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Effect of One-Dimensional Carbon Nanotube Loading on Dispersion and Mechanical Properties of Epoxy Nanocomposite Materials

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ABSTRACT

The remarkable mechanical, thermal, electrical and chemical properties of carbon nanotubes (CNT) make them ideal candidates as nanoadditive in light weight polymer composite. It is their extraordinary mechanical properties (exceptionally high tensile strength and stiffness) that has aroused particular interest and promoted research into the fabrication of nanotube based composite materials. CNTs reinforced nanocomposites were developed by solution mixing process, followed by external load-free molding and curing. To enhance the dispersion of nanoadditives in polymer matrix, they were chemically modified to introduce surface active functional groups. Nanocomposites were characterized by various techniques to analyze the effect of reinforcement on glass transition temperature (Tg), thermal stability and flexural strength. Mechanical properties of these composites were evaluated and it was observed that by suitable chemical treatment of the reinforcement, the flexural strength was significantly increased. Tg of the nanocomposites increased with increase in filler content. It implies that the high aspect ratio and good dispersion state of CNTs in the epoxy may promote the mobility reduction effect. The thermal oxidative stability was also significantly improved with the addition of CNTs due to the barrier effect of its rolled structure.

Keywords:— *Carbon nanotubes, nanocomposites, flexural strength, glass transition temperature.*

I. INTRODUCTION

The remarkable mechanical, thermal, electrical and chemical properties of carbon nanotubes (CNT) make them ideal candidates as nanoadditive in light weight polymer composite. It is their extraordinary mechanical properties (exceptionally high tensile strength and stiffness) that has aroused particular interest and promoted research into the fabrication of nanotube based composite materials. The key factor in preparing a good composite rests on good dispersion of the nanotubes, the control of the bonding between nanotubes and matrix and the density of the composite material. Besides the type of nanotubes, the synthesis modes (arc discharge, laser, chemical vapor deposition) are important variables since they determine the perfection of the structure and the reactivity of the surface.

II. LITERATURE REVIEW

Different methods have been used to efficiently disperse the CNTs in polymer matrices. CNTs are ultrasonicated in a solvent to disperse them before they are added to the polymer matrix or before another dispersion technique is applied [1-4]. Micro scale twin screw extruder [5-7], melt mixing [8-9] and coagulation are some mechanical methods that minimize the aggregate formation by applying appropriate shear to CNT polymer composite. Dispersion of CNTs is also done by using dispersants as processing aids in CNT polymer composites [10-11]. In another technique CNTs are chemically modified and functionalized to disperse them better in the polymer matrix [12-14]. Another method to disperse CNTs is by in situ polymerization in which CNTs are added while the polymerization is in process [15-16]. Ultrasonication is a very effective method used to disperse CNTs in a solvent before it is added to a polymer matrix. Safadi et al [17] dispersed MWNTs in toluene using an ultrasonic wand dismembrator at 300 W for 30 min. In another effort, Dufresne [18] performed centrifugation in addition to ultrasonication. Sandler et al [19] also used a twin-screw extruder to disperse MWNTs in a polyamide matrix. Du et al [20] used a new and versatile fabrication method called coagulation method to achieve a better dispersion of SWNTs in PMMA matrix without changing the structure of the SWNTs. Gong et al [21] have reported the role of surfactants which function as a dispersing agent in processing of CNT epoxy composites. Another method to disperse CNTs is by chemically modifying them. Mickelson et al [22] reported modifying SWNTs by fluorination.

III. EXPERIMENTAL PROCEDURE AND MATERIALS

The surface of the CNTs was modified with nitration mixture in order to disperse them homogeneously. CNTs were homogeneously dispersed in a solvent in two -stage process. Initially ultrasonic bath was used to disperse CNTs in low boiling point solvent. Later, it was sonicated by means of high intensity probe type sonicator.

The development of composites is done by means of simple mixing process. CNT reinforced polymer composites of various concentrations say 0.25, 0.5, 1 wt% have been developed. For the preparation of composite, one of the challenging tasks was dispersion. CNTs were sonicated in acetone for 30 minutes with the help of high intensity probe type ultrasonicater. Pre-calculated CNTs and epoxy AW-106 grade resin were carefully weighed and mixed together in a beaker. A high intensity, ultrasonic irradiation mixed the CNTs and resin for 1 hour. The beaker containing the mixer was sub merged in an ice bath to ensure that the CNTs were not damaged during the sonication process. Once the sonication was complete, epicure curing agent HV 953 was added and mixed using mechanical stirrer for about 10 min. the mix ratio of epoxy and curing agent was 4:1. The mixing of resin and curing agent initially produce highly reactive, volatile vapor bubbles, which creates voids and affect the properties of the final product. To reduce chance of voids, the liquid was pre heated to 80°C at vacuum conditions for 30 min, to reduce its viscosity. After the bubbles were completely removed the mixer was transferred to rectangular stainless steel molds and cured for 4 hours at 120°C. The neat epoxy samples were prepared by following the same procedure without addition of CNTs.

Viscosity and dispersion characteristics of CNTs dispersed in epoxy were measured by Rheometer (Brookfield, R/S Plus, United Kingdom). The dispersion of the CNTs in the slurry was studied with the help of Optical micrograph. Thermal analysis was done with the help of TGA (Thermo gravimetric Analysis). The mechanical properties of the prepared nanocomposites where characterized using 3 point bend test (Instron). Instron-5500R universal tester was used to measure the flexural properties of the prepared samples. The three-point bending tests were conducted at room temperature. A 2000 N load cell and a crosshead speed of 2.0 mm/ min were used for the bending tests. The dimension of the testing samples was approximately: 60 × 10 × 3 mm. All of the samples were polished with sandpaper to ensure that the surfaces were smooth before bending tests and more than three specimens of each sample were tested, while the mean values were calculated.

IV. RESULTS AND DISCUSSION

4.1 CNTs dispersion and rheological characteristics

Figure 1 shows images of stereo microscope for every 10 minutes of ultrasonication in the stages of dispersion. It indicated that the agglomerated CNTs are broken in the initial stages and well distributed in the later stages. The dispersion is better and CNTs are homogeneously distributed throughout the matrix at the end of 30 minutes of sonication.

Dynamic viscosity of CNT dispersed epoxy was illustrated in figure 2. The viscosity of the mixture has a direct correlation with the spatial and orientation distribution of CNT in the matrix. This can be used as a physical signal with which to monitor the quality of dispersion, as long as the interpretation of the rheological signal is calibrated. The viscosities of different compositions of CNT/epoxy resin are tested at room temperature and it was observed that with the increase in concentration of CNT, the viscosity increased. The decrease in dynamic viscosity with respect to shear rate was noticed implies that the fluid shows shear thinning behavior.

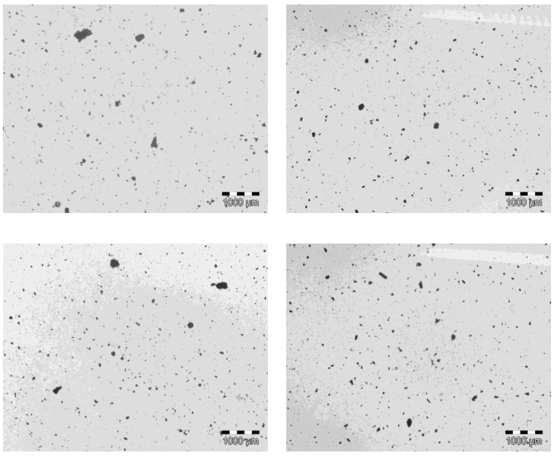

Figure 1: Microscopic images of CNTs dispersed in epoxy: (a) 10 minutes, (b) 20 minutes, and (c) 30 minutes

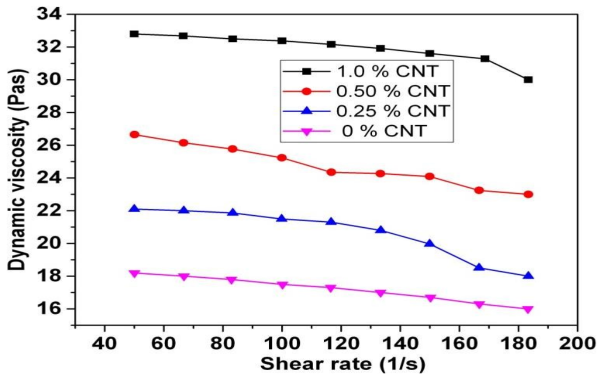

Figure 2: Dynamic viscosity of CNT dispersed epoxy

4.2 Mechanical properties

Due to the unique properties of CNTs they are being widely studied as a constituent of composite material. The practical applications of CNT composites have so far been largely limited by their poor processibility. The intrinsic Vander Walls attraction among the tubes in combination with their high surface area and high aspect ratio often leads to significant agglomeration of CNTs. The surface of CNTs is also non-reactive which makes it difficult in achieving efficient dispersion, as they mix and blend with the host matrix. To effectively use CNTs as intrinsic reinforcements in composite structures and ensure a good stress transfer between CNTs and the polymer matrix, uniform dispersion within the polymer matrix and improved nanotube/matrix wetting and adhesion are critical.

The variation in porosity of composite material with CNT loading was shown in Table1. The three points bending flexural test provides values for the modulus of elasticity in bending (Ef), flexural stress (σf), flexural strain (εf), and the flexural stress-strain response of the material. The main advantage of a three point flexural test is the ease of the specimen preparation and testing. The flexural strength, flexural modulus of different concentration of MWCNTs/ epoxy composites were calculated by three point bend test by using δf=1.5 (PL/bd2) and Ef= 0.25 (L3m/bd3) where σf = Stress in outer fibers at midpoint (MPa), εf = Strain in the outer surface (mm/mm), Ef = flexural Modulus of elasticity (MPa), P = load at a given point on the load deflection curve (N), L = Support span (mm), b = Width of test beam (mm), d = Depth of tested beam (mm), m = The gradient (i.e., slope) of the initial straight-line portion of the load deflection curve (N).

Table 1: Physical properties of CNT-epoxy composite materials

Type of composite	**Theoretical density(g/cc)**	**Observed density (g/cc)**	**Porosity (%)**
0.25 wt% CNT-Epoxy	1.698	1.050	38.2
0.5 wt% CNT-Epoxy	1.697	1.070	36.9
0 wt% CNT-Epoxy	1.725	1.140	28.9
1 wt% CNT-Epoxy	1.720	1.219	29.1

From Table 2 it is clear that the flexural stress and modulus keeps on increasing up to 1wt%. of CNTs reinforced epoxy composites. All flexural properties are increased at 1wt% CNTs reinforced composites. The addition of 1 wt% of CNTs in epoxy exhibited superior mechanical characteristics as compared to lower content of CNTs indicates that the threshold limit for such composite material is attained to transfer the load. The higher loading may lead to the decrease of flexural strength and modulus for higher CNT content can be interpreted by an increasing susceptibility of agglomeration of CNTs was noticed by other groups. Because of their high surface energy, large aspect ratio and with increasing viscosity it will be more difficult for the CNTs to disperse in the epoxy resin when their contents are higher, leading to less energy dissipating in the system.

4.3 Thermal properties

The following TGA curves describe the composite containing different content of MWCNTs and different carbon materials. We can observe that, they have slightly affected the thermal decomposing temperature of epoxy resin. The MWCNTs have a great effect on the onset decomposing temperature. Introducing MWCNTs to epoxy resin can increase the initial decomposing temperature of neat resin more or less. Because of the strong interaction between the epoxy resin matrix and MWCNTs, the diffusion of small molecules can be retarded under high temperature.

Table 2: Mechanical properties of CNT-epoxy composites

Sample	**Flexural Strength (MPa)**	**Flexural Modulus (GPa)**
Neat epoxy	48	1.59
0.25 wt% CNTs	65.3	1.98
0.50 wt% CNTs	72.89	2.18
1 wt% CNTs	75.85	2.63

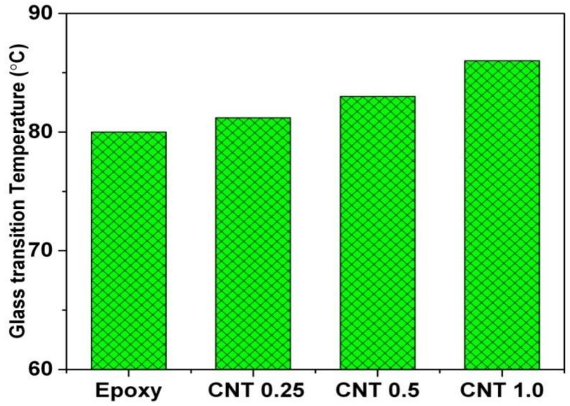

Figure 3: TGA curves of different reinforcement carbon nanomaterials

From above TGA curves of different reinforcement carbon nanomaterials; we observed that the high surface area of CNTs results in a high viscosity of the CNT/epoxy mixture, making bubbles and agglomerates in the matrix when adding higher contents of MWCNTs, which will affect their dispersion in the epoxy resins. Their relatively bad dispersion may result in some defects in epoxy matrix, which can quicken the thermal decomposition rate of the matrix.

V. CONCLUSIONS

The dispersion is better and CNTs are homogeneously distributed throughout the matrix at the end of 30 minutes of sonication. The Dynamic viscosities of different compositions of CNT/epoxy resin are tested at room temperature and it was observed that with the increase in concentration (% wt.) of CNT, the viscosity increased. The decrease in dynamic viscosity with respect to shear rate was increased. The high surface area of CNTs results in a high viscosity of the CNT/epoxy mixture, making bubbles and agglomerates in the matrix when adding higher contents of MWCNTs, which will affect their dispersion in the epoxy resins.

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