

Interactions between the glass fiber coating and oxidized carbon nanotubes



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ABSTRACT

Chemically oxidized multiwall carbon nanotubes (MWCNTs) were deposited onto commercial E-glass fibers using a dipping procedure assisted by ultrasonic dispersion. In order to investigate the role of the fiber coating (known as “sizing”), MWCNTs were deposited on the surface of as-received E-glass fibers preserving the proprietary coating as well as onto glass fibers which had the coating deliberately removed. Scanning electron microscopy and Raman spectroscopy were used to assess the distribution of MWCNTs onto the fibers. A rather homogeneous coverage with high density of MWCNTs onto the glass fibers is achieved when the fiber coating is maintained. Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS) and nuclear magnetic resonance (NMR) analyses of the chemical composition of the glass fiber coating suggest that such coating is a complex mixture with multiple oxygen-containing functional groups such as hydroxyl, carbonyl and epoxy. FTIR and XPS of MWCNTs over the glass fibers and of a mixture of MWCNTs and fiber coating provided evidence that the hydroxyl and carboxyl groups of the oxidized MWCNTs react with the oxygen-containing functional groups of the glass fiber coating, forming hydrogen bonding and through epoxy ring opening. Hydrogen bonding and ester formation between the functional groups of the MWCNTs and the silane contained in the coating are also possible.

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1. Introduction

The recent advances in nanoscience and nanotechnology have motivated the development of hierarchical composites which combine the multifunctional characteristics of nanoscale fillers with the structural properties of traditional microscale fibers [1–5]. The addition of nanostructured fillers to polymer composites can improve their interlaminar and/or intralaminar properties [6,7]. In particular, the addition of carbon nanotubes can provide enhanced mechanical, electrical, thermal and sensing capabilities to polymer composites [2,4,8–11]. In an attempt to exploit the multifunctional characteristics of such nanoscale fillers for advanced composites, many studies have focused on placing carbon nanotubes (CNTs) or carbon nanofibers within the polymer matrix. However, the addition of nanotubes within the polymer matrix increases its

processing viscosity difficulting composite manufacturing. Additionally, due to the high surface area of CNTs and the van der Waals attractive forces among them, their addition to the matrix tends to form aggregates which may act as stress concentrators inducing premature matrix cracking [12,13]. Other alternatives for introducing CNTs into structural composites imply placing them onto the fiber surface [14–16] or directly growing them onto the fiber [5,17,18]. Problems associated with direct growth of CNTs onto micrometer fibers include a reduction in the ultimate strength of the fibers as a result of their thermal degradation during the CNT synthesis [5,18]. Among the methods for placing the CNTs onto fibers, the most common are covering the fibers with coatings containing CNTs [12,19], dipping the fibers in a liquid solution containing CNTs, and depositing CNTs onto the fibers assisted by an electric field [1,14,20–23]. Dip coating, electrochemical and electrophoretic deposition approaches have been extensively used for depositing CNTs onto carbon and glass fibers [1,3,6,24–26]. Some efforts to homogeneously deposit CNTs onto engineering fibers using either dipping or electrophoretic deposition require fiber [10,21] and/or CNT [2,16] functionalization. Other research

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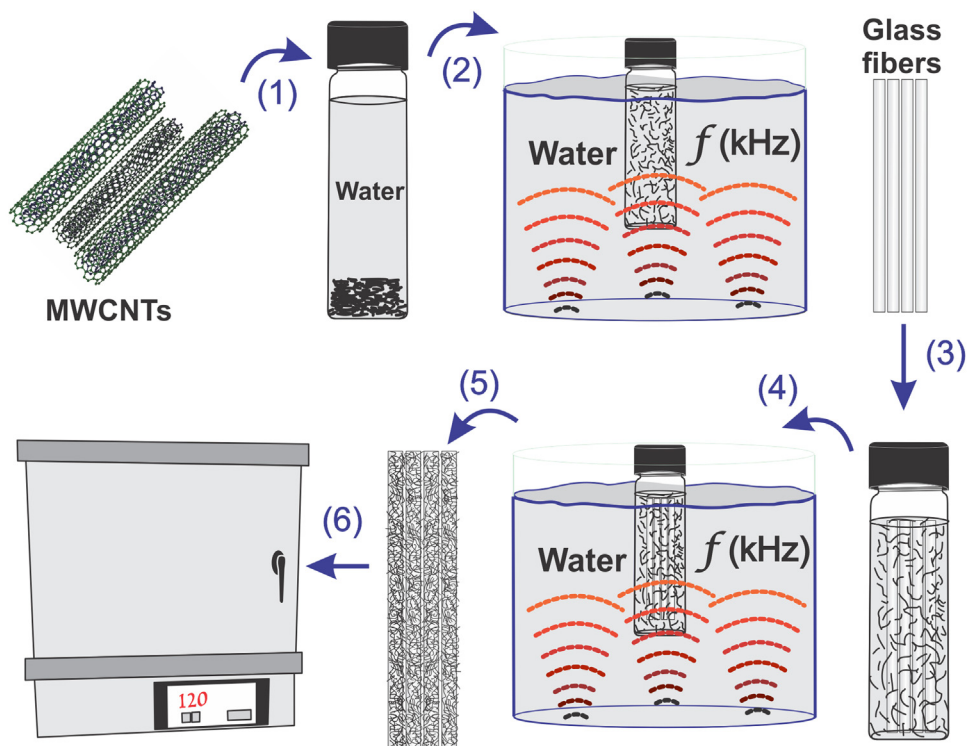


Fig. 1. Ultrasonic procedure for depositing MWCNTs onto glass fibers.

efforts employ surfactants in the suspending medium [2,14,16] or use home-made fiber coatings [12,27]. In spite of these advances, additional efforts must be conducted to obtain a more homogeneous deposition of CNTs onto engineering fibers with high density of CNTs, especially for large scale purposes. Additionally, the role of the fiber coating in the CNT-to-fiber deposition process is not understood, since in most cases the fibers are used after deliberately removing their coating [3,5] or simply downplaying the role of such a coating. Therefore, the current work investigates the interactions between the coating of commercial E-glass fibers and the functional groups existing at chemically oxidized multiwall carbon nanotubes (MWCNTs), upon their deposition onto the fibers' surface. Acid oxidized MWCNTs are deposited onto either as-received commercial glass fibers (containing a proprietary fiber coating) or onto glass fibers whose coating has been deliberately removed. The deposits of MWCNTs onto the fibers are analyzed by scanning electron microscopy (SEM) and Raman spectroscopy. The chemical composition of the glass fiber and its coating are characterized in order to identify the chemical groups that are prone to react with the functional groups of the oxidized MWCNTs. A mixture of MWCNTs and fiber coating, which was extracted from the fibers with deposited MWCNTs, is analyzed to better understand the chemical interactions among the functional groups of the MWCNTs and the reactive functional groups on the glass fibers' surface.

2. Materials and methods

2.1. Materials

Commercial E-glass fibers (Poliformas Plásticas S.A de C.V. Mérida, Mexico) with an average diameter of $15\ \mu\text{m}$, density of $2.54\ \text{g}/\text{cm}^3$ in the form of fiber tows (~ 4000 filaments per tow) were used in this study. A group of fibers were used as-received (containing a proprietary coating or "sizing"), and for a second group the proprietary coating was removed by Soxhlet extraction in acetone. Commercial MWCNTs (Cheap Tubes Inc., Vermont, USA) with

Table 1

Identification of glass fibers with and without deposited MWCNTs.

Label	Glass fiber condition
CC	Containing the coating (as-received).
CR	With the coating removed.
CC-MW	CC with deposited MWCNTs.
CR-MW	CR with deposited MWCNTs.

purity $> 95\%$, $30\text{--}50\ \text{nm}$ outer diameter, $5\text{--}10\ \text{nm}$ inner diameter, and $1\text{--}6\ \mu\text{m}$ length were used. The MWCNTs were oxidized using a solution of $\text{H}_2\text{SO}_4/\text{HNO}_3$ at $3.0\ \text{M}$ for 2 h, following the procedure reported in Ref. [28]. It has been shown that this acid oxidation treatment generates hydroxyl and carboxyl functional groups on the CNTs walls and ends [28,29].

2.2. Deposition of MWCNTs onto glass fibers

MWCNTs were deposited onto as-received glass fibers (labeled "CC", for "coating-containing") as well as onto fibers whose coating was removed (labeled "CR", for "coating removed") following the procedure depicted in Fig. 1.

The procedure used to deposit MWCNTs onto glass fibers, Fig. 1, consisted of adding 5 mg of MWCNTs to 40 mL of distilled water (1) and dispersing the MWCNTs into the water for 2 h using an ultrasonic bath (2) operated at 42 kHz and 70 W. Then, 1 g of glass fibers was immersed in the MWCNT/water mixture (3) and such mixture was further agitated in an ultrasonic bath for 3 h (4), reaching temperatures $\sim 60^\circ\text{C}$. Finally, the glass fibers with deposited MWCNTs were removed from the closed container (5) and dried in a convection oven at 100°C overnight (6). Two groups of fibers were produced in this way as identified in Table 1. Fibers labeled CC-MW used as-received glass fibers (CC) as a substrate for MWCNT deposition while fibers labeled CR-MW used fibers whose coating was removed (CR). Notice that the deposition method used in this work utilizes water (to avoid dissolution of the fiber sizing), and maintains the glass fibers under ultrasonic dispersion into

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