Structural health monitoring of composites from carbon nanotube coated e-glass fibre

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Abstract: As composite materials are rapidly replacing traditional materials in many industries, improved methods of identifying their damage are in development. With nanotechnology-based sensors, it is possible to efficiently detect damage without the time-extensive processes of scanning the structure. This research investigated the development of a nanomaterial-based sensor for health monitoring of composite structures. To develop the sensor, carbon nanotube/epoxy mixture was coated on a strand of e-glass fibre to be adhered onto a fibreglass composite specimen. Tensile testing of the specimens provided data on the actual strain, which was correlated with the experimental differential resistances measured by a multimeter. The experimental resistance data was calibrated with the actual strain data. Ultimately, the experimental sensors created a sample of gauge factors, which represents 91.24% probability of replicating the observed range of gauge factors by using the same manufacturing procedures, providing a valid alternative and consistent method to detecting composite damage.

Keywords: composites; strain gauge; carbon nanotubes; CNT; e-glass fibre; health monitoring system.

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1 Introduction

The rising popularity of composite materials has influenced the aerospace, automotive, and buildings industries to creating structures with such advanced materials. Manufacturers are substituting traditional materials with modern composite materials for benefits of high strength to weight ratios and non- corrosive properties. However, most companies are currently using a dated health monitoring system that consists of utilising transducers to monitor transmitted waves generated by ultrasonics. This method is time intensive and can be improved with accelerated methods by using an advanced system such as nanotechnology- based sensors to refrain from bonding strain gauges throughout the structure (Abot, 2017; Luo et al., 2019). By exploring methods to detect damage, rather than diagnose damage, in composite structures, critical failure can be efficiently prevented, hence a reduction in time and costs will be achieved while improving the safety of users, occupants, and nearby civilians.

The objectives of this research entail investigations on material selections, manufacturing process, and testing of the sensor. Calibration of the manufactured sensors was performed through comparisons between the experimental sensor data to that from a traditional extensometer adhered to the same test specimen. The results were verified for accuracy, validity, and the feasibility based on the practicality of the manufacturing process.

2 Background

Recent studies have explored the development of a structural health monitoring to predict strain, damage, and residual strength of composites (Wang et al., 2018; Balaji and Sasikumar, 2017; Barber et al., 2009; Sebastian et al., 2014). For example, 2% by weight

reduced graphene oxide (rGO) was mixed into Araldite LY 556 epoxy and HY 951 hardener with a sonication method and the resulting mixture was rolled onto e-glass fibres. By coating the strand of e-glass fibre with conductive rGO, the piezoresistance relating the strain to the force applied was observed when a tensile test was performed (Balaji and Sasikumar, 2017). A similar experiment has also been performed with the same method but using carbon nanotubes (CNT) instead of rGO. Investigators grew CNTs aligned on alumina fibre cloth using a chemical vapor deposition method. The cloth was made into a laminate using West Systems Epoxy Resin 105 and Hardener 206. This sensor was then subjected to an impact test at which a steel ball was dropped at a set height (Raghavan et al., 2009).

The aforementioned sensor type was also subjected to monatomic tests, cyclic fatigue tests, and load controlled incremental loading/unloading tensile test, which were compared to a controlled strain gauge data set (Aly et al., 2016). Another method of creating such sensor used a buckypaper film that was rolled onto the surface of the test sample. This sensor was used for monitoring delamination through a cyclic fatigue test, where a significant difference in electrical resistance between loaded and unloaded conditions was observed as a result of the crack separating and closing (Kravchenko et al., 2018).

Typically, tensile testing was performed during the initial phases of the studies. However, once the tensile tests have proven to be successful, three-point bend tests and vibrational tests have been examined. Once all the tests have been perfected on a coated single strand of fibre, a gridded weave layout were prototyped to be able to locate impacts (Sasikumar et al., 2019). In most tests conducted on the experimental sensors, terminals at each end of the sensors were probed with a multimeter to read the resistance when subjected to a load. Both two- and four-point probe electrical resistance measurements investigated, but typically a two-point probe was sufficient as the resistance readings noticed were large, in the kilo-ohms range. A four-point probe was only used if small resistances were read and high sensitivity of the multimeter was needed (Raghavan et al., 2009; Aly et al., 2016; Inam et al., 2014).

The theory behind coating a strand of fibre comes from the way a strain gauge works (Alexopoulos et al., 2010). A strain gauge simply functions as a wire, when stretched, or tensioned, the resistance increases, as shown in equation (1):

$$R = \frac{\rho L}{A} \tag{1}$$

where R is the resistance, ρ is the resistivity, L is the length, and A is the cross-sectional area of the wire.

Once a sensor that reads resistances from a multimeter is created, the electrical strain (ε_E) could then be determined by equation (2) and compared to the mechanical strain (ε_M) , from equation (3), or collected through the data acquisition machine attached to the tensile testing machine. With the electrical strain plotted against the mechanical strain, the gauge factor (*GF*) could be determined from equation (4) by taking the slope of the linear plot.

$$\varepsilon_E = \frac{\Delta R}{R_0} = \frac{R - R_0}{R_0} \tag{2}$$

$$\varepsilon_M = \frac{\Delta L}{L_0} = \frac{R - L_0}{L_0} \tag{3}$$

$$GF = \frac{\varepsilon_E}{\varepsilon_M} \tag{4}$$

The gauge factor is the ratio between the electrical strain to mechanical strain. This ratio represents the sensitivity of the sensor. In addition, a stress-strain curve, generated from data collected from a strain gauge attached on the opposite side of the specimen was plotted on the same figure as the electrical- mechanical strain curve to provide a controlled data set (Balaji and Sasikumar, 2017; Aly et al., 2016; Kravchenko et al., 2018). Plotting two data sets on a single plot allowed for confirmation that the stress-strain curve and the electric-mechanical strain curve are both linear, as testing was conducted in the elastic region of the test coupon.

3 Methods

3.1 Materials Selection

The materials selected consisted of carbon nanotubes (CNT) as the sensing medium due to its high conductivity and e-glass fibre as the base strand coated with CNT. e-glass was selected due to its desirable electrical insulation properties as well as its high strength and stiffness and relatively low cost when compared to s-glass fibres. West Systems 105 Epoxy Resin and 206 Slow Hardener were chosen for their non-conductive properties and long cure time of 10–15 hours, providing ample time for coating the fibres. Properties of the multi-walled carbon nanotubes are presented in Table 1 (Sky Spring Nanomaterials, Inc., n.d.). Silver paint was used for creating the two terminals at the end of each sensor. Copper foil was then wrapped around the ends of the probes to serve as nodes for attaching alligator clamps connected to a multimeter. This setup provided access to data collection. An NP130HF thermoset fibreglass Composite Sheet, donated by Norplex Micarta, was designated as the test sample. Properties of the Norplex Micarta NP130HF are presented in Table 2 (Norplex-Micarta, n.d.). An extra benefit of using this fibreglass composite sheet as the test material is that it maintained the material properties of the strand of e-glass fibre sensor base.

MWNTs -COOH functionalised 95% properties				
Property	Specification			
Purity	> 95 %			
Content of -COOH	2.0 wt%			
Outer diameter	10-20 nm			
Inner diameter	3–5 nm			
Length	5–30 nm			
Electrical conductivity	> 100 s/cm			
Bulk density	0.27 g/cm ³			
True density	21 g/cm ³			
Manufacturing method	Catalytic CVD			

 Table 1
 Physical properties of multi-walled carbon nanotubes

Source: Sky Spring Nanomaterials, Inc. (n.d.)

Table 2 Norplex-Micarta NP130HF fibreglass coupon material properties

Norplex-Micarta NP130HF material properties				
Thickness	1.587 mm			
Length	22.86 cm			
Width	2.54 cm			
Elastic modulus	25,510.6 MPa			

Source: Norplex-Micarta (n.d.)

3.2 Manufacturing processes

The experimental setup began with the addition of carbon nanotubes to epoxy resinhardener. Five parts of West Systems 105 Epoxy Resin and one-part West Systems 206 Slow Hardener by weight were mixed with 2% of carbon nanotubes by total weight. This mixture was then coated onto a strand of e-glass fibre by dipping the fibre strand into the mixture and placing it between vacuum bags. A squeegee method was used to create an even coat of conductive mixture onto the e-glass fibre. Once fully coated, the tacky strands of coated fibre were then dragged along powdered carbon nanotubes to adhere extra nanoparticles on the surface of the sensor. This sensor was then compressed between two flat plates and left for 15 hours to cure. Once cured, the ends of the sensors were painted with silver paint to create the terminals and left to dry. An initial test to measure resistance by probing both ends with a multimeter proved the conductivity of the sensor. The magnitude of the initial resistance measured were in the order of mega-ohms.



Figure 1 Confirmation of conductivity in sensors (see online version for colours)

 Table 3
 Specimen and sensor dimensions

Sample dimensions							
Specimen -	Coupon			Sensor			
	Width	Length	Thick	Width	Length	Thick	
0	2.603 cm	22.86 cm	0.1587 cm	0.6960 cm	10.2108 cm	0.08382 cm	
1	2.520 cm			0.6680 cm	9.3142 cm	0.07366 cm	
2	2.583 cm			0.5131 cm	9.4463 cm	0.08128 cm	
3	2.476 cm			0.5359 cm	9.6520 cm	0.07874 cm	

Next, the test coupon was trimmed from the NP130HF thermoset fibreglass Sheet to make 2.54 cm by 22.86 cm specimens. One sensor was centred and adhered to each coupon using West Systems 105 Epoxy Resin and 206 Hardener under compression and allowed time for curing. Once cured, a tab of copper foil was wrapped around the coupon and sensor at the areas where silver paint was applied to allow for the alligator clips from the multimeter to reach the terminals, as shown in Figure 1. The dimensions of the samples created are presented in Table 3. It should be noted that Specimen 0 was the first working sensor created during the early manufacturing processes while Specimens 1 through Specimen 3 were created in attempts to replicating the results from Specimen 0.

3.3 Testing procedures and data collection

Tensile testing was determined as the first step of testing these sensors. First a blank specimen without any CNT sensors was tested to 5,000 N to ensure that the material was still within the elastic range. Since an extensometer was made available, a strain gauge was not needed for data collection. Being within the range of elasticity was confirmed by plotting the stress-strain curve and verifying the linearity of the relationship. Therefore, a

maximum load of 5,000 N was applied on the coupon during tensile testing. Although the majority of past experiments used a strain gauge to collect a controlled data set, an extensiometer was primarily used to provide the control needed to compare to the experimental CNT sensor created. To collect data from the CNT sensor, the multimeter logged resistances between the two terminals. Each specimen was tested for five trials.

4 Results and discussion

4.1 Data analysis

Once tensile testing was performed, the stress-strain curve of each trial was generated. Before plotting the electrical-mechanical strain curve from the CNT sensor, the electrical strain was computed using equation (2) from the resistance data logged. The two curves plotted for each specimen are shown in Figures 3 through Figure 6. In addition, a correction factor was incorporated into the third data set which was plotted to correct the electrical strain curve to match the stress-strain curve. The slope of the stress-strain curve is known as the Modulus of Elasticity, while the slope of the electrical-mechanical strain curve is the GF. Both values were determined and are presented in Table 4 with their mean and standard deviation for each specimen and for all samples and trials. It was observed that the gauge factor determined for Specimen 0 had the highest variability, as it was an initial experiment. However, the variability was minimised once the manufacturing procedure was solidified. Figure 7 depicts a plot with all the average electrical-mechanical strain curves, centred at the origin for each specimen, with the slope representing the gauge factor.



Figure 2 Testing apparatus (see online version for colours)

Specimen	Trial	Extensometer (controlled) modulus of elasticity (MPa)	CNT sensor gauge factor	
0	1	29,483	6.81	
	2	27,066	2.46	
	3	28,822	3.43	
	4	30,291	4.36	
	5	30,056	4.24	
	Mean	29,143.6	4.26	
	Std. dev.	1,292.7	1.62	
1	1	34,128	3.64	
	2	35,303	3.22	
	3	30,821	3.32	
	4	27,610	2.81	
	5	31,946	3.21	
	Mean	31,961.6	3.24	
	Std. dev.	3,004.0	0.30	
2	1	31,564	3.83	
	2	30,846	3.34	
	3	30,759	3.35	
	4	30,656	3.36	
	5	30,680	3.29	
	Mean	30,901	3.43	
	Std. dev.	378.0	0.22	
3	1	32,164	4.08	
	2	32,900	3.55	
	3	32,709	3.54	
	4	32,723	3.39	
	5	32,785	3.53	
	Mean	32,656.2	3.62	
	Std. dev.	285.3	0.27	
Total mean		31,165.6	3.64	
Total std. dev.		2,035.8	0.87	

 Table 4
 GF determined with sample mean and standard deviations





Figure 4 Specimen 1 sample plot (see online version for colours)







Figure 6 Specimen 3 sample plot (see online version for colours)



Figure 7 Average electrical-mechanical strain of CNT Sensors with gauge factors (see online version for colours)



4.2 Statistical analysis

With the raw experimental data and gauge factors tabulated, a box plot was created for the gauge factor of each sample tested, as shown in Figure 8. It is visually apparent that the samples created after the initial success of specimen 1 resulted in a smaller spread that is concentrated at its mean modulus of elasticity and gauge factor. The consistency in the span of specimens 1–3 proves the consistency in the testing technique. However, the slight variations in the average values at which the modulus of elasticity and gauge factors were concentrated at show the inconsistency in manufacturing methods.

The gauge factors observed were between the range of 2.46 and 6.81 with an average gauge factor of 3.64. This gauge factor falls in the range of a typical metal foil strain gauge and aligns with Alexopoulos et al.'s results (2010). In comparison, Alexopoulos observed a gauge factor of 3.14 when loading the specimen to 83% of fracture stress, which is approximately a 14.75% difference from the mean of all the samples tested. However, when Alexopoulos et al. (2010) loaded the specimen to 100% of fracture stress, a gauge factor of 3.43 was noticed, which resulted in a 5.94% difference from the mean of all the samples tested in this study. With an overall average standard deviation of 0.87, a z-test statistical method for a Gaussian distribution was appropriated by using equation (7) to find the x-value at the minimum and maximum values of the gauge factors and equation (8) to find the probability of the gauge factors observed of the population as follows:

$$Mean(GF) = 3.64\tag{5}$$

$$Std. \, dev(GF) = 0.87 \tag{6}$$

$$Z = X \frac{Mean(GF)}{Std.\,dev(GF)} \tag{7}$$

$$P(\operatorname{Min}(GF) \le X \le \operatorname{Max}(GF)) = P(Z(\operatorname{Min}(GF)) \le Z \le Z(\operatorname{Max}(GF)))$$
(8)

$$Z(\operatorname{Min}(GF)) = \frac{2.46 - 3.64}{0.87} = 1.356$$
(9)

$$Z(\operatorname{Max}(GF)) = \frac{6.81 - 3.64}{0.87} = 3.644$$
(10)

$$P(\operatorname{Min}(GF) \le X \le \operatorname{Max}(GF)) = P(Z \le 3.644) - P(Z \le -1.356)$$
(11)

$$P(\operatorname{Min}(GF) \le X \le \operatorname{Max}(GF)) = 0.9999 - 0.0875$$
(12)

$$P(\operatorname{Min}(GF) \le X \le \operatorname{Max}(GF)) = 0.9124 = 91.24\%$$
(13)

Figure 8 Box plots of the gauge factors determined for each specimen (see online version for colours)



Gauge Factor

175

It was determined that the probability that the gauge factor of any test sample will fall within the range observed in this study is 91.24%, assuming a Gaussian distribution. The probability density on a standard normal distribution curve where the mean is set to a z-score of zero is represented in the shaded area in Figure 9.





4.3 Future recommendations

For future attempts, it is highly recommended to troubleshoot the trials performed by contemplating possible flaws in synthesising the carbon nanotubes into the resin, as well as the proportions of nanoparticles and the epoxy. The effect of the electrode area at the terminals should be examined. Also, there is a need to conduct some testing to ensure that a solid connection to the nanotubes exists. The procedures should be attempted with different sensing mediums, such as graphene oxide or buckypaper. In addition, a method of electrophoretic deposition should be tested for loading the nanosensors on a substrate such as a sheet of tissue paper or fabric to yield an evenly dispersed sensor, similar to buckypaper. If the initial resistance ratings were lower, the sensors would be more sensitive. The differences in characteristics and behaviour of the sensors should be studies with varying the lengths and thicknesses of the sensors. Another method that may yield positive results would be creating a paste with carbon nanotubes infused into a syringe where the sensors are drawn into the material utilising the sensor.

Once the manufacturing process has been fulfilled and consistent resistance readings observed with calibrations from tensile testing accomplished, calibrations should be performed through bend testing and fatigue testing. Finite element analysis through Solidworks, Ansys, or Abaqus may also be included as an additional form of validation. Future research should also consider reproducing the same results but with the sensors lined up in a gridded pattern with multiple nodes with an ability to locate the point at which a force is acting on the grid. Lastly, the gridded sensor should be examined when embedded within a sandwich of composite layers.

5 Conclusions

Creating a manufacturing process for a health monitoring sensor coated with nanoparticles provide a number of challenges to learn from. Performing multiple experiments provided an insight on how the various manufacturing methods resulted in the creation of a more effective sensor for aerospace, automotive, and building structure applications. The manufacturing of CNT sensor was proven a viable concept. The gauge factors determined were between the range of 2.46 and 6.81, which fall between 84.67% of the accepted range of gauge factor 2 to 5 for a typical commercial strain gauge. With an overall average standard deviation of 0.87, the probability that the gauge factor of any test sample will fall within the range observed is 91.24%, assuming a normal Gaussian distribution. The method used in this investigation to create the CNT sensors showed consistency of readings through tensile loads. Further studies are warranted to optimise the sensor and to test its sensitivity under the effects of bending, vibrations, and fatigue.

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