Composites Part B 85 (2016) 15–23

Contents lists available at ScienceDirect

Composites Part B

journal homepage: www.elsevier.com/locate/compositesb

Improving mechanical properties of high volume fraction aligned multi-walled carbon nanotube/epoxy composites by stretching and pressing

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A R T I C L E I N F O

Article history:
Received 24 May 2015
Received in revised form
19 August 2015
Accepted 16 September 2015
Available online 26 September 2015

Keywords:
A. Polymer-matrix composites (PMCs)
A. Nano-structures
B. Mechanical properties
D. Mechanical testing

A B S T R A C T

Aligned multi-walled carbon nanotube (CNT) sheets produced from aligned CNT arrays were used to develop high volume fraction CNT/epoxy composites. Stretching and pressing techniques were applied during CNT sheet processing to straighten the wavy CNTs and to enhance the dense packing of CNTs in the sheets. Raman spectra measurements showed better CNT alignment in the CNT sheets and the composites after stretching and pressing. Aligned CNT/epoxy composites with CNT volume fraction up to 63.4% were developed using hot-melt prepreg processing with a vacuum-assisted system. Stretching and pressing of the CNT sheets enhanced the mechanical properties of high volume fraction CNT/epoxy composites considerably. Stretching and pressing increased tensile strength of the composites by 32% and elastic modulus of the composites by 27%. Applying stretching and pressing is effective for production of superior CNT sheets with high alignment and dense packing of CNTs, thereby supporting the development of high-performance CNT composites.

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1. Introduction

Carbon nanotubes (CNTs) have highly desirable mechanical, electrical, and thermal properties [1–4]. Their excellent mechanical properties along with their low density make CNTs attractive as a potential reinforcement material for next-generation advanced composites. The advanced composites used for aerospace structures comprise a high volume fraction of aligned stiff fibers embedded in high-performance polymers [5]. Vertically aligned CNT arrays have been developed for the production of high volume fraction aligned CNT-reinforced polymer composites [6–9]. Furthermore, Wardle et al. [10] fabricated high volume fraction aligned CNT/epoxy composites using mechanical densification of vertically aligned CNT arrays, followed by capillarity-induced wetting with unmodified epoxies. However, the composite length was restricted drastically because of limited height of the CNT arrays. Therefore, long-aligned CNT sheets have been created recently from vertically aligned CNT arrays using solid-state drawing and winding techniques [11–13]. Highly oriented aligned CNT sheets have been particularly promising for the development of high volume fraction CNT composites with high performance.

High volume fraction CNT composites based on aligned CNT sheets have attracted great interest because they are envisioned as a revolutionary advanced composite material for a host of demanding applications [14–17]. The high volume fraction allows the properties of CNTs to dominate the composite properties [10]. However, several reports have described that the waviness, entanglement, and poor packing of CNTs in the sheets degraded the

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http://dx.doi.org/10.1016/j.compositesb.2015.09.012
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mechanical properties of their composites [18–22]. Therefore, mechanical stretching has been applied to the CNT sheets to straighten the wavy CNTs and to enhance dense packing of CNTs, thereby improving the strength and stiffness of CNT-reinforced composites [23–25]. Nevertheless, the handling of the CNT sheets without resin for mechanical stretching is generally difficult because of static electricity [15]. Consequently, Nam et al. [26] proposed a simple press-drawing process by which pressing was applied directly where a CNT web enters the winding roll to create superior aligned CNT sheets with high strength and stiffness. These CNT sheets are effective for the production of high-performance CNT composites.

Recently, a stretch-winding technique has been applied to fabricate high volume fraction CNT-reinforced polymer composites with high strength and stiffness [16]. For this study, aligned CNT sheets were produced from vertically aligned CNT arrays using a novel combination of stretch-drawing and press-winding techniques. These techniques can reduce the waviness and entanglement of CNTs considerably, and can increase dense packing of CNTs in the sheets, thereby improving the properties of aligned CNT composites. High volume fraction CNT composites based on epoxy resin and aligned CNT sheets were developed using hot-melt prepreg processing with a vacuum-assisted system (VAS). The mechanical properties of the high volume fraction CNT/epoxy composites were studied. The CNT volume fraction was estimated through thermogravimetric analysis (TGA) data. Field emission scanning electron microscopy (FE–SEM) (SU8030; Hitachi Ltd., Tokyo, Japan) was used to investigate the microstructural morphologies of the CNT sheets and their composites.

2. Experimental procedures

2.1. Materials

A B-stage epoxy resin sheet covered with release paper and plastic film was obtained from Sanyu Rec Co. Ltd. (Osaka, Japan) with the recommended cure condition of 130 °C for 2 h. The areal weight of the B-stage epoxy resin sheet with density of 1.2 g/cm^3 was controlled to approximately 12 g/m^2. Vertically aligned multi-walled CNT arrays with about 0.8 mm height were grown on a bare quartz substrate using chloride-mediated chemical vapor deposition [7]. Fig. 1a portrays a vertically aligned CNT array used for this study. An FE–SEM micrograph showing horizontally aligned CNTs drawn from the CNT array was inserted in Fig. 1a. A scanning transmission electron microscopy (STEM) image showing the high quality of CNTs is presented in Fig. 1b. As-grown CNTs examined in this study have mean diameter of 22 nm (see Fig. 1b inset).

2.2. Processing of aligned CNT sheets

The main purpose of our strategy for fabricating high-strength and high-modulus CNT composites is to create superior aligned CNT sheets before embedding them into a polymer matrix. Pristine aligned CNT sheets have been produced from vertically aligned CNT arrays using drawing and winding processes [12,15]. Although most of the CNTs are aligned unidirectionally, many wavy and entangled CNTs were observed in the pristine CNT sheets. Therefore, mechanical stretching has been applied to the aligned CNT sheets to straighten the wavy CNTs in the sheets [25]. In addition, a stretching system to stretch CNT webs during the CNT sheet processing has been reported recently by Wang et al. [16]. The CNT webs traveled horizontally and passed through a stretching system including a pair of stationary rods. For our study, stretched CNT sheets were produced from aligned CNT arrays through drawing and stretch-winding processes. Furthermore, pressed CNT sheets were created from aligned CNT arrays using drawing and press-winding techniques, as presented by Nam et al. [26]. Moreover, a new combination of both stretching and pressing was proposed to develop stretch-pressed CNT sheets for additional improvement of the composite properties. Fig. 2 depicts a schematic showing the processing of an aligned CNT sheet using drawing, stretching, winding, and pressing techniques. Pristine, stretched, pressed, and stretch-pressed 300-ply aligned CNT sheets were used for composite fabrication.

2.3. Production of aligned CNT/epoxy composites

Composites made of an epoxy resin film and aligned CNT sheets were developed using hot-melt prepreg processing with the VAS. This method maintained the alignment of CNTs during epoxy resin impregnation [25,26]. First, an aligned CNT/epoxy prepreg was prepared by stacking 300-ply CNT sheets with 20 mm width and 40 mm length on an epoxy resin film. Then, the prepreg was set between two release films (WL5200; Airtech International Inc., CA, USA). Next, the prepreg was pressed under 0.5 MPa pressure for 5 min at 100 °C using a test press (Model MP-WNL; Toyo Seiki Seisaku-Sho Ltd., Tokyo, Japan). Subsequently, the prepregs were peeled from the release films and release paper. Finally, the prepregs were cured at 130 °C for 2 h under 2 MPa in the VAS to produce the composites. The pristine, stretched, pressed, and stretch-pressed CNT/epoxy composites were fabricated for comparative assessments.

2.4. Thermogravimetric analysis

The thermal degradation behaviors of epoxy resin, CNTs, and their composites were analyzed up to 800 °C in argon gas at a flow

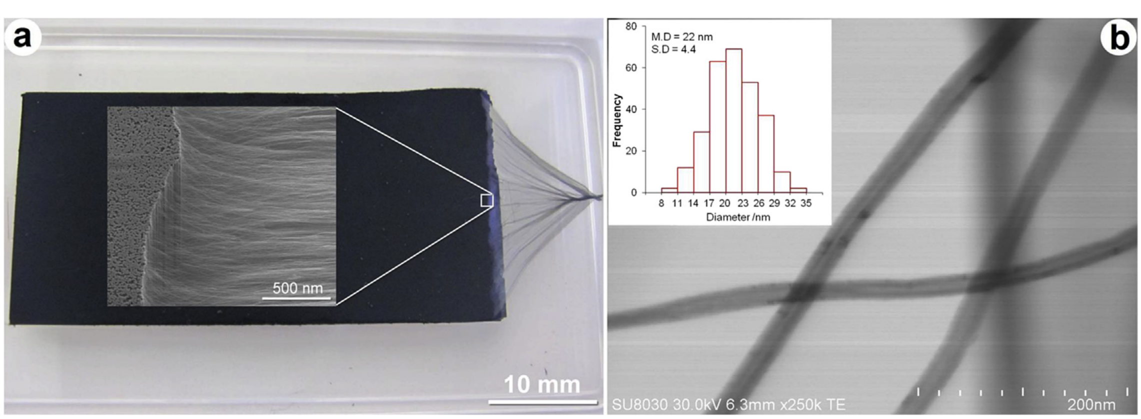

Fig. 1. (a) Vertically aligned CNT array and an inserted FE–SEM image showing horizontally aligned CNTs. (b) A STEM image and diameter distribution of CNTs.

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