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Experimental Investigation for Multi Walled Carbon Nanotubes in Polymer based Matrix for Structural Applications

Shankar A. Hallad[1], N. R. Banapurmath[2], Anand M. Hunashyal[3], N. H. Ayachit[4], A.S.Shettar[5]
Center for Material Science[1,2,3]
Department of Mechanical Engineering[1, 2]
Department of Civil Engineering [3, 5]
Ranichannamma University Belgaum[4]
B.V. Bhoomaraddi college of Engineering and Technology Hubli, Karnataka, India
nr_banapurmath@rediffmail.com

Abstract
The present paper investigates the behaviour of polymer matrix beam reinforced with multi-walled carbon nanotubes (MWCNTs) to study mainly the strength aspects for structural applications. The addition of MWCNTs in the control beams was varied at 0.25, 0.5, 0.75 and 1 percent by weight of polymer matrix. Dispersion of MWCNTs was carried out using ultrasonic energy. Composite beams were tested under flexure in order to evaluate their mechanical property such as load–deflection criteria. These results were then compared with those from plain polymer beams. The present work also investigates the optimum percentage of MWCNTs that gives the best results in terms of both enhanced properties and economical aspects. Scanning electron microscopy and Energy dispersion X-ray spectroscopy was conducted to examine the interfacial surface adhesion between the MWCNTs and the polymer matrix. Reinforcement of polymer beams with MWCNTs 0.75% by weight of the polymer matrix showed enhanced results in terms of increased flexural strength by 65% compared to plain control beams.

Key Word and Phrases
Epoxy Resin, Three Point Loading, SEM, EDS, Sonication, MWCNTs, Nano-Composites.

1. Introduction

Carbon nanotubes (CNTs) have been considered as one of the effective reinforcement nanoparticles for polymer based composites owing to their outstanding mechanical properties as well as high aspect ratio [1], [2]. However, their results in reinforcing a polymer matrix so far has been poor, which was revealed by many researchers to two main issues: (1) the difficulty of dispersing CNTs in polymer matrix (2). Insufficient bonding at the nanotube/polymer interface. It has been identified from long time that the mechanical properties of polymer materials can be enhanced by fabricating composites that are imbibed with different volume fractions of one or more reinforcing phases. With the passage of time, practical realization of composites has begun to shift from micro-scale composites to nano-scale composites, because of the unique blending of mechanical, chemical and physical properties of nanofillers – fillers with a characteristic dimension below 50nm. The idea of dispersing nanoparticles is due to the large enhancement in the specific surface area and interfacial area they present to the matrix phase.

As traditional composites use over 35 wt% of reinforcing phase, the dispersion of just a few milligrams of nanoparticles into polymeric matrix could lead to drastic changes in their mechanical properties with added functionalities. Fiber-reinforced composites have emerged as a major class of structural material and are either used or being considered as substitutions for metals in many weight-critical components in aerospace, automotive, and other industries. In this work, we propose to reinforce the adhesive layer through the homogeneous dispersion of only a small fraction of carbon nanotubes (CNTs). CNTs are regarded as one of the most promising reinforcement materials for the next generation of high-performance structural and multifunctional composites [3]. These molecular scale tubes of graphitic carbon have outstanding mechanical, thermal and electrical properties. In fact, some CNTs are stronger than steel, lighter than aluminum and more conductive than copper [4]. Theoretical and experimental studies have shown that CNTs exhibit extremely high tensile modulus (1 TPa) and strength (150 GPa). In addition, CNTs exhibit high flexibility, low density (1.3–1.4 g/cm3) and large aspect ratios (1000s). Due to this unique combination of physical and mechanical properties, CNTs have emerged as excellent candidates for use as reinforcing agents in polymeric materials to yield the new generation nanocomposites. Perhaps the most remarkable improvement in the tensile modulus and yield strength of a polymer through the dispersion of CNTs was observed by Liu et al. [5]. By dispersing only 2 wt% of multi-walled carbon nanotubes (MWCNTs) in a nylon-6 matrix Liu et al. observed an increase of approximately 214% in the tensile modulus and 162% in the yield strength. They attributed these impressive improvements in the stiffness and strength to a uniform and fine dispersion of the CNTs and good interfacial adhesion between the nanotubes and matrix which were assessed using SEM. In the present work the mechanical performance of polymer beam reinforced with MWCNTs is compared with mechanical performance of plain beams for load v/s deflection criteria, based on the performance we can conclude that polymer reinforced beam yielded best results as compared to plain beams. This could be due to the mechanical joggling of the nanotubes at the fiber/polymer interface.

2. Experimental Programme

The properties of the MWCNTs used in this case are given in Table 1; the MWCNTs were of industrial grade with a purity of greater than or equal to 95 percent. The specimen characteristics have been mentioned in Table 2 .The specimen reference has been mentioned in Table 3.Uniform dispersion of MWCNTs against their agglomeration due to Vander Waals bonding is the first step in the processing of nano composites. Dispersion is a critical issue while mixing CNTs in either water or organic solvents. The method of sonication, duration of sonication and method of casting the specimens were maintained uniformly throughout. Different predefined amounts of MWCNTs were added to polymer matrix as shown in Table 3 and the whole mixture was kept in an ultrasonicatior for 120 min to achieve uniform dispersion in the matrix.

At the first stage of the experiment, small-scale experimental testing was conducted to investigate the efficiency of uniformly dispersed, randomly oriented CNTs as reinforcement for epoxy composites. Single-point bending tests on 40 mm x 12 mm x 6 mm beams were carried out as per the ASTM standard to compare the load v/s deflection responses of plain epoxy beams and CNT reinforced epoxy beams.

3. Preparation of Specimens

The CNTs employed in this work were industrial grade MWCNTs with a purity of 95 wt% and a concentration of 0.25%, 0.50%, 0.75% and 1% by the total weight of the epoxy matrix. Hardner is added by 10% the weight of epoxy matrix to initiate the crystallization of epoxy resin. The final product was placed in 40mm x 12mm x 6 mm wooden molds in layers, which were compacted by using a tamping rod. The specimens were then cured in a room for 48 hrs before being removed from the molds.

Table 1: Properties of the MWCNTs used for study

Specifications	Dimensions
Diameter	10–30 (nm)
Length	1–2 (mm)
Purity	0.95 (%)
Surface area	350 (m^2/g)
Bulk density	0.05–0.17 (g/cm^3)
Density	1.8 (g/cm^3)
Tensile strength	3500 (N/mm^2)
Length of fiber	5 (mm)
Fiber thickness	0.3 (mm)

Table 2: Specimen characteristics utilized for experimental work

Characteristics of specimen	Particulars
Size	40(mm) x 12(mm) x 6(mm)
Epoxy resin	L-12
Hardner	K-6
Amount of MWCNTs	0.25, 0.5, 0.75, and 1 per cent by Weight of epoxy

Table 3: Details of the test specimen

Sample No.	Specimen Reference	Constituents	Percentage of MWCNTs by weight
1	PE	Plain epoxy	Nil
2	A1	Plain epoxy+MWCNTs	0.25
3	A2	Plain epoxy+MWCNTs	0.50
4	A3	Plain epoxy+MWCNTs	0.75
5	A4	Plain epoxy+MWCNTs	1.00

Table 4: Specimen size used for the structural scale tests

Sl. No	Type of test conducted	Size of specimen
01	Flexural test (Beam) a) Three point load	40(mm) x 12(mm) x 6 (mm)

4. Three Point Load Test on Beams

The mechanical performance of the hybrid nano-composite material reinforced with MWCNTs in polymer based matrix was evaluated by fracture mechanic test. Beam specimen of size 40mm x12mmx6 mm were tested by three–point loading test as shown in (Fig.1). Six replications were made for each nano-composite specimen tested. A hydraulic closed loop testing machine was used. For experimental accuracy, ASTM D2344M was followed to determine the average values of the flexural strength for polymer beams. The equipment used for the three-point load test is shown in (Fig.2). The specimen size and type of test conducted is shown in Table 4.

Fig. 1 Sample placements for three point load set-up

Fig. 2 Equipment used for three- point load set-up

5. Results and Discussions

Evaluation of optimum percentage of MWCNTs required for reinforcing plain epoxy beams based on three point loading test .We evaluate the optimum percentage of MWCNTs by wt % of epoxy required to reinforce the plain epoxy beams, which gives highest structural efficiency in terms of load carrying capacity. Hence the flexural behavior of multiwall carbon nanotubes reinforced in epoxy beams is investigated. Composite beams were tested under flexure (Three point loading) to evaluate their mechanical properties such as strength, deflection criteria etc. The results obtained were compared with the results of the tests using control beams. The designation of the test specimen used is shown in Table 5.

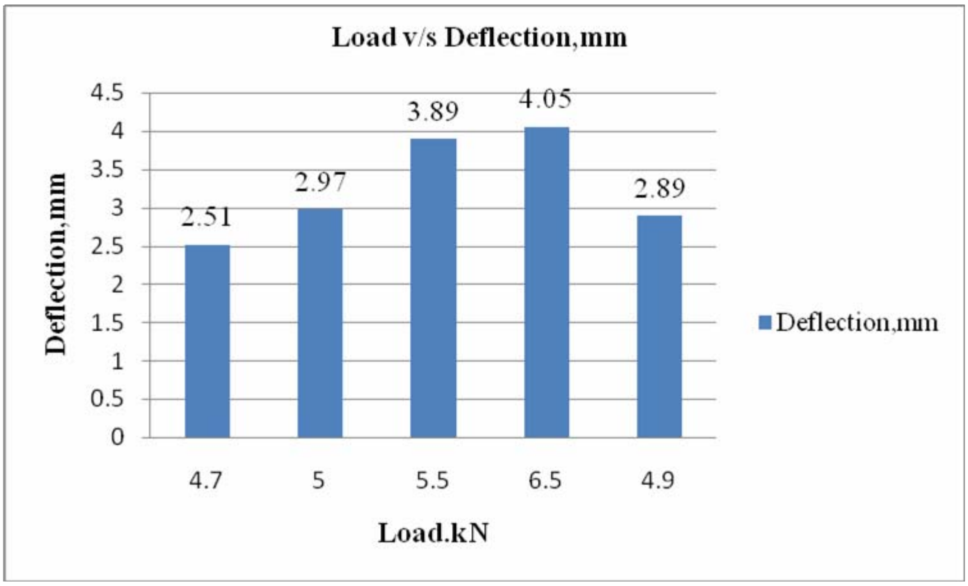


Fig. 3 Load-Deflection curves for different proportions for MWCNTs reinforced epoxy composite beams subjected to three point loading test

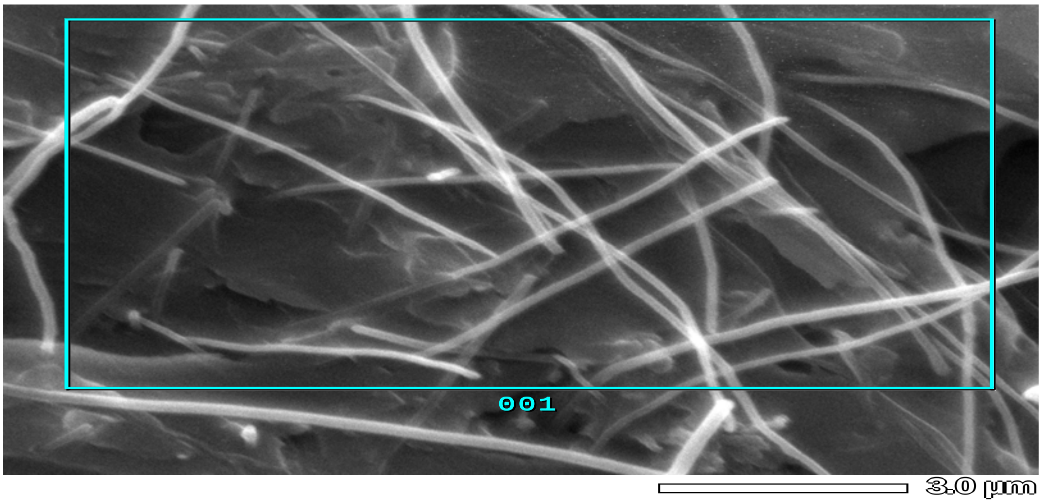
Table 5: Test results for MWCNTs reinforced in epoxy composite beam subjected to three point loading test: Ultimate load

Sl.no	**Specimen Reference**	**Ultimate load in (kN)**	**Maximum deflection in (mm)**
1	Plain epoxy (PE)	4.7	2.51
2	PE + 0.25 wt. % of MWCNTs* (A1)	5.0	2.97
3	PE + 0.50 wt. % of MWCNTs* (A2)	5.5	3.89
4	PE + 0.75 wt. % of MWCNTs* (A3)	6.5	4.05
5	PE + 1.0 wt. % of MWCNTs* (A4)	4.9	2.89

*Note-wt. % w.r.t. to epoxy

The aim here is to determine the optimum percentage of MWCNT dosage and its effect on the ultimate load-carrying capacity of the epoxy beam under three-point loading. Fig. 3 shows the load–deflection curves for composites with different percentages of MWCNTs in the epoxy matrix. The variation of ultimate load was studied keeping the PE beams as reference. The ultimate load followed an increasing trend up to 0.75 wt. % of MWCNTs (A3) showed a maximum ultimate load, since the composite at greater MWCNTs contents has a tendency to undergo large deflections; the MWCNTs provide additional toughness to the composite. Deflections observed showed that it is maximum in (A3) case and that the ultimate load of the material is also high.

For higher MWCNTs contents the composite has a tendency to undergo large deflections and the fibers provide additional toughness to the composite. By this the energy absorbing capacity of the composite generally increases because greater amount of load has been carried as the MWCNTs resists the crack propagation. Therefore **(**A3) is considered to be optimum from both deflection and strength criteria. This suggests that the energy-absorbing capacity of the composite generally increases, because a greater amount of load is carried as the CNTs resist the crack propagation. Fig 3 shows variation of load with deflection for various control beams considered. From Fig 3 it follows that reinforced polymer beam shows higher resistance for deflection when compared to plain polymer beam. This could be due to load transferring ability of the fibers was found to be improved due to the mechanical joggling of the nanotubes at the fiber/polymer interface. As the % of CNTs increased in polymer based matrix the strength further increased , this is observed till 0.75% by weight of CNTs this could be due to the high surface area of nano-particles attracts the polymer molecule which reduces the mobility of polymeric chains and hence causes increase of viscosity in the polymer matrix. For 1% CNTs by weight in polymer based matrix the strength decreased drastically this could be due to increase in the nanotube waviness may be an additional mechanism limiting the modulus enhancement of nanotube-reinforced polymer beams. It has been observed that even slight nanotubes waviness significantly reduces the effective reinforcement when compared to straight nanotubes.



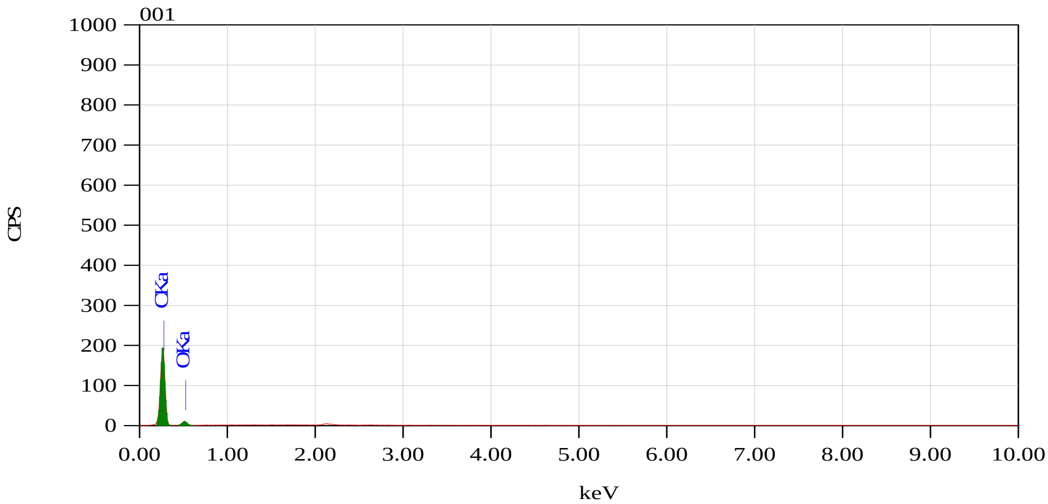


Fig.4 SEM images EDS spectrum of CNTs with 0.25% by weight

Fig 4 shows a SEM micrograph and EDS spectrum of the CNT/epoxy composite with 0.25 vol% CNTs, from which it follows that CNTs act as bridges across pores and cracks. This indicates that high bonding strength between the CNTs and epoxy matrix is achieved. This is consistent with other results published in the literature [6, 7].Experimental observations revealed that the MWCNT-reinforced epoxy composite beams showed increased strength compared with plain epoxy beams. The nano-level reinforcement significantly improved the flexural strength of the beams [7, 8]. The results showed an increase in the load carrying capacity of the composite beams compared with the reference beams.

6. Conclusions

Polymers reinforced with carbon nanotubes is an actively researched area and the hybrid nanocomposites thus developed has been used in different structural applications such as fuel systems components in cars, windmill blades, cables, tethers, beams etc.

From the above experimental results it can be concluded that polymer beams reinforced with MWCNTs samples developed in-house showed optimum results for mechanical properties as compared to plain beams. This could be due to the mechanical joggling of nanotubes at the fiber/polymer interface, the high surface area of nanoparticles which attracts the polymer molecule and thereby reduce the mobility of polymeric chains and hence causes increase of viscosity in the polymer matrix. The elastic extension nature of the polymer matrix also lead to the telescopic extension nature of CNTs which is a greatly deserved factor for the enhancement of strength by transferring the load carrying capacity to CNTs.

Further work in this research area involves development of new hybrid nano-composites using both nano and micro-materials that could address both micro/nano level aspects. Accordingly use of different nano-particles like carbon nano fibers (CNFs), Graphene oxide at nano-level and carbon fibers (CFs) at micro level with natural fibers could also be effectively used for structural applications.

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