Studying the Mechanical Properties of Epoxy Carbon Nanotubes Composite

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Abstract
In the present work a modified chemical vapor deposition method (CVD) was adopted for the production of carbon nanotubes (CNTs). Scanning Electron Microscope (SEM) was used for identification of these of carbon nanotubes. The identification revealed two types of carbon nanotubes Single wall (SWCNTs) and nanocoils tubes. These nanotubes were successfully employed in the development of bulk composites, where commercial epoxy resin has been used as matrix, while mixed phase of carbon nanotubes served as reinforcement phase. It was observed that a substantial increase in all mechanical properties: hardness and Young modulus as well as the thermal properties from which Tg curves were recorded, consequently Glass transition temperature Tg was determined.

Key words: CNTs, Epoxy resin, Mechanical properties thermal properties, Tg.

Introduction
Carbon nanotubes are promising to revolutionize several fields in material science. Nanotubes have a wide range of unexplored potential application in various technology such as aerospace, energy, automobile, medicine and chemical industry (1). Polymeric nano composite (PNCs) represent a radical alternative to conventional filled polymer where the reinforcement is on the order of few nanometers. Uniform dispersion of these nanoscopically sized filler particles produces ultra-large interfacial area per volume between the nanotubes and matrix polymer. Therefore, new combination of properties derived from the nanoscale structure of nanotubes which gives the opportunities to overcome the problems associated with conventional reinforcement polymers and epitomizing the promise of nanoengineering materials(2). Since the discovery of carbon nanotubes (CNTs) by Iijima in 1991(3, 4) increasing attention has been paid to the newly emerging material and many investigators have endeavored to fabricate advanced (CNTs).

Composite material which exhibit remarkable mechanical and electrical properties (5). One of the most intriguing application of CNT is the polymer/CNT nanocomposites(6). Hersam et.al (7), Laurie et.al (8) Ruiz et al (9), Fiege et al (10), Gomes et al(11), Cai et al (12)The high mechanical, electrical and thermal properties of CNTs makes them ideal candidate as fillers in light weight composite. It is their extraordinary mechanical properties exceptional high tensile strength and stiffness that has aroused of particular interest composite material (13). On the other hand Ren et al (14) reported a two fold increase in the tension fatigue strength for an aligned (SWNTs /Epoxy composite) are found in comparison to typical carbon fiber/epoxy composite. Also significant improvements in the mechanical properties of epoxy/SWCNTs nanocomposite were illustrated by a 50.8/ increase in storage modulus Liao et al (15). The main objective of the present work is to prepare a composite material using commercial epoxy resin reinforced with a locally synthesis mixed phase of carbon nanotubes (SWCNTs+ nanocoils). The effects of addition of mixed phase of carbon nanotubes on different mechanical and thermal properties are studied.

Experimental Materials
Carbon NanoTubes CNTs
Consists of single wall CNTs (SCNTNs) and nanocoils CNTs used as reinforcement materials, it was by prepared by modified (CVD) process: it will be discussed later.

Resin
Epoxy resin typeNitofill (EPLV) from (Fosroc) was used as a matrix.

Hardener
Type Nitofill (EPLV) from (Fosroc) was used with epoxy resin.

Procedure Preparation of Mixed Phase of CNTs
Acetylene C$_2$H$_2$/Ar mixture (50ml/500ml) was introduced into the reactor, acetylene decomposed when heated to 700 °C for a half an hour this
leading to catalytic growth of CNTs. Finally the gas was switched back to Ar and the furnace was allowed to cool to room temperature before exposing the product to the air. The (CNTs) yield was roughly calculated as follow:

\[ g(\text{mg/mg}) = \frac{C_0(\text{mg})}{B(\text{mg})} \]

Where \( C_0 \) is the weight of as prepared product sample mg and B is the weight of the catalyst precursor (Fe\(_2\)O\(_3\)/Al\(_2\)O\(_3\)) mg.

**Preparation of CNTs Composite**

1- The epoxy resin was mixed thoroughly with hardener in the percent of (3:1) then the (CNTs) was mixed carefully with the epoxy resin using a mechanical Hotplate stirrer type (DaianLabtech Co. LTD).
2- The mixture was transferred to a suitable mold made of plastic with the dimensions of 30mm×15mm, different molds were prepared in order to test different mechanical and thermal properties.
3- The molded samples were left for 24 hours to cure the sample and complete the cross linking reactions.
4- The cured samples were subjected to grinding and polishing in order to prepare them for testing.

**Characterization**

**Scanning Electron Microscopy (SEM):**
A Carl Zeiss-Supra with accelerator voltage 5KV (UKM Malaysia) was employed to observe the structure of carbon nanotubes (CNTs).

**Mechanical Properties**

**Compression Test**

Were carried out on disc shaped sample using bold – Harris no.36110.

**Hardness Test**

The hardness values were found out according to ISO868 by a hardness tester (shore D, TH210).

**Thermal Analysis**

Tg curves were recorded with Perkin-Elmer (NS20-2013) Thermal analysis. The Tg measurements were conducted with heat rate of 10 °C/min under atmospheric pressure from 30-160 °C.

**Result and Discussion**

Figures (1-2) show images of (SWCNTs) while figure (3) shows image of nanocoil (CNTs). The (CNTs) images obtained from in Scanning Electron Microscopy (SEM) shown in the previous figures were synthesized at high yield. Our observation suggests two types of (CNTs): single wall nanocarbon tubes (SWNTs) and nanocoil tubes. The modified (CVD) method leads to increase in yield (SWNTs) and produces a nanocoil at the same time. The high yield may be attributed to the effect of modification on slowing down the C\(_2\)H\(_2\) gas velocity, which gives more chance to gas to decomposed in high yield through the tube reactor. Our results agree well with suggestion given by Kenji et.al\(^{[15]}\) based on similar observation. The Spectrum shown in figure (4) represent the chemical analysis of the (CNTs) sample obtained by electron diffraction spectroscopy (EDS) of SEM system showing the majority are carbon (94%) and a little amount of Al (0.32%) and Fe (2.22%) and the remainder is oxygen which formed (3.33%).

**Results of Nanocomposite:**

Young's modulus, hardness, Tg and thermal conductivity of (CNTs), Epoxy nanocomposite were determined. The stress-strain curve of samples with and without (CNTs) are shown in figures (5and 6). It is observed that all the mechanical properties are improved when (CNTs) are introduced into the composite. Effective utilization of (CNTs) in composite applications depends strongly on the ability to homogeneously dispense them through matrix without destroying their integrity. It is clearly seen that the modulus of elasticity increases by a significant value when a small amount of (CNTs) blended with Epoxy Resin (0.44g/40g). This may be attributed to the role played by mixed phase of (CNTs) in improving load transfer from matrix to (CNTs), load transfer play a key role in mechanical properties of composite if the adhesion between the matrix and the (CNTs) is not strong enough to sustain high loads, load storage and then increasing the Young's modulus.

The hardness test using Shore-D showed an improvement of (46%) in hardness value for the nanocomposite. This enhancement can be ascribed to reinforcing ability of mixed phase of (CNTs).

The Tg curves of both the blank and nanocomposite samples are shown in figures (7-8). The Tg value has shifted from 33.4°C for blank sample to 64.42°C for nanocomposite samples. These values revealed a clear improvement in thermal stability of nanocomposite as compare to pure non-blended resin. The higher Tg of nanocomposite is due to good dispersion of (CNTs) within the resin matrix. Our results are in good agreement with the results obtained by Andrew and Weisenbergert\(^{[17]}\).

**Conclusion**

1-SEM results revealed mixed phase of (CNTs) which consists of two types of (CNTs) were obtained (SWCNTs and nanocoil).
2. Blended (CNTs) with epoxy resin enhances all mechanical properties such as: Young modulus, hardness.
3. Great change in Tg values has been obtained when epoxy resin was blended with (CNTs).

References

Figure (1): SEM Image of (SWCNTs)
Figure (2): SEM Image of (SWCNTs)

Figure (3): SEM Image of Nanocoil Produced in This Work
Figure (4): Chemical Analysis of (CNTs)

Figure (5): Stress – Strain Curve of Epoxy Resin Sample
Figure (6): Stress – Strain Curve of Epoxy Resin Reinforced with (CNTs)

Figure (7): Tg Curve of Blank Sample (Epoxy Resin)
دراسة الخصائص الميكانيكية لمتراكبات الايبوكسي مع الانابيب الكربونية النانوية

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الخلاصة

في هذا البحث تم تطوير طريقة الترسيب الكهربائي الكيميائي (CVD) لانتاج انابيب النانوكرابون، وتم استخدام تقنية المايكروسكوب الماسح الاليكتروني (SEM) لتشخيص الانابيب المنتجة في هذا البحث وكانت هذه الانابيب النانوية على نوعين: احادية الجدار (SWCNTs) والحمزونية (Nanocoils). تم استخدام طور الانابيب النانوية ونجاح طور تقوية لانتاج مواد مركبة، مادة الأساس فيها هو الايبوكسي التجاري.

بعد اتمام عملية الخلط وتكوين المادة المتراكبة تبين وبعد القيام باختبارات الخواص الميكانيكية زيادة ملحوظة في جميع تلك الخواص مثل الصلالة ومعامل بوينك كما تم قياس الخواص الحرارية والتي تم تسجيل منحنى Tg لها قبل وبعد عملية التدعيم ومن ثم تعيين Tg.

الكلمات الدالة: CNTs, الانابيب الكربونية النانوية, الايبوكسي, الخواص الميكانيكية, الخواص الحرارية, Tg.