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A comparative study of a self strain-monitoring carbon nanotube film and carbon fibers under flexural loading by electrical resistance changes

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Abstract. With the increased use of composites for load-carrying structures, the ability to obtain strain and damage information is critical to maintain reliable structures in the field. A promising class of multifunctional composites with the ability to self-sense strain and/or damage is possible through the use of carbon fibers and carbon nanotubes. This paper presents a comparative study of two sensors for fiber-reinforced polymer composites: the sensors are the carbon fibers themselves, and a non-structural carbon nanotube (CNT) film applied through spray deposition. The changes in resistance of the sensors are compared under monotonic and cyclic flexural loading. Within the limits of this study and the current CNT sensor configuration, the carbon fibers are shown to have a higher sensitivity for strain and damage sensing. However, the CNT film appears to track the performance of GFRPs reasonably well for tensile strains.

1. Introduction

The application of fiber-reinforced polymer (FRP) composite materials has impacted a wide variety of engineering structures, due to their low weight and tailorability to support specific loading conditions. With composites becoming increasingly common, low-cost fabrication processes have been developed in industry. However, one remaining challenge is the ability to sense and monitor damage within a composites structure. This challenge is further complicated by several complex failure mechanisms, including fiber fracture, delamination, matrix cracking and interlaminar shear failures, e.g. [1]. Unlike isotropic materials such as metals and polymers, these failures can easily go unnoticed in inspections routines, as these failure modes may not be discernable to the naked eye.

In order to address this challenge, several techniques and strategies have emerged to monitor strain and damage within a composite structure. The method of interest in this paper is to utilize electrically conductive constituent materials, with potentially piezoresistive qualities, to yield composites with multifunctional capabilities. “Multifunctional” refers to structural and non-structural functionality, such as sensing/activation, energy harvesting, and electrical conductivity, [2]. For the case of flexural loadings, electromechanical coupling between strain and surface resistance measurements can be obtained for conductive materials. Traditionally, metal foil strain gages give localized strain information. However, some of their limitations are that they can only be applied to the surface of a composite, and can only cover a limited sensing area. The application of multifunctional composites could provide strain and/or damage sensing capabilities that are not limited to the elastic range, as a composite could be monitored from its elastic regime until rupture. Through the use of carbon fibers and carbon-based nanomaterials, a much larger sensing area could be incorporated within a structure to utilize this multifunctional capability.

Carbon fibers (CF) have been widely used in the aerospace industry since the 1970s, and are slowly emerging also in wind turbines, for rotor diameters larger than 45 meters. One of the first investigations of the electrical properties of CF was completed by Owston, [3], who showed that CF reinforcements have the unique property of being intrinsically conductive. Later, studies monitoring resistance changes during monotonic tension, [4, 5], and cyclic flexure, [6, 7], were performed to demonstrate these sensing capabilities.

Carbon-based nanomaterials, in particular carbon nanotubes (CNT), have been utilized in several composite architecture configurations to create electrically conductive composites. CNT have been used to monitor damage when they are dispersed into matrix systems, [8-10], deposited onto glass fibers [11], and layer by layer fabrication [12, 13]. In this paper, a CNT-based thin film is investigated as a sensor, [14], this time under flexure loading. Films like this have been validated for uniaxial monotonic and cyclic tensile piezoresistivity, [15]. One of the main advantages of this film, as compared to other CNT composite architectures, is the ease of processing and scalability, through which it can be applied to existing structures' surfaces.

Most structures and beams are not subjected to pure uniaxial loadings. A far more common load experienced by structures and beams is bending. The work in this paper will compare resistance changes of a CNT thin film to the demonstrated self-sensing ability of carbon fibers, for FRP specimens under flexural loading. Resistance changes of both the tension and compression side of a coupon are monitored simultaneously.

2. Materials and methods

2.1. Composite specimen preparation

A vacuum-assisted resin transfer method was utilized to fabricate carbon fiber-reinforced polymer (CFRP) coupons, using carbon unidirectional fibers (Torayca T700SC 12K, Soller Composites, NH, USA). The CF have an electrical resistivity of 1.6×10^{-3} Ω cm with a filament diameter of 7 μm. In order to investigate the sensing properties of the CNT films, glass fiber-reinforced polymer (GFRP) composites were also prepared, with unidirectional E-glass fibers (Vectorply E-LR 0908, AL, USA). A LAM125/LAM237 epoxy/hardener system (Pro-set, MI, USA) was infused to obtain the CFRP and GFRP coupons, using a 100:28 resin:hardener ratio. Before infusion, the epoxy mixture was degassed for 25 min, to remove air bubbles and reduce voids within the coupons. Both composite laminates were cured at room temperature for 14 hours, and then post-cured at 82 °C for an additional 8 hours in a convection oven. Individual coupons were cut to size with a water-cooled diamond saw, to nominal dimensions of 183 mm length × 25.4 mm width. The layup orientation was $[0]_{13}$ for the CFRP samples and $[0]_{10}$ for the GFRP samples, with the zero degrees plies aligned with the span of the samples. The number of layers for both fiber architectures was chosen to maintain a 40:1 support span to thickness ratio following "Procedure B" of ASTM D7264 [16].

2.2. CNT thin film fabrication

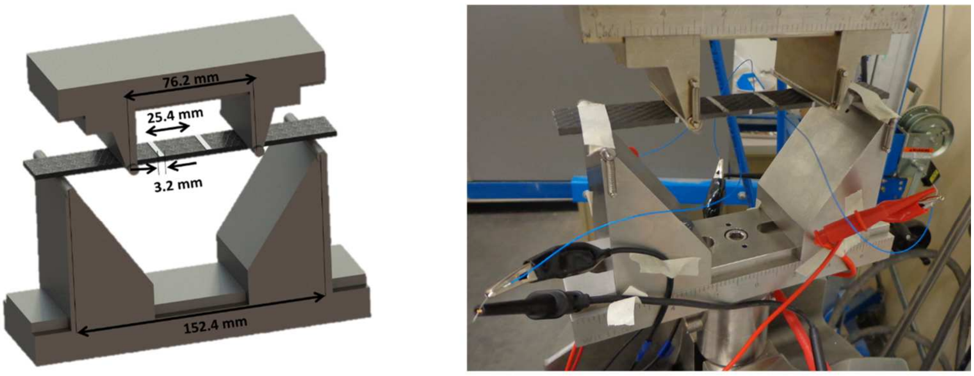
The carbon nanotube (CNT) film applied by spray deposition in this study has been successfully employed as a sensor in GFRP samples, [14]. The constituents of this film can be categorized into three components: ink, latex, and water solution. The ink consists of 1 wt.% of multiwall carbon nanotubes (MWCNT) (SMW 200, SouthWest NanoTechnologies), 2 wt.% of poly(sodium 4-styrenesulfonate) (PSS) (~1M M_W, Sigma-Aldrich) and a small amount of N-methyl-2-pyrrolidinone (NMP). MWCNTs form an electrically conductive network within the film. The PSS helps to decrease contact resistance among adjacent MWCNTs while maintaining dispersion of the nanotubes. In lieu of the MWCNT being functionalized through a chemical treatment, the NMP solvent is used to improve dispersability of the MWCNT in the solution. This ink solution is prepared by first adding the PSS into deionized water, then adding the MWCNT, and finally adding the NMP to help aid dispersion. Then, the MWNT/PSS/NMP mixture is sonicated for 60 min using an S-450D Branson tip sonicator operating at 225 W and 20 kHz. After the MWCNT are dispersed in the solvent solution, a poly(vinylidene fluoride) (PVDF) latex-based solution is added to the mixture, to promote adhesion with the FRP coupons. Finally, the MWCNT/PSS/NMP/PVDF mixture is sprayed on the coupons using an airbrush inside a fume hood, as seen in Figure 1. For CFRP samples only, an additional epoxy layer was applied to the cured sample before the spraying of the CNT sensor, in order to ensure insulation of the electrical properties between the CNT film and the conductive carbon fibers. The film was left to dry at room temperature for one hour. Since the coupons were to be subject to flexural loading, the CNT thin film was sprayed onto both sides of the coupons to monitor their tension and compression responses.

Figure 1. Spray deposition of CNT film onto glass fibers.

2.3. Test setup and instrumentation

An MTS 810 hydraulic test frame with a 244 kN load cell was used to apply force on a four-point bending fixture (WTF-FL-52 Wyoming Test Fixtures, USA), as shown in Figure 2a. A quarter point loading was utilized with a support span of 152.4 mm and loading span of 76.4 mm. Monotonic flexural loading was applied at a displacement rate of 1 mm/min. Cyclic flexural loading was applied at 0.01 Hz with a constant deflection amplitude of ±1.5 mm.

The instruments that measured the electrical resistance from the electrodes applied to the coupons (bottom and top) consisted of two digital multimeters (DMM) (Agilent 34401A and 34410A). An internal 4-wire setup was implemented to account for contact resistance between the electrodes and the sensors and the resistance of attached wires (see Figure 2b). Each DMM collected data for a single channel using Agilent's Benchvue data acquisition software. Each DMM was allocated for sensing one of the samples' sides (one DMM for tension, one for compression). Cross-head displacement and load *versus* time were collected from the MTS, while both DMMs collected resistance values *versus* time. Post-processing of the data via linear interpolation correlated stress, strain and fractional resistance changes, meant as $(R-R_o)/R_o$, where R_o is the baseline value.



a) b)

Figure 2. a) Sketch of 4-pt fixture, with support span, loading span, and electrode dimensions, b) instrumented CFRP sample.

2.4. Electrode application

In order to monitor changes in electrical resistance, electrodes were applied directly to both types of sensors (CF and CNT film). Surface-based measurements were utilized in order to separately monitor the strain and damage due to compression and tension loads at the edges of the samples' depth. For the CF sensor, the surface of the coupons was slightly sanded prior to application of the electrodes in order to remove the epoxy matrix and expose the carbon fibers. Electrically conductive silver paint (Ted Pella) was applied over the coupons to create a conductive path among the CF tows, while silver epoxy (MG Chemicals 8331G) was used to attach 30 AWG copper wires. For the CNT film sensor, silver epoxy alone was used to create a conductive path between the film and the copper wire electrodes.

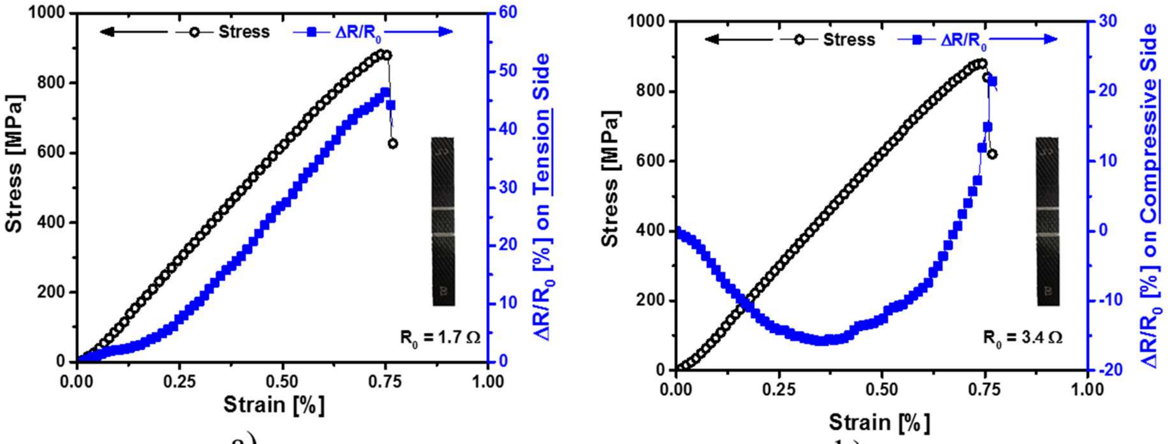
3. Results and discussion

3.1. Baseline resistance measurements

After instrumenting the samples with electrodes, baseline resistance values were recorded using a 4-wire measurement. Baseline resistance values of carbon fibers were on the order of ohms, while those of the CNT sensor were on the order of hundreds of ohms, for an electrode distance of 25.4 mm.

3.2 Monotonic flexural loading

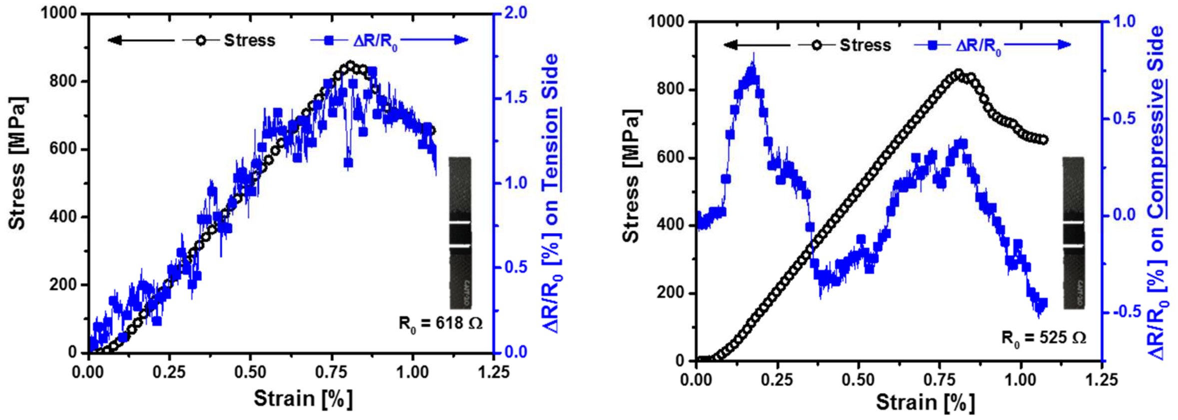
Stress and fractional resistance changes *versus* strain are shown in Figure 3 for a CFRP sample with CF sensor. Above and below the neutral axis of the samples are regions dominated by tensile and compressive strains. Surface resistance values are shown to increase with strain linearly up until failure on the specimen side with tensile strains, as seen in Figure 3a. At the failure point of the specimen, indicated by a sharp drop in the sample's stress, a corresponding drop in resistance can be seen in Figure 3a. Typically, fiber rupture will cause resistance to increase under tension [4]. However, under flexural loading, a CFRP composite fails in compression before tension. The side with compressive strains yields a nonlinear decrease in resistance values initially until approximately 0.3% strain, upon which the decreasing trend of resistance values changes directions. It is hypothesized that this transition, where resistance begins to increase, could be an early indicator of damage within the sample. The increase in fractional resistance in Figure 3b would indicate that the CFRP composite is capable to sense its own strain and damage.



a) b)

Figure 3. Monotonic flexural loading using CF as a sensor, in a CFRP sample a) under tension, b) under compression.

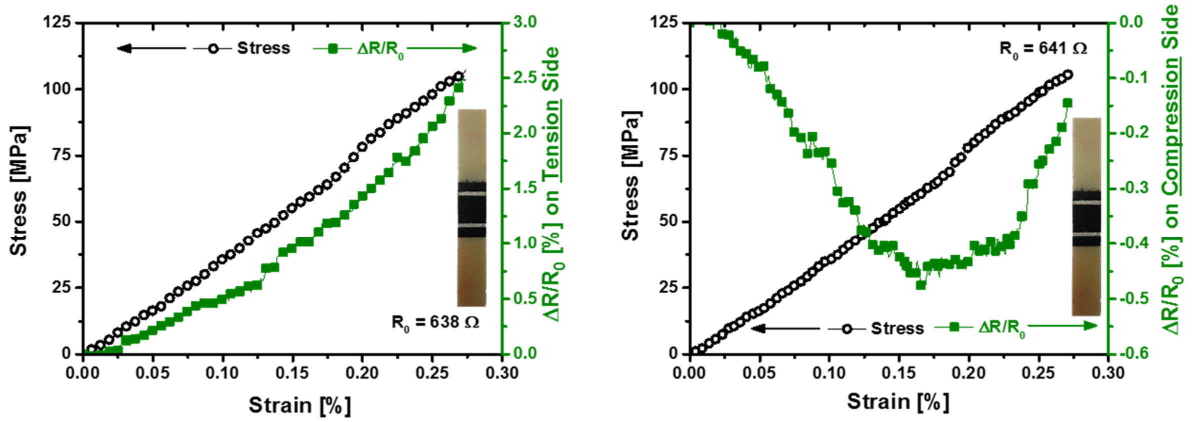
The gauge factor, *K*, i.e. the reversible change in fractional resistance changes per unit strain value, was classified as the region between 0.25% to 0.5% strain, for this study. For the few CFRP samples with CF sensors tested, *K* values were within reasonable agreement with the literature [17]. Figure 4 shows the response of the CNT sensor in one CFRP sample. There was noise in the tension side. The change in resistance was particularly inconsistent on the compressive side, among the samples. *K* was of the order of 1-3 under tensile strains, comparable to those of metal foil strain gauges, hence this sensor in its current configuration and CNT concentration exhibits a lower sensitivity to strain and damage with respect to the CF sensor.



a) b)

Figure 4. Monotonic flexural loading using the CNT film as a sensor, in a CFRP sample a) under tension, b) under compression.

To further study the sensing behavior of the CNT film under flexure, the film was also applied to GFRP samples. Figure 5 show similar trends on the tensile and compression side to trends found using CF as a sensor in CFRP specimens as seen in Figure 4. However, the maximum fractional change in resistance is found to be much lower. This results in the CNT film appearing less sensitive to strain induced from flexural loading. For the compressive side with the CNT film, again there is a transition point from decreasing to increasing resistance changes. In this case, the transition point is assumed not to be caused by damage within the specimen. However, it is possible that, at high strain levels, the film could be prone to damage, causing this transition in resistance changes.



a) b)

Figure 5. Monotonic flexural loading using a CNT film as a sensor, in a GFRP sample a) under tension, b) under compression.

3.3. Cyclic flexural loading

Cyclic flexural loading plots of displacement and fractional resistance changes *versus* time are shown for the CF (Figure 6) and the CNT sensor applied to GFRP (Figure 7). The samples are loaded for 5 cycles at three different displacement amplitudes. Resistance values before loading are shown at time *t* before zero, in order to indicate the amount of noise before mechanical loading.

The CF sensor under cyclic loading follows the same trends under monotonic loading for tensile and compressive strains. While the fractional resistance peaks decrease with loading cycles, the amplitude between peaks and troughs are consistent throughout all the cycles. At the third displacement amplitude of 9 mm, the fractional resistance changes no longer follow the trends found under monotonic loading. This may be attributed to samples' damage and/or the viscoelastic behavior of the resin.

For the CNT film sensor applied to GFRP samples, it can be seen that the resistance increases with loading and decreases while unloading. The amplitude of the fractional resistance changes indicates that there are no residuals within the film after repeated cycles. Comparing Figure 6 and Figure 7, the CNT films yields lower fractional resistance changes than the CF sensor. Under cyclic flexure, the CNT film applied to GFRP samples exhibits a response similar to that of Figure 7.

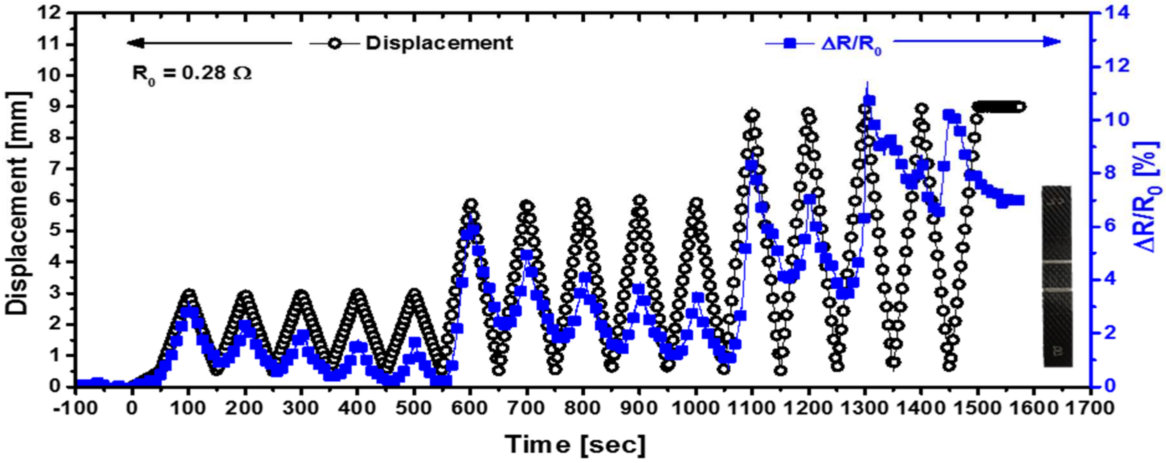


Figure 6. Cyclic flexural loading with increasing displacement amplitudes using CF as a sensor in a CFRP sample.

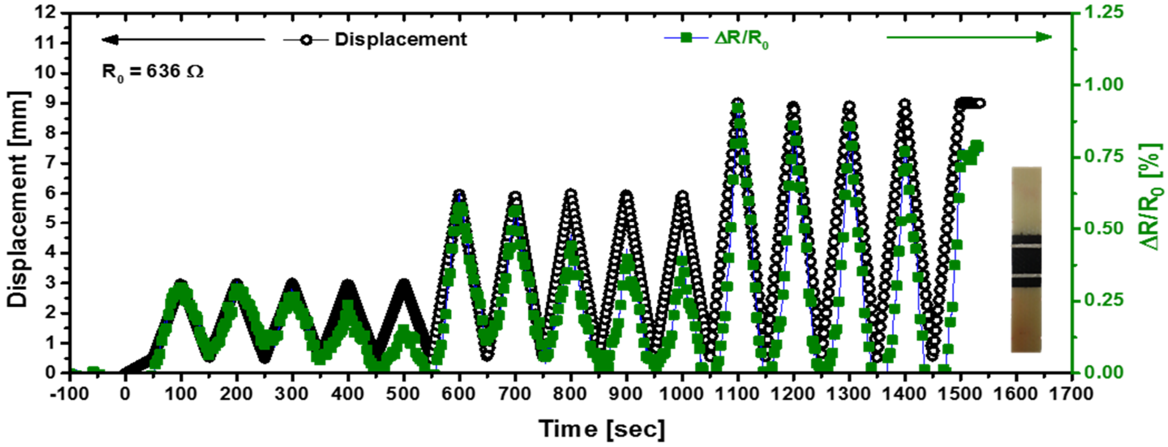


Figure 7. Cyclic flexural loading with increasing displacement amplitudes using a CNT film as a sensor in a GFRP sample.

4. Summary, conclusions and future work

CNT films and CF were investigated as resistance-based sensors under monotonic and cyclic flexural loading, with electrodes applied to the tensile and compressive surfaces in each sample. For the CNT films applied to GFRP samples and the CF in CFRP samples, resistance values increased with tensile strains and decreased with compressive strains. Under compressive strains, a transition point of decreasing-to-increasing resistance trends were found for both the CNT film on GFRP samples and the CF sensor. For CNT films applied to CFRP samples, the response exhibited a lot of scatter, and no straightforward, common trend was found among the tested samples. Overall, the CF exhibits a much higher sensitivity of resistance changes under both tensile and compressive strains. Furthermore, the CF multifunctional ability is shown to include both strain and damage sensing of the samples. However, the CNT film may allow the structural health of non-conductive GFRP to be monitored under flexure.

Future work will further investigate the performance of the CNT film in terms of its CNT concentration, to better tune the sensor. These structural health monitoring methods will also be applied to ~1.2 m long carbon/epoxy I-beams with different orientations in the flanges and web, for a proof of concept applied to an actual structure.

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