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ORIGINAL ARTICLES

Experimental Study On Tensile Behavior Of Multi Wall Carbon Nanotube Reinforced Epoxy Composites

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ABSTRACT

The present study is concerned with a method for producing a reinforced polymer by introducing Carbon Nanotube into the polymeric host. It aims to investigate the tensile characteristics of Multiwall Carbon Nanotubes (MWCNTs) reinforced epoxy composites. Tensile test specimens of the composite were fabricated by increasing concentration of surface modified MWCNTs using molding method at room temperature. The investigation clearly exhibits the 1.25wt.% of epoxy/MWCNTs nanocomposites have enhanced tensile characteristics. These results suggest that targeted chemical modification of the carbon nanotube surface is an effective way to enhance the mechanical properties of carbon nanotube–polymer composites. The optimum loading of MWCNTs in polymeric host has been evaluated.

Key words: MWCNTs; Epoxy; Nanocomposites; TEM; SEM; Tensile; Polymerization.

Introductions

Fabrication of hybrid composite has the maximum attention in mechanical application currently by using varieties of nanomaterials. Due to exceptional mechanical, electrical and thermal Properties of Carbon nanotubes (CNTs), it has become an ideal fillers for polymer nanocomposites for various structural and functional applications (Xie XL, *et al*., 2005; Ajayan PM, *et al*., 1994). Factors influencing the properties of CNT nanocomposites have been extensively studied and the development of nanocomposites with much improved mechanical and functional properties have been reported (Ma PC, Kim JK, Tang BZ, 2007; Li J, *et al*., 2007; Geng Y, *et al*., 2008). The mechanical and fracture properties were improved after addition of small quantities of carbon nanotubes to the matrix (Seyhan T, *et al*., 2008; Cho J, Daniel I.M., 2008). Allaou state that the Young's modulus and the yield strength have been doubled and quadrupled for composites with respectively 1 and 4 wt.% nanotubes, compared to the pure resin matrix samples (A. Allaoui, *et al*., 2002). It is also well established that the quality of composite components depends on the processing route adopted for fabrication. Selecting right filler is important; Herna´ndez-Pe´rez experimental investigation states that the nanotube morphology and impurity content can significantly affect the effective properties of the resulting composite (A. Herna´ndez-Pe´rez, *et al*., 2008). Guadagno and Vertuccio concludes that good dispersion and interpenetration of the carbon nanotubes in the epoxy matrix having higher elastic modulus, particularly at high temperatures (Li J, *et al*., 2007). Many studies are in favor of using MWCNTs as filler in polymeric host for improved tensile properties (Wei-Jen Chen, *et al*., 2010; M.Paramsothy, *et al*., 2011; Toshio Ogasawara, *et al*., 2011; Daniel R.Bortz, *et al*., 2011; M.T. Kim, *et al*., 2011; L.Guadagno, *et al*., 2011). Still the basic problems remain stacked in making uniform dispersion of the mixture blend, handling procedures, and optimum quantity to get the desired result. Not much attention has been given in handling process and way of using MWCNTs. The MWCNTs have shown dispersion problem and difficulties are noticed during fabrication process because of aggregation problem leading non uniform solution. In this study, there is a description of process for the uniform distribution of MWCNTs in polymeric host and the fabrication of nanocomposites for tensile characteristics studies. The MWCNTs with surface modification is desirable in such critical problems of agglomeration to achieve the homogeneous mixture and uniform product otherwise. The covalent functionalization of MWCNTs can be considered as one of the easy process for the uniform dispersion in polymeric host matrix. Covalent functionalization of CNTs can be achieved by either direct addition reactions of reagents to the side walls of nanotubes or modification of appropriate surface-bound functional (e.g., carboxylic acid) groups on the nanotubes (J. N. Coleman, *et al*., 2006; J. N. Coleman, *et al*., 2006; Prabhpreet Singh, *et al*., 2009).

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Materials and Method

Materials:

Qtubes [purified multiwall carbon Nanotube] was received as gift pack from Quantum Material Corporation of 95%purity.The TEM (*Philips-TECHNAI FE 12*) images of purified MWCNTs Fig.1 and carboxylic group functionalized MWCNT-COOH is shown in Fig.2. The specific surfaces of MWCNTs area were found to be 350m^2/g. Araldite CY230-1 was used as standard epoxy resin in this study. Firstly, the MWCNTs were functionalized with carboxylic group under proper conditions.

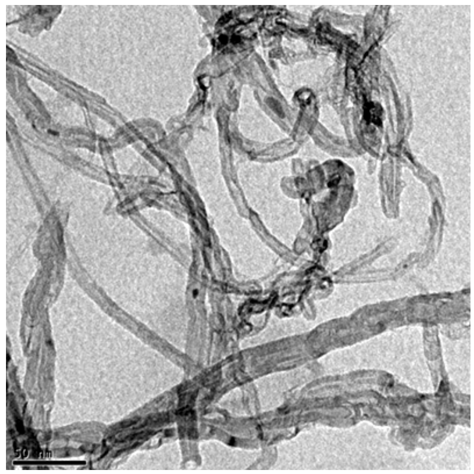

Fig. 1: TEM images of purified MWCNTs

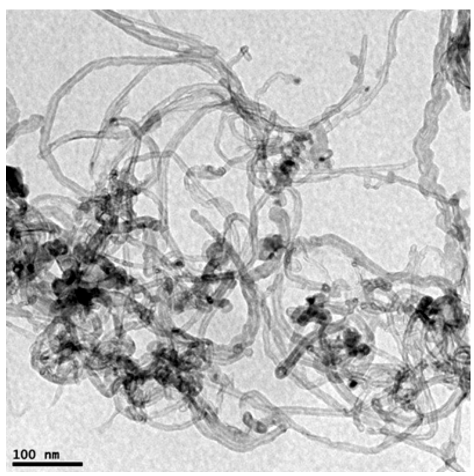

Fig. 2: TEM images of functionalized MWCNTs-COOH

As produced MWCNTs were dispersed and modified in concentrated H_2SO4/HNO_3 (1:3v/v) solution at 110°C for 60minutes under stirring condition. The modified MWCNTs were dried in a vacuum oven at 100°C for 12 hours. This carboxylic group functionalized MWCNTs were further treated with Bisphenol A (BPA). Bisphenol A [4-Dihydroxy-2, 2-Diphenyl Propane] obtained from resil polymer in the form of grains, was firstly mixed in Methyl Ethyl Ketone (MEK)[20%] by stirring. When Bisphenol A dissolved in MEK, the MWCNTs were incorporated in the ratio of 4:1[CNT: Bisphenol A] on continuous stirring. The stirring continued for another 2 hours. The suspension was left for 8 hours in the oven at 60°C. The solvent MEK evaporated and the paste was obtained containing the MWCNTs with BPA at its surface. The dried powder of MWCNTs was obtained by keeping it in oven for overnight at 100°C. The surface engineered MWCNTs were taken in liquid epoxy resin in prescribed amount to make nanocomposites with different weight fraction of MWCNTs.

Sample preparation:

Functionalized MWCNTs were reinforced into the epoxy resin and sonicated for 30min. The aradur HY 951 as a hardener manually mixed with epoxy/MWCNTs with the ratio of 10:1. Using laser cutting machine the desired dog bone shape of mould was prepared and the composition was poured in the mould. The mould was kept in the oven at 100°C for 12 hours for curing. The knurling is introduced in the cured specimen in both the ends with the dimension of 25mm for the purpose of gripping.

Tensile Test:

Schematic diagram of dog bone shape tensile specimen shown in Fig.3. Tensile test of epoxy/MWCNTs specimens after polishing is shown in Fig.4 were tested at room temperature 33°C and RH 47% at a constant cross head of 1mm/min in universal testing machine (UTM)[PMT 1C] Fig.5. The machine is connected with data acquisition system as per ASTM standard E 1856 and the hardware unit to initialize the parameter of loading rate, gauge length, width and thickness of the specimen. The cross pitch scares gripping jaws are fitted to the top and bottom head of the machine.

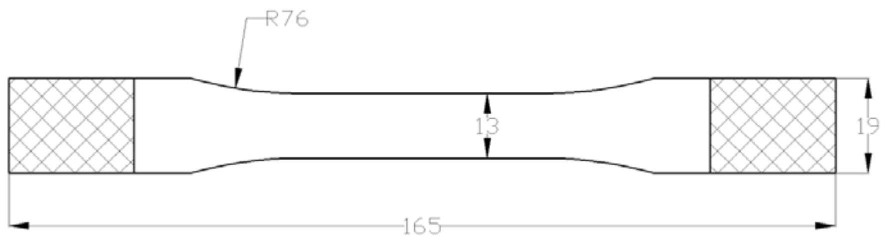

Fig. 3: Schematic diagram of dog bone shape tensile specimen

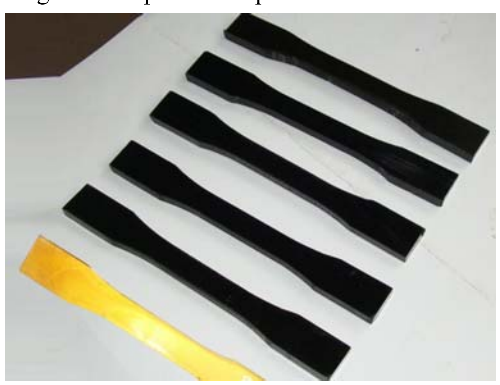

Fig. 4: Dog bone shape pure epoxy and MWCNTs specimen after polishing

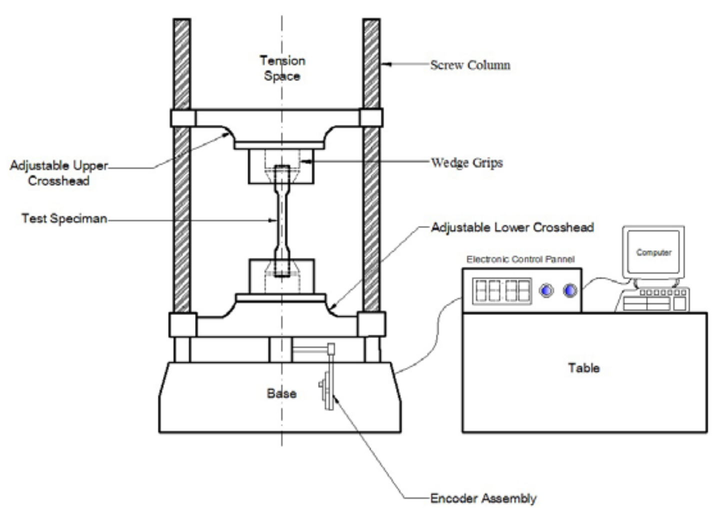

Fig. 5: Schematic diagram of Tensile Testing Machine

The dog bone shaped specimen with length of 165mm, width 19mm, gauge length 57.15 mm and thickness of 7 mm were fitted in the machine and loads are applied gradually with the increment of 0.1KN. The displacement was measured by high resolution encoder with the accuracy of 0.6µm. The strain values were measured by using extensometer.

Result and Discussion

It has been shown that acid functionalization improves the interfacial bonding properties between the CNTs and a polymer matrix. The carboxylic functional groups have been shown to give a stronger nanotube–polymer interaction, leading to enhanced values in Young's modulus and mechanical strength. (Junbo Gao, *et al*., 2006; G. Sui, *et al*., 2008; Siu-Ming Yuen, *et al*., 2008; Siu-Ming Yuen and Chen-Chi M. Ma, 2008; Jen-Tsung Luo, *et al*., 2008; Siu-Ming Yuen, *et al*., 2008; Asif Rasheed, *et al*., 2006; 2006; Nanda Gopal Sahoo, *et al*., 2006; K. K. Wong, *et al*., 2007). BPA causes cyclone addition reaction leading easy mixing with epoxy resin and that is why basically it has been severely used as monomer in the making of epoxy goods. This functionalization provided stable dispersions of CNTs in a range of polar solvents, including water. An advantage of the phenolic functionalities is that they allow post-functionalization of the MWNTs with other molecules that can be employed in preparing customized products. In fact, the functionalized MWCNTs increase the compatibility with epoxy matrix due to formation of an interface with stronger interconnections. The mechanism has probably followed because of cross linking among MWCNT-COOH, Epoxy and BPA as per the presentation in Fig. 6. The tensile test results of pure epoxy and epoxy/MWCNTs

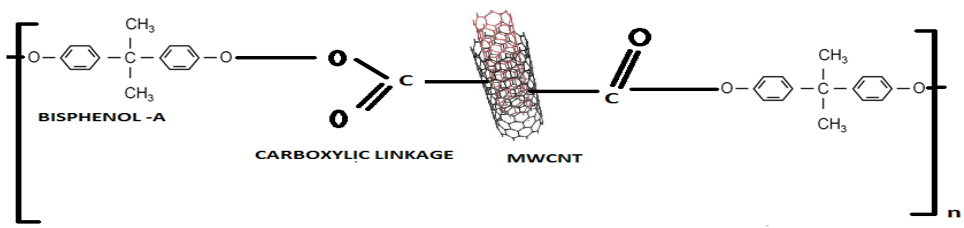

Fig. 6: Schematic presentation of linkages among CNT – Carboxy – Bisphenol entities

nanocomposites as shown in table 1. The investigation clearly depicts that the stress level is increased by adding considerable amount of MWCNTs. The load carrying capacity of 1.25wt.% get increased 4.42 times higher than pure epoxy as shown in Fig.7. However the tensile strength also increased by considerable amount and the percentage of elongation clearly represent that the displacement value gradually decreased when increasing the MWCNTs content in the epoxy matrix shown in Fig.8. Moreover the nanocomposites 5wt.% also exhibits similar enhanced performance but brittle fracture occur on the specimen due to the excessive content of MWCNTs

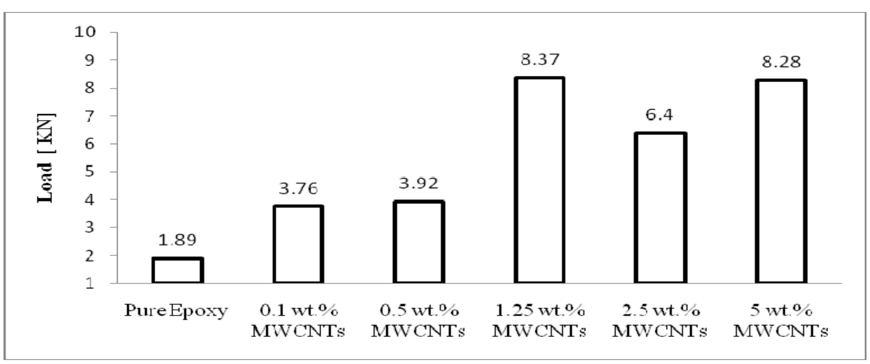

Fig. 7: Variation in load carrying capacity of pure epoxy and MWCNTs nanocomposites

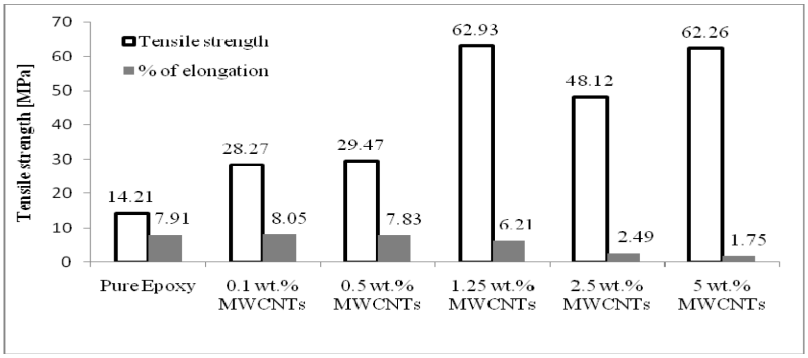

Fig. 8: Tensile strength and % of elongation of pure epoxy and MWCNTs nanocomposites

dispersion in the matrix. The 0.1wt.%, 0.5wt.% and 2.5wt.% MWCNTs nanocomposites also performed well and shown that the load carrying capacity of the nanocomposites gradually increased. In addition there is a significant decrease in the displacement value and percentage elongation of nanocomposites. In case of displacement value the nanocomposites with 2.5% MWCNT contents have proven to be best nanocomposites with 5% MWCNTs has shown the lowest percentage elongation. The nanocomposites with 1.25% wt MWCNTs epoxy has shown the best performance. The enhancement in Breaking load, peak load and stress value of 1.25% MWCNTs in comparison to base matrix of neat epoxy have been noted as 6.11,4.42,4.42 times respectively. Although the result is misleading towards the weight concentration of MWCNTs, still the interest comes with 1.25% MWCNTs content composite because of other facets and economical point of view. Thus study is suggestive to optimize the fabrication by taking 1.25% MWCNTs content composite for futuristic

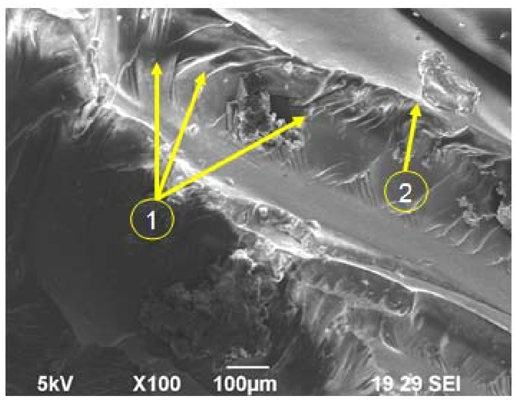

Fig. 9: SEM image of pure epoxy fracture surface. (1) Hyperbolic open in fracture surface, (2) Pieces of epoxy pull out from the matrix.

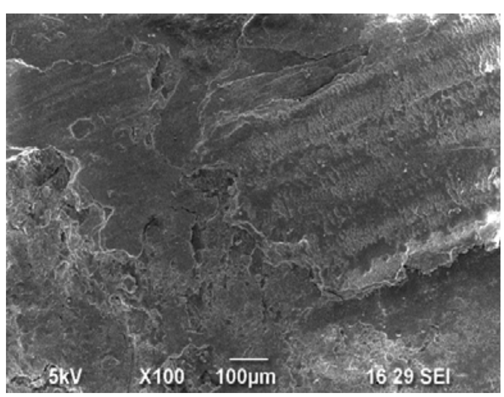

Fig. 10: SEM images of 1.25wt.% epoxy/MWCNTs fracture surface. Fracture initiate from bottom left to top right

development in the domain. It has been seen that optimum load capacity must be managed. The excessive load of MWCNTs causes brittleness due to agglomeration of fillers. However this can be further avoided by increasing the sonication rate and shear mixing rate. On the other hand the lower amount of MWCNTs as filler are inadequate to bear the load transfers in the matrix. At the breaking load, the sample has been examined by the scanning electron microscopy [*JEOL JFM 6390*] suggesting the uniform distribution of MWCNTs in case of best performer test sample (1.25%). The image Fig.9 clearly show that the hyperbolic opens occur on the pure epoxy fracture surface and also pieces of epoxy pull out from the matrix. The morphology of the 1.25wt.% MWCNTs fracture region clearly debits that no pull out in the matrix and also the fracture initiated from bottom left to top right shown Fig.10.

Conclusion:

A method for the fabrication of reinforced epoxy composite was conducted by introducing carbon nanotube into a polymer to provide a homogeneous mixture of the host and matrix solution. The covalent functionalization is the suitable way to make homogenous mixture in polymeric solution. The optimization of MWCNTs load has been evaluated as 1.25% by weight. There is significant improvement in the ultimate tensile strength which valued 6.11 times better than the neat epoxy. There were considerable enhancement in breaking load, peak load & stress value. Also, there is remarkable decrease in percentage elongation and displacement value 78% & 68% respectively of 1.25% MWCNTs containing composite than the neat epoxy. Thus the fabricated nanocomposites acted as super performance materials which seek many immediate application leading mechanically robust, tough and strong goods. Further this study is suggestive for respective application area such as fishing gears, safety belts, conveyor belts, sewing thread, protective clothing, man mad fibre, cement paste.

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