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Composites: Part A 35 (2004) 1225–1232

**composites**

Part A: applied science  
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# Processing and property investigation of single-walled carbon nanotube (SWNT) buckypaper/epoxy resin matrix nanocomposites

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Received 16 April 2003; revised 20 August 2003; accepted 1 September 2003

## Abstract

Due to their extraordinary high modulus and strength, single-walled carbon nanotubes (SWNTs) are considered by many researchers as the most promising reinforcement materials for use in high performance composites. However, fabricating SWNT-reinforced composites with good tube dispersion and high SWNT loading is a great challenge since SWNTs have a strong tendency to form bundles or ropes and rapidly increase the viscosity during processing. In this research, a new method was developed to fabricate nanocomposites with preformed SWNT networks and high tube loading. SWNTs were first dispersed in water-based suspension with the aid of surfactant and sonication. Through a filtration process, SWNTs were fabricated into thin membranes called buckypapers to form networks of SWNT ropes. The tube/resin impregnation of the produced buckypaper was realized by infiltrating acetone diluted epoxy resin (Epon 862/EPI Cure W system) along the thickness direction. A hot press molding process was used for curing to produce the final nanocomposites of multiple layer buckypapers with high SWNT loading (up to 39 wt%). Dynamic mechanical analysis (DMA) results show that the storage moduli of the resulting nanocomposites were as high as 15 GPa. The DMA results also indicate that the nanotubes had a strong influence on the composites damping properties. The through thickness permeability of the resulting buckypapers with nanoscale pore structure was also measured. AFM and SEM observations show that the SWNTs have a good dispersion in the buckypaper and nanocomposites. The research results show that the proposed buckypaper/resin infiltration approach is capable of fabricating nanocomposites with controllable nanostructure and high SWNT loading, which are important for developing high performance nanotube-based composites.

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**Keywords:** A. Nano-structures; B. Mechanical properties; Buckypaper

## 1. Introduction

A single-walled carbon nanotube (SWNT) can be thought of as a single graphite sheet rolled into a tube and capped by two hemisphere fullerenes. Recent investigations have shown that SWNTs possess remarkable mechanical properties, such as exceptionally high elastic properties, large elastic strain and fracture strain sustaining capability, exceeding those of any previously existing reinforcement materials of composites [1–6]. For example, both theoretical and experimental results have shown that SWNTs have a high elastic modulus in the range of 500–600 GPa [7]. The estimated maximum tensile strength of SWNTs is close to 200 GPa [8], which is 40 times higher than IM7 carbon

fiber. Carbon nanotubes also shows great flexibility compared to conventional fiber reinforcements [9,10]. Since SWNTs are considered by many researchers as the most promising reinforcement material for next generation high performance structural and multifunctional composites [11,12], strong interests exist in developing SWNT-reinforced nanocomposites [13–17].

Traditionally, researchers fabricated composites by directly mixing the SWNTs into polymers and then using casting or injection techniques to make the final nanocomposites. However, SWNTs have a strong tendency to form bundles and aggregate together because of their high surface area and the strong van der Waals interaction. Their very stable chemical characteristics and lack of functional sites on the surface make efficiently dispersing SWNTs into polymer matrix and controlling the final nanostructure of the nanocomposites during the processing difficult, particularly for

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high SWNT loading (>10 wt%) composites. Some researchers chemically modified the nanotube surface to obtain better nanotubes dispersion in polymer matrix [18,19]; however, the chemical modification may destroy the pristine structure of the SWNTs and decrease maximum modulus and strength by 15% [20]. Park et al. [21] tried an in situ polymerization method to effectively disperse as-pristine SWNT bundles into polyimide with the aid of sonication. Besides the dispersion issue, the rapidly increasing viscosity of the polymer made high SWNT loading in nanocomposites difficult.

In this research, a new technique approach was developed to manufacture SWNT-reinforced composites. The SWNTs were first dispersed into water with the aid of Triton X-100 surfactant and sonication to form a well-dispersed and stable suspension. The SWNTs suspension was filtrated to form buckypapers, which were composed of uniform SWNT rope networks. Buckypapers with preformed tube networks have a macroscale dimension and can be handled as conventional fiber mats to attain controllable reinforcement dispersion and volume content. The low viscosity resin solution was allowed to infiltrate the buckypapers to impregnate the SWNT rope networks. A hot press molding process was used for molding and curing to produce the final composites. Nanocomposites with preformed tube networks and high SWNTs loading (up to 39% by weight) were manufactured.

## 2. Experimental study

The SWNTs used in this study were produced by Carbon Nanotechnologies Inc. (CNI) (Houston, TX) under the brand name 'BuckyPearls™', since the SWNTs look like small black pearls. According to the CNI technical data sheet and report [22], the individual tubes are about 0.8–1.2 nm in diameter and 100–1000 nm in length. The BuckyPearls™ are purified SWNT products with a residual metal content of 3–12 wt%. The bulk density of the BuckyPearls™ is 0.4 g/cm<sup>3</sup>. The SWNTs were used without further purification.

### 2.1. Preparation of SWNT buckypaper

Buckypapers are thin (10–50 μm) membranes of nanotube networks produced by multiple steps of tube dispersion and suspension filtration. Since we can use aqueous suspension and a number of different water-based surfactants, relatively good tube dispersion can be expected in buckypapers. The formed tube networks in the buckypapers were kept in the composite fabrication operation and transited into the final solid nanocomposites. In other words, the buckypaper provided a nanotube reinforcement structure for the final nanocomposites.

In this study, buckypapers were prepared by grinding BuckyPearls™ with a little water using a mortar and pestle and sonicating for some time with a Sonicator 3000

(Misonix Inc.), a thick black paste was formed. A selected surfactant and more deionized water were added into the paste and conducted 30–200 min sonicating depending on suspension concentration. The multi-step sonication produced a stable ink-like suspension, which was found to remain stable for several weeks. The final concentration of the suspension was 10–200 mg SWNT/L. The suspension was filtrated through a filter with the aid of vacuum to fabricate the buckypaper. Following filtration, the buckypapers were thoroughly washed with deionized water to remove the dispersion surfactant. The buckypapers were carefully peeled off from the filter and dried overnight at room temperature and then dried in a vacuum oven.

### 2.2. Permeability test of SWNT buckypaper

The through thickness (*z*-direction) permeability of the produced buckypapers was measured to predict the resin infiltration time. Deionized water was used as the working fluid in the permeability test. The buckypapers acted as filter membranes. The buckypapers were placed inside a filter and sealed. Deionized water was added to flow under full vacuum through the buckypaper along the thickness direction. The water flow rate, buckypaper thickness, buckypaper surface area and vacuum pressure were recorded to calculate the *z*-direction permeability of buckypapers with 10–50 μm thickness and nanoscale pore structure. The *z*-direction permeability  $K_z$  (saturated permeability) was calculated by the following equation derived from Darcy's law: [23,24]

$$K_z = \frac{Q\eta L}{AP} \quad (1)$$

where:

$Q$	flow rate;
$\eta$	viscosity of water;
$L$	thickness of buckypaper;
$P$	vacuum pressure;
$A$	surface area of buckypaper.

### 2.3. Resin infiltration of SWNT buckypaper

The resin infiltration was performed under the same conditions of the permeability test; however, an epoxy resin solution was used as working fluid instead of water. Epoxy resin used for this research was Epon862 and the curing agent was EPI Cure W (Shell Chemical Company). Epon862 and EPI Cure W were mixed at a weight ratio of 100:26.4. The viscosity of the mixed resin was 2700 cp at room temperature. Due to the nanoscale pore structure of the buckypaper, the viscosity was too high to efficiently impregnate the buckypaper at room temperature. To decrease the viscosity, the mixed resin was diluted with acetone. The diluted resin system was allowed to flow through the buckypaper along its thickness direction for several hours depending on

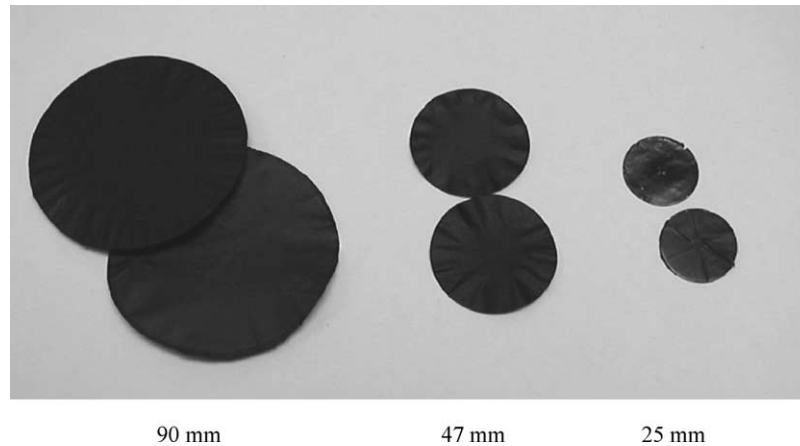


Fig. 1. Single-walled carbon nanotube buckypaper.

the thickness of the buckypaper. After infiltration, the impregnated buckypaper was soaked into Epon862 resin system overnight.

#### 2.4. Nanocomposites fabrication

SWNT buckypaper/epoxy resin nanocomposites were prepared using a hot press machine manufactured by Carver Co (Model #3925). Several resin-impregnated buckypapers were stacked to fabricate relatively thick ( $\sim 0.5$  mm) bulk composite samples. Following the instructions from the Shell Co., the buckypaper/epoxy nanocomposites were cured at  $177^\circ\text{C}$  for 2.5 h under pressure, cooled to room temperature, post-cured in the oven for another 2 h at  $177^\circ\text{C}$  and cooled to room temperature inside the oven. Three or five layers of impregnated buckypapers were stacked together to provide an adequate thickness of about 0.1–0.5 mm. Both the weight of buckypaper before the resin infiltration and the weight of the final composites were recorded to calculate the final tube weight percentage.

#### 2.5. Characterization

The nanostructures of buckypaper and buckypaper/epoxy nanocomposites were characterized using a scanning electron microscope (SEM) (Joel 6400F, Joel Co.) and an atom force microscope (AFM) (Dimension 3000, Digital Instrument Co.). The mechanical properties and damping behavior of the nanocomposites were tested with a dynamic mechanical analyzer (DMA) (DMA2980, TA Instrument Co.). The test mode was tensile (film) mode. The sample was heated to  $350^\circ\text{C}$  at  $5^\circ\text{C}/\text{min}$ . The frequency was 1 Hz.

### 3. Result and discussions

#### 3.1. Buckypaper and nanostructure

Fig. 1 shows the SWNT buckypapers produced in this research. Buckypapers have good strength and flexibility to

allow for handling like traditional glass fiber mat. The SEM and AFM images of SWNT buckypaper are shown in Figs. 2 and 3. The image of buckypapers showed that the tube network was composed of continuous SWNT ropes, which were the result of tube self-assembly by van der Waals force during buckypaper filtration [25]. The rope size and porous structure of the buckypapers were uniform, indicating very good tubes dispersion in the suspension. The ropes had an average diameter about 30–60 nm. The buckypaper nano-scale structures had pores with an average opening around 100–200 nm, which are much smaller than those in traditional glass fiber and carbon fiber fabrics or mats [26]. Since the porous structure of the buckypaper were going down to nanoscale, it is necessary to check the comparability between buckypaper pore size and the dimension of the resin/curing agent molecules. As shown in Fig. 4, under the minimum energy condition, the molecular dynamic simulation revealed that the Epon 862

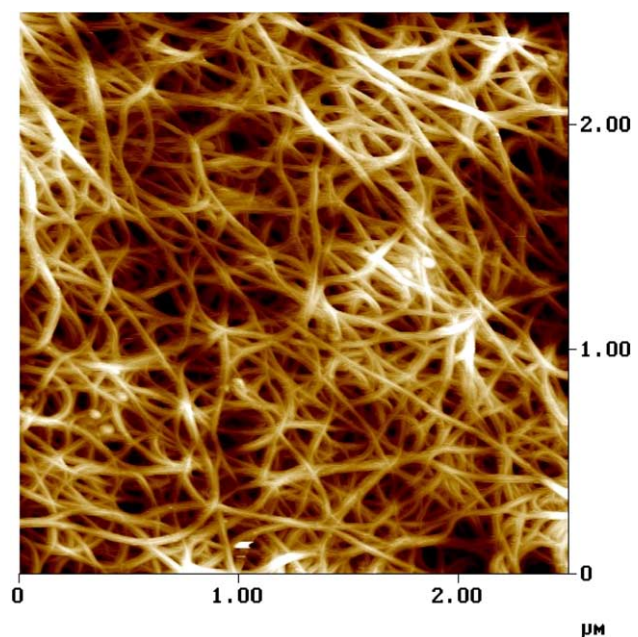


Fig. 2. AFM image of buckypaper surface.

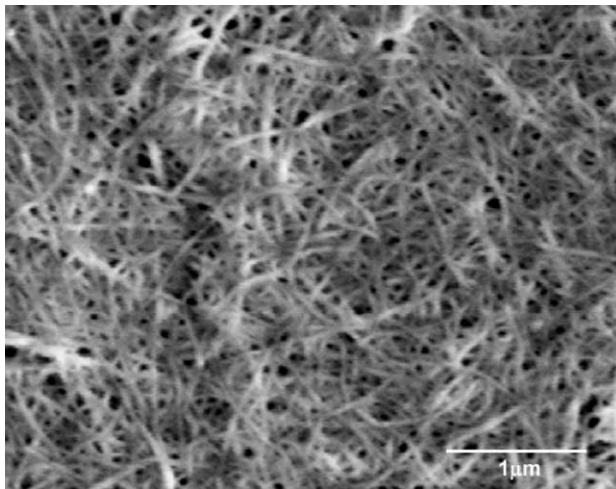
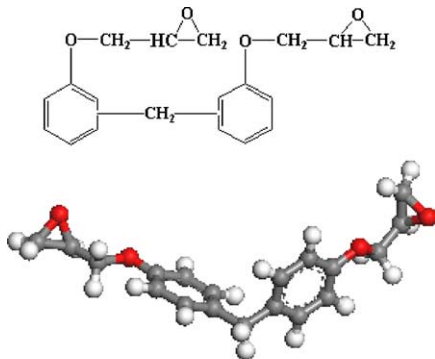
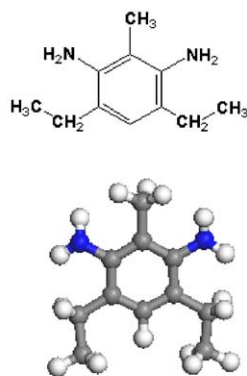


Fig. 3. SEM image of buckypaper surface.

epoxy resin and curing agent molecules have the approximate dimensions of  $23 \text{ \AA} \times 9 \text{ \AA} \times 6 \text{ \AA}$  and  $6.7 \text{ \AA} \times 6.5 \text{ \AA} \times 1.8 \text{ \AA}$ , respectively. The simulation result indicated that both resin and curing agent molecules could penetrate into this nanostructure and form a 3D cross-link network of resin matrix [26].



(a) Epon862 molecule



(b) EPI Cure W molecule

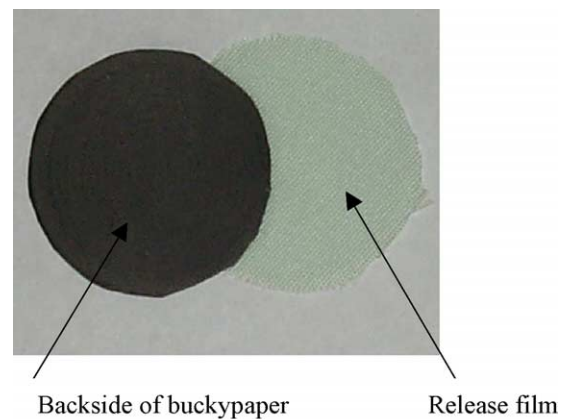
Fig. 4. Chemical structures and molecular models of EPON 862 resin and EPI Cure W under minimum energy.

Table 1  
 $K_z$  of buckypaper

Buckypaper thickness (m)	$2.54 \times 10^{-5}$	$3.12 \times 10^{-5}$	$3.57 \times 10^{-5}$
$K_z$ ( $\text{m}^2$ )	$3.358 \times 10^{-19}$	$1.105 \times 10^{-19}$	$1.442 \times 10^{-19}$
Average $K_z$ ( $\text{m}^2$ )	$1.968 \times 10^{-19}$		

### 3.2. Permeability and impregnation of buckypaper

A filtration system was used to impregnate the epoxy resin system into the buckypaper. Since in-plane resin flow and wetting are almost inapplicable due to nanoscale pore structure and extremely high flow resistance, the buckypapers had to be infused with resin and impregnated along their thickness direction. Under this condition, the  $z$ -direction permeability is a key parameter. The results of  $z$ -direction permeability test on the resulting buckypaper are shown in Table 1. The average value of buckypapers'  $z$ -direction permeability was about  $2 \times 10^{-19} \text{ m}^2$ . Compared to normal glass fiber reinforcement fabrics or mats ( $V_f = 60\%$ ), the permeability of buckypaper was 8–10 orders lower [27]. Therefore, infusing the resin through



(a) before resin infiltration



Resin covered backside of buckypaper

(b) after resin infiltration

Fig. 5. Buckypapers before and after resin infiltration.

Table 2  
Nanocomposites samples

Sample no.	Number of buckypaper layers	Composites weight (mg)	Composites thickness (mm)	SWNT content (wt%)
A	5	457.91	0.198	28.1
B	3	222.55	0.114	31.3
C	3	486.65	0.220	37.7
D	3	534.33	0.246	39.1

buckypapers in the in-plane direction, as in conventional RTM and VARTM process of fiber reinforced composites, is almost impossible.

The minimum infiltration time to completely infuse and impregnate the entire thickness direction of buckypapers can be calculated by Eq. (2), which is derived from 1D Darcy's Law:

$$t = \frac{\eta L^2}{K_z P} \quad (2)$$

where:

- $t$  infusion time;
- $\eta$  viscosity of resin;
- $L$  thickness of buckypaper;
- $P$  vacuum pressure.

The viscosity of Epon 862/EPI Cure W system was 2700 cp at room temperature, which means under full vacuum, completely impregnating a buckypaper of 40  $\mu\text{m}$  thickness at room temperature would take more than 60 h.

During such a long time, the Epon 862/EPI Cure W system will partly gel, which increases the viscosity and time to impregnate, making the process infeasible. Acetone was used to dilute and decrease the viscosity. However, the acetone content cannot be too high since sufficient resin must remain within the tube network after the evaporation of acetone to keep the desired resin content in the final nanocomposites. The viscosity of the diluted resin solution used in this research was below 100 cp at room temperature. Applying Eq. (2) shows the time for diluted resin to completely impregnate through a 40  $\mu\text{m}$  thickness buckypaper at room temperature was reduced to about 2 h. In this research, the infiltration time was chosen to be 5 h to ensure the buckypaper was completely impregnated, which was indicated by observing a liquid resin layer on the opposite surface of the buckypaper during infiltration. Fig. 5 shows the buckypaper before and after resin infiltration. In order to improve efficiency of the resin infiltration for producing large composite parts, higher infiltration pressure should be used in the process.

### 3.3. Buckypaper nanocomposites

The manufactured buckypaper-reinforced composites are listed in Table 2. The composites had a diameter of 47 mm. Direct mixing resin with SWNTs with less than 1 wt% loading will result in extremely high viscosity. High viscosity causes dispersion problems and makes manufacturing high quality composites difficult. Using the proposed method, the high viscosity issue can be avoided, and

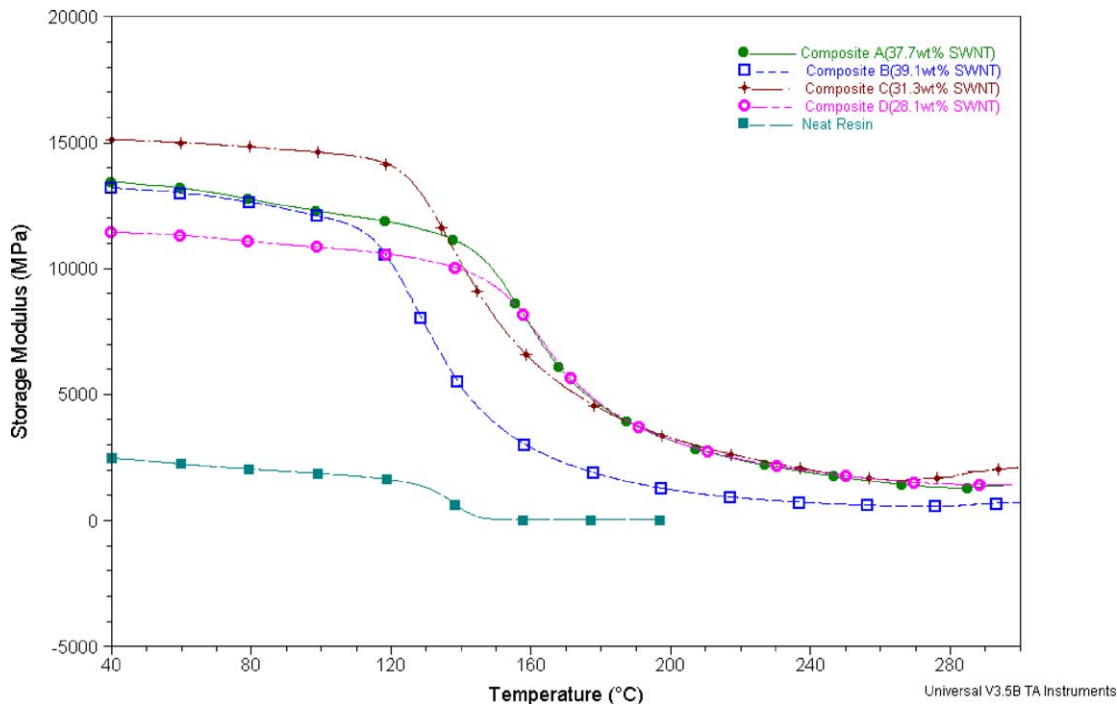


Fig. 6. DMA storage modulus curves of nanocomposites.

Table 3  
DMA storage modulus test results

SWNT loading (wt%)	Storage modulus (GPa)	Increase (%)
0	2.55	0
28.1	11.45	349
31.3	15.10	492
37.7	13.49	429
39.1	13.24	419

the SWNT loading of the resulting composites can be as high as 39 wt%.

### 3.4. Dynamic mechanical properties

The storage modulus results of the composites are shown in Fig. 6 and Table 3. The storage moduli of the resulting nanocomposites were dramatically increased (by 349–492% of the neat resin modulus). However, the storage moduli of the composites are significantly less than that predicted by the rule of mixtures for a random orientation individual SWNT-reinforced composites. We believe that the poor load transfer between tube/tube in the ropes and tube/resin matrix is the major reason. Particularly, the formation of large tube rope in the composites could significantly decrease the load transfer in the composites. For example, Salvetat et al. experimentally showed that when the SWNT rope diameters increased from 3 to 20 nm, the elastic and shear modulus of SWNT ropes could be decreased from 900 GPa to around 100 GPa due to possible tube slippage in the ropes [28]. Therefore, the actual nanotube rope-reinforced composites may demonstrate poor mechanical properties. To realize

individual tube dispersion in the buckypaper and composites to achieve better load transfer is an ongoing research effort for this study. Another notable phenomenon of the DMA results is that there is no obvious trend of the influence of tube loading on the storage modulus. We believe that the nanostructures and mechanical properties of the nanotube-reinforced composite are highly dependent on tube dispersion, which directly influences molecular interactions of tube/tube and tube/resin in the materials. Such molecular interactions will play a critical role in load transfer and interfacial bonding that determine mechanical properties of the nanocomposites. The variations in tube dispersion in the resultant composites could be the major reason for this phenomenon.

The DMA  $tg\delta$  curves are shown in Fig. 7. It can be seen that the  $T_g$ s of the resultant nanocomposites did not have obvious change. The damping behavior of the buckypaper reinforcement nanocomposites is different from that of the traditional fiber-reinforced composite. There is no obvious  $T_g$  peak on the  $tg\delta$  curve. After glass transition, the  $tg\delta$  continuously increases rather than going back to the base line. The damping behavior reflects the value of energy dissipation of molecular movement and interaction. For the cured resin, usually below the  $T_g$ , the resin molecule segment cannot completely move. The very low  $tg\delta$  value indicates no significant energy loss. During the glass transition, the molecule segment absorbed enough energy and began to move; however, the moving space inside the polymer was too tight, so more energy was required to move the molecules. A temperature higher than  $T_g$  resulted in enough free space inside the resin to let the molecule segments move freely, causing the  $tg\delta$  to again decrease.

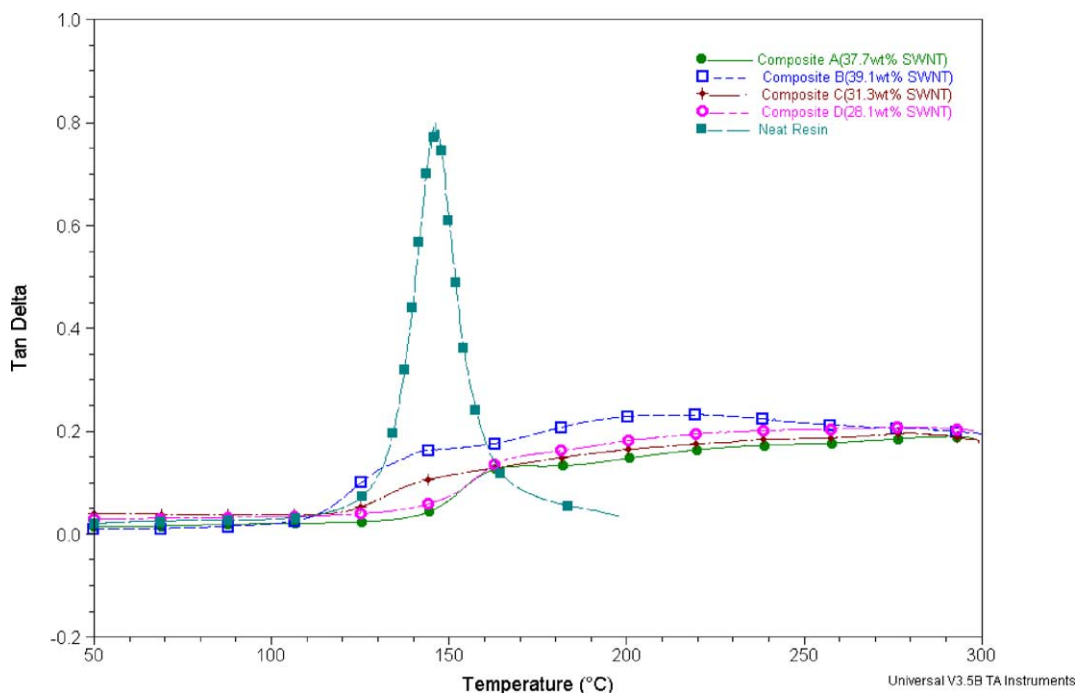


Fig. 7. DMA  $tg\delta$  curves of nanocomposites.

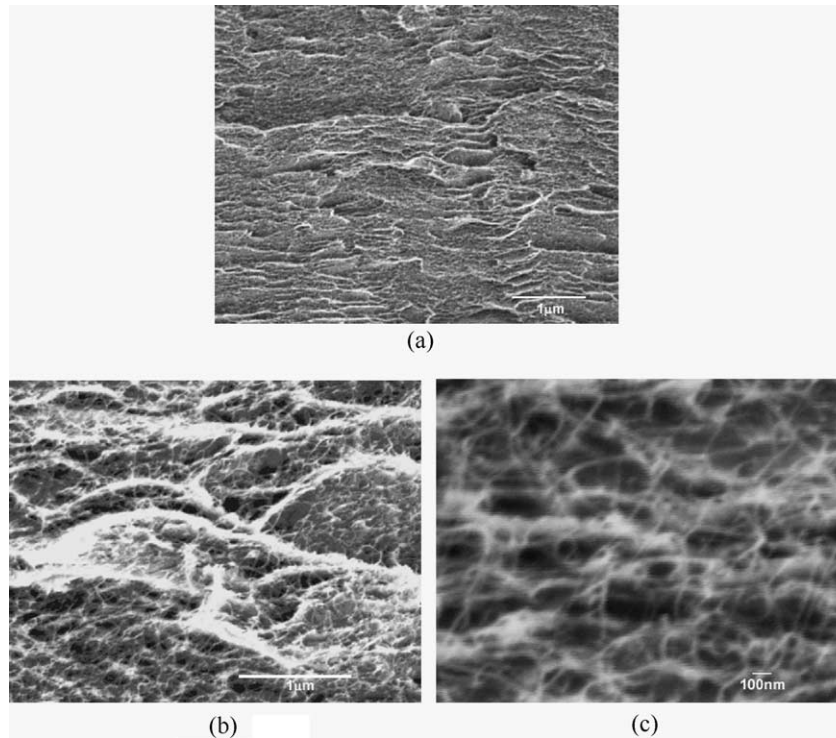


Fig. 8. SEM images of fracture surfaces of the composites.

For traditional carbon fiber and glass fiber reinforced composites, both fiber and porous structures are at microscale and only a very small portion of the resin molecules are able to directly interact with reinforcement. The damping behavior of the carbon fiber and glass fiber reinforced composites are almost the same as that of the neat resin. However, for the buckypaper-reinforced composite, SWNTs have extremely large surface area and they are at the same scale of the resin molecules. Interactions between tubes and resin molecules are expected to be intensive. Therefore, the resin molecules segment movement may intensively interact with the SWNTs and consume energy. These intensive molecular-level interactions may be the major reason to result in high  $tg\delta$  for the buckypaper-reinforced composites.

### 3.5. SEM observations of nanocomposites

Fig. 8 shows the SEM image of the fracture surface of the 39.1 wt% SWNT loading composite. This sample was fractured under tensile force. Fig. 8a shows that epoxy resin evenly impregnated throughout the buckypaper, which indicates good resin impregnation. The fracture surface of the nanocomposites was coarse, which may imply that the composites have good toughness because of the presence of nanotubes [9,10]. Fig. 8b and c shows that the SWNTs ropes were spread over the surface and continuous and evenly distributed networks were formed in the composites with such a high tube loading. It also indicates that the preformed

tube networks of the buckypapers are successfully transited into the final nanocomposites.

## 4. Conclusions

A new fabrication technique was developed in this research to produce SWNT-reinforced epoxy resin matrix nanocomposites. SWNTs were first fabricated into buckypapers to create continuous SWNT rope network as reinforcement skeleton in the final nanocomposites. The buckypapers produced with multiple dispersion and filtration steps have relatively uniform ropes size and porous structure. Because of the extremely low buckypaper permeability, the epoxy resin (Epon 862/EPI Cure W system) was diluted with acetone to decrease the viscosity to be efficiently infiltrated through buckypaper along its thickness direction, which facilitates resin/tube impregnation. A hot press molding process was used to make the final nanocomposites.

The produced nanocomposites had a SWNT loading up to 39%. AFM and SEM images showed that the epoxy resin in the buckypapers was adequately infused into nanoscale networks of the SWNTs. The storage moduli of the resulting composites were as high as 15 Gpa, which is 429% higher compared to neat resin modulus. The DMA results showed that the presence of SWNTs significantly affects the damping properties of the composites due to possible intensive molecular interactions between tubes and resin molecules. However, the mechanical properties of the resulting

composites were still lower than expected, which requires further investigation. The research results show that the developed buckypaper/resin infiltration technique of pre-forming tube network in buckypapers and using resin infiltration is feasible to produce high SWNT loading nanocomposites.

### Acknowledgements

The authors would like to acknowledge the support from the Air Force Research Laboratory and Florida State University Research Foundation.

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