Damage monitoring of adhesively bonded composite-metal hybrid joints using carbon nanotube-based sensing layer

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ABSTRACT
Improving mechanical properties and decreasing costs have significantly increased the use of fiber composites in automotive, aerospace, and civil engineering applications. Structural composites are bonded to traditional metallic materials in a variety of applications, and mechanical fasteners often cannot be used due to the low bearing strength of composites. With the increasing use of adhesives in load-bearing structures, novel techniques are required for monitoring the structural integrity of adhesive joints. Previously, carbon nanotubes (CNTs) have been added to adhesives and resins to create in-situ sensors, but the increased viscosity and potential for galvanic corrosion remains a challenge. In this research, a piezoresistive carbon nanotube-based sensing layer is embedded in a composite/steel adhesive joint for damage sensing. The use of a thin sensing layer with low-fiber volume fraction enables the use of existing adhesives without causing any major changes in the physical properties of the adhesives or the curing cycle and reduces the chances of galvanic corrosion. Different approaches of using an adhesive layer and a nonconductive fabric are investigated for insulation of the sensing layer. The nonconductive fabric approach for insulating the specimen yields better mechanical properties as there are no weak interfaces in the adhesive bondline. Additionally, it is more convenient for scaling up for field applications as the adhesive is cured in one stage. The sensing layer can not only be used to detect incipient damage in the joint, but also identify different modes of failure.

1. Introduction
Advances in the processing science and cost reduction of fiber reinforced polymer composites have increased their use in a variety of fields such as automotive, aerospace and civil infrastructure. Increased use of composites in complex components combined with the manufacturing constraints due to intricate geometries requires joining different composite parts to themselves and, in many cases, to other conventional materials like aluminum and steel. Adhesive bonding plays a crucial role when fiber composites are used for strengthening of steel structures such as bridge girders [1] and the effectiveness of this strengthening approach depends on the integrity of the composite/steel bond.

The widely used methods for joining materials are mechanical fastening and adhesive bonding. Drilling holes to use fasteners reduce the cross-
sectional area of the structure and introduces local stress concentrations. In fiber composites, drilling holes could lead to damage to the continuous reinforcing fibers and can also cause edge delamination [2]. Furthermore, composites have low bearing strength, resulting in fastener-induced damage at low loads. Adhesive bonding provides a higher strength to weight ratio compared to mechanical fasteners [3, 4]. The joints are more durable because the stress is distributed across the bond area. Adhesive bonds have superior fatigue resistance, improved visual appearance and enhanced aerodynamic performance when compared to joints with fasteners.

While adhesive bonding offers many advantages, some of the key challenges when working with adhesives are the requirement of complex fixtures, difficulty in maintaining uniform thickness, elaborate surface preparation and environmental interaction factors such as moisture and temperature. A critical shortcoming of adhesive joints is their inability to be disassembled, which eliminates visual inspection of joint integrity. Therefore, many researchers have characterized mechanical properties and failure modes of adhesively bonded joints [5, 6]. Studies have also addressed the effect of different parameters such as adhesive selection, surface treatment, bonding pressure and adherend thickness [7, 8]. In addition to ongoing research for improving the quality of adhesively bonded joints, it is necessary to develop nondestructive testing methods for monitoring the condition of critical joints.

Nondestructive techniques such as acoustic emission can be utilized but the data analysis is complex and the interpretation of results is not quantitative. Ultrasonic inspection can be used for providing information on local damage but is difficult to use for providing real-time data [9, 10]. Fiber Bragg grating sensors have shown potential for monitoring the condition of adhesively bonded joints [11, 12], but embedding the sensors without creating defects is a challenge. An array of sensors is required to monitor a joint with a large surface area, which further increases the complexity.

Due to their extraordinary properties, carbon nanotubes are of particular interest in a variety of applications such as improving mechanical properties [13–15], self-sensing composites [16, 17], structural health monitoring [18–22] and flexible sensors [23, 24]. Thostenson et al. [25] added carbon nanotubes to an epoxy resin while manufacturing a composite to monitor the health of mechanically fastened composite joints and the characterization of the damage mode. The high aspect ratio (length/diameter) of carbon nanotubes allow creating electrically conductive composites at a very low volume fraction of carbon nanotubes [26, 27].

Lim and coworkers [28] introduced CNTs in epoxy adhesive for damage sensing of hybrid composite/steel joints. In order to monitor the fatigue performance of adhesive joints both Mactabi et al. [29] and Kang et al. [30] dispersed carbon nanotubes in adhesives. While the use of carbon nanotubes has given promising results in the laboratory setting, potential challenges such as galvanic corrosion, increased viscosity of adhesive and manufacturing challenges remain to be addressed. Ahmed et al. [20] used a piezoresistive sensing layer embedded in the adhesive between composite and steel for integrated strengthening and sensing methodology for steel structures. Insulation of the sensing layer is critical to avoid galvanic corrosion and electrical shorting. The sensing layer was isolated using a thin layer of adhesive which involved a complicated multi-stage curing process in fabricating the lap shear joint. This led to a reduction in the maximum shear stress of the specimens and could create manufacturing complexities when implemented at larger scales and in the field.

In this research, damage monitoring of a single-lap composite/steel joint is investigated using a carbon nanotube-based sensing layer. The sensing layer is manufactured by depositing carbon nanotubes on a nonwoven aramid fabric with randomly oriented fibers. The aramid fabric has low fiber volume fraction and acts as a carrier and creates a backbone for the carbon nanotubes to form an electrically conductive network which acts as a piezoresistive sensor. Single-lap joint configurations tested in tension are analyzed under monotonic tension and progressively increasing cyclic loads. To electrically isolate the sensor, an adhesive insulated approached, established in our prior work and a novel technique of using a nonconductive fabric for insulation of the
sensing layer is investigated, keeping in mind the scalability and manufacturing feasibility for the field application. The resistance data are analyzed to detect incipient damage and distinguish between different failure modes.

2. Experimental

In order to investigate the sensing response and the effect of the sensing layer on the bond strength, modified single lap shear joints were manufactured. The joints were manufactured using steel and carbon fiber composite substrates and the carbon nanotube sensing layer was embedded in the adhesive bondline. The sensing layer needs to be electrically isolated from the steel and carbon fiber composite to prevent sensor shorting and galvanic corrosion. Three types of specimens were manufactured: (1) Control specimens without sensing layer (control), (2) sensing layer insulated with a thin layer of adhesive (adhesive insulated) and (3) sensing layer insulated with a nonconductive fabric (fabric insulated). Figure 1 shows the schematic diagram of the specimens with the sensing layer insulated using different approaches.

2.1. Materials and manufacturing

A unidirectional carbon fiber composite was manufactured using T300 carbon fiber pre-impregnated with epoxy resin (Cytec Industries). Eight plies of the prepreg were assembled and cured in an autoclave at 200°C for 5 h under a pressure of 0.55 MPa (80 psi) to make a 30 cm x 30 cm laminate. The composite was then cut into 100 mm x 25 mm pieces. Low carbon, precision ground steel was machined on a milling machine to the same size as that of the composite specimens. A 2-part epoxy adhesive (Hysol 9309.3 NA, Henkel) was used for joining the steel and the carbon fiber composite.

The carbon nanotube sensors were fabricated by depositing carbon nanotubes onto a nonwoven aramid fabric (20601–50 g/m², Technical Fiber Products) with randomly oriented fibers, which acts as a carrier fabric for the nanotube sensing network. For the sensing layer, the carbon nanotubes were deposited using a commercially available carbon nanotube sizing (SIZICYL®—Nanocyl, Belgium) which was diluted with ultra-pure water at a mass ratio of 1:2 (sizing: ultra-pure water). To ensure uniform mixing, the sizing and ultra-pure water was mixed using a centrifugal mixer (THINKY® ARM-310) at 2000 rpm for 150 s followed by sonication for 30 min using an ultrasonic bath (Branson® 1510). The aramid fabric was dip coated for 20 min and then dried in a convection oven for 30 min at 150°C. Figure 2 shows the aramid fabric before and after coating with carbon nanotubes. The carbon nanotubes are uniformly deposited on all the fibers in the fabric as shown in the scanning electron micrograph in Figure 3, creating an electrically conductive network which enables piezoresistive sensing. For fabric insulated specimens, the sensing layer is isolated from the steel with a secondary nonconductive fabric using a lower areal density fabric of the same nonwoven aramid fibers (20601–17 g/m² Technical Fiber Products). When
embedded in the adhesive joints, the layer thicknesses of the aramid fabric are approximately 230 and 600 \( \mu m \) for the 17 g/m\(^2\) and 50 g/m\(^2\) aramid fabric, respectively.

### 2.2. Model joint system preparation

A modified version of the single-lap joint specimen in ASTM D5868 [31] was made using the carbon fiber composite and the steel. All specimens were manufactured using similar procedures and conditions, and because the adhesive layer thickness is an important factor in joint strength, a bondline thickness of 2 mm was maintained for all specimens to enable direct comparison. The hybrid fiber composite-metal joints were assembled using a carpenter’s vice along with custom machined spacers for maintaining a constant bondline thickness. The surface of the carbon fiber composite was prepared with silicon carbide sandpaper (320 grits). A sandblaster was used to abrade the steel to remove the oxide layer and roughen the surface, and both the steel and composite substrates were degreased with acetone. To ensure uniform mixing of the adhesive and the hardening agent, a centrifugal mixture was used at 2000 rpm for 150 s. After coating, the sensing layers were cut into 38 \( \times \) 25 mm pieces. Although the bond area is 25 mm \( \times \) 25 mm, the extra length of the sensor is included for the applying electrodes to measure the electrical resistance. The excess length of the sensor was masked using a high-temperature tape to enable the application of electrodes after curing the adhesive.

For the specimens with sensing layers, two different manufacturing approaches were used to electrically isolate the sensing layers. Figure 4 shows the schematic diagram of the processing method of both specimens. The key difference for both specimens is the different approaches for insulation of the sensor and the two-step curing of the adhesive insulated as compared to the one-step curing for the fabric insulated specimens. For the adhesive insulated specimens, a two-step curing method was used. First, a thin layer of the adhesive was applied to the bond area on the steel substrate and cured at 85 \( ^\circ \)C for 1 h. This layer acts as the insulating layer preventing galvanic corrosion and the electrical shorting of the sensor as established in our prior work [20, 21]. Next, the sensor was placed on the cured adhesive layer, and additional adhesive was applied and the carpenter’s vice with spacers was used to maintain the bondline and cured for another hour at 85 \( ^\circ \)C. For specimens where the sensing layer is isolated using a fabric, curing was accomplished in a single step. A lower areal density nonwoven aramid fabric was placed on the steel surface prior to the sensing layer and then adhesive was applied to both layers in the bond area followed by clamping in a vise and curing.

After curing the joints, end tabs made of an electrically insulating glass/epoxy laminates were attached to all the specimens for electrical isolation and alignment of the specimens in the load frame. The tape covering the masked regions of the sensing layer was removed and conductive silver paint (Flash-Dry 04999-AB, SPI. Supplies) was used to apply 6 mm wide electrodes across the width of the specimen. After drying, electrical lead wires were attached using conductive silver epoxy (40-3900, Epoxies Etc. USA).

### 2.3. Mechanical and electrical characterization

A displacement-controlled screw-driven load frame (Instron 5567) was used to load the specimens under tension at a crosshead displacement rate of 6.35 mm/min (0.25 in/min), which is half of the recommended displacement of ASTM D5868 to allow a longer duration test to match the sensing response to acoustic emission activity. The specimens were tested under both monotonic and incremental cyclic loading and global deformation was measured using displacement of the crosshead. A Keithley 6430 sourcemeter was used to supply a constant source voltage of 20 V to measure the electrical response of the sensors during both the monotonic and cyclic tensile experiments. An acoustic emission system (MISTRAS Group, Inc., Princeton Junction, NJ) was also used to record acoustic events during each experiment following the procedure described by Gao and coworkers [32].
- A 60 kHz narrow band resonant sensor with high sensitivity having an operating frequency range of 35-100 kHz was used. To avoid the recording of background noise from load frame and other sources in the testing laboratory, a threshold of 30 dB was utilized for filtering the noise.

3. Results and discussion

Figure 5 shows the joint shear strength of all the specimens. The control specimens, without a carbon nanotube sensing layer, have an average shear strength of 14.4 MPa. The adhesive insulated specimens have an average shear strength of 9.7 MPa—a 31% reduction when compared to the control specimens. Ahmed et al. also reported a 23% decrease in the ultimate strength for similar specimens. This is likely because of the reduced number of chemical bonds at the interface created by multistage curing of both adhesive layers. For example, Wang et al. [33] reported a significant decrease in tensile, bending and impact properties of specimens with an epoxy-epoxy interface where both epoxy layers were cured separately. The fabric insulated specimens have an average shear strength of 14 MPa, not a large reduction in the average joint shear strength, although there is a slightly higher experimental scatter.

Figure 6a,b shows the stress-displacement, resistance and acoustic emission response under monotonic tensile loading to failure of adhesive insulated and fabric insulated specimens, respectively. For both specimens, there is little initial acoustic activity. The increase in electrical resistance in these regions is linear and results from the elastic piezoresistive response of the sensing layer under tensile deformation. At the nanoscale, the piezoresistive response is due to the increase in the nanotube-nanotube tunneling gaps as the fibers in the sensing layer are stretched in tension. The change in the contact resistance due to the change in the tunneling gap results in a significant change in the electrical resistance of the sensing layer.

For the adhesive insulated specimen (Figure 6(a)), at a displacement of about 0.35 mm, there is a sharp change in electrical resistance that corresponds directly to a sharp change in the cumulative acoustic hits and large spikes of acoustic hits are observed. Correspondingly, the stress-displacement data shows a slight discontinuity at that displacement. The acoustic activity and resistance change data indicate that there is likely some damage that occurred at the additional interface of the adhesive layer caused due to the two-step curing of the specimens. In contrast, the fabric insulated specimen (Figure 6(b)) shows an overall continuous resistance change where the slope follows the applied stress. At a displacement of 0.3 mm and 0.5 mm, increased acoustic activity is observed, which corresponds with very minor disturbances in the resistance curve. These acoustic peaks are almost an order of magnitude
magnitude smaller than the one observed for the adhesive insulated specimen where the resistance curve spikes. Just before failure in the fabric insulated specimen, spikes in resistance are observed which correspond directly with large amounts of acoustic hits. Figure 6c shows the stress-displacement and acoustic emission response for a control specimen without carbon nanotube sensing layer.

The control specimens have failure stress of \( \frac{14}{24} \) MPa, similar to the fabric insulated specimen, slightly higher than the adhesive insulated specimen. The acoustic emission peaks are seen at a displacement of 1 mm and after, and they are significantly smaller when compared to peak observed at 0.35 mm for the adhesive insulated specimen. Figure 7 shows photographs of the joint fracture surfaces observed for the different specimens. For the adhesive insulated specimens (Figure 7(a)), a mixed failure is observed, a partial debonding at the adhesive/steel interface and propagation into the sensing layer at the interface formed by the separately cured adhesive layers. There are clear remnants of the sensing layer on both substrates. Although the failure surface for the adhesive insulated specimen is rougher, the joint strength is lower due to the presence of the weak interface because of the two-step curing. For the specimens insulated by the fabric layer, fracture at the composite/adhesive interface was observed and no damage is introduced into the sensing layer (Figure 7(b)). This failure mechanism is the same as observed in the control specimens with no sensing layer. The failure surface for control specimen is shown in Figure 7c, which looks very similar to the failure surface observed for the fabric insulated specimens. Figure 8 shows the scanning electron micrographs of the damage to the sensing layer for the adhesively insulated specimen. On the fiber surface it can be observed that the carbon nanotube coating has been stripped off and higher magnification images reveal fibrillation and transverse splitting of the aramid fibers. For the fabric insulated specimens, the one step curing of the adhesive bondline, without an additional interface within the adhesive bond, prevents the failure from occurring at the sensing layer interface, resulting in higher shear strength. Based on the different types of resistance curves observed for the two specimens, the amount of resistance change and the number of disturbances and non-linearities can give information about the type of damage occurring which can be used to predict the failure mode before the eventual failure.

In order to better understand the accumulation of damage in the adhesive joints for the two different methods of insulating the sensing layer from the steel substrate, cyclic loading was used to examine the sensing response to damage progression in the joints. In all cases, the failure modes were similar to those observed in the monotonic tests, where failure in the adhesive insulated specimens showed fracture within the sensing layer and the fabric insulated specimens showed debonding at the composite interface.

Figure 9 shows the transient stress, resistance change and acoustic activity during progressively increasing cyclic loading for both specimens. The specimens with the adhesive insulation (Figure 9(a)) show a highly nonlinear sensing response after the first two load cycles, with a number of sharp jumps and irregularities during both loading and unloading. The nonlinearity also corresponds directly to acoustic activity, indicating that the sensor is detecting damage that is likely occurring within the adhesive layer. There is also a permanent resistance
change observed after the end of second cycle, which continues to increase after each cycle. Although the damage to the sensing layer may cause a higher change in resistance and provide direct information about the damage, but the adhesive bond loses reliability due to the weak interface and the non-linearity could possibly affect the repeatability of the sensor. In contrast, the sensing layer that is insulated by the fabric layer (Figure 9(b)) shows a more linear sensing response that tracks well with the applied stress with very slight acoustic activity in each cycle until the final load cycle where the failure of the joint occurred. There is a slight permanent resistance change after the 9th cycle, which corresponds to more accumulated acoustic emissions. In addition, it is observed that the load/unload behavior becomes slightly nonlinear after this point. It is possible that the sensing layer is detecting the incipient damage in the joint. In addition, nonlinearity at higher loads may be due to combined shear and peel stresses that are occurring in the adhesive due to the eccentricity of the joint.

In order to examine the sensing response, cycles were examined individually. For both specimens, the sensing response of the second cycle (Figure 10) and the last full cycle prior to failure (Figure 11) are highlighted. In the second cycle for the adhesive insulated specimen (Figure 10(a)), there is a spike in the resistance change curve along with corresponding acoustic activity at a stress of approximately 2 MPa. All of the acoustic activity is observed in the loading segment, and there is a permanent resistance change under the unloaded condition. For the fabric insulated specimen (Figure 10(b)) the resistance response tracks well with the applied stress and no permanent resistance change is observed when the specimen is unloaded, indicating that there is likely no damage in the sensing layer. Also, little acoustic activity is measured when compared to the adhesive insulated specimen.

Figure 11a,b shows the last complete loading-unloading cycle before failure for the adhesive insulated and fabric insulated specimens, respectively. The resistance change curve for the adhesive insulated specimen shows a lot of inflections and spikes that also correspond to acoustic emissions, likely due to the damage occurring in the sensing layer. For just this cycle alone, the cumulative acoustic
The number of hits for the adhesive insulated specimen is an order of magnitude higher, about 6,000 as compared to about 800 for the fabric insulated specimen. The irregularities in the resistance change curve, higher acoustic activity and permanent change in resistance indicate more damage at the sensing layer for the adhesive insulated specimen.

4. Conclusions

This research establishes a novel technique of using carbon nanotube-based sensing layers for damage monitoring of adhesively bonded composite-metal hybrid joints. This technique has the potential for detecting damage and evaluating different failure modes based on the resistance change behavior of the sensing layer. The applicability of this technique for damage sensing is shown through real-time measurements during monotonic and progressively increasing cyclic tensile loading. Two approaches for the insulation of the sensing layer have been considered by using: (1) a thin layer of adhesive and (2) a nonconductive fabric. For both approaches the sensor is able to accurately detect damage formation corresponding to increased acoustic activity. The nonconductive fabric approach leads to higher joint strength as compared to the adhesive insulated approach, and the use of a nonconductive fabric is more convenient and scalable for large scale field applications since it involves a one-step curing process for the adhesive. The carbon nanotube-based sensing layer is able to detect deformation and
incipient damage. Due to the distinct resistance response, this technique could be used to distinguish between different kinds of failures. Lastly, the carbon nanotube-based sensing layer holds promise for applications in damage sensing of adhesive joints when composite materials are used for strengthening and rehabilitation of civil infrastructure.

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Disclosure statement

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