

# Carbon Nanofiber Reinforced Carbon/Polymer Composite

M. Joshi\* and A. Bhattacharyya

Department of Textile Technology, Indian Institute of Technology

Hauz Khas, New Delhi – 110016, India

\* mangala@netearth.iitd.ac.in

## ABSTRACT

Conventional carbon fiber reinforced phenolic resins are widely used to prepare carbon/carbon composites due to their high carbon yield. Property enhancement of carbon fabric/phenolic composites is possible through carbon nanomaterial dispersion in the matrix. The effect of carbon nanofiber dispersion in phenolic resins/carbon fabric composites was investigated. The dispersion efficiency in different systems and conditions was studied and the effect of nanofiber concentration on the properties determined. Carbon nanomaterial incorporation influences the mechanical properties of the composites and gives better thermal stability. Nanofiber dispersion shows better adhesion of phenolic resin to the fabric under fracture surface study.

**Keywords:** nanocomposites, carbon nanotubes, carbon nanofiber, phenolic resins, composite

## 1. INTRODUCTION

Phenolic resin is extensively used for fabric reinforced carbon-carbon composite due to its highest yield of carbon after carbonizing. Carbon fiber finds use in both filament and fabric form as reinforcement in polymeric matrices for high performance structural applications [1]. Textile structured composites plays an important role in preparation of carbon-carbon composites [2] and exhibit in plane balanced properties. Consequently they find uses in many high-end applications where their special properties can be taken advantage of e.g. spacecraft re-entry frames. Carbon nanotubes have been successfully used to improve the wear properties of carbon-carbon composites [3]. The use of carbon nanotubes in phenoxy matrix also results in significant rise in dynamic mechanical properties [4]. In the present study, carbon nanofiber is dispersed into phenolic resin before using it as matrix for carbon fabric composites. Although literature mentions studies on carbon nanotube in phenolic resins [3], no study using nanofiber-reinforced phenolic resins as matrices for carbon/polymer composites has been reported.

## 2. EXPERIMENTAL

### 2. 1. Raw material used

1. Carbon fabric – 3k x 3k, Plain weave, 195 gm/m<sup>2</sup> (Surabhi International, Kanpur, India.)
2. Carbon nanofiber – Pyrograf III (PR24 AGLD) (dia.: 60 – 150nm).
3. Non-ionic surfactant – Polyoxyethylene 8 lauryl ether (30% w/v), (Loba Chemie)
4. Methanol – Solvent for resin, AR grade
5. Phenolic resin –Prepared in house.

### 2. 2. Processing

#### 2. 2. 1. Resin preparation

The resin was prepared by the condensation of phenol and formaldehyde and refluxing for 1.5 hrs. at 70°C.

#### 2. 2. 2. Dispersion of carbon nanofiber

1.0% carbon nanofiber (on weight of resin preform) was dispersed into the resin through three different processes:

- a) Sonication at 20 KHz for 2 hrs.
- b) Mechanical stirring with 0.2% non-ionic surfactant (on volume basis) for 2 hrs.
- c) Mechanical stirring with 0.2% non-ionic surfactant (on volume basis) for 1 hr. and subsequently sonication treatment of 20 KHz for 1 hr.

Thin films were prepared and observed using optical microscope. The best method of dispersion was chosen for composite preparation.

#### 2. 2. 3. Preparation of the composite

The fabric was impregnated by dipping in the resin preform and drying overnight. Then it was taken for compression-molding using conditions:

- a) Curing temperature: 150°C
- b) Curing time: 2 hrs.
- c) Force: 3 Metric ton

The average resin fraction on weight basis after curing = 45%

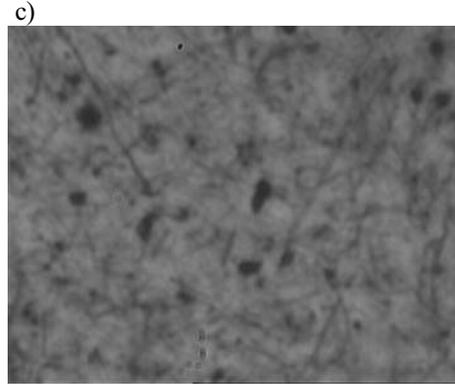
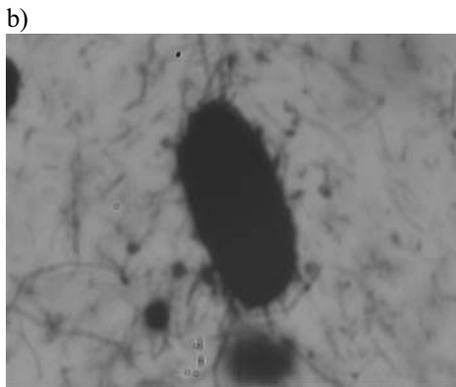
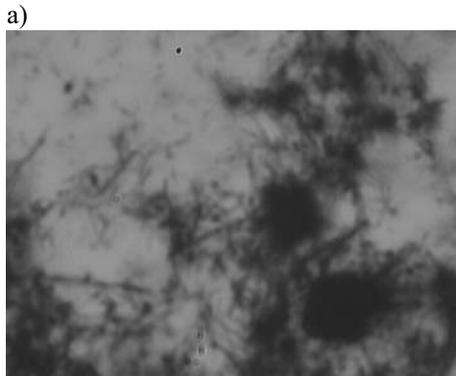
### 3. CHARACTERIZATION

Nanofiber dispersion was observed with a LEICA optical microscope in transmission mode. The tensile (ASTM D3039-76) and flexural tests (ASTM D790) were carried out on a Zwick Z010 tensile tester. Three-layered fabric composites were made for flexural test purpose. All samples were tested warp way. Dynamic mechanical analysis was carried out on a Perkin-Elmer DMA7 at 1 Hz, 200 mN static load and 160 mN dynamic load from  $-30^{\circ}\text{C}$  to  $200^{\circ}\text{C}$ . Thermal conductivities were measured on an ALEMBETA instrument. The fracture surface of the composite was studied with a LEICA microscope.

### 4. RESULT & DISCUSSION

#### 4. 1. Dispersion of Carbon Nanofibers

The nanofibers were dispersed into the resin and the cured films were observed through transmission mode of LEICA. Among the three processes the use of both surfactant and Sonication process was found most suitable (Figure: 1).



**Figure 1: 1.0% nanofiber dispersion by three different methods** (full scale  $100\ \mu\text{m} \times 100\ \mu\text{m}$ ); a) Sonication for 2 Hrs, b) Surfactant (0.2%), stirring for 2 Hrs, c) Stirring with Surfactant (0.2%) for 1 Hr & Sonication for 1Hr.

Care was taken to minimize the amount of surfactant in this process, as surfactant acts as an impurity in the system. It has been observed that the use of 0.1% surfactant is sufficient to have satisfactory level of dispersion of the nanofiber when the sonication time is 1.5 hrs.

#### 4. 2. Tensile Testing

The tensile properties of the produced composites were estimated and were given in Table 1 below.

**Table1. Tensile properties of prepared samples**

Samples	Modulus (GPa)	Force at break (MPa)	Strain at break (%)
Carbon/Phenolic resin composite	7.98	285.9	3.98
With 0.5% nanofiber	8.31	290.0	3.26
With 1.0% nanofiber	8.48	309.8	3.29
With 1.5% nanofiber	8.66	320.4	3.16

The addition of nanofiber improves the tensile properties. 1.5% nanofiber addition into the matrix results in 8% increase in modulus and 12% increase in breaking strength of final composite. Nanofiber imparts brittleness into the matrix, which results in reduction of strain at break. The strength improvement is significant considering the fact that the modulus and strength of nanofiber is only slightly higher than the carbon fiber. The improvement is more due to the change in

interfacial behavior than the conventional mixture rule.

### 4. 3. Flexural testing

The flexural properties of the produced composites were estimated and were given in Table 2 below.

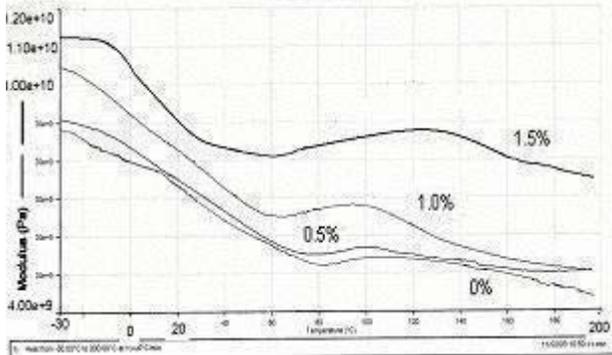
**Table 2. Flexural properties of prepared samples**

Samples	Modulus (GPa)	Force at break (MPa)	Strain at break (%)
Carbon/Phenolic resin composite	51.4	654.4	2.01
with 0.5% nanofiber	53.8	688.6	1.77
with 1.0% nanofiber	55.5	648.2	1.60
with 1.5% nanofiber	57.5	695.9	1.27

Result shows the improvement in modulus and breaking strength by 11.5% and 9%, respectively. Decrease in strain at break shows the brittleness due to higher interaction.

### 4. 4. DMA

Three point rectangular bending test on single-layer fabric composites were carried out from  $-30^{\circ}\text{C}$  to  $200^{\circ}\text{C}$ . Figure 2 reveals that the storage modulus remains more stable over the temperature range. The storage modulus increases from 8.8 GPa to 11.2 GPa with the incorporation of 1.5% nanofiber into the matrix at  $-30^{\circ}\text{C}$ . Heating up to  $200^{\circ}\text{C}$  does not cause sharp fall in modulus when carbon nanofibers are dispersed into the matrix. Similar effect is observed for loss modulus



**Figure 2:** Storage modulus of composite samples with different nanofiber fraction in matrix

### 4. 5. Thermal conductivity

Table 3 shows that thermal conductivity values increased with the increase in nanofiber content. The thermal conductivity of carbon nanofiber ( $20 \text{ Wm}^{-1}\text{K}^{-1}$ ) is higher than the phenolic resin ( $0.8 \text{ Wm}^{-1}\text{K}^{-1}$ ); hence nanofiber dispersed into the matrix enhances the conduction of the composite.

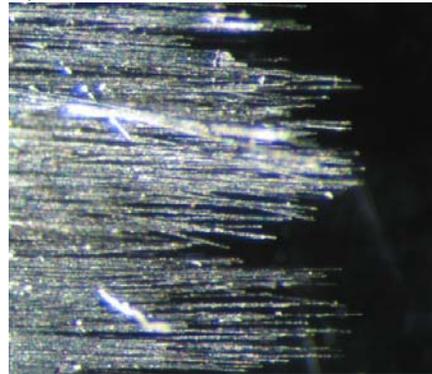
**Table 3. Thermal conductivity of composites**

Parameters	Nanofibre content			
	0%	0.5%	1.0%	1.5%
Thermal conductivity ( $\lambda$ ) $\text{Wm}^{-1}\text{K}^{-1}$	$52.2 \times 10^{-3}$	$56.7 \times 10^{-3}$	$67.3 \times 10^{-3}$	$70.9 \times 10^{-3}$

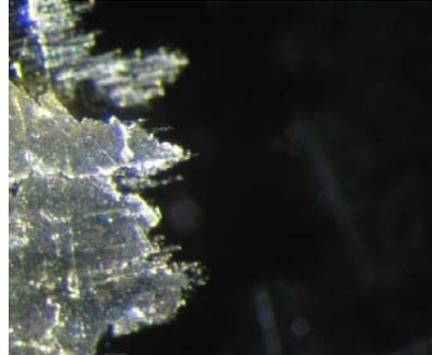
### 4. 6. Fracture surface study

The reflection mode pictures in LEICA microscope indicate that nanofibers increase the adhesion between the matrix and the carbon fiber. Figure 3 indicates that the carbon fibers of the reinforcing fabric are not fully exposed in the fracture surface when carbon nanofiber is present in the matrix, which indicates better adhesion between fiber and matrix.

a) Full scale 1mm x 1mm



b) Full scale 1mm x 1mm



**Figure 3:** Fracture surface of composite with a) 0% nanofiber, b) 0.5% nanofiber

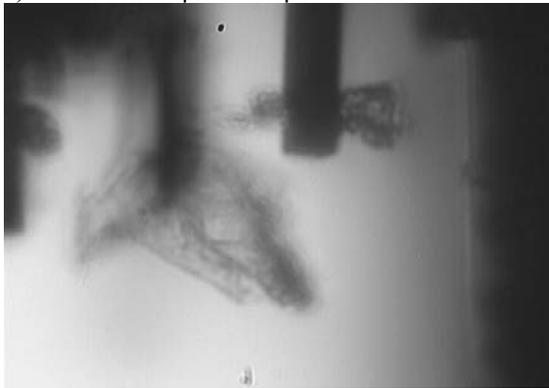
In transmission mode at higher magnification, it was evident that the nanofiber dispersion into the

matrix is satisfactory at the fracture surface (Figure 4a). The Phenolic resin carbon fiber bond strength is not very good as is evident from Figure 3a. But after incorporation of the carbon nanofiber into matrix some fringing from the original fiber occurs (Figure 4b) which results in better load transfer of the resin to the carbon fiber. The carbon nanofiber may act as the coupling between the carbon fiber and the resin. The poor bond strength of phenolic resin is somewhat overcome with carbon nanofiber into the matrix.

a) Full scale 100 $\mu$ m x 100 $\mu$ m



b) Full scale 100 $\mu$ m x 100 $\mu$ m



**Figure 4:** a) Nanofiber at fracture surface (proper dispersion), b) Adhesion of resin with help of nanofiber on fiber

## 5. CONCLUSION

The use of surfactant is helpful for proper dispersion of the nanoparticle into the matrix. The use of non-ionic surfactant is reported for dispersion of carbon nanotube in epoxy matrix [5]. It has been also found true for carbon nanofiber in phenolic resin as investigated in the present study. Sonication can minimize the amount of surfactant to some extent. The combined process of surfactant-assisted sonication has been found most

suitable for dispersion of carbon nanofiber. Incorporation of carbon nanofiber into phenolic resin matrix of carbon composite enhances its mechanical and thermal properties significantly. It has been observed that nanofiber alters the interface behavior of the carbon fiber and phenolic resin matrix. Thus may give better load transfer into the reinforcement and results in a synergistic effect of nanofiber into the property alteration beyond common mixture rules.

The dynamic mechanical analysis shows that the introduction of the nanofiber into the matrix enhances its stability under wide range of thermo-mechanical condition.

Carbon nanomaterial reinforced polymeric matrix composites with carbon fibre reinforcement are examples of three phase nanocomposite systems – something that has not been studied in great detail before. The interfacial interactions and the stress transfer mechanisms would also be very relevant in the presence of the second reinforcing phase- the fabric. The result of the excellent interphase properties is obtained in a carbon-carbon composite because of its uniphase nature. So the current study has to be extended to carbonization of this composite to find the change in properties because of presence of carbon nanofibers in the phenolic resin.

## 6. REFERENCES

1. N. K. Naik, S. I. Tiwari and R. S. Kumar: An analytical model for compressive strength of plain weave fabric composites; *Composites Science and Technology* **63** 5 (2003), pp. 609-625.
2. T.W. Chou and F.K. Ko, Editors: *Textile structural composites*; Elsevier, Amsterdam (1989), pp. 209-263.
3. Dae-Soon Lim, Jeong-Wook An and Hwack Joo Lee: Effect of carbon nanotube addition on the tribological behavior of carbon/carbon composites; *Wear* **252** 5-6 (2002), pp. 512-517.
4. H. W. Goh, S. H. Goh, G. Q. Xu, K. P. Pramoda and W. D. Zhang: Dynamic mechanical behavior of in situ functionalized multi-walled carbon nanotube/phenoxy resin composite; *Chemical Physics Letters* **373** 3-4 (2003), pp. 277-283
5. S. Cui, R. Canet, A. Derre, M. Couzi and P. Delhaes: Characterization of multiwall carbon nanotubes and influence of surfactant in the nanocomposite processing; *Carbon* **41** 4 (2003), pp. 797-809