



Hybrid nanoreinforced carbon/epoxy composites for enhanced damage tolerance and fatigue life



Joel S. Fenner, Isaac M. Daniel*

Robert R. McCormick School of Engineering and Applied Science, 2137 Tech Drive, Evanston, IL 60208, United States

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ABSTRACT

Hybrid nano/microcomposites with a nanoparticle reinforced matrix were developed, manufactured, and tested showing significant enhancements in damage tolerance properties. A woven carbon fiber reinforced polymer composite, with the polymer (epoxy) matrix reinforced with well dispersed carbon nanotubes, was produced using dispersant-and-sonication based methods and a wet lay-up process. Various interlaminar damage tolerance properties of this composite, including static strength, fracture toughness, fatigue life, and crack growth rates were examined experimentally and compared with similarly-processed reference material produced without nanoreinforcement. Significant improvements were obtained in interlaminar shear strength (20%), fracture toughness (180%), shear fatigue life (order of magnitude), and fatigue crack growth rate (factor of 2). Observations by scanning electron microscopy of failed specimens showed significant differences in fracture surface morphology between the two materials, related to the differences in properties and providing context for understanding of the enhancement mechanisms.

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1. Introduction

Composite materials reinforced by nanoparticles (nanocomposites) are a prominent topic of recent and ongoing composites research. The improvement of material properties that can be obtained from relatively small quantities of nanoparticles has been a strong motivation for further exploration, leading to optimistic predictions of greatly enhanced mechanical properties across the board for such materials. In general, these broad predictions have often failed to materialize, and there is some disparity in the results obtained to date.

Various studies involving nanoparticle enhancement have shown specific increases in static strength [1–5], fracture toughness [4,6–10], and fatigue life [11–15], among other properties, confirming the notion that nanoparticles can have impressive benefits at low concentrations. The resulting enhancements can vary widely depending on the type of property under investigation, the choice of materials, nanoparticle type, concentration and dispersion, and processing methods. Static strength improvement can vary, for instance, from the results of Bekyarova et al. [2] with a 20% increase in tensile strength of a carbon fiber/CNT composite, to those of Iwahori et al. [1] where increased tensile and

compressive strengths from 2% to 10% came at the expense of stiffness reduction. Other properties more indicative of structural survivability, such as fatigue life, can vary from results by Manjunatha et al. [14] with an almost order-of-magnitude improvement through the addition of silica and elastomeric nanoparticles to a glass fiber composite (at rather high concentrations near 10%), to those of Grimmer and Dharan [12] with an improvement of 2.5 times in a similar composite with only 1% CNTs.

Improvements in damage tolerance properties are expected from the introduction of nanoparticles due to additional energy absorbing mechanisms that arise [7]. Addition of carbon nanotubes, for example, provides an additional source of energy absorption through processes such as frictional nanotube pullout, nanotube fracture and microcrack bridging (Fig. 1). This behavior on the nanoscale is reflected on the macro-scale through increased fracture toughness, improved impact damage tolerance, higher residual compressive strength, and extended fatigue life.

Despite these anticipated and attractive enhancements, the manufacture of composites containing nanoparticles remains a challenge, as it is often difficult to produce quantities of material with a good dispersion of nanoparticles [16,17], a reasonable volume fraction, and a small void content [7]. Small changes in processing or materials can cause wide variation in final material properties, exacerbating the debate over the overall effect of nanoparticles on bulk material properties [7,18–24].

* Corresponding author. Tel.: +1 847 491 5649; fax: +1 847 491 5227.

E-mail address: imdaniel@northwestern.edu (I.M. Daniel).

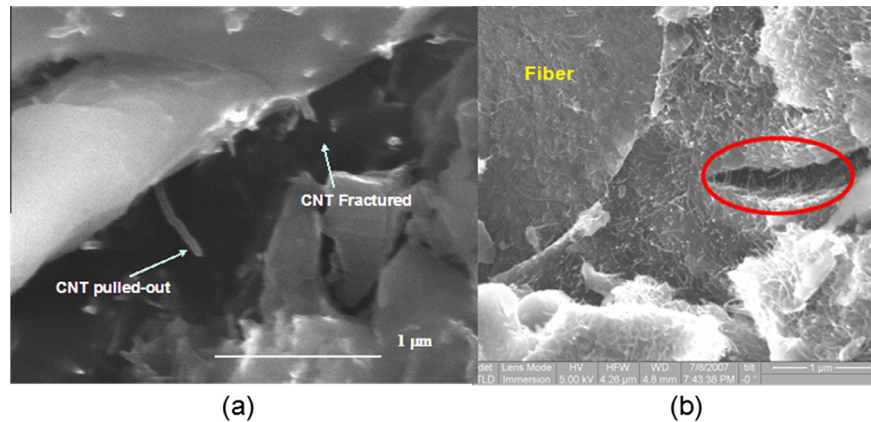


Fig. 1. Mechanisms of energy absorption in hybrid nano/microcomposites (a) nanotube pullout and fracture and (b) microcrack bridging by nanotubes [3].

Evaluation and quantification of the effects of nanoparticles is not always a consistent and definitive proposition. For example, improvement in basic strength (tensile, compressive, or shear), though relevant, is often quantitatively modest, and hence is not always a good measure of enhancement and should not be examined alone [1]. Fracture toughness is a more meaningful parameter to consider, as it describes more directly the ability of the material to tolerate sudden damage or absorb energy during destructive processes, and hence improve the survivability of the material in service [25]. Fatigue life, and related fatigue-fracture crack growth rate, are material properties associated with more long-term or progressive failure processes, and hence describe the ability of a material to survive in service over long periods [26].

Given these considerations, this study focuses on the development, processing, and testing of hybrid multi-scale nano/microcomposite materials to demonstrate and evaluate the enhancements in damage tolerance and energy absorbing properties. The work describes a comparative study with one basic material – a typical woven carbon/epoxy composite – and examines the effect on final properties of the introduction of short multi-walled carbon nanotubes into the matrix during manufacture. A discussion is given, with plausible explanations of the observed results.

2. Material processing

The material investigated was a carbon fabric/epoxy composite with the matrix reinforced by multi-walled carbon nanotubes (CNTs). The major mechanical reinforcement was provided by a 5-harness satin weave carbon fabric preform (AS4 fibers, 6 k tows, Hexcel AGP370-5H). The matrix was a Bisphenol-A epoxy resin (DGEBA, Huntsman GY 6010) cured with an anhydride hardener (methyltetraphthalic anhydride, Huntsman HY 917) and an additional amine accelerator (1-Methylimidazole, Huntsman DY 070). The nominal mixture ratio was 100:90:1 (resin:hardener:accelerator) by weight. This particular epoxy resin chemistry was chosen for its ability to tolerate mild temperature excursions (~60 °C), prior to introduction of the accelerator, without appreciable shortening of pot life, allowing greater flexibility in processing. The nanotube reinforcement was provided by short multi-wall carbon nanotubes (Helix Material Solutions) of 1–2 μm length and 10–30 nm outer diameter. A polyester block copolymer supplied in solution with a weakly volatile organic solvent (Disperbyk-2150, BYK Chemie) was used to facilitate dispersion of the CNTs. This particular dispersant was chosen based on prior work that demonstrated its effectiveness in dispersing carbon nanotubes (Fig. 2) [27]. A nanotube particle loading of 0.5 wt% of the epoxy resin

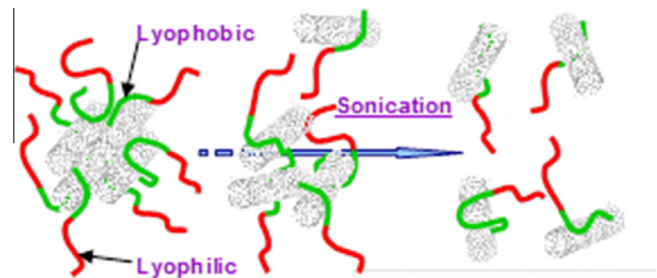


Fig. 2. Enhancement of nanotube dispersion with copolymer dispersant [27]. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

was chosen based on earlier evidence suggesting a near optimum enhancement of matrix dominated properties (compressive strength, interlaminar shear strength) for this material system (Fig. 3) [27].

A weighed amount of DGEBA was combined with 0.5 wt% of nanotubes and 0.5 wt% of copolymer solution. The materials were mixed together thoroughly, and then vacuum degassed at an elevated temperature (80 °C) to remove the organic solvent. The anhydride hardener was added and mixed thoroughly, followed by further vacuum degassing. The resulting mixture was then sonicated (90 W at 20 kHz for 30 min) to disperse the nanotubes. Just prior to composite infusion, the amine accelerator was added to spur the polymerization reaction, followed by a final degassing stage. Infusion of the resin mixture into the carbon fiber preform was carried out layer by layer in a wet layup process. The wetted preform stack was also subjected to final degassing and then placed into a mold for curing.

The construction of the mold (Fig. 4) allowed for careful control of finished laminate thickness and controlled removal of excess resin during curing. This approach was developed and adapted to avoid previously encountered problems related to a marked increase in resin viscosity due to the presence of nanotubes and a filtration effect on nanotubes encountered in VARTM processing. Elevated temperature curing was carried out in a two-step cycle: 30 min at 80 °C, 60 min at 150 °C, with heating at a rate of 2 °C/min. The same procedure was used for the reference (without CNTs) and nano-reinforced (hybrid) composites. After curing, composite plates were rough cut into specimens by means of diamond-abrasive cutting wheels, and wet-polished with SiC abrasive papers to final dimensions and smoothness.

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