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Study of Mechanical and Morphological Properties of Aligned and Randomly Oriented Carbon Nanotube Composites

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Abstract:

Randomly oriented multi wall carbon nanotubes (RCNTs) and Aligned carbon nanotubes (ACNTs) added into the epoxy resin were mixed by sonication and then cured to obtain nanocomposite samples. The mechanical properties of the composites were characterized in terms of flexural tests and hardness tests. Scanning electron microscopy studies were employed to investigate the deformation mechanisms and crack propagation of the nanocomposites. Compared to random carbon nanotube-based composites, a significant increase in modulus and hardness was obtained with aligned carbon nanotube composites due to good load transfer from matrix to CNTs and effective bridging of cracks.

***Keywords*: A. Carbon nanotube; B. Epoxy; C. Composite; D. Mechanical property**

1. Introduction

Polymer composites containing carbon nanotubes (CNTs) have emerged as advanced multifunctional materials in view of exceptional mechanical, thermal and electrical properties associated with CNT [1]. They have become attractive structural materials not only in the weight-sensitive aerospace industry, but also in the marine, armor, automobile, railway, civil engineering structures, and sporting goods industries because of their high specific strength and specific stiffness. Different polymer/CNT nanocomposites have been synthesized by incorporating carbon nanotubes (CNTs) into various polymer matrices, such as polyamides [2], polyimides [3–5], epoxy [6], polyurethane [7–8] and polypropylene [9–11]. The presence of the nanotubes can improve the properties of polymers as well as add multi-functionality to polymer based composite systems [12-14]. Among the resins, epoxy resins have good stiffness, specific strength, dimensional stability and chemical resistance, and show considerable adhesion to the embedded filler [15].

Many studies have been conducted on multiwalled carbon tube (MWNT) based composites earlier and recently on aligned carbon tube (ACNT). MWNT composites have shown improved result in tensile modulus and yield strength [16]. However some studies reported decrease in flexural strength and pull out of CNTs in those composites [17]. In order to detect orientation and deformation of the CNTs in the nanocomposites, the tensile behaviour of both random and aligned MWNTs/Polystyrene nanocomposites was investigated by Thostenson and Chou [18]. They found the aligned CNTs composites showed more improved mechanical properties than random CNTs composites. Therefore, the comparison of different CNTs to obtain an efficient composite is very important. The purpose is to compare the suitability of these fillers in a polymer matrix that can be aimed for structural applications in aerospace/ automotive industries. In view of this in the present study, two types of nanotubes were dispersed in epoxy matrix. The mechanical properties of CNTs reinforced epoxy composites were measured using the flexural and hardness tests. Fracture behaviour and crack propagation of the nanocomposites are studied by scanning electron microscopy. The pure epoxy samples were also prepared and subjected to the tests for comparison.

2. Experiments

A. Materials

Multiwall carbon nanotubes (MWCNTs) used for the preparation of nanocomposites was obtained from MER corporation, USA. They are produced by arc plasma method (purity 95%, length 1-5 µm and diameters 20-70 nm). SEM morphology of the products (Fig. 1) was carried out with a "JEOL JSM-6480 LV Scanning Microscope". They are highly entangled and randomly oriented.

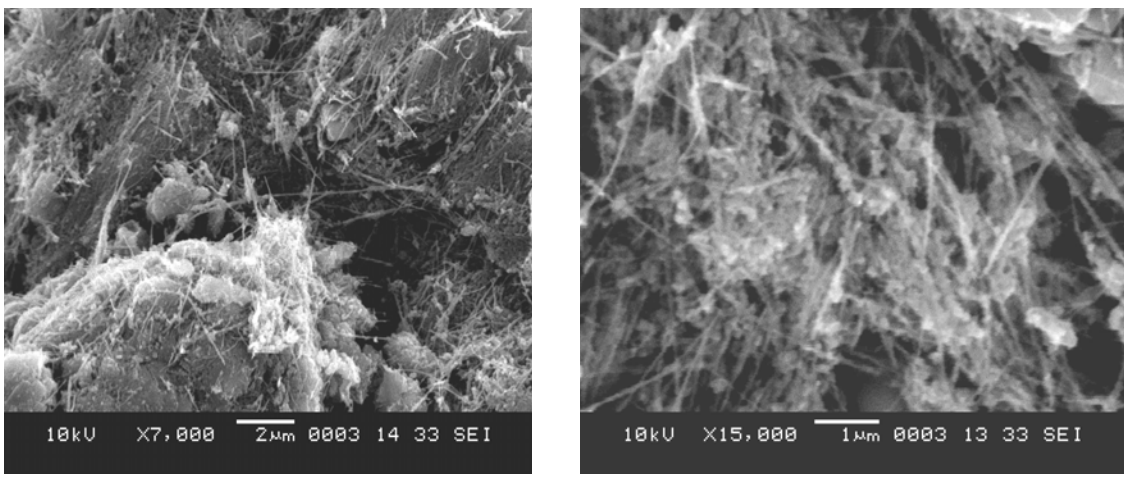

Fig.1 SEM of Random carbon nanotubes (RCNTs)

Aligned carbon nanotubes (ACNTs) used for the preparation of nanocomposites was obtained from ARCI, Hyderabad, India. They are produced by chemical vapor deposition method diameters 10-20 nm. SEM morphology of the products (Fig.2) was carried out with a “JEOL JSM 5410 Scanning Microscope".

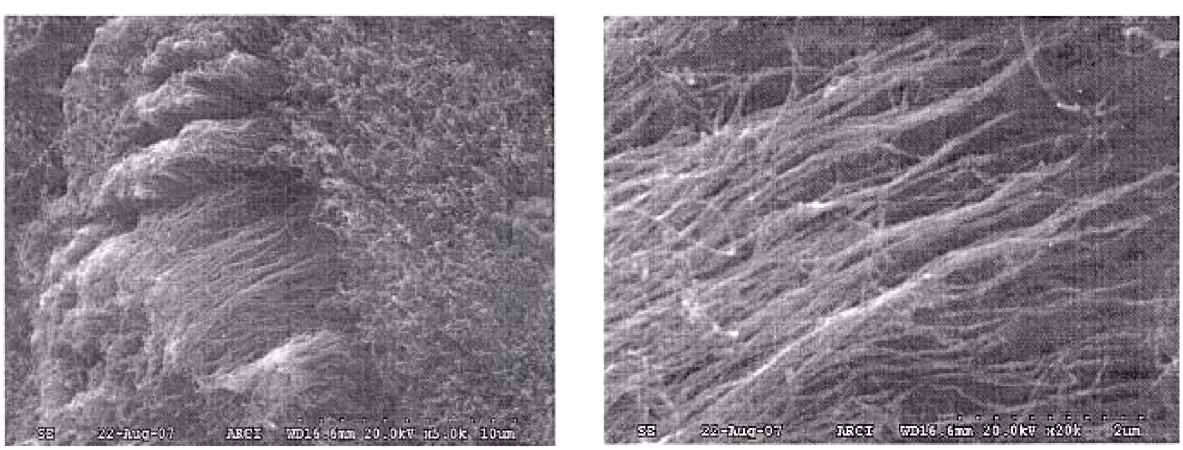

Fig.2 SEM of Aligned carbon nanotubes (ACNTs)

Epoxy polymer matrix was prepared by mixing epoxy resin (Ciba-Geigy, araldite LY-556 based on Bisphenol A) and hardener HY-951 (aliphatic primary amine) in wt. ratio 100/12. Epoxy resin (5.3-5.4 equiv/kg) was of low processing viscosity and good overall mechanical properties.

B. Nanocomposite preparation

Two types of nanocomposites involving Randomly oriented multiwalled carbon nanotubes (RCNTs) and aligned multiwalled carbon nanotubes (ACNTs) were prepared. First the nanotubes of both types were treated in ethanol for the deagglomeration of the tube bundles. The treated tubes (0.5 %) were then added to the epoxy resin and sonicated for 2hrs at room temperature.Hardener was added to that mixture and stirred. Then the mixture was poured into a mold and cured under vacuum at 90^0 C for 10 hrs. Pure resin samples have also been prepared for comparison purpose.

C. Flexural strength test

From each sample, five rectangular specimens were taken for three-point bend test as per ASTM D790 (width=2.7cm, thickness=0.7cm, span=11.2cm, length=12cm). Flexural tests were carried out at ambient temperature using Instron 1195 keeping the cross-head speed 2 mm/min. Flexural modulus of each sample was determined from the average value of five specimens.

D. Hardness of nanotube composites

The hardness of all samples was measured using a micro-hardness tester. A total of 10 points on each of the nanotube composites were measured in order to get average readings. The unit and magnitude of the hardness are defined by Vickers hardness, Hv and determined by measuring the average diagonal length, d of the indentation (mm).

E. Surface topography characterization

Scanning electron microscope (JEOL JSM-6480 LV) was used to conduct the dispersion behaviour and fracture surface topography characterization. After mechanical test fracture surfaces were coated with a thin platinum layer.

3. Results and Discussion

A. Flexural measurements

Flexural modulus of pure resin, RCNT composite & ACNT composite are shown in Fig.3. Both the composite samples are showing greater modulus than pure resin sample that is attributed to the high mechanical strength of CNT.

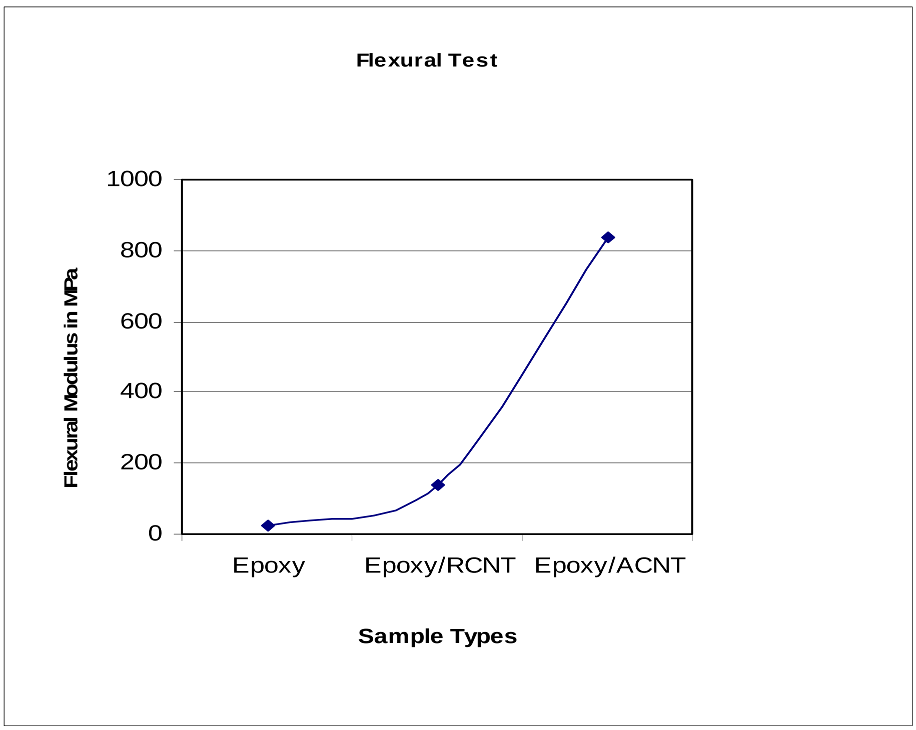

Fig 3 Flexural moduli of resin as well as composite samples

The flexural modulus was found to be 136.86 MPa in case of epoxy/RCNT composite which is about six times than the flexural modulus of plane epoxy sample (24.52 MPa). Increase in modulus is more pronounced in epoxy/ACNT composite i. e. 837.42 MPa which is more than six times that of epoxy/RCNT composite and thirty four times that of epoxy sample. This may be due to efficient load transfer from matrix to aligned CNT in axial direction. Local stiffening due to nanotubes results in improved load transfer at the fibre/matrix interface [18]. It had been reported that the increase in elastic modulus between the random and aligned nanocomposite is a consequence of the nanotube orientation, not polymer chain orientation.

B. Hardness measurements

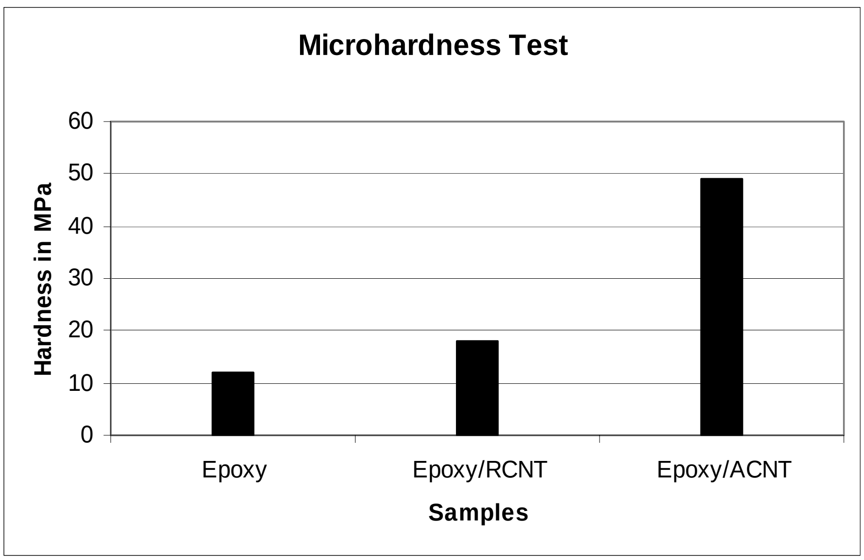

Fig. 4 Hardness of epoxy and epoxy/ nanotube composites

A considerable enhancement of hardness is observed by the nanocomposites in comparison to pure resin sample (Fig.4). Pure resin samples showed hardness of 12 MPa. Epoxy/RCNT had hardness value of 18 MPa which is 50% more and epoxy/ACNT had 49 MPa that is about four times that of epoxy sample. High strength and long nanotube reinforcements may result in forming a network structure that improves the hardness of the composites.

C. Surface topography analysis

The investigation of the fractured surfaces to analyse the micro deformation and crack propagation mechanism has been carried out using Scanning electron microscope (SEM). The micrographs corresponding to nanocomposites obtained by incorporating RCNTs and ACNTs are presented. From Fig.5a & b, the fracture surface of epoxy/RCNT appears to be rough than epoxy/ACNT composites.

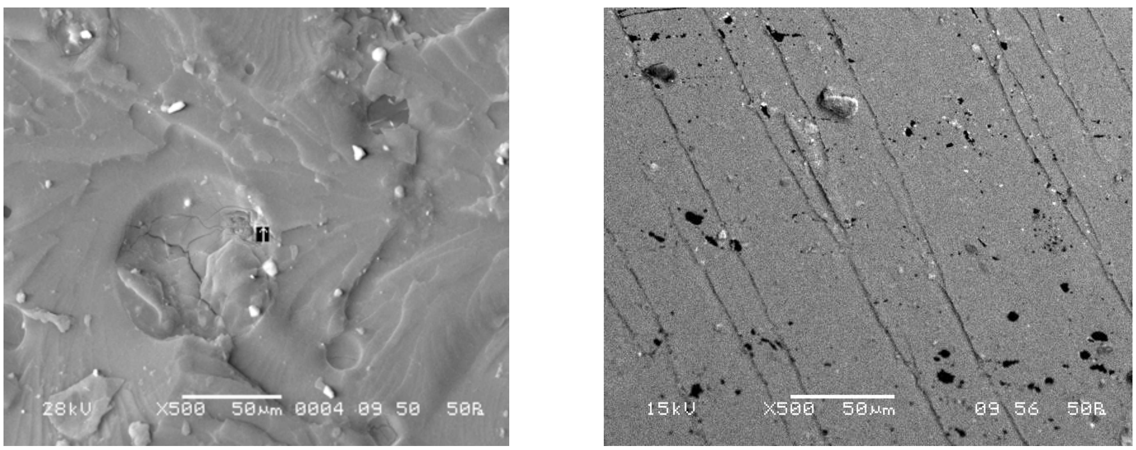

Fig.5 Fracture surface of (a) epoxy/RCNT (b) epoxy/ACNT composite sample

The resultant failure mechanism of the epoxy/RCNT interface was analysed by observing the crack propagation regions within the composite (Fig. 6a). An agglomeration of several carbon nanotubes was observed in the fracture surface near the crack region. At a low stress level, the agglomerated particle increases the stiffness of the material, but at a high stress level, the stress concentration caused by the agglomerated particle initiates a crack, which make the sample fail quickly. Some nanotubes were observed to be pulled out which might be the result of a poor interfacial bonding between the nanotubes and matrix. Therefore, the nanotubes inside the composites could not take up the load, which resulted in the decrease of flexural strength of the nanotube composite beams [19]. In comparison to that in case of epoxy/ ACNT composites, no crack was observed though the surface was seen to undergo less intensive fracture with smaller crack lengths in vertical direction (Fig. 6b).

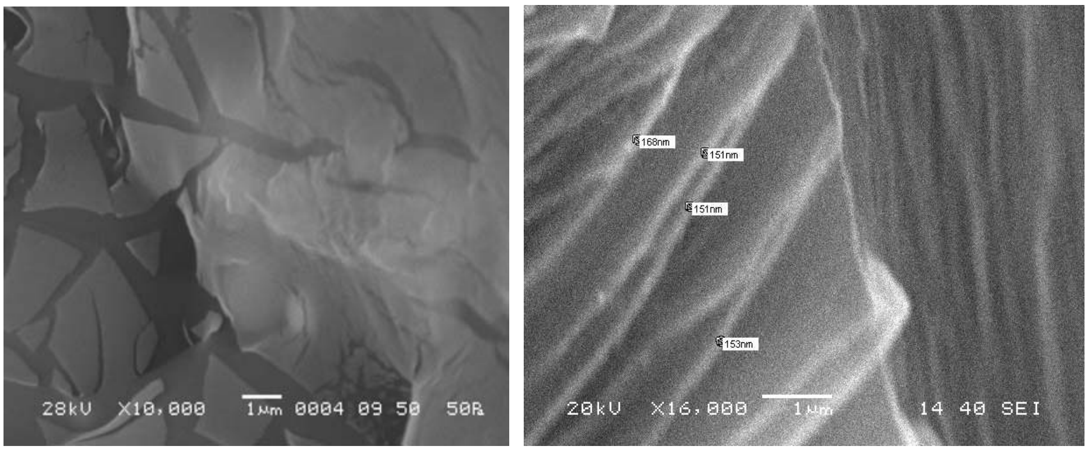

Fig.6 Crack features of (a) epoxy/RCNT (b) epoxy/ACNT composite sample

Higher magnification showed a crack interacting with the nanotube reinforcement. RCNT matrix pullout was observed along with extension and bridging of RCNTs across the crack (Fig.7a). In epoxy/ACNT composites, the cracks were spanned by the nanotubes causing enhanced resistance to the crack propagation process. The bridging of the nanotubes as a mechanism of inhibiting the crack initiation in polymer and ceramic based nanocomposites has been well illustrated in literature [20-24].

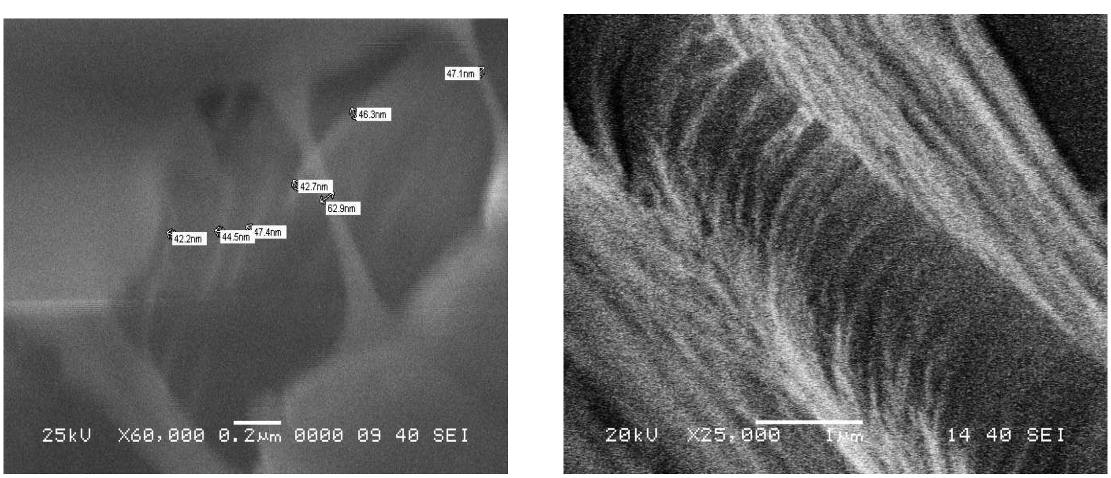

Fig.7 Bridging mechanism of (a) epoxy/RCNT (b) epoxy/ACNT composite sample

From the above discussion, the evident difference between fracture surfaces indicated that the reinforcing role of nanotubes in the two kinds of polymer nanocomposites was different. The mechanical tests described above further supported this.

4. Conclusions

All the composite samples demonstrated enhanced mechanical properties than pure resin samples that were attributed to addition of high strength nanotubes. In addition, aligned nanotube composites resulted in significantly improved flexural modulus and hardness indicating that there is efficient load transfer between the polymer matrix and the nanotube reinforcement along axial direction. Reduction in flexural modulus and hardness value in epoxy/ RCNT composites was due to formation of agglomerates of nanotubes inside polymer matrix that reduced reinforcing effects of the CNTs by acting as flaws in the resin. Investigation of fracture surface in nanocomposite revealed that narrower crack-tips underneath the advancing cracks were more efficiently bridged by the nanotubes in epoxy/ACNT resulting in an increased resistance against crack propagation.

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