



Short communication

## Enhancing mechanical properties of an epoxy resin using “liquid nano-reinforcements”

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## ARTICLE INFO

## Article history:

Received 28 October 2008

Received in revised form 11 January 2009

Accepted 13 January 2009

## Keywords:

Nanofiller

Hybrid

Epoxy resin

Nanocomposite

Mechanical properties

## ABSTRACT

“Liquid nano-reinforcements” and hybrids of nanofillers with different size were introduced in this study to simultaneously improve the reinforcement and toughening of an epoxy resin. The flexural strength and breaking strain of the epoxy composites with 0.3 wt% reactive carbon nanofibers (CNFs) and 0.2 wt% carbon nanotubes (CNTs) increased by 45% and 64%, respectively, exceeding that of the epoxy nanocomposites singly reinforced by the same (0.5 wt%) content of CNFs. The fracture surface of the hybrid nanocomposites indicated the high and broad resistance to fracture. Dynamic mechanical analysis showed there was an obvious increase in storage modulus and glass transition temperature for the hybrid nanocomposites compared to that of the neat epoxy resin.

Published by Elsevier B.V.

### 1. Introduction

The increasing proliferation and application of advanced polymer composites requires increasingly higher and broader performance resin matrices. To comprehensively improve mechanical properties such as strength, modulus and toughness simultaneously is particularly advantageous for aircraft applications where multi-axial loading and long-term durability are characteristic of designs. However, it has always been challenging to enhance toughness without sacrificing strength and modulus or the reverse [1–3]. In fact this inverse relationship has always plagued material enhancements using traditional approaches to structural composites, such as toughening by adding elastomer [4,5]. Carbon nanotubes (CNTs) and carbon nanofibers (CNFs) with excellent mechanical properties have been widely used as reinforcements for polymer composites. However, many reports indicate that carbon nanofiller reinforced polymer composites were weaker or only slightly stronger than the neat polymers [6–8]. And, strengthening and toughening seemed contradictory in many studies [1–3,9].

The goal of this work is to demonstrate that nano-enhancements to polymeric composites can show improvements in both toughness and strength related properties without sacrificing one for

the other. In other words the inverse relationship can be transformed to a direct relationship through the introduction of the nano-phase constituents. To make full use of material characteristics of different fillers, the combinations of different reinforcing fillers have been introduced into the preparation and application of composite materials. The advantages of hybrid systems include the combined materials properties of each component, more design choices, cost effectiveness, etc. [10–12]. But the hybrid concept has rarely been considered for nanocomposites up to the present [3]. Recently, Avilés and co-workers found the reinforcing effect of two types of CNTs with different size and aspect ratio in polymer materials exhibited a notable difference [6]. The typical diameters of multi-walled CNTs are in 10–20 nm range, and that for CNFs are in 50–200 nm range. The difference between the diameters of CNTs and CNFs make them exert reinforcing effects in the different nanoscale levels. In addition, the hybrids of CNFs and CNTs may avoid the aggregates of single nanofillers due to the high concentration.

Epoxy resin is a widely used polymer matrix for advanced composites with its good stiffness, dimensional stability and chemical resistance. But with the increasing demand for higher mechanical properties in aircraft and industrial applications, the strength, modulus and toughness of epoxy resins need to be further improved to match the advanced reinforcing fibers, such as T1000<sup>®</sup> carbon fiber. Despite some aggressive researches have been devoted to reinforcement of epoxy resin with CNTs or CNFs, the experimental

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results are deficient [2,6,13,14]. Some desirable reinforcing and toughening researches were focused on rubbery epoxy resin [14,15], and the valuable studies on the epoxy resin used for structural materials are still urgently needed. In our previous studies, we developed a kind of reactive graphitic carbon nanofibers (r-CNFs), which showed effective enhancement in the strength and toughness for polymers [16,17], and in this paper, the concepts of “liquid nano-reinforcements” and hybrids of CNFs and CNTs are introduced to realize the improvement of reinforcing and toughening epoxy resins simultaneously.

## 2. Experimental details

### 2.1. Materials

The reactive CNFs (r-CNFs) were developed through the reaction of the cut amino-linkerized herringbone CNFs by digital sonifier with butyl glycidyl ether (BGE). The formation process of r-CNF is shown in Fig. 1(a). The detailed synthesis procedures were described in [17,18]. In our previous studies, the  $H^+$  in the reactive group  $OH^-$  has been shown reactive to form covalent bonding with epoxy resin [17,18]. The r-CNFs are 50–150 nm in diameter and 1–5  $\mu\text{m}$  in length (shown as insert in Fig. 1(b)). The amino-functionalized CNFs (Fig. 1(b)) provided by Vanderbilt University were blended with BGE, and treated using a digital sonifier operating at 70 W and 180 min elapsed time. Then the excess BGE was removed by using a vacuum oven and the final mass ratio of r-CNFs to BGE was optimized into 1:1. We found that the r-CNFs existing in such small amount of BGE forming the solution, the so-called “liquid nano-reinforcement”, could facilitate the easy dispersion and reaction of the r-CNFs in the epoxy. Therefore, the r-CNFs were added into the epoxy in the form of liquid nanofiller. The multi-walled CNTs used in this study were provided by Zyvex Co. The average

diameter of the CNTs is 20 nm, and lengths are from 100 nm to 1  $\mu\text{m}$  (Fig. 1(c)). The epoxy, EPON<sup>®</sup> 862, was used in this paper.

### 2.2. Preparation of the epoxy nanocomposites

The neat epoxy resin was prepared by mixing the epoxy resin with the EPI-CURE Curing Agent W at room temperature for 2 h, degassing the mixture under vacuum conditions, and curing via a thermal cycle (120 °C for 1 h and 177 °C for 2 h). For preparation of the r-CNF/epoxy and r-CNF/CNT/epoxy composites, the carbon nanofillers were added into the epoxy resin and the mixture was stirred at 60 °C for 3 h. Then, the curing agent was added and an ultrasonic cleaner was used for further mixing of the nanofillers at room temperature for 2 h. Subsequently, the mixture of nanofillers and epoxy was degassed under vacuum condition, followed by the same thermal curing schedule as that of the neat epoxy.

### 2.3. Characterizations of the materials

An MTS load frame was used for the three-point bending test of neat epoxy and composites according to ASTM standard D790-00. The speed of the cross-head was 1 mm/min. Five specimens were measured for each case, and the average and standard deviation values of flexural strength, modulus and ultimate strains were calculated. The microstructures of r-CNFs and CNTs were observed using JEM-200CX Transmission electron microscope (TEM) produced by NEC Company, Japan. The microstructures of fracture surface of the nanocomposites were observed using S-3500N model scanning electron microscope (SEM) produced by Hitachi Company, Japan. The dynamic mechanical analysis (DMA) of the neat epoxy and the nanocomposites was carried out with a Rheometrics Scientific DMTA 3E Dynamic Mechanical Analyzer in the three-point bending mode at a frequency of 1 Hz. The specimens were heated from room temperature to 200 °C at a heating rate of 5 °C/min.

## 3. Results and discussion

### 3.1. Mechanical properties of the r-CNFs/epoxy nanocomposites

The flexural properties of the neat epoxy and nanocomposites in this study are shown in Table 1. It can be seen that the r-CNFs resulted in effective reinforcing effect to the epoxy matrix. The flexural strength, modulus and breaking strain of the epoxy systems increased by 32%, 9% and 70%, respectively, when 0.3 wt% r-CNFs were added. Modulus enhancement is lower than that of strength due to the involvement of the reactive diluent BGE. The optimum ratio of r-CNFs to BGE is 1:1 gained for the epoxy resin Epon<sup>®</sup> 862 through our extensive prior experimental studies. It is worth mentioning that the breaking strains of the epoxy were dramatically increased with the addition of the r-CNFs, i.e. a 70% increment occurred for the 0.3 wt% r-CNFs reinforced epoxy. This large enhancement in breaking strain is greatly significant for structural composite matrix materials, and combined with 32% increase in strength, it denotes a significant improvement in toughness of the epoxy nanocomposites. Therefore, the addition of the liquid nanofiller: r-CNFs in BGE with ratio 1:1 is effective for simultaneously strengthening and toughening the epoxy. The morphology of the fracture interface of neat epoxy and the composite specimens was observed by scanning electron microscopy, as shown in Fig. 2. Compared to the smooth fracture surface of the neat epoxy (Fig. 2(a)), the fracture surface of the r-CNF/epoxy composites (Fig. 2(b)) presented ridges, rivers and plastic deformation predominantly. These more obvious 3-dimensional patterns came from the changes in fracture plane due to the existence of r-CNFs, which are associated with the greater area for absorption of fracture energy

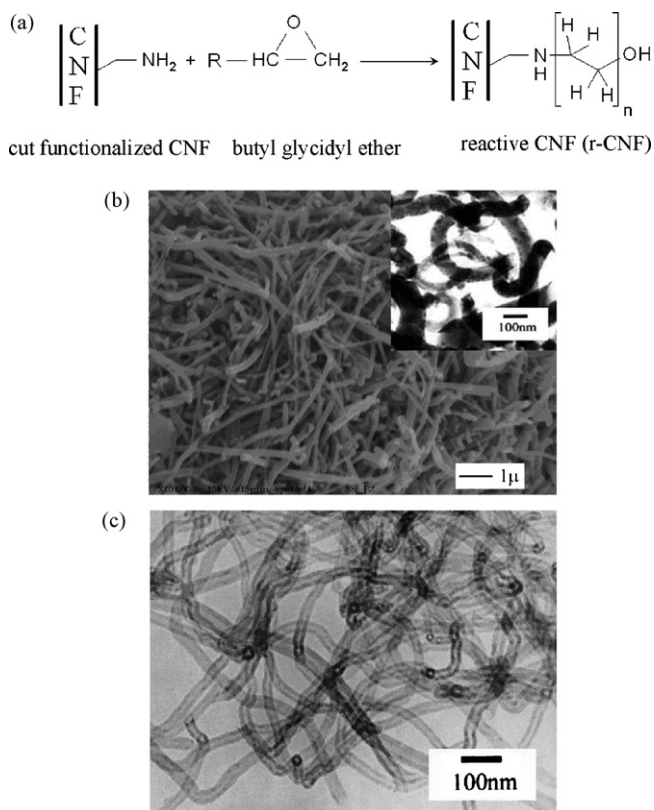


Fig. 1. The carbon nanofillers used in experiment: (a) formation of an r-CNF; (b) SEM micrograph of CNFs; (c) TEM micrograph of CNTs.

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