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Nanoscale structure and local mechanical properties of fiber-reinforced composites containing MWCNT-grafted hybrid glass fibers

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ABSTRACT

Carbon nanotubes (CNTs) were grown from the surface of glass fibers by chemical vapor deposition, and these hybrid fibers were individually dispersed in an epoxy matrix to investigate the local composite structure and properties near the fiber surface. High-resolution transmission electron microscopy revealed the influence of infiltration and curing of a liquid epoxy precursor on the morphology of the CNT "forest" region, or region of high CNT density near the fiber surface. Subsequent image analysis highlighted the importance of spatially dependent volume fractions of CNTs in the matrix as a function of distance from the fiber surface, and nanoindentation was used to probe local mechanical properties in the CNT forest region, showing strong correlations between local stiffness and volume fraction. This work represents the first in situ measurements of local mechanical properties of the nano-structured matrix region in hybrid fiber-reinforced composites, providing a means of quantifying the reinforcement provided by the grafted nanofillers.

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1. Introduction

Fiber-reinforced plastics (FRPs) have taken a central role in engineering design for the last 20–30 years, due to their ease of processing, tailorable properties, and weight savings compared to metal alloys [1]. While these composite materials have remarkable properties along the fiber directions, a common shortcoming of FRPs is failure in matrix-rich interlaminar regions where stress transfer between load-bearing fibers is less efficient and neat polymer properties dominate [2]. Even though composites often incorporate approximately 60 volume percent fibers, local matrix-rich regions persist and are of critical concern in commercial applications where failure can cause catastrophic damage. Hybrid composites, which differ from traditional FRPs by the incorporation of nanofillers into the matrix phase, exhibit multi-scale reinforcement leading to increased stiffness, strength and toughness and are often imbued with multifunctionality in the form of enhanced electrical and thermal conductivity or barrier properties depending on the choice of nanoparticle reinforcement [3,4].

Multi-scale composites [5] can be classified into two types of systems, as shown in Fig. 1. One system entails independent dispersion of the nanofillers throughout the polymer matrix, resulting in a "mixed inclusion" system. Much research has focused on mixed inclusion systems through the incorporation of various types of nanoparticles [6–10], rubber particles or elastomeric block copolymers [11,12], or combinations of the above [13–16] into the matrix. Alternatively, nanofillers can be chemically linked to the fibers, often by growing carbon nanotubes (CNTs) or carbon nanofibers (CNFs) from the fiber surface by chemical vapor deposition prior to infiltration by the polymer matrix, resulting in a hybrid-fiber composite system [17]. Alternatively, catalyzing fiber surfaces and directly attaching loose CNTs on CNFs to the outer surface of parent fibers can achieve the same hybrid-fiber effect [18–20]. The region of CNTs or CNFs on hybrid fibers has been referred to as the "forest" region due to the dense packing, preferential alignment, and similar lengths of the nanoparticles [21–23]. Upon infiltration and curing of a polymer resin to form a hybrid-fiber composite system, the forest region can act as an anchor to further strengthen the fiber–matrix interface and more efficiently transfer stress, as determined by single-fiber pull-out experiments [24–26] for similar hybrid-fiber systems.

Both types of hybrid composite system demonstrate multi-scale reinforcing phases spanning approximately three orders of magnitude in size, yet most characterization techniques investigate macroscopic properties, thereby obfuscating the influence of each reinforcing phase. In order to independently probe the influence of the nanoscale phase, some studies have investigated nanocomposite

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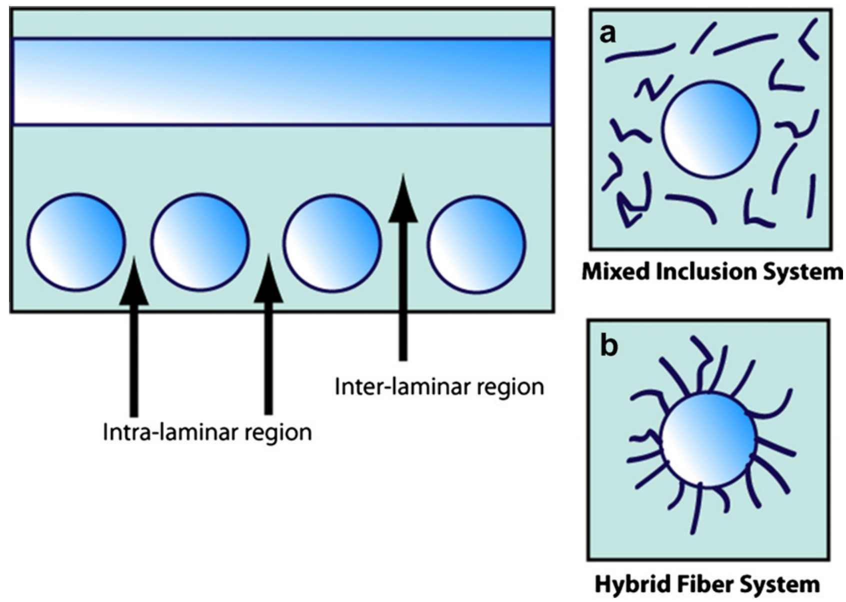

Fig. 1. Schematic illustration of the two types of multi-scale fiber-reinforced composite systems: (a) mixed inclusion system, showing micro- and nano-scale fibers independently dispersed through the polymer matrix, and (b) hybrid-fiber system, showing a traditional fiber with grafted nanoparticles embedded in the polymer matrix.

systems containing a polymer matrix and nanofiller and drawn comparisons to analogous mixed inclusion systems containing traditional fibers [27,28]. However, such studies typically have limited analysis of the interactions between the different reinforcing phases and their individual contributions to the mixed inclusion composite system properties. Other recent work has investigated model hybrid fiber systems by growing CNT or CNF forests from flat substrates followed by subsequent infiltration and curing of an epoxy precursor [29]. While these studies shed valuable insight into the behavior of hybrid-fiber composites, the morphology of CNTs or CNFs grown from a curved substrate such as carbon or glass fibers can deviate significantly from that of an ideal forest [30] depending on factors such as nanoparticle geometry, concentration, alignment, and waviness, particularly after infiltration of a polymer resin. The in situ morphology of the forest region in a hybrid-fiber composite system, the central focus of our study, plays a key role in determining local strength and stiffness enhancements of the matrix by the nanofiller.

In this paper, we use a combination of high-resolution transmission electron microscopy and nanoindentation techniques to explore, in situ, the nanostructured morphology and local mechanical properties of the forest region in a hybrid-fiber composite system containing glass fibers with grafted multi-walled carbon nanotubes (MWCNTs) embedded in an epoxy matrix. Although the waviness of CNTs in composites has been studied either through experiments on model systems [31] or through modeling [32,33], no studies to date have been dedicated to investigating the influence of in situ morphology of the nanoscale reinforcement in hybrid-fiber composites on local properties. This approach enables deeper understanding of nanoscale effects in multi-scale composite systems in order to ascertain the efficiency of the nanofiller reinforcement and to aid predictions of macroscopic behavior through modeling.

2. Materials and methods

Multi-walled carbon nanotubes (MWCNTs) were grown on glass fibers from a commercial source pre-coated with a nickel iron particle catalyst in a continuous process. Fibers were plasma pretreated and drawn through a chemical vapor deposition (CVD) chamber with flowing carbon-based forming gases and nitrogen at temperatures greater than 600 °C.

Prior to being cured in an epoxy matrix, Hybrid fibers were imaged using scanning electron microscopy (SEM) by use of a Hitachi S-3400N-II at an accelerating voltage of 5 kV and a working distance of ~6 mm.

Hybrid-fiber composites were prepared by suspending individual hybrid fibers in a DGEBA-based epoxy consisting of EPON 828 resin (≈52 wt%), a nadic methyl anhydride (NMA) hardener (≈46 wt%) and Ancamine K61B (≈2 wt%) as catalyst. The epoxy precursors were mixed by hand and degassed under vacuum until all volatiles were released. The mixture was transferred to an open mold with the hybrid fibers and cured for 60 min at 70 °C and 80 min at 150 °C under vacuum. The cured plaque was sectioned perpendicular to the fibers by a Struers Accutom 5 precision saw with a diamond-coated blade, and embedded in a room temperature cure epoxy medium. Serial polishing of each specimen was carried out perpendicular to the fiber direction using a Buhler AutoPolisher down to a final step with a polishing cloth and a 0.05 μm colloidal silica suspension to obtain a flat mirror-like surface.

Mechanical properties were carried out on polished surfaces via quasi-static nanoindentation on a Hysitron Triboindenter with a diamond Berkovick tip, which resolves an elastic modulus from Herzian contact principles and purely elastic unloading [34,35]. Care was taken to space indentations sufficiently far away from the fiber surface (at least 1 μm) to ensure that the experimental data was not influenced by the glass fiber, as discussed in the Supporting Information.

High-resolution transmission electron microscopy (HRTEM) of ~90 nm thick specimens sectioned using an ultramicrotome was carried out on a JEOL JEM-2100F FEG FasTEM. A series of 34 HRTEM images (each with a ≈1-μm^2 field-of-view) spanning the forest region around a glass fiber were acquired and stitched together using an open source program, Hugin, to create a high-resolution image. This master image (10,400 pixels × 9600 pixels) allows for comparison of morphology to indentation data with sub-nanometer resolution (1 pixel = 0.48 nm^2). The resulting gray-scale image was converted to a binary image by noise filtering and a singular conversion threshold using MATLAB. Local volume fractions were

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