Composites Science and Technology 81 (2013) 69–75

Contents lists available at SciVerse ScienceDirect

Composites Science and Technology

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

Mechanical and interfacial evaluation of CNT/polypropylene composites and monitoring of damage using electrical resistance measurements

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ARTICLE INFO

Article history:
Received 11 December 2012
Received in revised form 27 March 2013
Accepted 1 April 2013
Available online 6 April 2013

Keywords:
A. Nano composites
B. Electrical properties
C. Crack
D. Life prediction

ABSTRACT

Carbon nanotube (CNT)/polypropylene (PP) composites were compounded using a solvent dispersion method to more uniformly disperse the filler. A twin screw extruder was then used to manufacture specimens. The effect of low CNT concentrations on the mechanical and interfacial properties of PP were investigated using tensile and impact tests as well as a pull-out test of a microdroplet of the composite, on a single fiber. Low concentrations of CNT resulted in small, but significant increases, in Young's modulus, impact strength and interfacial adhesion. The increase in these mechanical properties is attributed to good reinforcing effects of the CNT filler. Measurement of the change in electrical resistance during bending and fatigue loading was used to monitor internal damage in the CNT/PP composite specimens. For CNT/PP composites with low CNT concentrations, these resistance measurements provide useful insight into internal failure, during cyclic loading.

1. Introduction

Polypropylene (PP) is one of the most widely used semi-crystalline thermoplastics for general purpose applications. Its attractiveness, as a potential matrix polymer, is based on its low price, favorable Physical/mechanical properties such as good stiffness, lightweight, good weathering ability, and design flexibility [1]. During a composite's service life, damage might initiate in the polymer matrix in the form of microcracks and/or delaminations. Frequently, inspection and evaluation is needed to detect such damage that may lead to failure. The development of techniques for damage detection and monitoring of structural integrity is, therefore, crucial to expanding the uses and applications of such composites [2].

Recently, due to their high aspect ratio and low density, nanoscale reinforcing particles have attracted considerable attention by polymer scientists and engineers [3–6]. Carbon nanotubes (CNTs) also have attractive properties for producing conductive composites, with a minimum of additional constituents. An advantage of CNT as a reinforcement material, is a large surface area to volume ratio which improves adhesion with the polymeric matrix, thereby, enhancing composite properties [7,8]. However, due to the strong intermolecular van der Waals interactions among the nanotubes, which can lead to the formation of aggregates, it is very difficult to uniformly disperse CNT in a polymer matrix [9]. Good dispersion of the CNTs in a polymer matrix is a prerequisite to optimize CNT as an effective reinforcing filler for polymer composites [10–14]. The development of favorable polymer composites based on thermoplastic matrices, while maintaining properties similar to those thermosetting polymer composites, would be very advantageous for many industrial needs. Minimizing the filler percentage while retaining good polymer properties would reduces costs. The high aspect ratios of CNTs shows promise of significantly enhancing the mechanical properties of polymers at low filler concentrations [15–17].

The introduction of CNTs into a polymer matrix is a promising approach for damage monitoring by measurements of resistance change [18,19]. The use of such electrical resistance measurements, as a nondestructive means of damage monitoring, is based on the premise that changes in the dimensions of the matrix affects the conductive paths within the composite material [20–24]. Changes in the network structure of the percolated CNTs owing to mechanical strain and/or the development of microcracks causes resistance changes, which can provide valuable information about the structural integrity of the composite [25–28].

In the research reported here, mechanical and interfacial properties of CNT/PP composites, with low filler content, were investi-

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http://dx.doi.org/10.1016/j.compscitech.2013.04.001

gated and compared with those of the neat PP matrix material. The electrical resistance monitoring of damage in the CNT/PP composites was performed while specimens were being exposed to cyclic loading.

2. Experimental

2.1. Materials

Multi-wall carbon nanotubes (MWCNTs, IlJin Nanotech Co., Korea), produced by chemical vapor deposition (CVD) were used as the reinforcing and sensing material. According to the producer, the purity of the MWCNT was better than 95%. The MWCNT are characterized by a relatively wide range of diameters (10–25 nm), and lengths (5–10 μm). Polypropylene (M1400, LG Chemical, Korea) was used as the thermoplastic matrix. Xylene (Samchun Pure Chemical Co., Korea) was used as the CNT dispersion-solvent. Glass fibers (RS2200KT-111A, Owens Corning Inc., USA) with an average diameter of approximately 16 μm were used as reinforcing fibers.

2.2. Methodologies

2.2.1. Fabrication process of CNT/PP composites

Fig. 1 shows schematically the fabrication process for the CNT/PP composites. The two processes used in the fabrication of the composite test specimens were: nanofiller dispersion and injection molding. To retain the inherent properties of PP, CNTs were added to the PP matrix at the low concentration of 0.5 wt%. In the dispersion step, the CNT was mixed with the xylene dispersion solvent in a beaker. As outlined in Fig. 1, the fabrication process started with (1) Stirring a mixture of PP and xylene heated to 210 °C and exposing this mixture of CNTs/xylene to 6 h of sonication, to obtain a uniform dispersion, (2) the mixture of the products from step 1 were then exposed to an additional hour of sonication at 210 °C and (3) the solvent was removed by a 24 h evaporation process at 60 °C in a vacuum oven. This material was then pelletized and the resulting blended granules were injection-molded using a co-rotating intermeshing twin-screw extruder (Bau-Tech, Korea). This extruder has a screw diameter of 19 mm and a distance between screw axes of 18.4 mm with a *L*/*D* ratio of 40. The screw speed was 150 rpm, and the residence time for the melt was about 3 min. The samples, for the mechanical testing, were injection molded at a temperature profile of 190/190/200/200 °C.

2.2.2. Mechanical properties measurement

The dimensions of the dog-bone specimens for the tensile test were 33 × 6 × 3 mm. Tensile tests were conducted using a Universal Testing Machine (LR 10K, Lloyd Co., UK) according to ASTM D638, at a test speed of 50 mm/min. Notched Izod impact strength tests were conducted, using an Izod impact instrument, according to ASTM D256. The impact speed and impact hammer energy were 3.5 m/s and 6.8 J, respectively. The dimensions of typical molded Izod impact test specimens were 63 × 12.7 × 3 mm. To provide for statistically meaningful results, a minimum a 10 samples were tested for each test condition.

2.2.3. Evaluation of the dispersion of the CNT/PP composites

The volumetric electrical resistance of the CNT/PP composite specimens was measured by the four-probe method using a multimeter (HP34401A), as shown schematically in Fig. 2. Electrical contact points, located at regularly spaced intervals, along the specimen using embedded copper wire without silver paste, were used to determine the electrical volumetric resistivity. The volumetric resistivity is the resistance per unit volume of the bulk material, and was used to provide a measure of the state of disper-

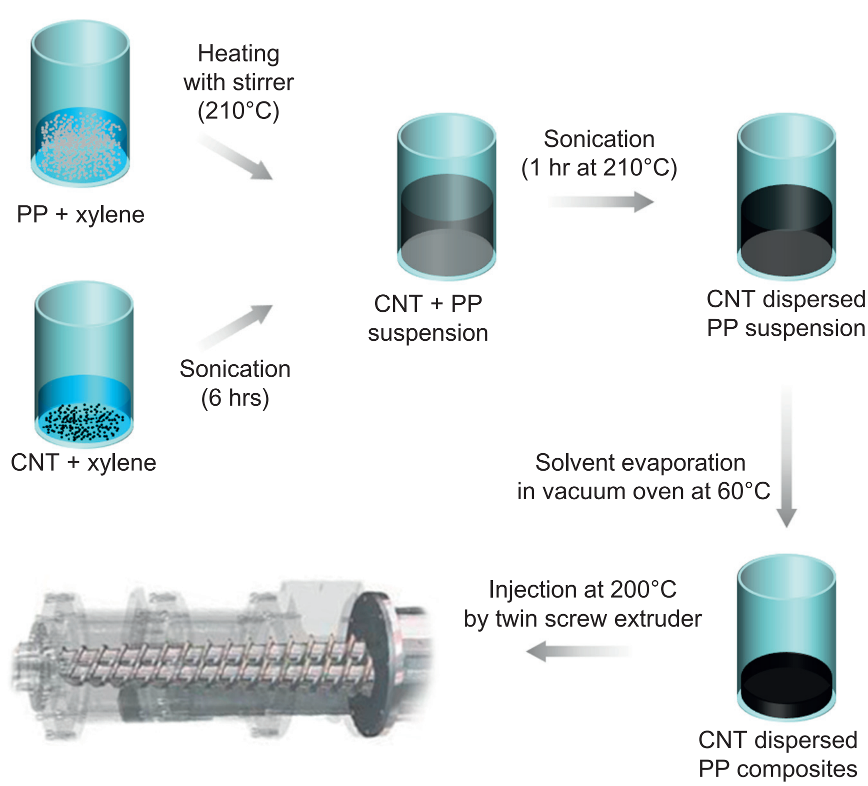

Fig. 1. Schematic outline of the fabrication process for CNT/PP composites.