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Influence of Heat Treatment on Wear and Hardness Properties of MWCNTs Reinforced Epoxy nanocomposites.

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Abstract

Nanocomposite of carbon nanotube add to epoxy resin material of weight fraction (0.25, 0.5, 0.75 1.0, 1.25, 1.5, 1.75 , 2 & 2.5 wt. %) were fabricated by dispersing within an epoxy resin using a Ultrasound machine followed by mechanical stirring. The samples were heat treated at temperature (80 °C for 3 hrs) The mechanical properties of the composites were investigated. Wear and hardness properties measurements indicated higher wear rate and hardness with increasing concentration of MWCNTs . The MWCNTs significantly improved the wear resistance and hardness when compare than the pure epoxy. These note show too after heat treatment of composite with (80 °C for 3 hrs).

Keywords: Epoxy, MWCNT, Nanocomposites, Heat treatment .

تأثير المعاملة الحرارية على خاصية البلى والصلادة لمتراكبات أنابيب الكاربون النانوية المدعمة براتنج الايبوكسي

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الخلاصة

تم تحضير متراكب نانومتري من مادة الايبوكسي مضاف اليه انابيب الكاربون النانوية متعدد الجدران وبنسب وزنية (0.25، 0.5 ، 1.75 ، 1 ، 1.25 ، 1.5 ، 1.75 ، 2 ، 2.5) % بتفريق داخل راتنج الإيبوكسي باستخدام جهاز الموجات فوق الصوتية متبوعاً بالتحريك الميكانيكي . العينات تعرضت لمعاملة في درجة حرارة (80 درجة مئوية ولمدة 3 ساعات) وتم التحقيق في الخواص الميكانيكية للمركبات . قياسات خصائص البلى والصلادة لمتراكب النانومتري أشارت إلى معدل أعلى للبلى والصلادة مع زيادة تركيز انابيب الكاربون النانوية . اضافة الكاربون النانوي ادى الى تحسين كبير في مقاومة البلى(التأكل) والصلادة مقارنة بعينات الايبوكسي النقي . وكذلك تظهر بعد المعالجة الحرارية للعينات عند (80 درجة مئوية ولمدة 3 ساعات) .

Introduction

Polymer matrix composites with carbon nanotube (CNT) reinforcement have become popular in structural applications because of unique atomic structure, very high Aspect ratio and extraordinary properties like strength and flexibility of CNT [1].

Acarbon nanotube is the most famous nanomaterial since its discovery in 1991 by Japanese scientist Sumio Iijima. Carbon nanotubes are miracle molecules with unique characteristics due to their small size, high surface area (from 50 to more than 110 m^2/g), electrical conductivity, stronger

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than steel (50-100 times) with only one-sixth its weight and others[2].The main applications of CNTs are polymer composites, electronics, hydrogen storage (like hydrogen storage), sensors (like gas sensor), and biological application (like drug delivery and cancer therapy nano gold)[3].

The high bond strength of the constituent carbon–carbon bonds of multi-walled carbon nanotubes (MWNTs) are the reason behind its outstanding mechanical properties. On the other hand, epoxy resins are well established thermosetting matrices of advanced composites, displaying a series of interesting characteristics, which can be adjusted within broad boundaries [4].

Epoxy resins are used in a variety of applications since their properties , such as thermal stability , mechanical and engineering response , low density and electrical resistance, can be varied considerably. [5].

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.[6] Ayatollahi et al.[7] investigated the effect that changing carbon nanotube aspect ratiohad 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 aim of study

The aim of this work is to improve the mechanical properties of the epoxy resin by adding different concentrations of MWCNTs and studying the effect of heat treatment on the nanocomposites characteristics.

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 ◦C , mixed viscosity 1.0 Poise at 35 ◦C; and the nanoparticle fillers are MWCNTs manufactured by NANOSHEL LLC USA.

Table 1- Materials properties

Materials	Length (μm)	Diameter (nm)	Purity %
MWCNT SHORT	5-15	15-25	> 98

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 (0.25 , 0.5 , 0.75 , 1 , 1.25 , 1.5 , 1.75 , 2 & 2.5 wt.%) use direct mixing process by added MWCNT to epoxy resin. The mixture was solicited for (1 half hrs.) by ultrasonic path and then being stirred for (3 hrs.) to disperse the MWCNTs in polymer matrix . Using vacuum system (10-2 bar) to remove the bubble before molding then the hardener added to mixture and stirred for (4 minute). The mixture casted in glass molds . The samples left for (72hrs) before pulling out and left in the vacuum chamber for (14) days before to improve curing conditions . And it was heat treated in oven for (3 hours at 80 ◦C) and is less than the degree of polymer melting. Then kept in the oven for (24 hours) and was tests .

3. CHARACTERIZATION OF MWCNTS AND EPOXY COMPOSITES TESTING .

3.1.Wearing Test

The sliding wear tests were carried out using an indenter-on-plate configuration as shown in Figure-1. The diameter of the sliding track was 40 “mm”. The indenter was made of stainless steel with a diameter of 3. 0 “mm”. The sliding tests were carried out with a load of 9 N, and the sliding speed of the counterpart had a fixed velocity. The testing time was 10 minutes. Mass loss was measured by differentiating the weight of the wear The wearing characteristic was assessed according to the weight loss.

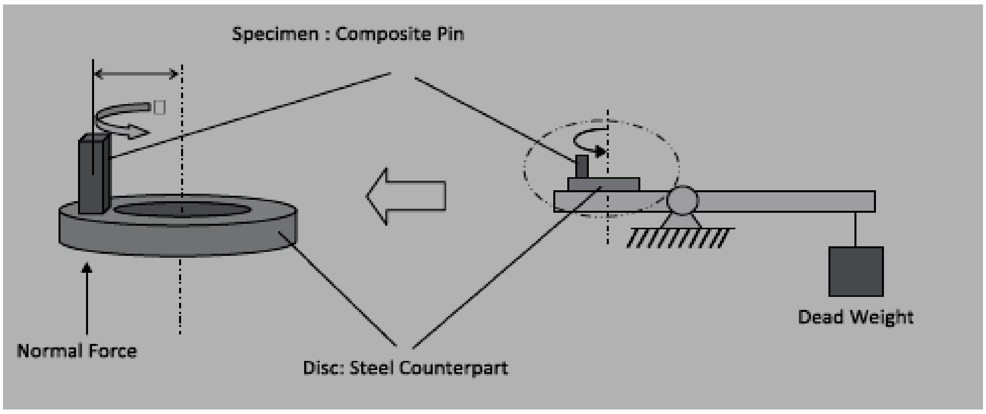

Figure 1- The pin-on-disc wear test.

3.2. Hardness test

The hardness tests were carried out according to (ASTM D 2240) by using Shore D Hardness tester (HT-6510 D, Time Group INC Company) as shown in Figure-2. The test specimens (40mm in diameter and 4mm in thickness) are shown in Figure-2. The specimens were tested by pressing the indenter of the instrument which is a needle of a sharp head into the specimen surface so that the result appears on the digital screen attached with the instrument**.**

Results and Discussion

The FT-IR spectra of EPx, EP / CNT nanocomposites material were illustrated in Figure-2 and Figure-3 it that the hydroxyl-stretching band of epoxy resin appears at 2927.94 cm^{-1}, Figure-3 (EP/CNTs) illustrates that the absorption peak at 2954.95 cm^{-1}. The characteristic of –OH stretching, which is the unreactive CNTs OH groups in inorganic networks.

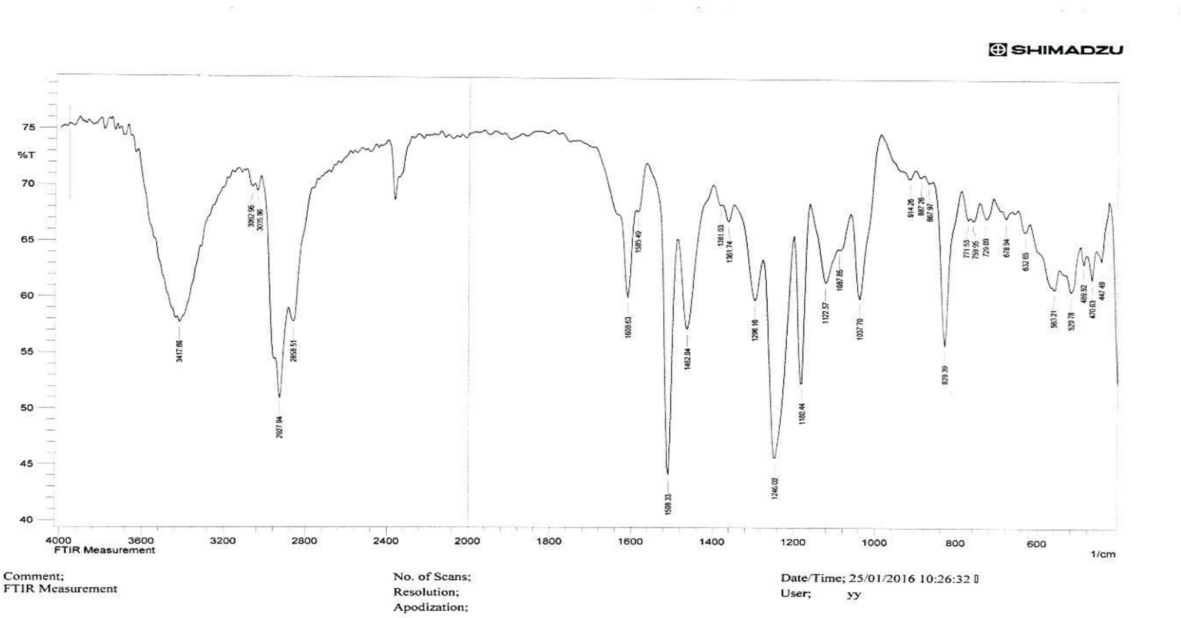

Figure 2- FTIR spectrum of epoxy resin**.**

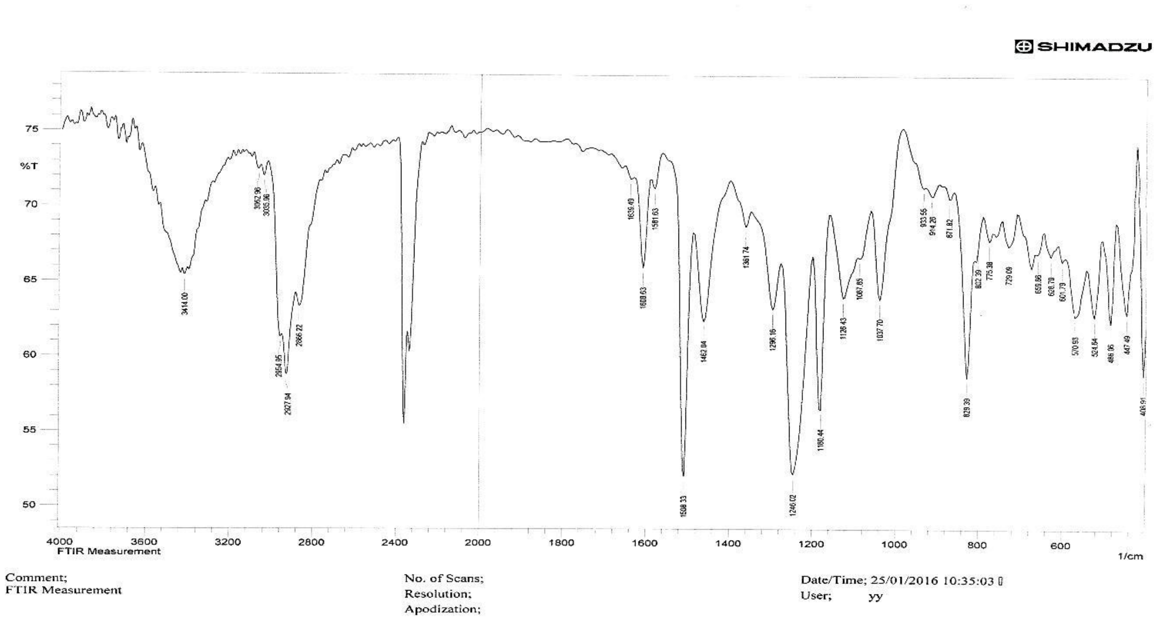

Figure 3- FTIR spectrum of epoxy /MWCNTs nanocomposites

Table 2- Hardness value

MWCNTs%	**Hardness (before heat treatment)**	**Hardness (after heat treatment)**
0.00%	**83.3333**	**71.6666**
0.25%	**84.3333**	**83.3333**
0.50%	**84.6666**	**83.3333**
0.75%	**85**	**84**
1.00%	**85.3333**	**83.3333**
1.25%	**85.6666**	**83.6666**
1.50%	**85.6666**	**85**
1.75%	**85.6666**	**84.6666**
2.00%	**85.6666**	**84.6666**
2.50%	**86**	**82**

Figure-4 shows the relationship between the weight loss of the epoxy (EP / MWCNTs) composites and the concentration of CNT. The wear rates(weight loss) are plotted as a function of CNT weight concentration with constant applied pressure about 7 N. The wear results show that the reinforced specimens have better wear resistance that of the pure epoxy,
The high value wear resistance at 2.5%is minimum weight loss. while the low value at 1.0 % maximum weight loss.

Is This is probably due to the fact that epoxy can easily remove at sliding surfaces (contact area) but in the composite case the CNT act as a rough surface relative to the counter face against which they slide[8].The behavior of weight loss with concentration before and after heat treatment was same behavior for all concentration , The heat treatment of composite with 80 ^{o}C at 3hrs increase the weight loss which means the reduce of wearing resistance because the composites convert from brittle and small ductile to more ductile.

The heat treatment affect on the polymer bonds by broken the bonds because of the degradation of polymer with heat treatment .

Figure -5 shows the relationship between the hardness of the pure epoxy and (EP / MWCNTs) composites with concentration of CNT. shows that of MWCNTs the hardness was increased with increase MWCNTs [9]. Increase of hardness values may be 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 two atoms in the material.[10] , and it is clear the effect of CNT to improve the hardness of composites.
The lower values of hardness recorded after heat treatment becouse the degradition on surface of polymer composites.

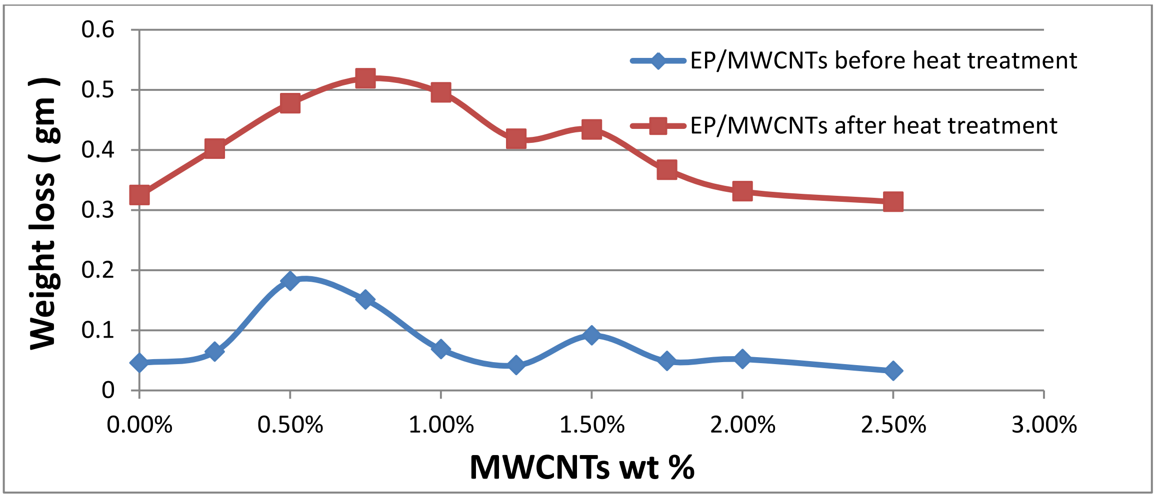

Figure 4- The behavior of wearing weight loss (/gm) vs. before & after heat treatment.

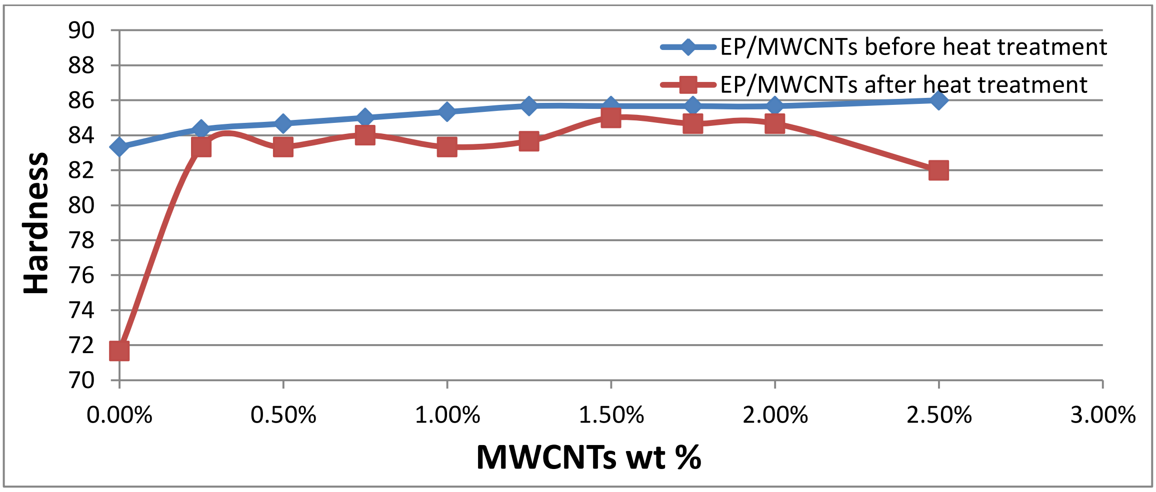

Figure 5- *The behavior of Hardness vs. before & after heat treatment.*

Figure- 6.show Atomic Force Microscopy (AFM) observation uniformity and three-dimensional surface profile of 1% MWCNTs nanocomposite the average roughness 1.74 and average diameters Figure-7. show Atomic Force Microscopy (AFM) observation uniformity and three-dimensional surface profile of 2% MWCNTs nanocomposite the average roughness 0.756 and average diameters . This shows that the material surface coarser less corrosion resistance.

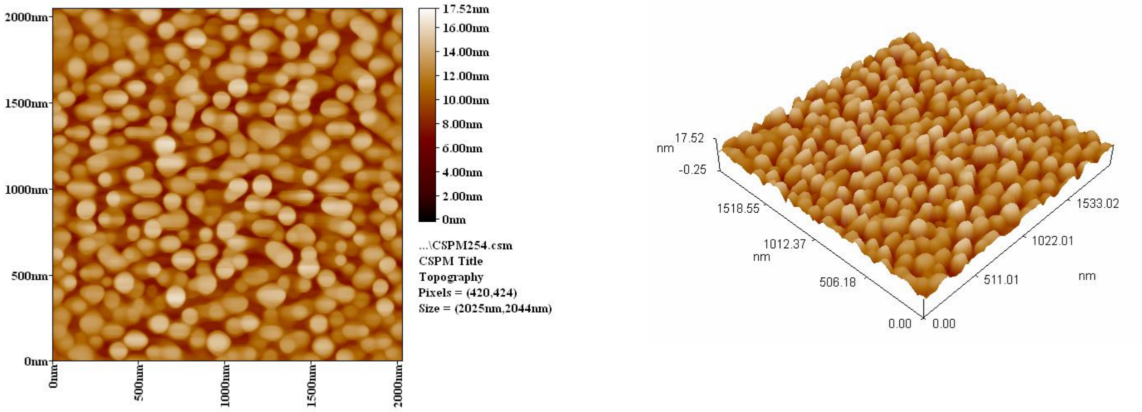

Figure 6- AFM micrograph uniformity and a three-dimensional surface profile of (1 % wt) CNTs in the epoxy nanocomposite.

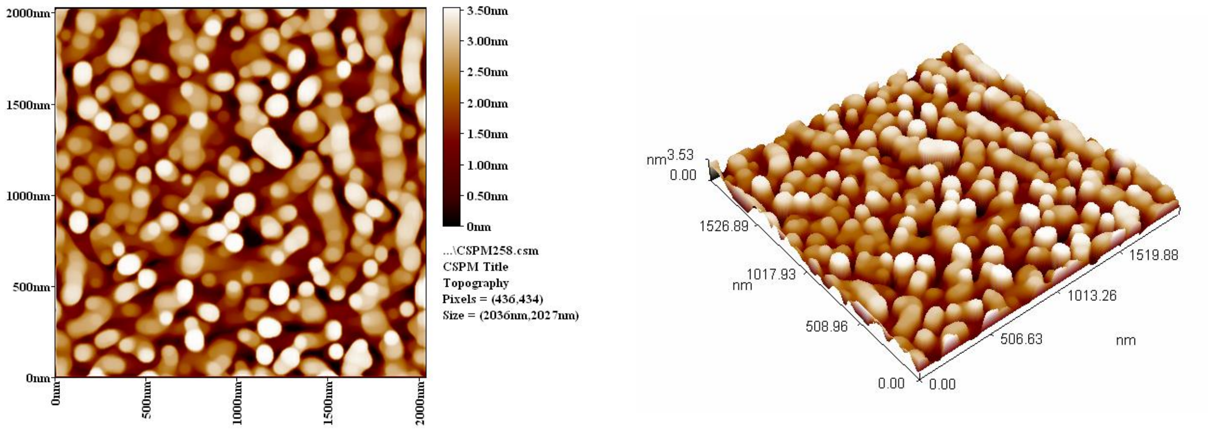

Figure 7- AFM micrograph uniformity and a three-dimensional surface profile of (2 % wt)CNTs in the epoxy nanocomposite.

Conclusion

1. Reinforced specimens have better wear resistance than the pure epoxy at high concentration of CNT. The incorporation of MWCNTs significantly decreases the wear rate of epoxy.
2. Reinforced specimens have better hardness than the pure epoxy . It is clear from nano CNT nano composites enhanced the hardness resistance of pure epoxy.
3. The heat treatment improved the wear resistance of nano composites but the hardness was decreased.

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