Analysis and comparison of CNF and ACNT reinforced epoxy composite using microscopic technique

Smrutisikha Bal

Central Instrument facility, National Institute of Technology, Rourkela, Odisha, India.

Email: balss@nitrkl.ac.in

Abstract:

Numerous research efforts have been undertaken regarding the mechanical and electrical behavior of epoxy matrix by the introduction of various carbon nanofillers. In the present work, a simple but novel route regarding preparation of nanocomposites has been reported in terms of curing them at low temperature. The purpose is to investigate the effect of reinforcing strategies on properties of prepared composites using different nanomaterials that are again supplemented by microscopic investigations. Flexural properties, hardness and electrical conductivity of nanocomposites improved significantly than epoxy. Among the two nanocomposites, ACNT composite demonstrated enhanced results that were again confirmed from micro graphical analysis. CNF composite exhibited comparatively lower mechanical result but almost equivalent electrical result with ACNT composite.

Key words: carbon nanotubes/ carbon nanofibers/ epoxy/ nanocomposite

Introduction

Epoxy matrix composites with Carbon nanofibres (CNF)/ aligned carbon nanotube (ACNT) reinforcement have become popular in structural applications because of their extraordinary mechanical and physical properties like strength and flexibility as well as for ballistic protection [1]. In this work, the nanocomposites have been prepared using sonication method with very low content (0.75 wt.%) of CNFs and of ACNTs in the epoxy matrix. Additionally, the nanocomposites were prepared for the first time at low temperature in refrigeration process [2]. Flexural modulus & strength, hardness and electrical conductivity of resin as well as nanocomposite samples were examined and variations in different properties between the respective cases were observed. The purpose of this study is to optimize the conventional method of manufacturing composites and examine their effect on mechanical and electrical which were again supplemented by electron microscopy.

Materials & methods

Carbon nanofibers (CNFs) used for this experiment are of 95% purity, 10–40 µm length and 200–500 nm diameter (Fig.1a). Aligned carbon nanotubes (ACNTs) are 10– 40 nm in diameter (Fig.1b), 5–15 µm long and >95% purity. 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).

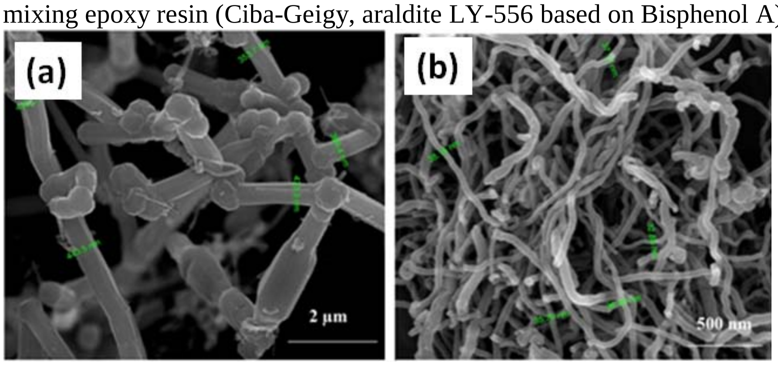

Fig.1 SEM of CNFs and ACNTs showing their diameter (a & b)

Flexural tests were accompanied according to ASTM D790–00 on an Instron 5967 testing machine. The hardness of all composite specimens was measured using a micro-hardness tester by the Vickers hardness test method. The electrical properties of the neat and nanophased epoxy were measured by using a Keithley electrometer with sample dimension (8 mm X 5 mm X 4 mm). Microscopic investigation was done through Field emission scanning electron microscope (FESEM: Nova NANOSEM 450) after conducting flexural test.

Results and Discussion

Increase in flexural properties was found in the order Epoxy < CNF/epoxy< ACNT/epoxy (Table 1). Increments in nanocomposites w.r.t epoxy is also shown in percentage. Fracture micrographs of the specimens reveal partial/complete pull out of CNF from the resin signifying ineffective interfacial adhesion between CNF and epoxy polymer (Fig.2a). The debonding occurred due to crack initiation in the matrix. However, very less number of pull out of nanotubes have been noticed in case of ACNT/epoxy composites (Fig.2b). This means that strong interfacial adhesion occurs between the aligned nanotubes and host matrix. The propagation of cracks is obstructed by the nanotubes which is the cause behind the increment in properties of the nanocomposites with respect to the neat epoxy. For ACNT/epoxy composites achievement of significant modulus and strength value may be attributed to local stiffening that occurs due to the aligned CNT at the interface which can facilitate efficient load transfer from matrix to aligned CNT in axial direction [3, 4]. Further, low temperature treatment results relatively better curing of epoxy resin, which allows more interaction with hardener by delaying the settling time [2]. This facilitates improvement in dispersion in nanocomposites. Hardness results agreed well with flexural test results with the confirmation that ACNT composite samples had improved mechanical properties than CNF composite

Table 1. Flexural properties and hardness of specimens

Sample	Epoxy	CNF/epoxy	ACNT/epoxy
Flexural modulus (MPa)	4501	5350 (19%)	8973 (99%)
Flexural strength (MPa)	66	81 (23%)	137 (107%)
Hardness (MPa)	12	17 (42%)	30 (150%)

samples with the same wt% of nanofiller. Nanofillers having high modulus and strength as well as high crystallinity can increase the hardness of the matrix phase of the composites.

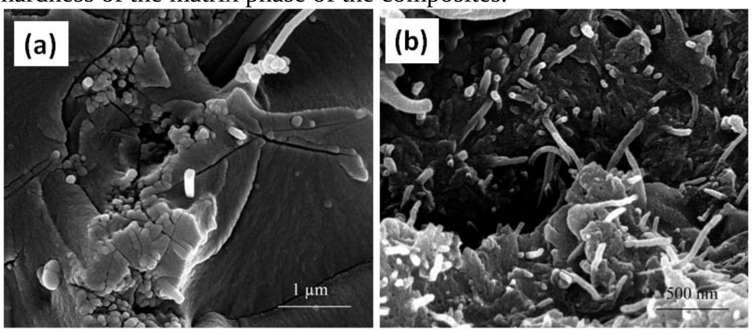

Fig. 2 debonding in CNF/epoxy (a) adhesion in ACNT/epoxy (b)

Demonstrable improvement in electrical conductivity was found in CNF/ ACNT reinforced composites. Conductivity of these nanocomposite samples are 5-6 orders higher to that of resin sample (Table 2). This was due to the aggregated phases that form a conductive three-dimensional network throughout the whole sample (Fig.3 a & b). Alignment of nanotubes in a particular direction in the epoxy polymer helps in forming effective network in the ACNT composite and thereby assists in conducting electricity effectively [5]. Even if the CNFs do not touch each other directly, conductivity of the CNF composites is achieved as long as the distances between them are lower than the hopping distances of the conducting electrons. As the materials with electrical conductivity between 10^{-6} S/cm and 10^{-2} S/cm are treated as semiconductors, the nanocomposites presented in this work can be utilized for electrostatic discharge and electromagnetic interference shielding applications [6].

Table 2. Electrical conductivity of specimens

Sample	Conductivity in S/cm
Epoxy	9.0×10^{-9}
ACNT/epoxy	2.9×10^{-3}
CNF/epoxy	1.8×10^{-4}

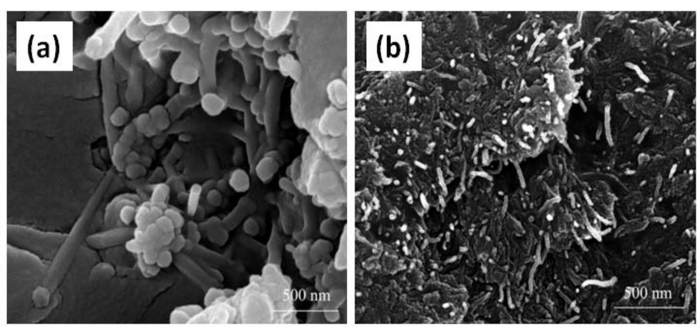

Fig. 3 Network formation in CNF/ epoxy (a) & ACNT/ epoxy (b)

Conclusions

The results obtained from mechanical and electrical studies in this work are well supported by microscopic demonstration. Although flexural and hardness values of ACNT/epoxy sample are much higher than CNF/epoxy sample, electrical conductivity in those samples offer little variation. Strong interfacial adhesion between ACNT and epoxy matrix facilitates higher flexural modulus and hardness. Network formation of nanoparticle inside the matrix of both nanocomposites is observed in micrographs that confirms electrical conductivity.

Acknowledgments

The author would like to thank Naval Research Board (DRDO), Govt. of India for the financial support to carry the research work.

References

1. H.D. Wagner, O. Lourie, Y. Feldman, R. Tenne, Appl.Phys. Lett. **72,** 188-190, (1998)
2. S. Bal, Mater & Design., 31: 2406-2413, (2010)
3. Y. S. Song and Youn, J. R. Youn, 43(7), June, 1378-1385, (2005)
4. N. M. Zulfi, A. A. Bakar and W. S. Chow, J Rein Pla & Comp. 32(22), November, 1715-1721, (2013)
5. Q. Wang, J. Dai, W. Li, Z.Wei, and J. Jiang, Comp. Sci. & tech., 68(7), June, 1644-1648, (2008)
6. L.R. Xu, A.J. Rosakis, Int J Solids & Struct., 9, 4237–4248, (2009)