Structural Health Monitoring using Carbon Nanotube (CNT) Enhanced Composites

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ABSTRACT

Carbon nanotubes (CNTs) are being investigated as a means for large-scale inspection of composite structures. Several studies have shown that CNTs possess exceptional mechanical stiffness and strength, as well as excellent electrical conductivity and piezoresistivity. Thus, it has been proposed that CNTs could be used not only to reinforce composite structures for improved impact and delamination resistance, but also to enable structures with multifunctional capability. The present investigators have developed advanced fiber-reinforced polymer-matrix laminates with *aligned* CNTs grown *in-situ* coupled with a non-invasive sensing method. CNTs have the potential to eliminate problems observed with traditional composite inspection methods that use resistivity due to increased and more reproducible conductivity properties, and offering 3-D electrical continuity to the surface. Results are presented for both through-thickness and in-plane resistance, measured using patterned silver-ink electrodes. Lastly, a discussion is presented for the application of guided-wave approaches exploiting the piezoresistive properties of the composites with embedded CNTs.

INTRODUCTION

Advanced composite materials are being adopted increasingly in aerospace structure design due to their superior specific stiffness and strength, as well as their resistance to fatigue, corrosion and ability to greatly reduce part count. Composites present additional challenges for inspection however, due to their heterogeneity and anisotropy, and the fact that often damage occurs beneath their surface. Currently

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successful laboratory non-destructive testing and monitoring methods are impractical for in-service inspection of large-area integrated composite structures due to the size and complexity of the support equipment required. In addition, many components that need frequent monitoring typically reside in limited access areas that would require breaking of factory seals and calibrations to manually inspect. It is clear that new approaches for inspection are necessary. Structural Health Monitoring (SHM) implies the incorporation of a non-destructive evaluation system into a structure to provide continuous remote monitoring for damage. SHM has the overall goal of improving vehicle safety and reliability while reducing maintenance and inspection-based life-cycle costs.

During this research, the present researchers aim to resolve these issues by using carbon nanotubes (CNTs). Several studies have shown that CNTs possess exceptional mechanical stiffness (as high as ~1 TPa) and strength, as well as excellent electrical conductivity (~1000x copper) and piezoresistivity (resistivity change with mechanical strain) [1-7]. Thus, in principle, they could be used not only to reinforce composite structures for improved impact and delamination resistance, but also to enable multifunctional structures with SHM capability, which would be very desirable. However, significant challenges remain to be addressed before CNTs can be incorporated into composites to realize this dual role.

In the use of CNTs purely to enhance mechanical properties, technical and manufacturing issues have hindered the development of large-scale CNT-enhanced composite structures [8, 9]. Alignment, dispersion and adhesion of CNTs in polymer matrices are vital for structural composite applications, and numerous studies have reported on the difficulties in dispersing CNTs in polymers [4, 8, 9]. These difficulties are further exacerbated when CNT-enhanced matrices are introduced into typical aligned-fiber advanced composites, where the CNT-containing matrix must effectively impregnate a high volume fraction of advanced fibers. Due to issues such as agglomeration and poor dispersion, only marginal improvements in mechanical properties were observed for both nanocomposites, and hybrid composites (in the latter, advanced reinforcing fibers such as carbon are used, unlike in the former), when CNTs are introduced into the bulk matrix [10, 11]. Somewhat better results were achieved using nanoscale modification of the interface between composite plies, by growing CNTs on the surface of cloth or placing unaligned CNTs at low volume fractions on fibers (including at the ply interface) [12-14]. However, these approaches do not significantly improve electrical conductivity and thus cannot exploit the potential multi-functionality of the embedded CNTs. Electrically-conductive CNT hybrid composites have been designed by some research groups, but most have significant repeatability problems due to factors such as low volume-fraction and poor dispersion [15, 16]. Thostenson and Chou built CNT-enhanced advanced fiber-reinforced composites with improvements in electrical conductivity and had encouraging results in damage monitoring using passive in-plane electrical resistance measurements [17]. However, their approach to incorporating CNTs in the fiber-reinforced composite results in random CNT morphology, resulting in no significant improvement in mechanical properties such as stiffness and strength. Also, their proposed approach to SHM used only *in-plane* resistance measurement; while this is feasible for coupon-level testing, their approach would require a dense network of invasive wiring and instrumentation for large-area monitoring. Thus, a novel approach to incorporating CNTs in fiber-reinforced aerospace structural composites for SHM is needed, which can fully exploit all the potential advantages of CNTs for improving mechanical properties as well as enable SHM with a non-invasive electrode network.

To address these requirements, the present investigators have developed advanced multifunctional fiber-reinforced laminates with *aligned* CNTs grown in-situ [18] using a non-invasive electrode network for SHM. Laminates developed through MIT's Nano-Engineered Composite aerospace Structures (NECST) Consortium have already been demonstrated to have as high as 69% higher interlaminar shear strength and much greater electrical conductivity (10^8 x through-thickness & 10^6 x in-plane) compared to similar laminates without CNTs [19]. The Laminates are a three-part hybrid system, as shown in Figure 1: advanced fibers (diameter of order microns) organized in tows and woven, a thermoset polymer resin, and dense aligned CNTs (with mass fraction between 0.5 and 2.5%) organized within the polymer matrix. As illustrated in Figure 1, CNTs are organized radially around the existing micron-sized fibers to form "fuzzy fibers" (FF), and the polymeric matrix binds all the filaments (advanced fibers and CNTs) together to form a fuzzy fiber reinforced plastic (FFRP). The alignment and dispersion of CNTs within the dense array of woven tows and fibers in the cloth material is achieved by radial *in situ* growth of CNTs from the surface of the woven fibers. The CNTs reinforce the polymer matrix between the advanced fibers to provide enhanced strength and toughness, as well as an electrically conductive pathway, as illustrated in Figure 1.

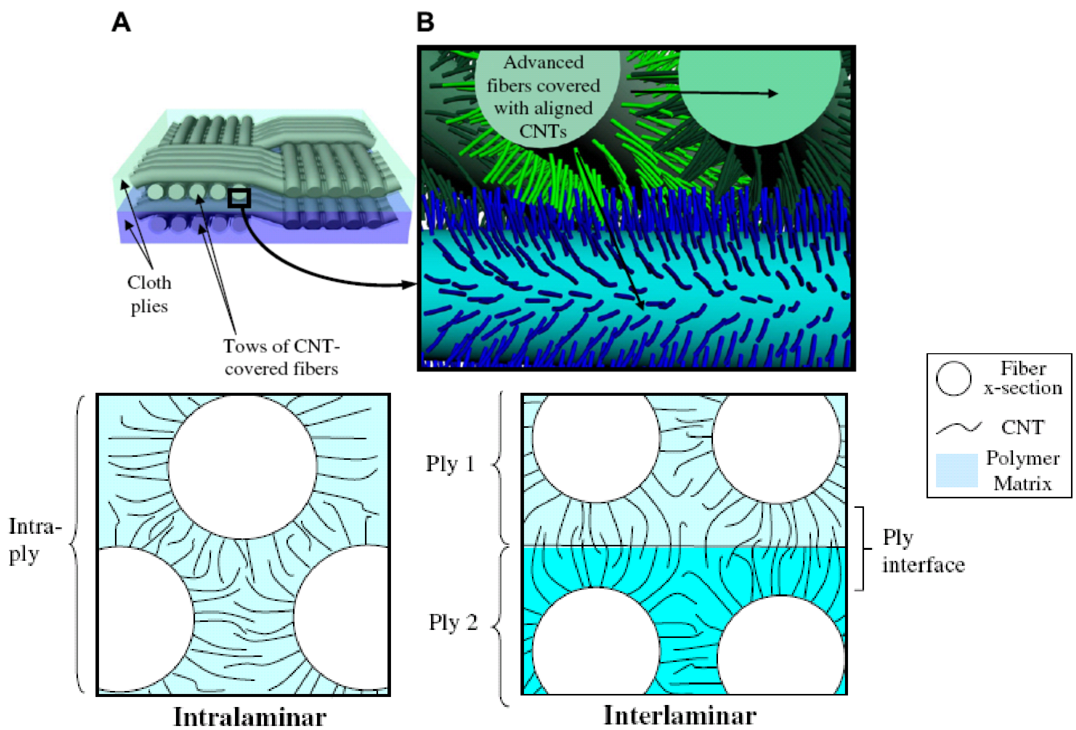

Figure 1: Illustration of fuzzy fiber reinforced plastic (FFRP) developed at MIT NECST: (A) Schematic of architecture composed of cloth containing fiber tows covered by CNTs, in polymer matrix. (B) Closer view of interface cross-section between 2 plies. CNTs grown on surface of each fiber interact with CNTs of nearby fibers achieving intralaminar (lower left) and interlaminar (lower right) reinforcement. Dimensions roughly to-scale except CNT diameter & volume fraction [19].

Resistive sensing methods—essentially putting a voltage across individual conductive fiber bundles and monitoring the change in resistance—have been investigated by previous researchers [17]. However, these methods have been hindered by several factors, including the fact that conventional carbon fibers have significant conductivity variability, changes have been small, and interconnection issues (*i.e.,* getting the measurement from the fibers without a free edge). This paper presents an experimental application of CNTs to potentially eliminate both problems with increased and more reproducible conductivity properties, and offering electrical break-out connections through 3-D leads brought to the surface.

EXPERIMENTAL APPROACH

The present investigators leveraged prior experience to instrument nano-engineered laminates from MIT NECST with a non-invasive silver-ink electrode grid and multiplexing micro-switches connected to compact hardware for *through-thickness* resistance measurement. The painted electrode grid, inspired by flat panel liquid crystal display (LCD) technology, use an "active" layer of electrode columns on one surface of the laminate as positive electrode, and on the other surface, another layer of electrode rows will act as "passive" ground (see Figure 2). Thus, by selecting a particular row and column, local through-thickness resistance measurements can be obtained for a grid of points over the structure. Furthermore, in-plane surface resistivity changes at a grid point can be obtained by probing resistance between 2 adjacent traces pairs closest to the point on either surface.

As one would intuitively expect, damage in the form of delaminations or cracks will affect the CNT link network around the affected zone in the structure, and correspondingly the local through-thickness (and in-plane) resistivity. Owing to the presence of the electrode network, by interpolating from the measurements at distinct grid points, high-resolution resistance maps over large structural areas can be obtained, allowing accurate localization of damage. The fact that this approach uses surface grids of non-invasive electrode networks for monitoring is the key factor that allows for easy scaling for larger structures, thereby keeping the mass and space penalty associated with the SHM system low, which is crucial in aerospace structures. However, static mechanical load and hysteresis effects could also affect resistance measurements, therefore results must be carefully examined to distinguish damage from these factors [17].

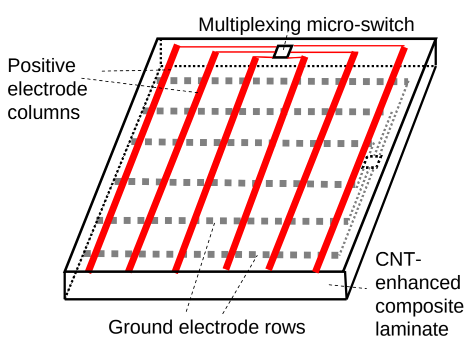

Figure 2: Proposed non-invasive silver ink electrode and future multiplexing switch network for resistance measurements over the CNT-enhanced composite laminate.

Specimen Preparation

The first step to manufacture FFRP laminates involves growing CNTs directly on fibers in an alumina fiber woven cloth using a modified thermal chemical vapor deposition (CVD) method developed in previous work [20]. A thick alumina fiber satin-weave cloth is first dipped in a solution of 50mM iron nitrate in isopropanol for 5 minutes and dried in air for at least 8 hours. This catalyst application method allows the catalyst to coat fibers inside the tows of the ply, ultimately distributing CNT growth to all areas of the woven ply. In a CVD furnace, the catalyst is conditioned to form nanoparticles, or seeds, from which CNTs grow radially aligned and perpendicular to the surface of the fibers. They extend into the matrix to provide reinforcement both within each ply (intralaminar) and between plies (interlaminar). The CNTs grown are typically 20 to 50 μm in length, longer than interlaminar spacing (~ 10 μm) and intralaminar spacing (~ 1-5 μm) [21].

Both FFRP and baseline laminates with no CNTs are fabricated using hand layup. For this sample set, the matrix system used is West Systems™ Epoxy, Resin 105 and Hardener 206. In hand layup, a ply is laid down on a sheet of non-porous teflon (GNPT) and epoxy is coated over the surface. The epoxy is wicked into the interior of the woven ply within a few seconds after which another ply is placed on top. In this work, once two plies are stacked, porous Teflon (PT), absorbent bleeder paper, and GNPT are placed over the laminate. A caul plate and vacuum are then used to provide pressure to the assembly to ensure uniform thickness. The sample is then left to cure for 12 hours at room temperature. The resulting specimens have less than 2% void fraction and a fiber volume fraction of ~50%. The resulting composites are trimmed with a diamond-grit wet cutting wheel [21].

A complete test matrix of 3 baseline and 8 FFRP specimens has been planned, however to date only 1 of each has been tested: 1 FFRP (114 x 25 x 3 mm) and 1 baseline alumina laminate (114 x 25 x 2 mm). Silver-ink electrodes were painted onto the specimen surfaces using simple masks. On the top plate surface, 14 parallel conductors were patterned in the short-dimension. On the opposite surface, 4 parallel conductors were patterned in the long dimension, so that they were perpendicular to the top side conductors when projected virtually through the thickness. These traces were all 1.5 mm wide and spaced 3 mm edge-to-edge.

Experimental Procedure

Prior to testing, the electrical resistance of each specimen was measured using a Fluke™ 189 model multimeter with resolution 0.01 Ω and accuracy of ±0.05%. Initial data showed significant variability when probing directly on the silver traces. Subsequently, as a first trial before implementing a multiplexing chip for trace switching, a procedure was developed to bond thick gauge wires to the traces using silver epoxy, resulting in < 1% change (< 0.1 Ω for ~10 Ω trace) across 10 repeat trials. Each of the specimens was centrally impacted with a 13 mm diameter steel ball at 75 ft-lbs using a guided dropped weight, calibrated to just initiate surface micro-cracking. Following impact, post-damage resistance measurements were collected again for each possible combination. Photographs of each specimen were also taken before and after impact under microscope in order to better document the true physical effect of the impact event on each of the specimens.

RESULTS

Following the impact, no damage was visible to the unaided eye for either specimen. Microscopy however revealed cracking on the back surface (opposite from the impact surface) of both the baseline and FFRP laminates, as seen in Figure 3 for the FFRP specimen. The original planned experiment had called for an analog multiplexer to switch electrode pairs for ease of measurement, however due to time constrains, the previously described instrumentation process was implemented for the presented results. Two sets of data were collected: 1) in-plane between each parallel pair of adjacent traces, and 2) through-thickness at each grid point created by the virtual intersection of the perpendicular top and bottom surface traces.

First, for the baseline laminate, all measured values for both in-plane pairs and through-thickness were higher than the range of the multimeter (5 MΩ) before and after impact. It was clearly evident that without the CNT enhancements, there was no suitable conductive path to form a closed circuit.

Next, data was collected for the in-plane resistance on the FFRP. For the short traces the average resistance was 9 Ω pre-impact. Following impact, while the outermost trace pairs on either side of the damage site along the x-axis showed $< 10\%$ change, the middle was consistently $> 100\%$, as seen in Figure 4(a). Minor changes $< 1\%$ were detected between long trace pairs. Finally, electrical resistance data was collected for the 56 through-thickness grid points, which averaged 20 Ω. As seen in Figure 4(b), a clear change of $> 100\%$ was detected in the impacted region. Along the x-axis, between the measurement points for the long traces (left edge in Figure 4(b)) and the damage site, $< 10\%$ change was observed. However on the opposite side of the damage site (right side of Figure 4(b)), a constant resistance offset was introduced due to the presence of cracks across the electrodes.

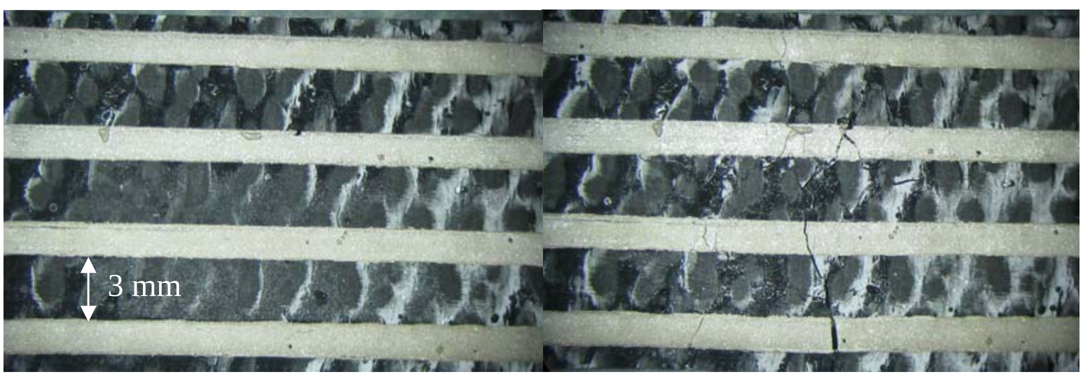

Figure 3: Before (left) and after (right) photographs of FFRP specimen impacted on opposite side

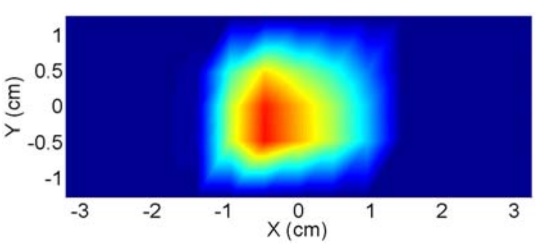

Figure 4(a): In-plane resistance % change image generated by multiplying & interpolating top/bottom adjacent parallel trace measurements

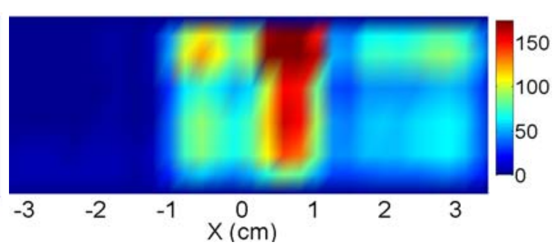

Figure 4(b): Through-thickness resistance % change image generated by interpolating grid point measurements

GUIDED-WAVE APPLICATIONS

Beyond resistivity, guided-wave (GW) approaches have been considered as a possible solution for CNT-based SHM, exploiting the piezoresistivite property of the embedded CNTs. If instrumented with a surface-bonded piezoelectric actuator for high frequency GW excitation, the response of the laminate could be captured through the local CNT piezoresistive response captured over an electrode grid using a similar resistance measurement setup with higher frequency acquisition system. This approach will enable full-field visualization of the GW as it propagates. This may allow easy detection and location of damage, which would scatter the GW field. In addition, since these use data at high frequency (>40 kHz), they are much less susceptible to static, structural dynamic operating or acoustic loads, the effects of which can be filtered out in the frequency domain. Similar efforts using piezos for GW excitation and laser vibrometers for sensing the GW field elsewhere have been successful in effective damage location [22]. However, with laser vibrometers being much bulkier and requiring some standoff distance, these are more suited to offline nondestructive evaluation than onboard SHM. In the method here, the multifunctional nature of the CNT-enhanced hybrid composite structure would be harnessed to sense this GW field for SHM without additional sensors.

CONCLUDING REMARKS

CNT-enhanced composites were fabricated, patterned with a silver ink electrode grid, and subsequently subjected to impact damage. Both in-plane and through-thickness electrical resistance measurements were collected. Clear changes were observed in both sets of data for grid lines close to the impacted zone of the specimen, demonstrating that these parameters were sensitive to damage in the structure. The peak changes were close to the center of the specimen near the impact site, and there was little to no change in values at points away from the damage zone. Overall, the barely visible impact damage caused significant changes, allowing for a full-field representation of the damage locations by interpolating the collected data. This demonstrates the potential of using this approach as an SHM solution, with the added benefit of CNTs reinforcing the structure.

It is interesting to note that the result do not show peak changes exactly at the center of the impact target. This may indicate that the parameters are sensitive to different damage modes. For instance, in-plane resistivity may be more sensitive to surface cracks, which tend to form at the edge of the impact zone. Conversely, through-thickness resistivity may be more sensitive to delamination, which would affect the CNT links across the specimen plies. In Addition, minor changes in traces resistance directly due to the compression force of the impact event could possibly have affected some data. These issues will be investigated in subsequent research.

Near-term research will aim to further validate this method with additional specimens in a more comprehensive test matrix, as well as to observe the effects of multiple progressive impact events. Furthermore, pre and post-damage tensile tests will be conducted to evaluate residual strength and stiffness for baseline and FFRP laminates. Future work will leverage direct-write technology to create more accurate electrode grid formations. Proof-of-concept studies will also be performed to investigate the viability of the presented GW approach to CNT-enhanced SHM.

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