.1054	NA
RAYLEIGH	0.12	0.1467	0.2001	0.3695	-0.6321
LEWIS-NIELSEN	0.12	0.3052	1.2115	1.6086	0.6342
NUMERICAL	0.12	4.3607	8.6015	14.963	31.925
EXPERIMENTAL	0.12	0.2	0.25	0.31	0.65

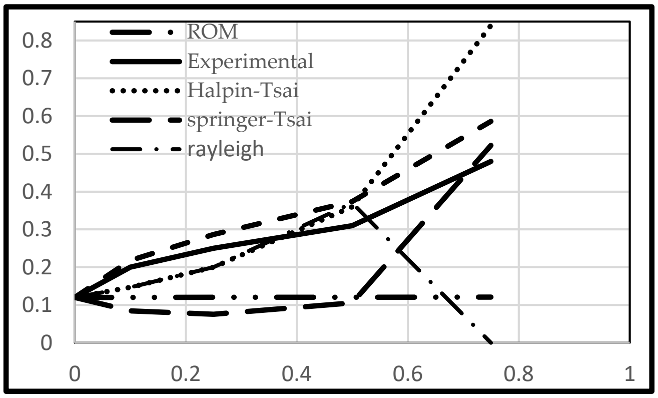


Figure 2. Thermal Conductivity values Numerical, Mathematical and experimental Model

3.2. Nanocomposite Epoxy Finite Element Model Results.

In COMSOL Multi-physics, Figure 3, it shows the created function unit that is based on the weight fraction of the MWCNTs in epoxy. It shows the function unit in the form of COMSOL mesh ready to study the effect of 0.5 W/m2 heat flux exposed to the lower edge for 180 sec.

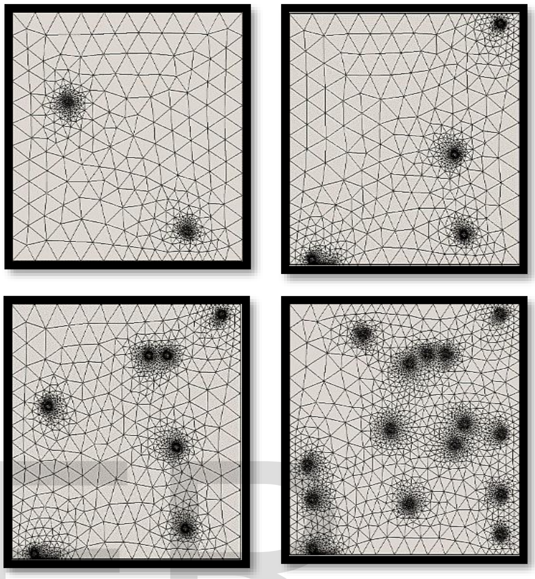
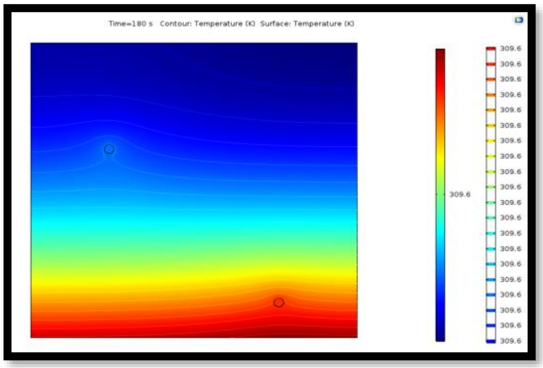


Figure 3 specimen mesh vs various CNT's percentage

In Figure 4, applying the heat flux for 180 seconds creates thermal graduation which shows the heat flow from lower edge to the upper one resulting for temperature difference. From Fourier's law, determination of the thermal conductivity (K) of the medium was made using the calculated heat flux (Q) and knowledge of the temperature distribution (T) across the distance (x) of a medium represented by $\alpha = f_c L^2 \quad m2s$ Equation 1 in section 3.3.



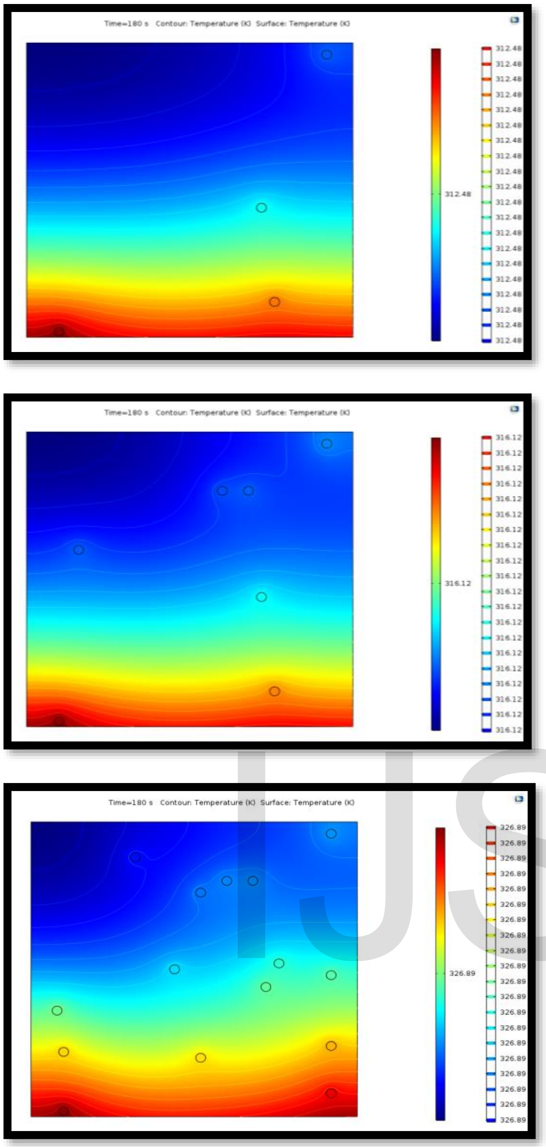


Figure 4 the thermal distribution through the functioning unit thickness

3.3. Thermal measurements

The PA technique was employed to investigate the thermal properties of the examined samples (thermal diffusivity, thermal effusivity and thermal conductivity). The theoretical background for the evaluation of thermal diffusivity from amplitude spectrum of PA signal is explained elsewhere. The thermal diffusivity (α) was calculated using the relation [13, 15].

$$\alpha = f_c L^2 \; {m^2}/{s} \qquad \text{Equation 1}$$

where (L) is the sample thickness and (fc) is the characteristic frequency. By knowing (fc) and (L) of the investigated specimen, the thermal diffusivity value can be evaluated.

The PA signal amplitude was recorded at various chopper frequency (f) for each sample (depth profile analysis). The plots of (ln PA) amplitude versus the (ln f) are given in Figure 5 (a–e) for mentioned MWCNTs weight percent. The distinct change in the characteristic frequency (fc) slope, where the crossovers take place can be easily observed. Using equation 1(α) was then calculated.

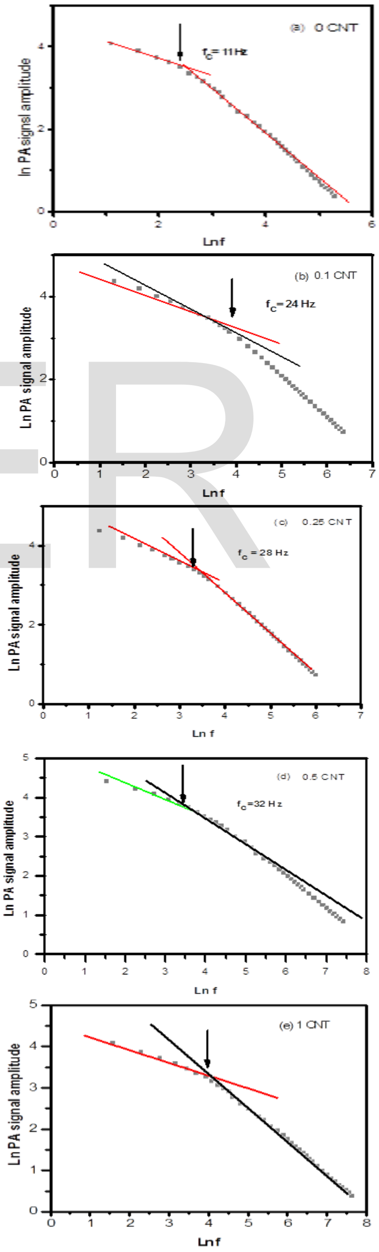


Figure 5 ln PA amplitude vs. ln f: a) 0 wt.%, b) 0.1 wt.%, c) 0.25 wt.%, d) 0.5 wt.%, and e) 1 wt.%

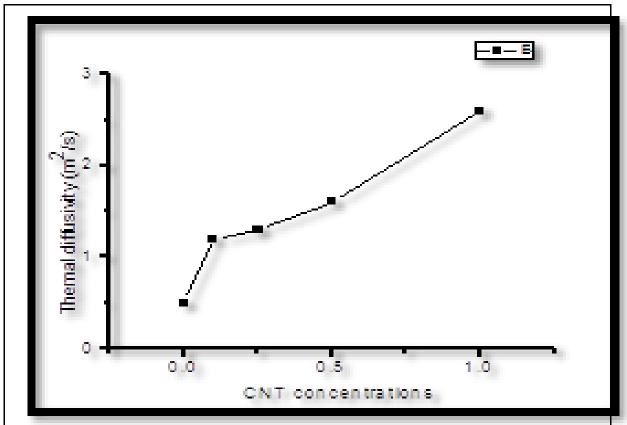


Figure 6 Thermal diffusivity of Epoxy/MWCNT versus MWCNT concentrations

The measured value for the epoxy nanocomposite specimen without MWCNTs addition was (0.5) m2/s which is very close to the reported value of (α) for epoxy. The measured values of epoxy/MWCNTs composite for different MWCNTs wt. % are displayed in Table 2. The variation of thermal diffusivity of epoxy/MWCNTs nanocomposite with various MWCNTs concentrations is given in Figure 6. The results showed that, there is an enhancement of (α) with increasing MWCNTs to the highest concentration (1%) compared with the neat specimen was (190%). The thermal effusivity (e) is also determined for the same samples using the PA technique where (for optically opaque thermally thick sample) the PA signal amplitude q is given by.

$q = \frac{A}{e.f}$ Equation 2

$$A = \frac{I_0 \gamma P_0 \alpha_g^{1/2}}{4\pi d_g T_0}$$

where (I0) is the incident Laser beam intensity, (T0) and (P0) are the ambient temperature and pressure respectively, (γ) is the ratio of gas (air) specific heats, (αg) is the gas thermal diffusivity and (lg) is the length of the gas column of the PA cell. The constant (A) can be eliminated by normalizing the signal measured from a specific sample to the signal obtained from the reference sample with a well-known effusivity [16].

Using $\alpha = f_c L^2$ $m2s$ Equation 1 and Si wafer as a reference sample, with a well-known thermal effusivity (16,060 Ws$^{1/2}$m^{-2}K^{-1} [3]. The constant factor, A, is calculated by normalizing the signal measured for the sample to that measured for Si. The obtained e values for samples with CNTs concentration from zero to 1 wt. % are also given in Table **1**, where the slopes of Si and the samples were obtained from Fig. 8 by linear fitting for the relation between (1/f) and PA amplitude (q). The results show that the values of (e) for epoxy/MWCNTs increase from 536.65 to 1334.49 (W.s$^{1/2}$ m^{-2} K^{-1}) as the concentration of MWCNTs increases from (0) to (1.0) wt. %.

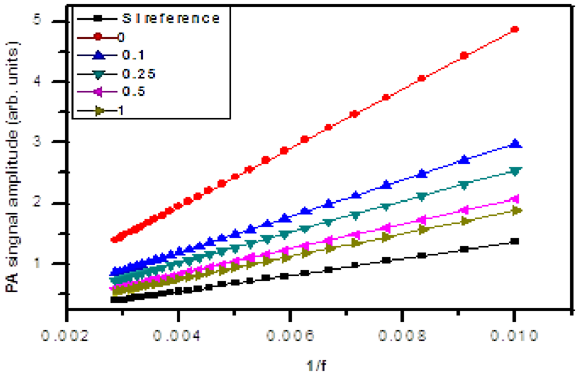


Figure 7 PA amplitude vs. 1/f : a) 0 wt.%, b) 0.1 wt.%, c) 0.25 wt.%, d) 0.5 wt.%, e) 1 wt.% and Si reference

TABLE 2.

THE CALCULATED VALUES OF THE DIFFERENT SPECIMENS

Sample	**Thermal Diffusivity (A) (10^{-6} M^2/S)**	**Thermal Effusivity (E) (W.S$^{1/2}$ m^{-2} k^{-1})**	**Thermal Conductivity(K) (W/M. K)**
0	0.5	536.65	**0.12**
0.1	1.2	577.35	**0.20**
0.25	1.3	693.37	**0.25**
0.5	1.6	775	**0.31**
1	2.6	1334.49	**0.65**

As mentioned, the thermal parameters $\alpha = {}^{k}/_{\rho c}$ and $e = \sqrt{k\rho c}$, therefore the values of (α) and (e) depending on (k) and (ρc) in a different manner. The variations of (ρc) versus MWCNTs content are weak. Consequently, the similar behavior of both quantities versus (Q Ds) loading clarify that the effect of (Q Ds) on these parameters is coming mainly from their effect on (k). The measured values of (α) and (e) which are (0.5×10-6 m2/s) and (535.65 Ws1/2m−2K−1), for the neat epoxy sample, are used to calculate its thermal conductivity ($k = e\sqrt{\alpha}$ = 0.12 W/m.K) which is very close to the reported value of (k) for Epoxy (0.2 W/m K). The measured values of (k) for our composite samples as a function of MWCNTs concentration are presented in Figure 8.

There is an increase in (k) with the increase of MWCNT concentrations.

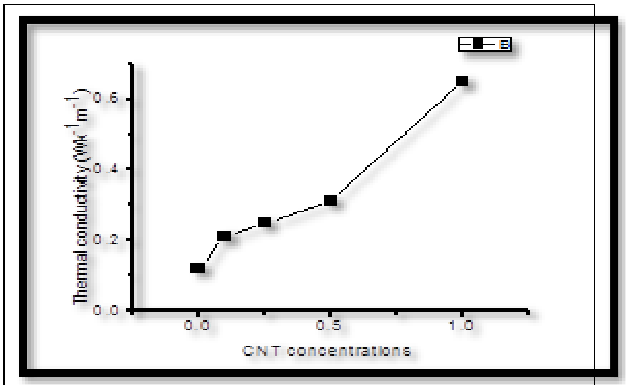


Figure 8 Thermal conductivity k of Epoxy/MWCNT versus MWCNT concentrations

The increase (k) is more significant about (180 %) at the highest MWCNT wt. %. Such an increase in (k) with the increase MWCNTs was reported by other authors [14]. In their work, they obtained an increase in (k) to about (150%) for PVC/MWCNTs. The reported thermal conductivity (k) of MWCNTs was about (6.0 W/m K) [17], [18]. Therefore, the significant increases of composite thermo-physical properties by adding a small amount of MWCNTs were predicted. Similarly, Jalila Al-Osaimi et. al. have made measurements on thermal properties of PMMA/SWCNTs and reported a significant increase in k (160 %) at (1 % SWCNTs) [13], [15]. Moreover, similar results were obtained by N. M. Al-Hosiny et.al [19]. They concluded that, incorporation of phase with high thermal conductivity (MWCNTs in our case) into the matrix with law thermal conductivity (Epoxy in our case) is accompanied by two opposites. Effects influencing heat transport in the composite. The first one is the appearance of the surface of the new phase increasing the number of interfaces, which increase of phonon scattering reduce the heat flow transport, thus resulting in an interfacial resistance. The second effect is the presence of the volume of the new phase with high thermal conductivity, which results in an increase of the heat flow. Our results that the increase of thermal conductivity with increasing of MWCNTs support these arguments, and showed that, the second effect is more dominant.

4. CONCLUSIONS:

In this research, micromechanical, numerical and experimental methods were performed to discuss the effect of adding various weight percent of MWCNTs to epoxy matrix improve the thermal conductivity of CFRP. Four small different weight fractions were added and studied.

In the micromechanical models, the nanocomposite thermal conductivity was dependent upon the thermal conductive values of the material constituents and its volume fraction. According to the micromechanical model it is clear that in small percentages (0.5) % is the optimum concentration. Generally, No model can be used for thermal predictions, the use of models was not adequate due to the extreme thermal conductivity differences between the fillers and matrix, and the low volume fraction content of the MWCNTs.

Numerically using COMSOL software package it is clear that the enhancement in the thermal conductivity was appeared by increasing the percentage of MWCNTs.

The thermos-physical parameters are obtained using (PA) technique. We demonstrate that (190%) increase in (k) with MWCNTs loading (1%). Our results show that, the presence of new phases with high thermal conductivity is more dominant. Finite element modeling proved to be the effective means for accurate predictions. This accuracy would largely be dependent on the appropriate model design to cover actual microstructure features and heat flux flow of the composites, limiting conductivity ratio effects.

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