

STRUCTURAL HEALTH MONITORING OF COMPOSITES FROM CARBON NANOTUBE (CNT) COATED E-GLASS FIBER

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ABSTRACT

This research investigated the development of a nanomaterial-based sensor for health monitoring of composite structures. To develop the sensor, carbon nanotube/epoxy mixture (2%wt CNT) was coated on a strand of E-glass fiber to be adhered onto a fiberglass composite specimen. The selection of E-glass fiber and fiberglass plate was largely due to its electrical insulating properties to demonstrate that the carbon nanotube is driving the sensing capabilities through its highly conductive nature. In addition, by adhering the coated E-glass fiber to a fiberglass coupon, the homogeneity and material properties were approximately maintained. Tensile testing of the specimen conducted through a Lloyd LD50 tensile testing machine provided data on the actual strain which was correlated with the experimental differential resistances measured by a multimeter, both at the same specified tensile loading conditions. With two sets of data, the experimental resistance data was calibrated with the actual strain data collected. 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: Mechanical Engineering, Aerospace Engineering, Structural Engineering, Composites, Strain Gauge, Carbon Nanotubes (CNT), Nanoparticles, Nanotechnology, Epoxy, Tensile Testing, E-Glass Fiber, Health Monitoring System, Mechanics of Materials, Manufacturing

1. INTRODUCTION

The rising popularity of composite materials has influenced the aerospace, automotive, and buildings industries to creating structures with such advanced materials. Although manufacturers are substituting traditional materials with modern composite materials for benefits in high strength to weight ratios and non-corrosive properties, most companies are currently using a dated health monitoring system that consists of utilizing 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. 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.

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The objectives of this research entail investigations in 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 sample test subject. The results were verified for accuracy, validity, and the feasibility based on the practicality of the manufacturing process was discussed.

2. BACKGROUND

Recent studies have explored the development of a structural health monitoring system to predict strain, damage, and residual strength of a composite by mixing 2% by weight reduced graphene oxide (rGO) into Araldite LY 556 epoxy and HY 951 hardener with a sonication method and rolling the mixture onto E-glass fibers. By coating the strand of E-glass fiber with conductive rGO, the piezoresistance relating the strain to the force applied was observed when a tensile test was performed [1]. A similar experiment has also been performed with the same methods but instead, using carbon nanotubes (CNT) instead of rGO. Investigators grew CNTs aligned on alumina fiber 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 [2]. This type of sensor was also subjected to monotonic tests, cyclic fatigue tests, and load controlled incremental loading/unloading tensile test with use of a tensile testing machine which were compared to a controlled strain gauge data set [3]. Another method of creating such sensor used a Bucky paper film that was rolled onto the surface of the test sample to monitor 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 [4]. Typically, tensile testing was performed during the initial phases of the study. 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 fiber, a gridded weave layout would be prototyped as a next step to be able to locate impact [5]. 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 were made possible, 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 [2][3][6].

The theory behind coating a strand of fiber comes from the way a strain gauge works [7]. A strain gauge simply functions as a wire, where when stretched, or tensioned, the resistance increases, as shown in Equation 1:

$$R = \frac{\rho L}{A} \quad (1)$$

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

Creating a sensor that reads resistances from a multimeter, 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.

$$\varepsilon_E = \frac{\Delta R}{R} = \frac{R - R_o}{R_o} \quad (2)$$

$$\varepsilon_M = \frac{\Delta L}{L_o} = \frac{L - L_o}{L_o} \quad (3)$$

$$GF = \frac{\varepsilon_E}{\varepsilon_M} \quad (4)$$

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 [1][3][7]. By 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, based on availability and short lead time, consisted of carbon nanotubes (CNT) as the sensing medium due to its high conductivity, E-Glass Fiber as the base strand coated due to its good electrical insulation properties, as well as its high strength and stiffness and relatively low cost when compared to S-Glass Fibers. West Systems 105 Epoxy Resin and 206 Slow Hardener were chosen for its non-conductive properties, as well as its long cure time of 10-15 hours, providing ample time for coating the fibers. Properties of the multi-walled carbon nanotubes are tabulated in Table 1 [8]. Silver paint was dedicated for creating the two terminals at the end of each sensor with copper foil wrapping around the ends for the probes to attached to when measuring resistance. The NP130HF Thermoset Fiberglass Composite Sheet, donated by Norplex Micarta, was designated as the test sample. Properties of the Norplex Micarta NP130HF is tabulated in Table 2 [9]. An extra benefit of using this Fiberglass composite sheet as the test material is that it maintained the material properties of the strand of E-Glass Fiber selected.

Table 1: Physical Properties of Multi-Walled Carbon Nanotubes [8]

MWNTs -COOH Functionalized 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/cu.cm
True Density	2.1 g/cu.cm
Manufacturing Method	Catalytic CVD

Table 2: Norplex-Micarta NP130HF Fibreglass Coupon Material Properties [9]

Norplex-Micarta NP130HF Material Properties	
Thickness	0.159 cm
Length	22.9 cm
Width	2.54 cm
Elastic Modulus	25.5 MPa

3.2 Manufacturing Processes

The experimental setup began with the synthesis of carbon nanotubes into epoxy resin-hardener. Five parts of West Systems 105 Epoxy Resin and one-part West Systems 206 Slow Hardener by weight was mixed with 2% of carbon nanotubes by total weight. This mixture was then coated onto a strand of E-glass fiber by dipping the stand of fiber into the mixture and placed between vacuum bags. A squeegee method was used to create an even coat of conductive mixture onto the E-glass fiber. Once fully coated, the tacky strands of coated fiber 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 given time 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.

Next the test coupon was trimmed to maximize the 0.159 cm thick, 45.7 cm x 45.7 cm sheet of NP130HF Thermoset Fiberglass Sheet from Norplex Micarta. 2.54 cm x 22.9 cm specimens were cut to make the most out of the resources obtained. One sensor was centered and adhered to each coupon with West Systems 105 Epoxy Resin and 206 Hardener under compression and allowed 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 tabulated 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.

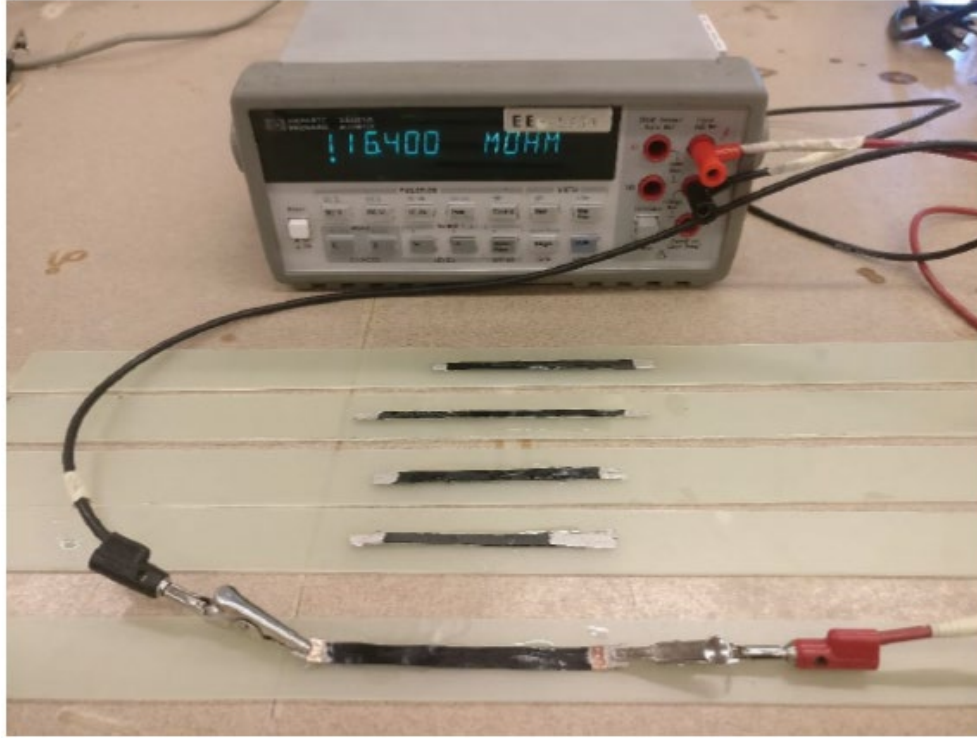


Figure 1: Confirmation of Conductivity in Sensors

Table 3: Specific and Sensor Dimensions

Sample Dimensions (Units in Inches)						
Specimen	Coupon			Sensor		
	Width (cm)	Length (cm)	Thick (cm)	Width (cm)	Length (cm)	Thick (cm)
0	2.60	22.9	.159	0.696	10.21	0.0829
1	2.52			0.668	9.31	0.0737
2	2.58			0.513	9.44	0.0812
3	2.48			0.536	9.65	0.0787

3.3 Testing Procedures and Data Collection

Tensile testing was determined as the first step of testing these sensors, but first a blank specimen without any CNT sensors was tested to 5000 newtons 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

5000 newtons was applied on the coupon during tensile testing. Although majority of past experiments used a strain gauge to collect a controlled data set, an extensometer 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.



Figure 2: Testing Apparatus

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 must be computed using Equation 2 from the resistance data logged. The two curves plotted for each specimen are shown below in Figures 3 through Figure 6. In addition, a correction factor was incorporated into the third data set 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 Gauge Factor. Both, which were determined are tabulated in Table 4 with its mean and standard deviation for each specimen and for all samples and trials. It was noticed that variability of the gauge factor determined for Specimen 0 had the highest variability, as it was an initial experiment. However, the variability was minimized once the manufacturing procedure was solidified. Figure 7 depicts a plot with all the average electrical-mechanical strain curves, centered at the origin for each specimen, with the slope representing the gauge factor.

Table 4: Gauge Factor Determined by

Specimen	Trial	Extensometer (Controlled) Modulus of Elasticity (MPa)	CNT Sensor Gauge Factor
0	1	29483	6.81
	2	27066	2.46
	3	28822	3.43
	4	30291	4.36
	5	30056	4.24
	Mean	29143.6	4.26
	Std. Dev.	1292.7	1.62
1	1	34128	3.64
	2	35303	3.22
	3	30821	3.32
	4	27610	2.81
	5	31946	3.21
	Mean	31961.6	3.24
	Std. Dev.	3004.0	0.30
2	1	31564	3.83
	2	30846	3.34
	3	30759	3.35
	4	30656	3.36
	5	30680	3.29
	Mean	30901	3.43
	Std. Dev.	378.0	0.22
3	1	32164	4.08
	2	32900	3.55
	3	32709	3.54
	4	32723	3.39
	5	32785	3.53
	Mean	32656.2	3.62
	Std. Dev.	285.3	0.27
Total Mean		31165.6	3.64
Total Std. Dev.		2035.8	0.87

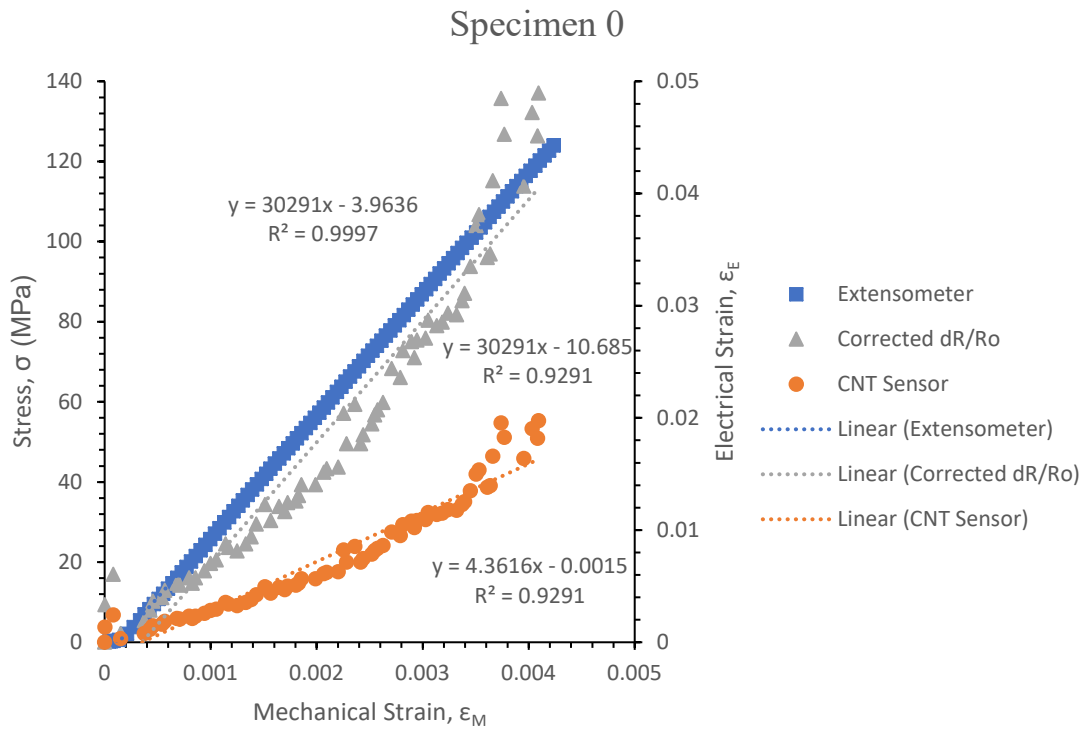


Figure 3: Specimen 0 Data Plot

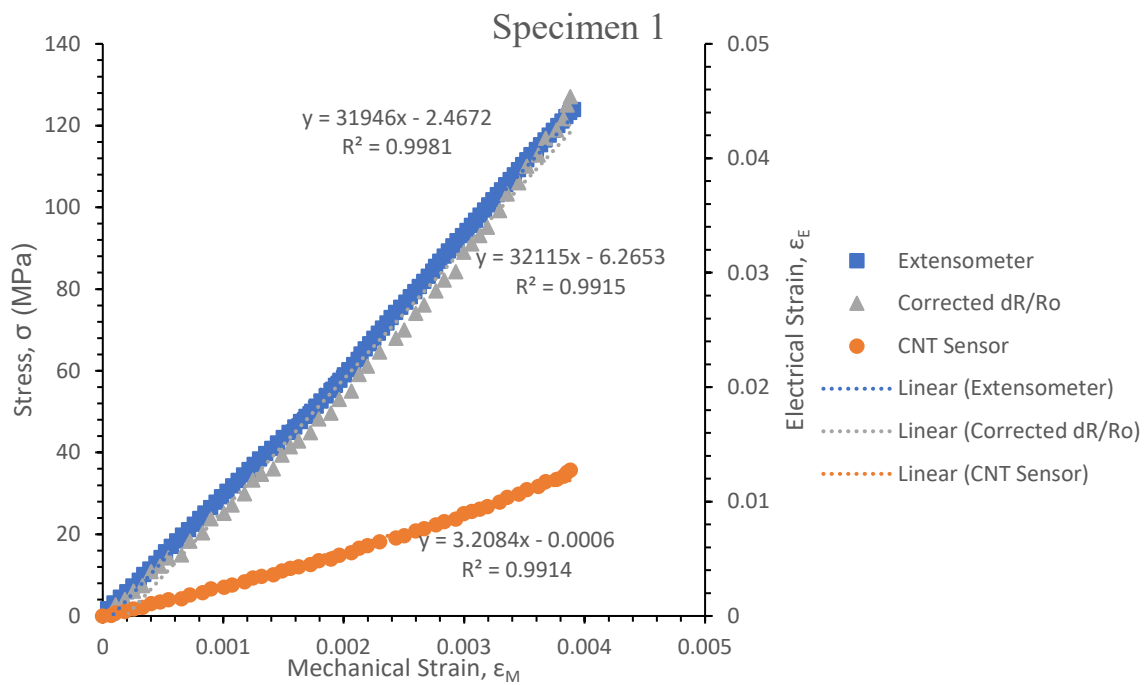


Figure 4: Specimen 1 Data Plot

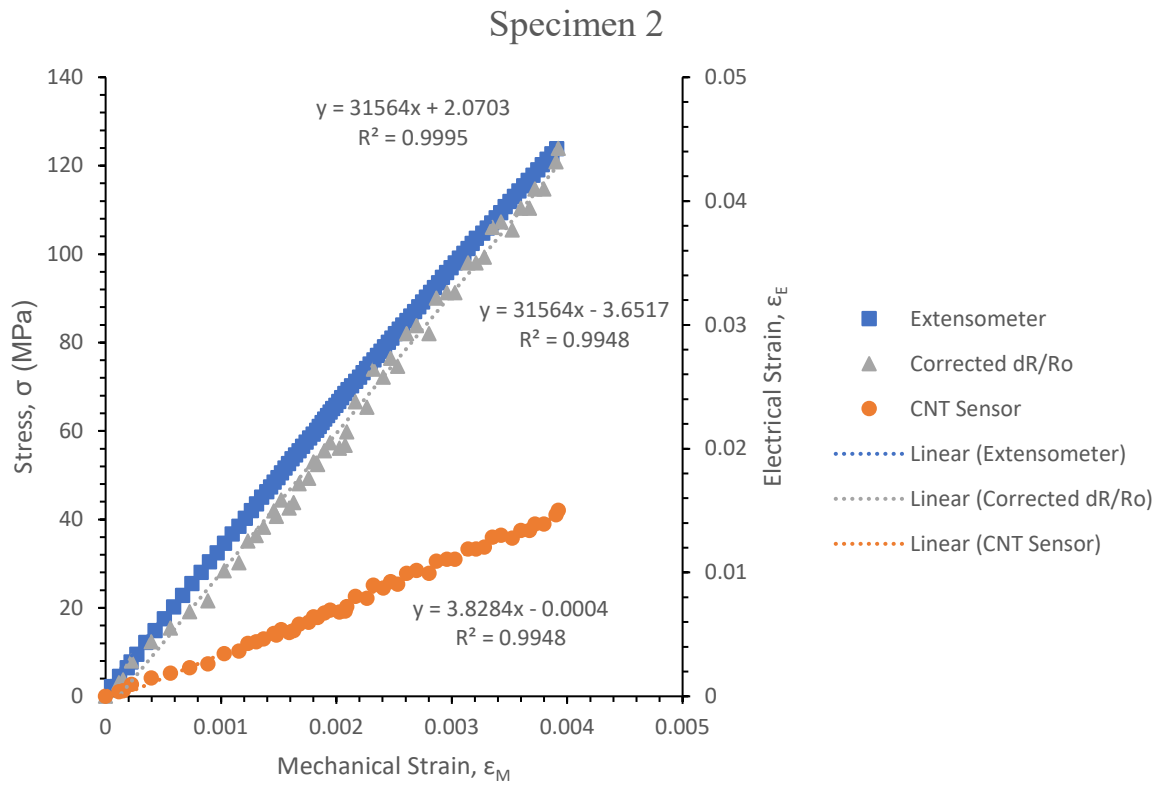


Figure 5: Specimen 2 Data Plot

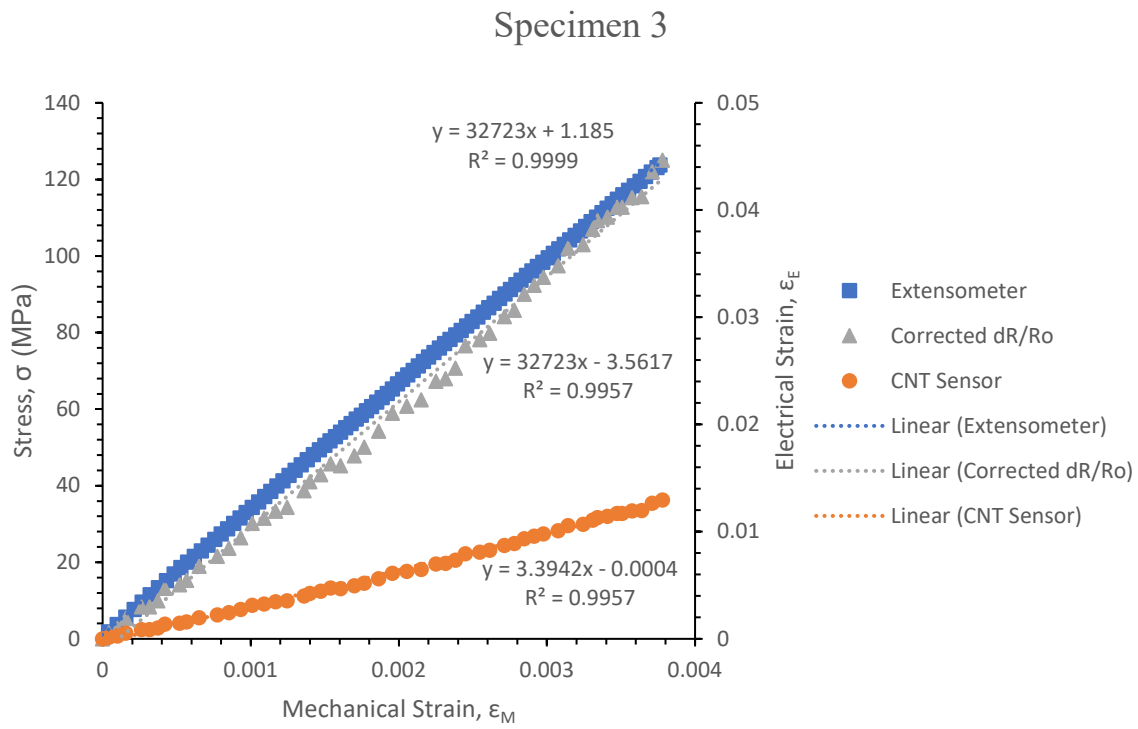


Figure 6: Specimen 3 Data Plot

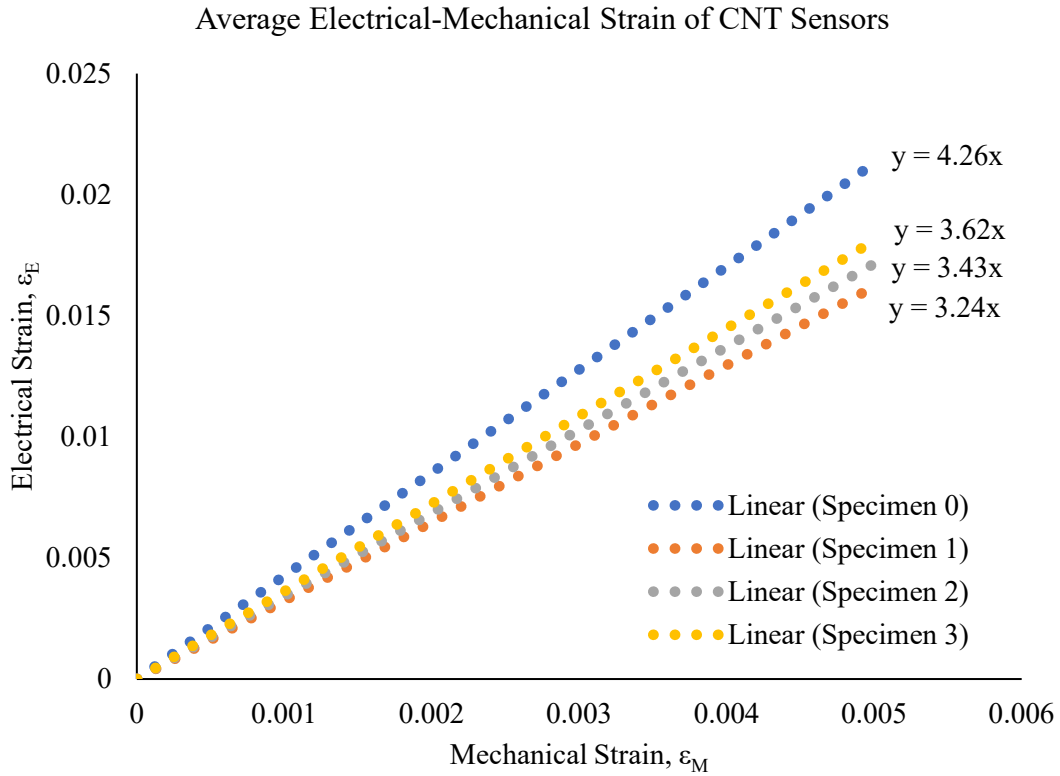


Figure 7: Average Electrical-Mechanical Strain of CNT Sensors with Gauge Factors

4.2 Statistical Analysis

With the raw experimental data and gauge factors tabulated, with the averages and standard deviation computed, a box plot was formed for gauge factor of each sample tested, as shown in Figure 8, respectively. 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 shows the inconsistency in manufacturing methods.

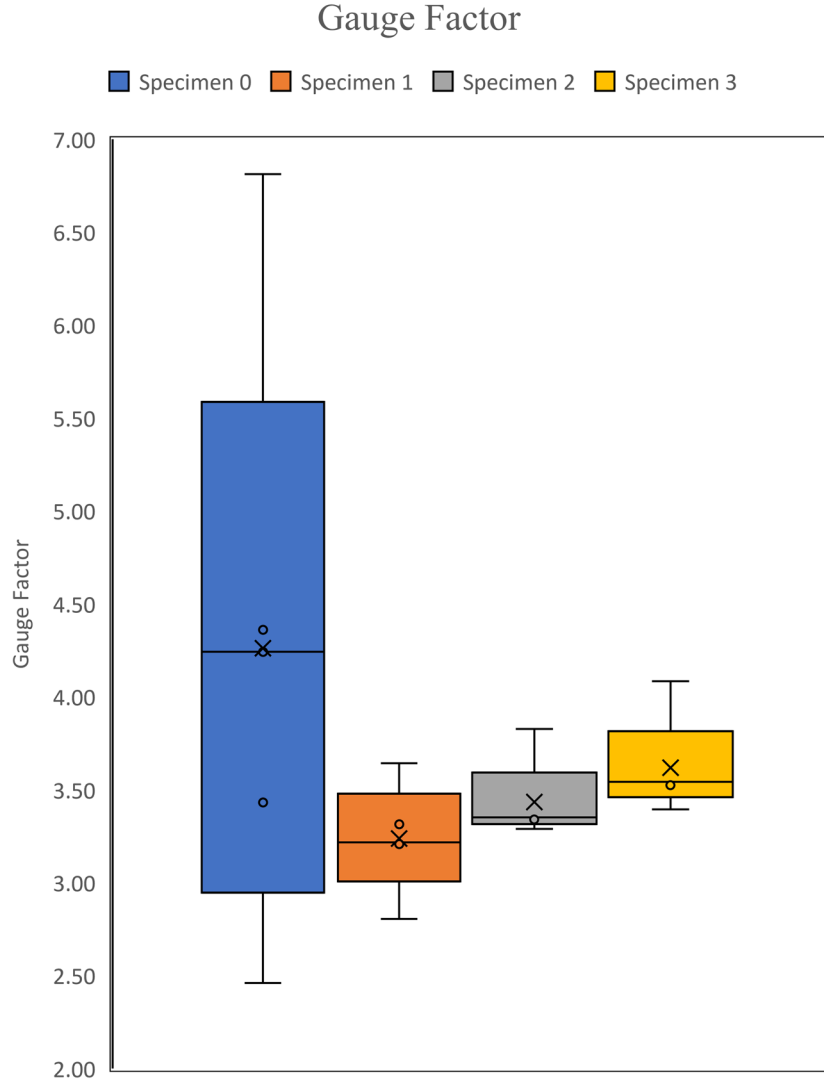


Figure 8: Box Plots of the Gauge Factors Determined for Each Specimen

The gauge factor observed were between the range of 2.46 and 6.81 with an average gauge factor of 3.64, which falls in the range of a typical metal foil strain gauge and aligns with Alexopoulos's results [7]. 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 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 [7]. With an overall average standard deviation of 0.87, a z-test statistical method for a Gaussian distribution is appropriated by using Equation 9 to find the Z-value at the minima and maximum values of the gauge factors and Equation 10 to find the probability of the gauge factors observed of the population as follows:

$$\text{Mean}(GF) = 3.64 \quad (5)$$

$$\text{Std.Dev}(GF) = 0.87 \quad (6)$$

$$Z = \frac{X - \text{Mean}(GF)}{\text{Std.Dev}(GF)} \quad (7)$$

$$P(\text{Min}(GF) \leq X \leq \text{Max}(GF)) = P(Z(\text{Min}(GF)) \leq Z \leq Z(\text{Max}(GF))) \quad (8)$$

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

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

$$P(\text{Min}(GF) \leq X \leq \text{Max}(GF)) = P(Z \leq 3.644) - P(Z \leq -1.356) \quad (11)$$

$$P(\text{Min}(GF) \leq X \leq \text{Max}(GF)) = 0.9999 - 0.0875 \quad (12)$$

$$P(\text{Min}(GF) \leq X \leq \text{Max}(GF)) = 0.9124 = 91.24\% \quad (13)$$

It was determined that the probability that the gauge factor of any test samples will fall within the range observed in this study is 91.24%, assuming a Gaussian distribution.

4.3 Future Recommendations

For future attempts, it is highly recommended to troubleshoot the trial performed by contemplating possible flaws in synthesizing the carbon nanotubes into the resin, as well as the proportions of nanoparticles and the epoxy. Examine the electrode area at the terminals and making sure that a solid connection to the nanotubes exists. Attempt the procedures with different sensing mediums, such as graphene oxide or buckypaper. In addition, if resources are available, try a method in electrophoretic deposition to plate 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. Study the differences in characteristics when varying the lengths and thicknesses of the sensors. Another method that may yield positive results would incorporate creating a paste with carbon nanotubes infused into a syringe where the sensors are drawn into the material utilizing the sensor.

Once the manufacturing process has been fulfilled and consistent resistance readings observed with calibrations from tensile testing accomplished, it would be a good idea to perform calibrations through bend testing and fatigue testing. Finite element analysis through Solidworks, Ansys, or Abaqus may also be included as an additional form of validation. To take this research further, consider reproducing the same results but with the sensors lined up in a gridded pattern with multiple nodes and be able to locate the point at which a force is acted on the grid. Lastly, repeat the gridded sensor but with this layout embedded within a sandwich of composite layers.

5. CONCLUSION

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 of how the various methods resulted in the creation of a more effective sensor in aerospace, automotive, and building structure applications. The infusion of 2% carbon nanotubes into epoxy by weight and application onto the strand of fibre was proven to be difficult based on the equipment available and lab facilities accessed while finding a data acquisition device compatible with the multimeter used could have allowed for more samples to be tested, which impacted the decisions made and results significantly.

The manufacturing of CNT sensor was proven to be a viable concept. The gauge factor determined were between the range of 2.46 and 6.81, falls 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 samples will fall within the range observed is 91.24%, assuming a normal Gaussian distribution.

With our method of creating these CNT sensors, it has been shown the consistency of readings through tensile loads. The authors plan to further study the effects of bending, vibrations, and fatigue on the CNT sensors.

6. REFERENCES

- [1] Balaji R, Sasikumar M. (2017). Graphene based strain and damage prediction system for polymer composites.
- [2] Raghavan, A., Kessler, S.S., Dunn, C.T., Barber, D., Wicks, S., & Wardle, B.L. (2009). Structural Health Monitoring using Carbon Nanotube (CNT) Enhanced Composites.
- [3] Aly, K., Li, A., & Bradford, P.D. (2016). Strain sensing in composites using aligned carbon nanotube sheets embedded in the interlaminar region.
- [4] Kravchenko, O.G., Pedrazzoli, D., Bonab, V.S., & Manzas-Zloczower, I. (2018). Conductive interlaminar interfaces for structural health monitoring in composite laminates under fatigue loading.
- [5] Sasikumar, M., Balaji, R., & Vinothkumar, M. (2018). Nanoparticles-coated glass fibre-based damage localization and monitoring system for polymer composites.
- [6] Inam, F., Bhat, B.R., Luhyna, N., & Vo, T. (2013). Comparison of structural health assessment capabilities in epoxy – carbon black and epoxy – carbon nanotube nanocomposites.
- [7] Alexopoulos, N.D., Bartholome, C., Poulin, P., & Marioli-Riga, Z. (2010). Structural health monitoring of glass fiber reinforced composites using embedded carbon nanotube (CNT) fibers.
- [8] Sky Spring Nanomaterials, Inc. (n.d.). MWNTs 95% 10-20nm. Retrieved from <https://www.ssnano.com/inc/sdetail/mwnts-95--10-20-nm/872>
- [9] Norplex-Micarta. (n.d.). NP130HF – Epoxy Glass Sheet. Retrieved from <https://www.norplexmicarta.com/grades/np130hf/>