

Study on mechanical and viscoelastic behavior of carbon nanotube (CNT) reinforced Epofine1564 nanocomposite

Puneet KUMAR^{1,*} and Jonnalagadda SRINIVAS²

¹ School of Mechanical engineering, Tel Aviv University, Tel Aviv, 69978, Israel

² Department of Mechanical Engineering, National Institute of Technology Rourkela, Odisha, 769008, India

*Corresponding author e-mail: puneetkumar@mail.tau.ac.il

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Abstract

This paper presents an experimental study to examine the mechanical and viscoelastic characteristics of multi-walled carbon nanotubes (MWCNTs) reinforced epoxy nanocomposite. The nanocomposite samples of varying volume fractions are prepared using solution mixing technique. A two-step mixing procedure with magnetic stir and bath type ultra sonicator is employed to acquire well dispersion of CNTs in epoxy resin. Three-point bending, tensile and impact tests are conducted on the prepared samples to measure the elastic modulus, strength and failure strain respectively. The morphological analysis of fractured samples is carried-out under field emission scanning electron microscopy (FESEM). The dispersion and reinforcing mechanism of CNTs in a polymer matrix are observed. The results show that a small amount of CNTs in a polymer matrix improves the stiffness and strength of the composite considerably. The agglomeration occurs beyond the 0.5 wt% of CNTs. Furthermore, towards thermal stability analysis, the improvement of viscoelastic characteristics with the addition of MWCNTs is studied using dynamic mechanical analysis test. Results are shown in the form of graphs and tables.

1. Introduction

Polymer nanocomposites are known for their high strength and conductivity. These materials are finding widespread applications in diverse engineering fields including aerospace, automobile, biotechnology and structural engineering. Carbon nanotubes are found to be attractive fibrous reinforcements in polymeric resins [1]. A high elastic modulus up to 1 TPa and tensile strength of approximately 60 GPa were reported by various theoretical and experimental studies [2-4]. Even with small amounts of CNTs, the polymer acquires significant improvements in mechanical, electrical and thermal properties [5-8]. Epoxy resins have good mechanical and thermal properties and are used as matrix materials in several nanocomposites. CNT reinforced polymer composites are found to be robust alternative as modern structural members. Therefore, different experimental studies are needed to predict their behavior under static and dynamic loading conditions [9-10]. Several experimental studies were found on mechanical characterization of CNT reinforced polymer composites but several challenges persist due to the limited control of CNT dispersion, alignment and interaction at interfaces. In situ polymerization, injection molding, solution mixing and press drawing are couple of procedures or techniques which are most widely utilized for the manufacture of polymer/CNT nanocomposites. The critical issues in selecting a fabrication method for these composites are ease of production, cost effectiveness and wastage minimization. Jindal et al. [11] presented a two-step procedure to fabricate the multi-walled carbon nanotubes (MWCNTs)-polycarbonate composite and performed nano indentation test to identify the elastic modulus and hardness. An enhancement up to 95% in elastic modulus and 200% in hardness was reported with the addition of 2 wt% CNT. Wu et al. [12] performed a layer-by-layer deposition to fabricate high performance CNT/polymer composite with controlled CNT dispersion. An epoxy based composite with the help of pristine and surface modified CNTs was fabricated by Ramana et al. [13] and carried-out the rheological and morphological examination to know the dispersion effect of CNTs on fractography. An improvement is also observed in flexural properties of modified CNT based composites. Vahedi et al. [14] investigated the effect of the addition of MWCNTs content on mechanical properties of epoxy resin and found maximum tensile strength and modulus occurred at the weight percent of 0.1 and 0.25 respectively. The Acrylonitrile Butadiene Styrene (ABS)/MWCNTs nanocomposite has been prepared using the solvent blending process [15]. A significant improvement has been noticed in elastic modulus ranging from 150 to 330% with the addition of MWCNTs (content varied 1 to 10%). Bansal et al. [16] fabricated polypropylene (PP)/MWCNTs composite using solution mixing method and characterized the elastic modulus and hardness. Fontananova et al. [17] developed a MWCNTs/ poly vinylidene-fluoride composite film with better interfacial interaction using functionalized CNTs. The results showed enhanced thermal, mechanical and transport properties of a polymeric film. Moumen et al. [18] investigated the impact resistance and damage evolution in CNT/epoxy laminated composite and observed that impact resistance of laminate reinforced with randomly oriented CNTs can be improved up to 15.6% at high strain rate. Saha and Bal [19] predicted the mechanical and thermomechanical properties of functionalized CNT based epoxy composite and observed 101%, 166%, and 61% improvements in flexural modulus, strength and storage modulus respectively with an addition of 0.75 wt% CNTs. Liu et al. [20] studied the mechanical characteristics of a reinforced polyurethane composite system. In another study [21], MWCNTs were used as reinforcements in polyvinylidene fluoride and polymethyl methacrylate base by melt mixing and hot press molding processes to prepare nanocomposite samples for mechanical characterization.

The viscoelastic analysis of CNT nanocomposites has received much attention over the past few years. While CNTs tend to deform elastically, the polymer matrix behaves in a viscoelastic manner, leading the composite to behave as viscoelastic. The response in creep and stress relaxation is one mode of viscoelastic study. The creep resistance of polypropylene nanocomposite with 1vol% MWCNTs was studied [22] and observed a notable reduction in creep deformation and creep rate. Plaseied and Fatemi [23] reported a reduction of creep strain with 0.5 wt% CNT in thermoset vinyl ester resin at 50°C. Jia et al. [24] conducted a significant experiment on CNT polypropylene nanocomposites towards finding viscoelastic properties. Another way to study viscoelasticity is by subjecting the material to variable loads at a moderately high frequency and observes their behavior during deformation. The analysis is conveniently carried-out with an oscillating sinusoidal load applied to the specimen at a particular frequency. Suhr et al. [25] conducted the shear tests on epoxy thin films containing densely packed MWCNTs and reported a significant increase of loss factor which they attributed to the frictional energy dissipation due to interfacial sliding at nanotube interface. With an effective medium theory, Pan et al. [26] illustrated the effect of interface conditions on viscoelastic characteristics of CNT nanocomposites. Although various studies have employed different polymeric resins for CNT reinforcements, use of Bisphenol-A Diglycidyl Ether (BDE) based resins as matrix material in nanocomposites is found to be very uncommon. Present work investigates the flexural, tensile and impact behavior of MWCNT reinforced epofine 1564 (modified BDE) low viscous resin based nanocomposite. Composite plates of different weight fractions are prepared and the required size samples are cut for tensile, flexure, impact and DMA (Dynamic Mechanical Analyzer) tests. Morphological study of fractured samples is carried-out using electron microscopy to understand the internal microstructure. Thermal stability tests are conducted on DMA to know the variation of storage and loss modulus as well as loss factor for the sample at different weight fractions. The results provide upper bounds for CNT weight fractions in epofine resins to minimize the agglomeration effects.

2. Experimental

In this section, fabrication procedure, materials used in fabrication and sample preparation techniques of MWCNTs/epoxy composite are explained briefly.

2.1 Fabrication of MWCNTs/epoxy composites

The experimental analysis was carried out by preparing five different kinds of sample with 0 wt%, 0.1 wt%, 0.3 wt%, 0.5 wt% and 1 wt% CNT content respectively. Four main materials that used in fabrication were multi-walled carbon nanotubes, pure epoxy, curing agent (Finehard-3486) and acetone. The complete fabrication procedure is depicted in Figure 1.

First, pre-calculated amount of CNT was mixed with 70 ml acetone at room temperature in order to minimize the agglomeration effect. The solution was kept for half an hour on magnetic stir at 400 RPM and oscillated by bath ultra-sonication waves for 30 min. Then, the required amount of resin was preheated to 120°C for 30 min and then mixed with the acetone-CNT solution. Now the total weight of the solution is measured and the solution was kept on magnetic stir (140°C and 600 RPM) for approximately one hour or till acetone gets evaporated completely. After that solution was carried for bath ultra-sonication and oscillated for 30 min at 70°C. During this whole process there may be a chance of air entrapped into the solution, so the solution was put into a vacuum state for degassing at 30 kg·cm⁻³ over 30 min. Then the solution was taken out from vacuum and curing agent is mixed in a ratio of 100:34 of weight as guided by the company. After mixing the curing agent, again this mixture is kept in vacuum for degassing for 10 min. A big casting mold was used to prepare a flat panel and mixture was poured into the mold. Finally, a nanocomposite specimen flat panel with large dimension was obtained from the mold. It seems that specimen's transparency decreases (becomes darker) with higher weight fraction of CNTs in the resin.

2.2 Materials

In this study, nanocomposite was prepared from an epoxy (Epofine-1564) resin and MWCNTs supplied by United Nanotech Innovation Private Limited, Bangalore, India. This resin has a liquid form made up of Bisphenol-A group and showed the density in the range of 1.10-1.20 g·cc⁻¹ and viscosity 1200-1400 mPa-S (measured at 25°C). Finehard-3487 was selected a curing agent which provides fast curing for such type

of resins. Epofine-1564/Finehard-3487 system has application in small rotating structures and recommended for the manufacture of composite windmill blades. MWCNT has an average outer diameter 5-20 nm, length 1-10 μ m with a bulk density of 0.20 gcc⁻¹ and purity more than 98% respectively.

2.3 Samples preparation

The test samples were prepared according to ASTM standards and were cut from the prepared large panel using a hacksaw as per test requirements. The tests under tensile loading were conducted according to the ASTM D638-03 with dog-bone shaped specimens. Samples with the configuration of length 100mm (included span length 40 mm), width 10 mm (included span width 6 mm) and thickness 3 mm respectively were prepared in tensile testing. All tests were completed at room environment and relative humidity of 57%. Bending specimens were prepared according to ASTM D790-02 with the length of 60 mm (included span length equivalent to 16 times the thickness), width of 12 mm and thickness of 3 mm

respectively. Charpy notched impact tests were carried out as per ISO 179-1 standard on the impact-testing machine. The specimens with the size of $55 \times 10 \times 5$ mm are used for testing. In tests, a hammer with a striking velocity of 5.0 m·s⁻¹ to 5.5 ms⁻¹, weight 18.75 kg, strike edge angle 300 ± 10 was used. The bulk specimen obtained from mold and test samples of MWCNTs/ epoxy are shown in Figure 2.

Scanning electron microscopy (SEM) images were obtained with a JEOL JSM-6084LV model microscope operating under vacuum (10-270 Pa) with a 30.0 kV accelerating voltage. FESEM images were obtained by Nova NanoSEM/FEI model with 1 kV equipped with EDAX. DMA was employed as per ASTM D4065-01 to determine the viscoelastic behavior of the nanocomposite samples as a function of temperature. DMA test was performed using TA (DMA Q800) instrument, operating in a three-point bending mode at an oscillation frequency of 1 Hz under controlled amplitude. The temperature was ramped from 40°C to 200°C, under controlled sinusoidal strain with a heating rate of 5°C·min⁻¹. The samples were in the dimension of 60×12.5×3 mm³.



Figure 1. Fabrication procedure of MWCNTs-epoxy nanocomposite specimen.



Figure 2. Bulk specimen obtained from mold and test samples.

3. Results and discussion

Mechanical and viscoelastic properties of MWCNTs/ epoxy composite system have been investigated in present study. A simple and economical fabrication process has been adopted in the study. Flexure and tensile behavior of MWCNT/polymer composite were initially evaluated through a three-point bending test and an axial tensile test respectively on a universal testing machine (Instron model 5967 with electromechanical test frame). A fixture comprises of two supports (6 mm in diameter and set 48 mm apart) and a loading platen mounted on an instrumented rig was used for three-point bending test. The arrangement is shown in Figure 3. The machine has a 30 KN servohydraulic bed equipped with a test data acquisition system. Its crosshead was run under controlled displacement mode at speed of 2.0 mm·min⁻¹. Five samples were prepared for each test with standard dimensions to confirm the accuracy and stability in results. The results have been discussed in three sub sections namely as mechanical characterization, morphological analysis and viscoelastic testing of MWCNTs/epoxy nanocomposites.

3.1 Mechanical characterization

There are three main test to investigate the mechanical behavior of an elastic material. Three-point bending test, axial tensile test and charpy impact test are employed here to examine flexural, tensile and impact properties of MWCNTs/epoxy composite. First, flexural stress and stain behavior has been obtained by threepoint bending test and depicted in Figure 4(a). It shows that stress-strain curve slop increases with the addition of CNTs in resin but a sudden drop is observed at high content of CNTs (1 wt%). This may be due to formation of CNTs bundles which results in to low interphase area and less load transfer from polymer matrix to filler. The variation of flexural modulus, strength, and elongation of nanocomposite samples with MWCNTs content are shown in Figure 4(b) and 4(c). It can be seen that the flexural properties increases with MWNTs content up to 0.5 wt%, but decreases after 0.5 wt%. Such a decrease in flexural properties at higher nanotube loading is probably due to the fact that, the nanotubes formed a cluster or agglomerate among themselves, resulting in a filler-filler interaction and a poor interface between MWNTs and epoxy matrix. The critical issues for achieving desirable properties of the CNT/polymer composites are to introduce a high content of CNTs with uniform dispersion and controlled alignment throughout the polymer matrix.



Figure 3. Experimental setup used for flexural testing (a) 3-point bending arrangement and (b) Machine set-up.

A tensile stress vs strain behavior of MWCNTs/ epoxy nanocomposite is depicted in Figure 5(a). The maximum slop can be observed for 0.5 wt% of MWCNTs reinforced nanocomposite. The effect of CNTs addition on tensile modulus, strength and failure strain of polymer nanocomposite is shown in Figure 5(b) and 5(c). It is obvious that Young's modulus and yield stress of nanocomposites are higher than those of pure resin, and tends to increase substantially with the MWCNTs content in composites. The effect of the 0.5 wt% MWCNTs was even more pronounced in tensile yield stress (Figure 5 (b)), where an average improvement of 35% was observed. This may be related to the more uniform dispersion of nanotubes in the matrix, leading to better interfacial properties.



Figure 4. Flexure properties of MWCNTs/epoxy composite with various wt % of CNT: (a) Flexure stress vs strain, (b) Variation of flexural modulus and (c) Variation of flexural strength and failure stain.



Figure 5. Tensile properties of MWCNTs/epoxy composite with different wt% of CNT: (a) Tensile stress vs strain, (b) Variation of Tensile modulus and (c) Variation of flexural strength and failure stain.

Figure 6 shows the impact properties of U-notched samples of MWCNTs/epoxy nanocomposite. The charpy impact strength of the notched specimens slightly increased as the MWNTs content increased up to 0.5 wt%. This increase is significantly larger for the samples with 0.3 wt%. This is due to the fact that notched impact behavior is controlled to a greater extent by factors affecting the propagation of fracture initiated due to stress concentration at the notch tip. Lowering of impact energy at higher nanotube content is due to the presence of few master-batch aggregates in the composite. Tensile testing involves high deformation, while impact strength is determined by local cracking.



Figure 6. Impact strength of pure resin and nano-composite samples.

Further, the elastic moduli of the nanocomposite samples are correlated with the dispersion degree, interfacial properties and curved shape of CNTs. CNTs are considered to be arranged in 3-D random orientations. In this regard, few modifications [27] are suggested to the conventional Halpin-Tsai model for accounting the nonlinear elastic behavior with volume fraction and is represented as:

$$E_{c} = \left(\left\{ \frac{3}{8} \right\} \frac{1 + \xi'_{L} \eta'_{L} V_{cnt}}{1 - \eta'_{L} V_{cnt}} + \left\{ \frac{5}{8} \right\} \left[\frac{1 + \xi'_{T} \eta'_{T} V_{cnt}}{1 - \eta'_{T} V_{cnt}} \right] \right] E_{m}$$
(1)

$$\eta_L' = \left\{ \frac{1}{(K_i K_w E_{cnt}/E_m) + \zeta_L'} \right\}$$

$$\eta_T' = \left\{ \frac{(K_i K_w E_{cnt}/E_m) - 1}{(K_i K_w E_{cnt}/E_m) + \zeta_T'} \right\}$$
(2)

$$\xi_L' = 2 \left(\frac{L_{cnt}}{D_{cnt}} \right) K_{agg}, \qquad \xi_T' = 2$$
(3)

where suffices cnt, m and c indicate the elastic and structural properties of CNT, polymer matrix and composite respectively. K_{i} , K_{w} , K_{agg} are the factors to accommodate the interphase, waviness and agglomeration effects. These factors depend on the geometric and manufacturing process of CNT reinforced polymer composite. The Young's modulus of the multi-walled nanotubes is considered to be uncertain (here $E_{cnt}=500$ and 1000 GPa) as it is not constant as in a singlewalled nanotube and the aspect ratio (L_{cnt}/D_{cnt}) is considered as 2000. Using E_m =2.841 GPa as obtained from experiments and applying the above equations for 0.1 wt% CNTs yielded the results shown in Table 1. The theoretical estimation of Young's modulus for 0.1 wt% nanofillers varied within 2.92-3.37 GPa and is in agreement with the experimental value. According to the literature, epoxy and other nanocomposites systems may yield results just as accurate as more sophisticated models.

The mechanical properties of polymer nanocomposites, especially the modulus, depend to a great extent on filler dispersion and interfacial interaction and are increased only when a good dispersion of the nanofiller and effective stress transfer at the polymer/ filler interface are guaranteed. Here, MWCNTs are able to reinforce the polymer matrix effectively due to their larger aspect ratio combined with high mechanical properties.

Table 1. Predicted result for 0.1 wt % of MWCNTreinforced composites.

Major influencing factors			Predicted modulus <i>E_c</i>	
Kw	Ki	Kagg	<i>E_{cnt}=1000</i> GPa	<i>E_{cnt}=500</i> GPa
0.2	0.2	0.2	2.99	2.92
0.2	0.4	0.4	3.12	2.99
0.2	0.6	0.6	3.25	3.06
0.2	0.8	0.8	3.37	3.12
0.2	0.4	0.8	3.12	2.99
0.2	0.6	0.8	3.25	3.06
0.4	0.2	0.2	3.12	2.99
0.6	0.2	0.2	3.23	3.05
0.8	0.2	0.2	3.35	3.12

3.2 Morphological analysis

Nanofiller's reinforcing efficiency in the epoxy resin is primarily determined by the degree of dispersion and changes in surface morphology during fracture. Therefore, morphological characterization is very important for the evaluation of the dispersion state of carbon nanotubes in the polymer matrix. In the present study, fracture surface analysis and dispersion of MWCNTs in epoxy resin were examined by using and Scanning Electron Microscopy (SEM) and Field Emission Scanning Electron Microscopy (FESEM) respectively. The SEM images of pure epoxy resin and nanocomposite samples containing 0.1 and 1 wt% MWCNTs are shown in Figure 7.

The observations come from SEM are for the fractured surfaces of composites and give first information about fracture mechanisms and the influence of particle modification on the fracture behavior. The fracture surfaces in Figure 7 (a) demonstrate a typical feature of brittle fracture behavior, thus accounting for the low fracture toughness of the unfilled epoxy. The surface is smooth, with highly directional deformation lines, which means that the crack propagated is not interrupted. However, the fracture surfaces of composites in Figures 7(b) and 7(c) seem to be comparatively rougher. The ridge patterns and river marks indicate that the composite has undergone a more ductile fracture. This means that the energy required for the propagation of the crack increased [19]. This is better understood with the obtained impact results, where it was shown that nanocomposites have greater impact strength compared to pure epoxy. The micrograph of FESEM for pure epoxy and MWCNT reinforced nanocomposites are as shown in Figure 8. It is observed from Figure 8(b) that the MWCNTs are

playing a role of bridging member and resisting for crack propagation as compared to pure epoxy specimen case as in Figure 8(a).

Figure 9(a) shows reasonably uniform dispersion and good distribution of the MWCNTs as observed with a small number of aggregates. A large amount of self-organized MWCNTs bundles was observed at high CNT content as shown in Figure 9 (b) on the failed surface. This indicates a significant part of the nanotubes was dispersed as nanotubes aggregate due to the imperfect mixing of the masterbatch.



Figure 7. Microscopic fractured surface analysis of MWCNTs/epoxy nanocomposite samples: (a) Pure epoxy (b) 0.1 wt% MWCNTs and (c) 1 wt% MWCNTs specimen



Figure 8. FESEM images of fractured MWCNTs/ epoxy nanocomposite bending samples: (a) Pure epoxy and (b) 0.1 wt% CNT.



Figure 9. FESEM analysis of fractured samples with low and high content of MWCNTs: (a) 0.1 wt% CNT (uniform dispersion) and (b) 1 wt% CNT (agglomeration).

3.3 Viscoelastic testing

Storage or elastic modulus is a measure of the stiffness of the sample. Figure 10 shows the storage modulus (E') against temperature for five different samples at a frequency of 1 Hz. From the graph, it is clear that pure resin sample has a minimum E' values, as it offers a lower degree of stiffness. An improvement in stiffness of all composite samples is observed at lower temperatures. As temperature increases, storage modulus decreases for all samples but a rate of decrement of E' is higher for 1 wt% CNT case.



Figure 10. Effect of CNT content on the storage modulus (E').

Loss modulus is considered to be the viscous response of the material and can be measured as energy loss under stress or deformation as heat/cycle [28]. The DMA curves of E" versus temperature are depicted in Figure 11. From E" plot it is seen that loss modulus also follows the very similar trend as storage modulus. The E" peak height is lower for pure epofine samples but with an addition of MWCNTs into epofine matrix, the loss modulus peak values increase as reported in the earlier literature. The increment in peak amplitude with an addition of CNT in the epofine matrix can be attributed to the increase in energy dissipation during the viscoelastic deformation of surrounding polymer of CNT. However, beyond 80°C, the loss modulus drops abruptly for 1 wt% CNT case due to agglomeration and non-homogenous dispersion in the two phase system.

Tan δ is the measure of the damping factor and it also specifies the elastic or viscous properties of a system. Its peak height is related to the internal energy dissipation of the matrix/filler interphase whereas peak position determines the glass transition temperature. Variation of Tan δ as a function of temperature is shown in Figure 12. It is observed that with 1 wt% CNT, the sample exhibits the highest peak value (about 0.94) at around 95°C and thereafter drops abruptly. It was also evident from the Tan δ plot that as the temperature increases damping increases to its maximum in the transition region and then starts to decrease in the rubbery region.



Figure 11. Effect of CNT content on the loss modulus (E").



Figure 12. The effect of CNT content on loss factor $(\tan \delta)$.

4. Conclusions

Multiwall carbon nanotubes (MWNTs) reinforced epofine (epoxy) resin nanocomposite materials have been prepared by solution mixing techniques by varying the filler weight fractions. Bath type ultra-sonication process was carried out along with magnetic stirring to promote the carbon nanotubes dispersion. Following conclusions have been drawn from the present study.

1. The morphological characterization based on scanning electron microscopy has confirmed that 0.1% MWCNTs exhibits best dispersion in the polymer matrix with present processing techniques and parameters.

2. The mechanical characterization has shown that both the tensile and bending moduli and strengths of the nanocomposites increase by the addition of nanotubes. An addition of 0.1% MWCNTs to the epoxy resin resulted in 20.5% and 14.85% increment in flexural strength and modulus respectively. It is observed that tensile elongation at break decreases with the addition of MWCNTs.

3. A slight improvement has been confirmed in impact strength with the addition of MWCNTs. Only 3% improvement in impact strength is observed for 0.1% MWCNT reinforced nanocomposite.

4. Thermal characterization with dynamic mechanical analysis revealed the enhanced thermal stability and remarkably improvement in dynamic properties (E' and E'') compared to neat epoxy sample. However, an increase in CNT weight fraction decreases the glass transition temperature and tan δ peak value, which resulted into low structure homogeneity and crosslinking density.

This work is limited to low percentage of CNT content in polymer matrix and work can be carried out for high CNT content with advance fabrication techniques under controlled parameters in future.

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