PROJECT REPORT

Monitoring Strain Using Carbon Nanotube Thread
Project # 3: Nanostructured Composite Materials

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Project # 3: Nanostructured Composite Materials:
Monitoring Strain Using Carbon Nanotube Thread

ABSTRACT

Carbon nanotubes (CNT) are excellent materials due to their high electrical, thermal, and mechanical properties; they are more flexible than plastic, weigh less than aluminum, they are stiffer and stronger than steel, and conduct electricity better than most metals. A thread spun from an array of CNTs could be used as a sensor to determine the strain, damage and failure in composite materials. Composite materials are commonly used in high performance structures where there is a need for a high stiffness and high strength at a low weight penalty. While these materials have incredibly high stiffness and strength in the plane of the laminate, cracks, delamination and other interlaminar failure modes could lead to their premature failure. The objective of this project is to evaluate the ability of carbon nanotube thread sensors to continuously monitor the state of strain in composite materials. The carbon nanotube thread is incorporated into the composites without altering the structure of the material and provides the state of strain in real time. The operation of the sensors is governed by electrical impedance spectroscopy. Several sensor configurations will be explored including short and long straight continuous sensors. The micron-size sensor threads will be integrated into composite materials (to form self-sensing composite materials) and the mechanical and electrical response of the composite material will be characterized. This transformational research is a first step in producing an integrated distributed sensor technology that will enable robust real-time health monitoring and condition-based maintenance of aerospace and other high-performance structures.

KEYWORDS: composite materials, carbon nanotube thread, sensors, strain, electrical impedance spectroscopy

1. INTRODUCTION

Often our understanding of the characteristic properties of materials is limited to density, and other basic parameters. However, when researching new materials, one must also consider their mechanical response; this involves determining or calculating the stress (σ) and the strain (ε). The stress-strain relation constitutes the mechanical response of the material and is determined by: (i) the elastic modulus (E) which is the tangent of the stress-strain curve in the initial linear regime; (ii) the ultimate stress of a material, which is the maximum force over a given area necessary to break the material; (iii) the ultimate strain or ductility. Strain is a non-dimensional quantity that is given by that is the total displacement of a material divided by its initial length, $\Delta l/l$, and the maximum strain defines how far it will stretch before it finally breaks. The electrical response of a material is typically given by the electrical conductivity, which defines the ability for a material to conduct electricity. Its inverse is the electrical resistivity, $\rho=RA/L$, where R is the resistance, A is the cross-sectional area, and L is the length; the higher the resistivity the less electron current a material is able to conduct.

Carbon nanotubes are nanoscale materials, ideal for use in composites, due to their very high surface area, very high strength and stiffness, very high ductility, thermal and electrical conductivity. Their drawbacks, however, are that they cannot be created in consistent quality, and they are worth their weight in gold. The potential of these materials far outweighs the drawbacks and they open up vast new opportunities for their use in many engineering applications.

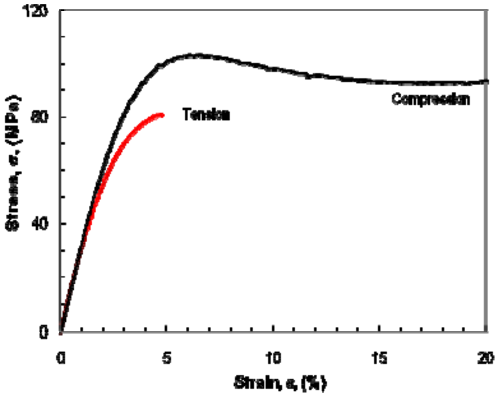


Figure 1. Stress- strain behavior of epoxy polymer under tension and compression respectively. *Note: the slope (E) is the same for both tension and compression, and strength and ductility are higher under compression*

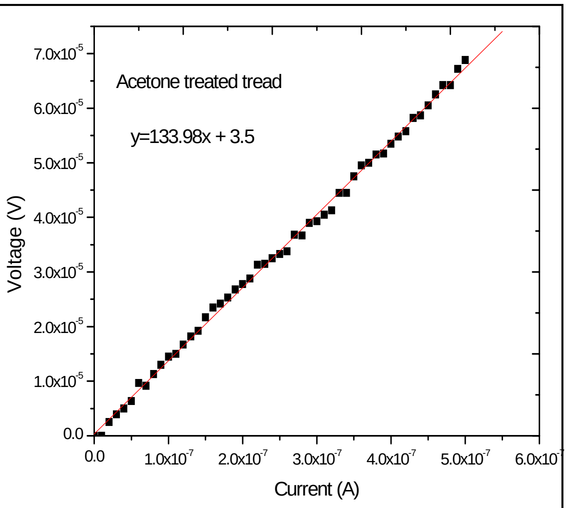


Figure 2. Voltage-current curve obtained by four- probe measurement of a CNT thread used to determine resistivity

2. LITERATURE REVIEW

Non-destructive evaluation (NDE) methods like ultrasound, laser vibrometry, C-Scan, X-ray, eddy current, or thermal wave imaging could detect the presence of hidden damage like corrosion, inner layer cracks, inner layer delaminations, and fatigue fractures. Many approaches for in-situ structural health monitoring (SHM) have also been proposed in the literature to provide more frequent monitoring for damage. The methods include vibration analysis, fiber-optic strain measurement, and stress wave propagation techniques. These techniques for in-situ SHM are not cost-effective for application to large structures, which have complex geometry and high feature density. Carbon nanotube, carbon nanotube thread and other carbon nanostructured materials constitute a new approach for sensing that can make SHM practical. Abot et al. are using carbon nanotube thread for sensing strain and monitoring damage in composite structures. The main advantages of CNT thread sensors are their small size, which allows embedding them into the structure without altering the strain and stress distribution that are being measured (Abot, 2009).

3. GOALS AND OBJECTIVES

The objective of this project is to evaluate the ability of carbon nanotube thread sensors to continuously monitor the state of strain in composite materials.

4. RESEARCH STUDY DETAILS

4.1 Background and introduction to Mechanics and Materials Engineering

Discuss concepts related to composite materials, carbon nanostructured materials, and sensing approaches.

4.2 Fabrication of carbon fiber composite with Toolfusion epoxy

Purpose: to create a sample for analysis of the makeup of a composite

- Toolfusion epoxy 10:2 ratio and woven 6k tow carbon fiber material
- Using a rotary blade, precisely cut 8 squares of woven carbon fiber using a pattern
- The woven fiber was saturated in epoxy layer by layer and stacked in a cork-dam mold. The mold was then put under a vacuum and pressed at 250 ° F
- Heat cures for 4 hours under these conditions

4.3 Measurement of Strain and Resistance in the Composite Sample Under 3-point Bending Loading

Purpose: to determine change in resistance and strain of a CNT thread embedded in polymer epoxy, through testing under 3-point bending loading

Result: A notable change in resistance did occur while stressing the sample with a load. The resistance increased as the stress increased and decreased as the load was removed.

4.4 Review Status of Experimental Project

Discuss scope of Project with Dr. Abot and design experiment: The goal of this project is to evaluate the ability of carbon nanotube thread sensors to continuously monitor the state of strain in composite materials. To do so we will fabricate a composite sample using 2 lengths of nanotube thread for sensing and a polymer matrix epoxy in which we will cast the sample.

4.5 Mechanical and Electrical Characterization of Composite Sample with Corresponding Analysis (Samples 1,2,3,4)

Purpose: Mechanically and electrically characterize composite sample instrumented with 2-thread sensor to evaluate the ability of CNT thread to work as a real-time strain sensor

A new polymer only sample (1) was created using tool fusion epoxy polymer. The resulting cured sample had too many bubbles and was unable to be used (Fig 13; Ap III.).

Two more samples were prepped, testing the initial resistance of the threads before embedded in epoxy, as well as their length and diameter. The first of the two (sample 4) was incorporated in a 3-part epoxy, but had too many bubbles, so the second sample (3) was prepared. This sample had bubbles but not to the extent of the previous two.

4.6 Mechanical and Electrical Characterization of Composite Sample Under Cyclic Loading with Corresponding Analysis

Purpose: To determine effect of number of cycles and stress level on CNT thread

Sample III was retested for resistance consistency and subjected to 4-point bending with the threads being stretched as the sample bent around the thread, thus shortening the threads. The maximum load was calculated for 30 MPa and the test was initiated. About 25 seconds into the test the sample snapped in half. Change in resistance and strain were recorded over time and the data was imported into the computer.

Conclusion: The samples had large numbers of bubble defects that were attributed to the old hardener used in the 3 part epoxy. It was determined that the initial calculations for load was incorrect due to a mistake with units, the load was miscalculated by a magnitude of 103. It was also noted that the Lab view software was collecting data points every 1/100th of a second resulting in an unmanageable 67k data points. It was decided that new samples would be created when a new batch of hardener arrived, and samples would be tested with a σ of 30MPa. The sample would be oriented as demonstrated in Fig. 3 so as to lengthen rather than shorten the threads. The Labview data collection software would be reconfigured to take one data point per second.

4.7 Fabrication of Composite Sample Using New Methodology and Thread Characterization (Samples II, III and IV)

Purpose: set CNT thread onto Teflon fabric and characterize mechanical and electrical properties of the thread alone, and then cast in a 3-part Epoxy for cyclic loading

Methods: sections of CNT thread were secured to a Teflon fabric using conductive epoxy in a patterned area drawn to fit in a silicon casting mold (Fig 3, 4). The CNT threads were cured for 2 hours and lengths and resistances were tested.

The cured samples were examined under the microscope to determine the diameter of the thread and detect anomalies. Sample IV was noted to have numerous thread anomalies, and it was decided that the sample was not used in cyclic loading (Fig 14,15; Apx III).

An epoxy polymer was mixed 100% GY6010 resin, 90% ARADUR 917 hardener, and 1% accelerator and allowed to degas at room temperature for 2 hours at -28PSI for 2 hours.

Sample II and III were then set into a Teflon mold using spray epoxy and cast in 3-part epoxy. Samples were heated to 150° C (302°F) and cured for 2 hours (Fig 19-22; Apx III)

Result: The samples have a large number of bubble defects in the matrix, and may not be suitable for testing; however, it was decided to try the samples to get initial data.

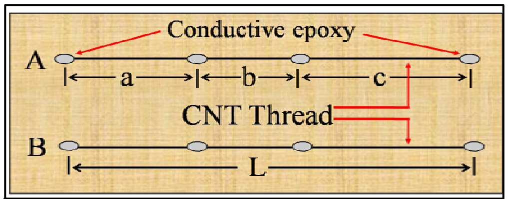


Figure 3: Sample schematic demonstrating the layout of the CNT thread and its bonding points on Teflon

Figure 4. CNT threads bonded to Teflon fabric using conductive epoxy

4.8 Characterization of New Sample Under 4-point Bending Cyclic Loading (Samples II, III and IV)

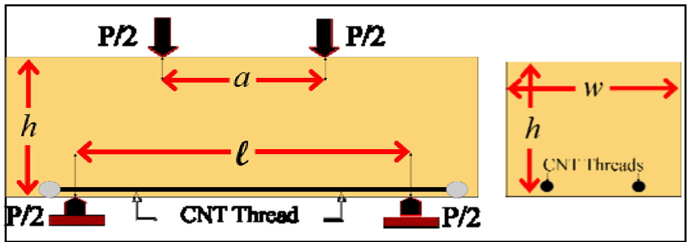


Figure 5: Diagram of 4 point bending, side and end view of composite sample

Purpose: the composite sample is subjected to repeated loading (fig 5) with 10, 15 and 20 MPa to determine if the CNT thread is sensitive to changes in stress.

Sample contains a large number of bubbles and holes. These defects can dramatically decrease the elastic modulus of the material so the initial stress was set for a maximum of 10 MPa. The first run did not have a maximum load defined due to a mistake setting the program; however, the sample reached a large total load before it was caught and there were no breaks. The sample was then stressed in cyclic 4-point bending (Fig 5) under increasing load, and the data was collected for resistance, load and stress, and analyzed.

4.9 Fabrication of Composite Ssample Using New Methodology and Thread Characterization (Samples V, VI, VII and VIII)

Fabrication of CNT thread sample was repeated as above with new hardener, and when samples were adhered to the mold it was noted that the spray epoxy that was used that left bubbles along the Teflon fabric. And attempt was made to remove the bubbles before casting in polymer matrix. Samples were mechanically and electrically characterized. (Fig 6)

The mold was preheated and pressurized to bond the mold to the base and the 3 part Epoxy was degassed for 2 hours. The degassed polymer matrix was poured into the mold and heat cured at 150C for 4 hours.

Despite the attempt at removing the bubbles it was still noted that there were a large number of bubbles in the sample. It was decided that these would not be used, and the next set of samples would not be bonded to the base with spray epoxy.

Figure 6. Image of CNT thread bonded to Teflon fabric with diameter measurements

4.10 <u>Fabrication of Composite Ssample Using New Methodology and Thread Characterization</u> (Samples IX-XII)

These samples were prepared using tape to fix the threads to the fabric before setting with conductive epoxy. This kept the threads strait, tight and prevented slipping while applying the conductive epoxy as seen in Fig. 4. Samples were tested for resistance and fixed in the mold with a surrounding seal of permabond adhesive, rather than the spray epoxy and heat and pressure cured while the 3-part polymer was degassing. It was noted that there were no bubbles when the polymer was added to the mold.

4.11 Mechanical <u>and Electrical Characterization of Composite Sample Under Cyclic Loading with Corresponding Analysis</u> (Samples IX-XII)

There were a few small bubbles around the edge of the samples that were removed by planning the edges. These samples exhibited no visual defects and were subjected to cyclic loading of 30MPa and the resistance, stress and loading data were collected, imported into excel and analyzed (Fig 7,8).

5. ANALYSIS; RESEARCH RESULTS

5.1 Experimental Program

The Data from sample 3 was used as a basis for understanding how the process of three point bending was used to assess the relationship between stress and strain and demonstrate the effects of changing strain on the resistance of a CNT thread in a polymer composite sample. From the analysis of sample 3 we determined that it would be better to use 4 point bending to create a section with no smear and simplify analysis.

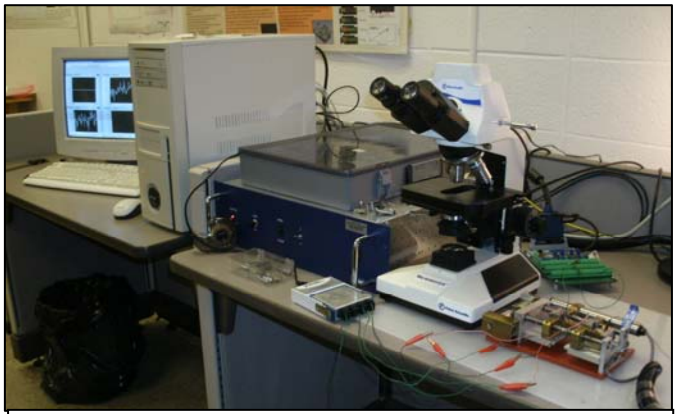


Figure 7: Apparatus for analyzing stress and resistance in bending load for composite sample

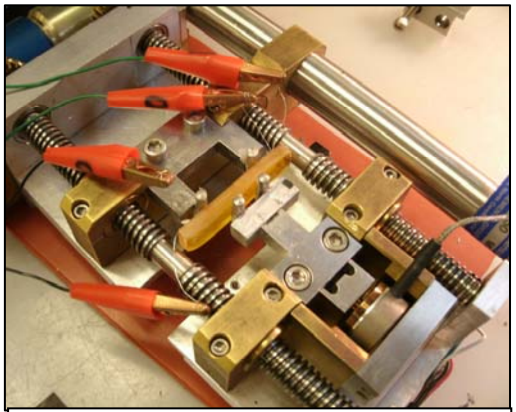


Figure 8: Sample in 4-point bending machine.

5.2 Nomenclature

L = Length (thread) [mm]
w = Width (composite) [mm]
h = Height (composite) [mm]
y = Height/2 [mm]
ℓ = Distance between supports (bending) [mm]
a = Distance between loading points (bending) [mm]
M = Moment [Nm]
E = Elastic modulus [Pa]
I = Inertia [mm^4]
P = Load [N]
ε = Strain
σ = Stress [Pa]
R = Resistance [ohm]
ρ = Resistivity [ohm*cm]
A = Cross sectional area (CNT thread) [mm^2]
F = Gage factor

5.2 Formulation:.

Below is given a list of constants and variables used in the analysis of the data

$$I = \frac{w * h^3}{12} \tag{1}$$

$$M = \frac{P * (l - a)}{4} \tag{2}$$

$$\sigma = \frac{M}{I} * y \tag{3}$$

$$\varepsilon = \frac{M}{IE} * y \tag{4}$$

$$\varepsilon = \frac{P * (l - a)}{4 * I * E} * y \tag{5}$$

$$F = \frac{1}{\varepsilon} * \frac{\Delta R}{R} \tag{6}$$

5.4 Data Analysis

This data was obtained from using 4-pt bending on sample XI, on July 24th. The load, displacement, and time came from the bending of the composite material. The resistance for thread A and B came from the resistivity occurring because of the bending.

Table 1: selected results from sample XI

Load (N)	Displ (mm)	Time (s)	Res A (ohm)	Res B (ohm)	Strain
253.81	0.3268	110	2005.95	2127.39	0.00751
259.296	0.3278	111	2006.10	2127.67	0.007673
266.193	0.3298	112	2006.19	2127.93	0.007877
274.407	0.3323	113	2006.42	2128.19	0.00812
282.698	0.3363	114	2006.64	2128.41	0.008365
290.788	0.3403	115	2006.72	2128.92	0.008605
297.902	0.3483	116	2007.10	2129.47	0.008815
303.202	0.3503	117	2007.46	2130.40	0.008972
304.132	0.3514	118	2008.13	2131.20	0.009
303.838	0.3488	119	2008.84	2132.20	0.008991
303.543	0.3463	120	2009.57	2133.09	0.008982
303.187	0.3514	121	2010.16	2133.97	0.008972
302.954	0.3478	122	2010.65	2134.58	0.008965
302.567	0.3478	123	2010.89	2134.89	0.008953
302.396	0.3483	124	2010.84	2134.78	0.008948
302.226	0.3453	125	2010.86	2134.89	0.008943
302.055	0.3458	126	2010.91	2134.76	0.008938
301.838	0.3428	127	2010.86	2134.72	0.008932
301.575	0.3433	128	2010.84	2134.71	0.008924
301.559	0.3403	129	2010.87	2134.79	0.008923
301.311	0.3423	130	2010.85	2134.60	0.008916
301.172	0.3448	131	2010.91	2134.64	0.008912
301.032	0.3458	132	2010.82	2134.63	0.008908

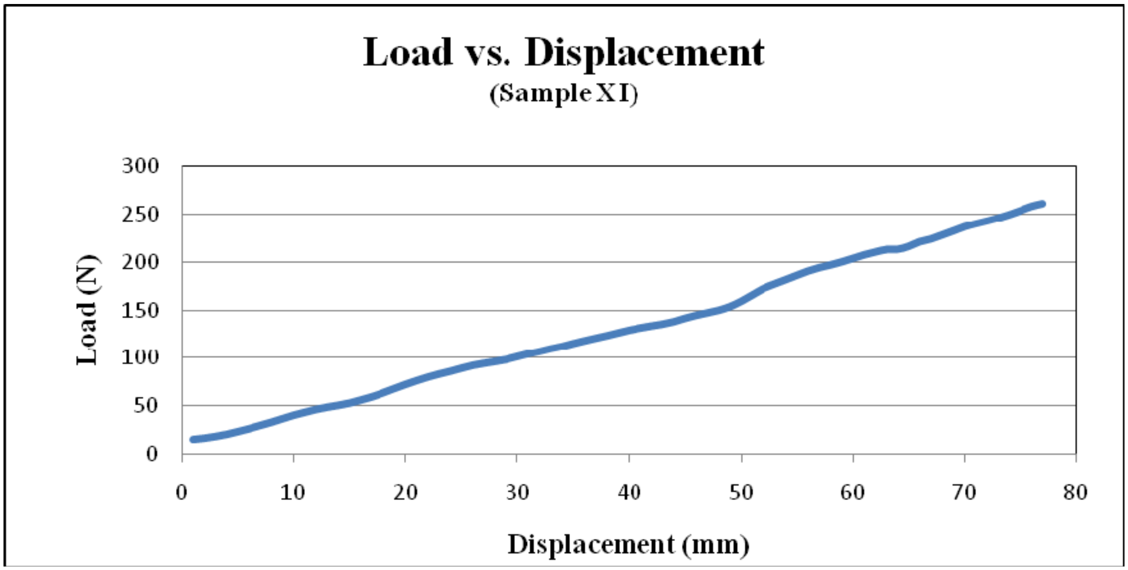


Figure 9: Sample XI, as the force increases the displacement increases in a linear fashion

As the load increases, the displacement increases. It has a linear tendency.

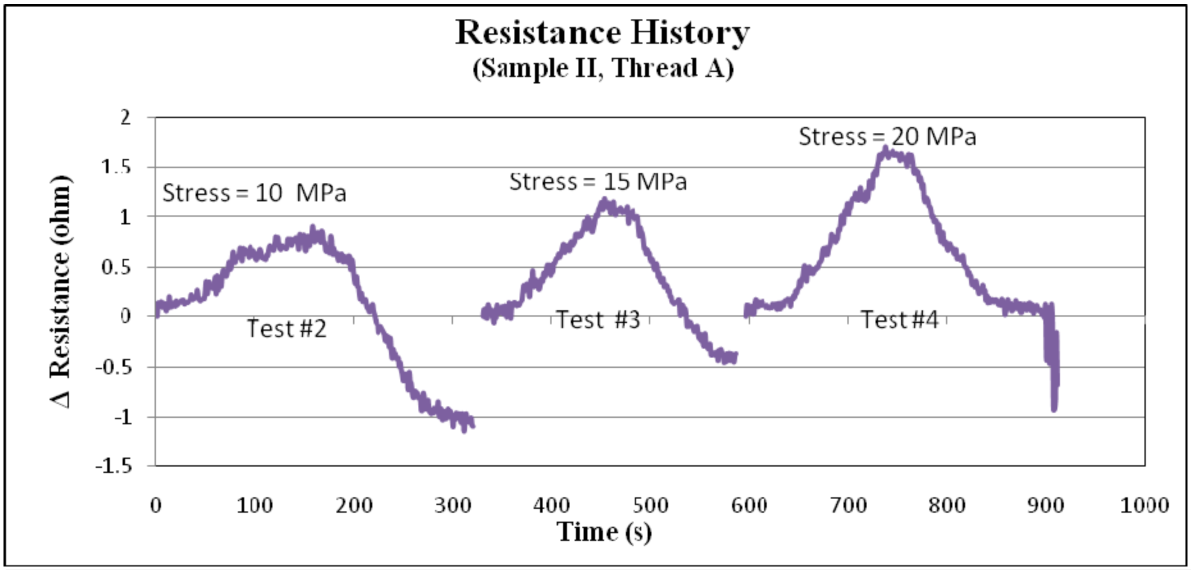


Figure 15: Delta Resistance history for three tests of Sample II; the change in resistance ($R-R_i$) over time in 4-point bending with increased loading over time

The stress increases for the sample over time. Rise and fall of resistance mirrored increase in stress over time.

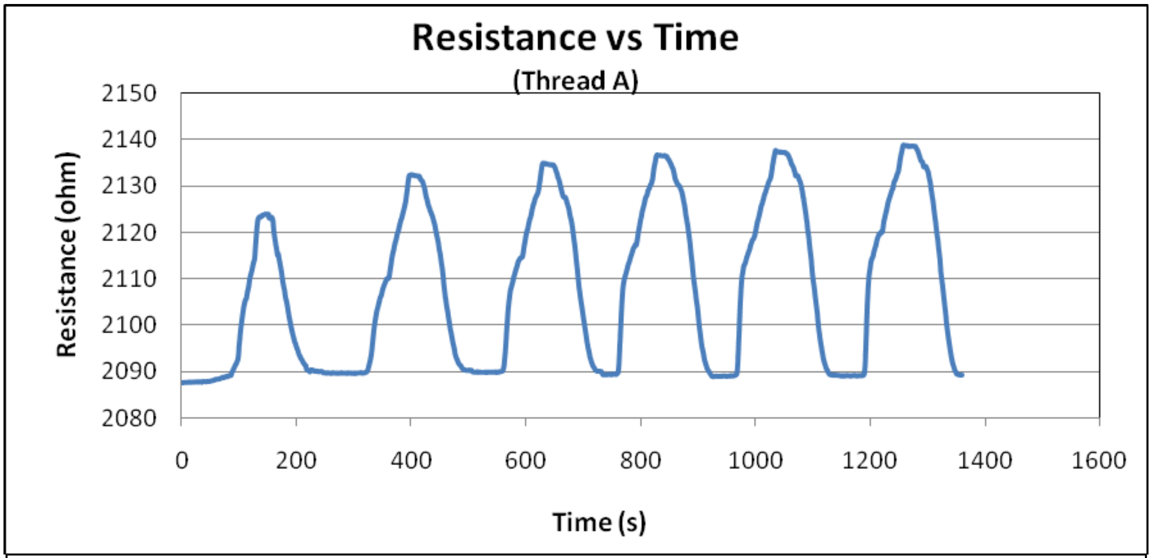


Figure 10: Resistance history over time: Sample XI; cyclic loading of sample with a maximum stress of 30 MPa, demonstrating the change in resistance over time through repeated loading.

This graph depicts several cycles applied to the sample. The resistance returns to where it started.

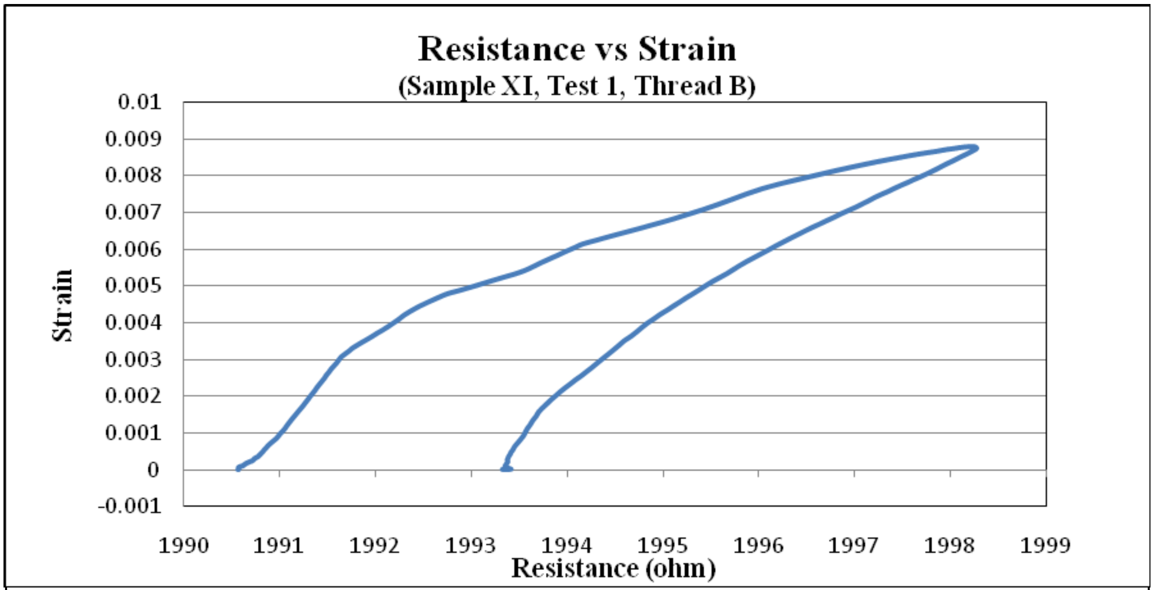
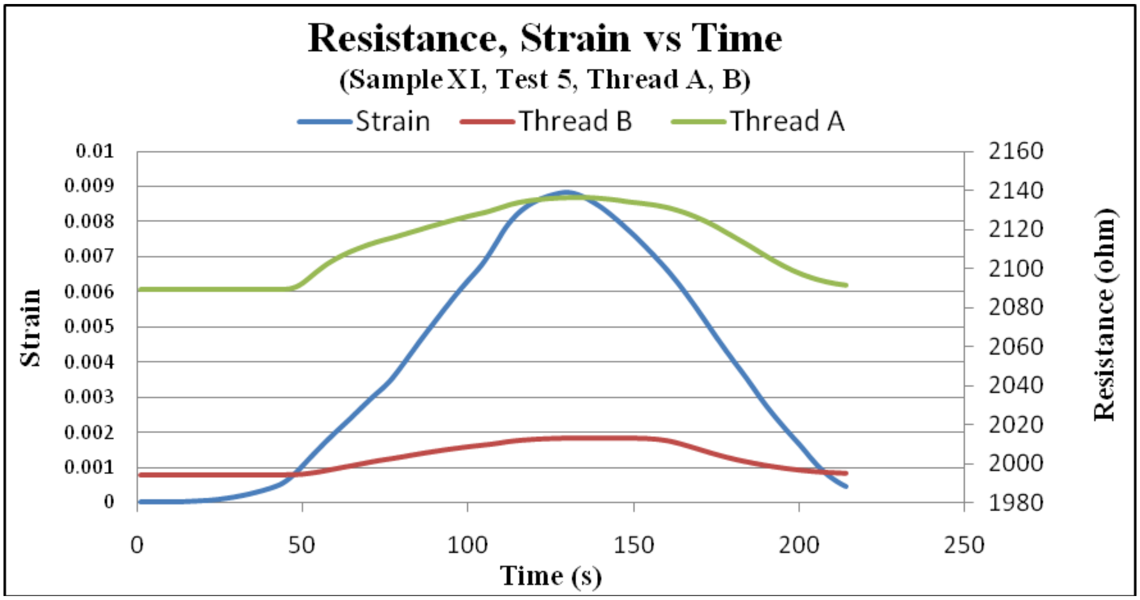


Figure 11: Resistance-Strain for test 1 of Sample XI

This is a large part of our findings because the CNT threads' resistance recovers after each cycle.

As the strain increased and then decreased, the resistance modeled it. The big difference is that



the resistance did not return exactly where it started.

From the graph, as the strain on the sample increased, the resistance of both threads, increased. As it decreased, the resistance followed. This signifies that the CNT threads are sensitive to the strain applied.

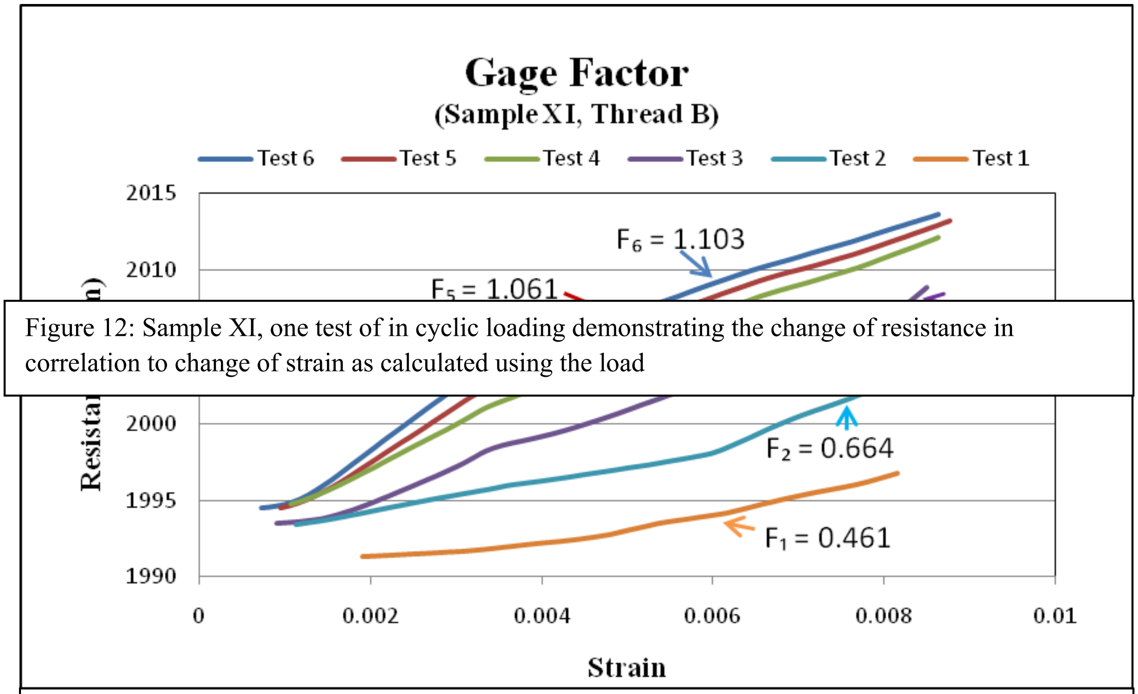


Figure 12: Sample XI, one test of in cyclic loading demonstrating the change of resistance in correlation to change of strain as calculated using the load

Figure 13: Sample XI, Gage factor(F) is calculated $\Delta R/R_{i*}\ \varepsilon$

b

Demonstrated by the increase in gage factor, it is evidenced that as the number of cycles increases, the resistance of the CNT threads increases.

5. CONCLUSIONS

Composite materials were instrumented with CNT thread sensors and mechanically and electrically characterized to evaluate the ability of sensors to continuously monitor the state of strain in composite materials

It was determined by experimentation that:

1. Resistance (resistivity) is recovered after one cycle
2. Strain Resistance curve exhibits a quasi-linear response
3. CNT thread is sensitive to the stress level in the material
4. Sensitivity of CNT thread increases slightly with number of cycles

6. RECOMMENDATIONS FOR FUTURE WORK

Studies will continue to explain the observed changes in the gage factor through combined morphology, spectroscopy and mechanical/electrical characterization of the CNT thread. This information will also be used to improve the CNT thread fabrication process.

8. ACKNOWLEDGEMENTS:

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Carbon Nanotube Thread: V Shanov PhD and M Shultz PhD

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9. BIBLIOGRAPHY

1. Daniel, I. M. and Ishai, O. *Engineering mechanics of composite materials*, *2nd ed.*, Oxford, New York, NY (2006).
2. Hyer, M. W. *Stress analysis of fiber-reinforced composite materials,* McGraw-Hill, New York, NY (1998).
3. Abot, J. L., Song, Y., Schulz, M. J. and Shanov, V. N. Novel carbon nanotube array-reinforced laminated composite materials with higher interlaminar properties. *Compost. Sci. Technol.* 68 (13): 2755-2760 (2008).
4. Kulkarni, M., Carnahan, D., Kulkarni, K., Qian, D. and Abot, J. L. Elastic response of a carbon nanotube reinforced fiber polymeric composite: a numerical and experimental study. *Compos. Part B-Eng.* (submitted).
5. Mallik, N., Schulz, M. J., Shanov, V. N., Hurd, D., Chakraborty, S., Jayasinghe, C., Abot, J. L. and Song, A. Study on carbon nano-tube spun thread as piezoresistive sensor element. *Adv. Mater. Res.* 67 (13): 155-160 (2009).
6. Kang, I., Maheshwari, G., Yun, Y. -H., Shanov, V., Chopra, S., Abot, J., Choi, G., Schulz, M. J. Nanoengineering of Sensory Materials, *Encyclopedia of Structural Health Monitoring*, edited by C. Boller, F. -K. Chang, and Y. Fujino, Wiley (2009).
7. Private communication with Dr Jandro Abot, July 2009

APPENDIX I: NOMENCLATURE

L = Length (thread) [mm]
w = Width (composite) [mm]
h = Height (composite) [mm]
y = Height/2 [mm]
ℓ = Distance between supports (bending) [mm]
a = Distance between loading points (bending) [mm]
M = Moment [Nm]
E = Elastic modulus [Pa]
I = Inertia [mm^4]
P = Load [N]
ε = Strain
σ = Stress [Pa]
R = Resistance [ohm]
ρ = Resistivity [ohm*cm]
A = Cross sectional area (CNT thread) [mm^2]

11. APPENDIX II: RESEARCH SCHEDULE (or Time schedule)

Table 2: Weekly research schedule

Tasks (corresponding to specific objectives)	**Week 1**	**Week 2**	**Week 3**	**Week 4**	**Week 5**	**Week 6**
(a) Research Background	🟦	🟦				
(a) Characterization of CNT thread		🟥	🟥	🟥	🟥	
(b) Fabrication of Composite Sample		🟥	🟥	🟥		
(c) Mechanical / Electrical Characterization		🟥	🟥	🟥	🟥	🟥
(d) Data Analysis				🟩	🟩	🟩
(e) Report/Presentation Preparation				🟩	🟩	🟩

APPENDIX III:

13.1 Anomalies

Figure 14: Toolfusion sample; note: this sample contained a large number of bubbles and was not suitable for testing

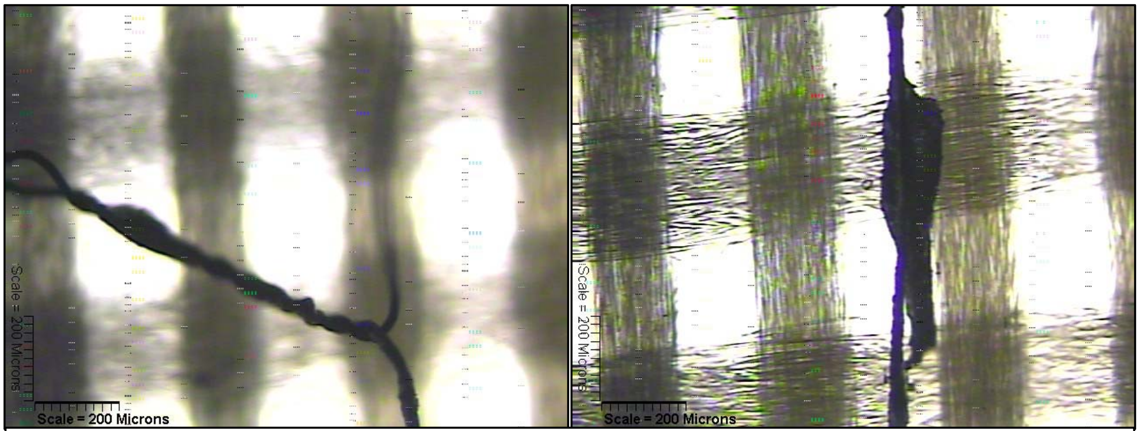

Figure 15a,b: loop and thread composition anomalies seen in fabrication process

3.1 Fabrication of Sample:

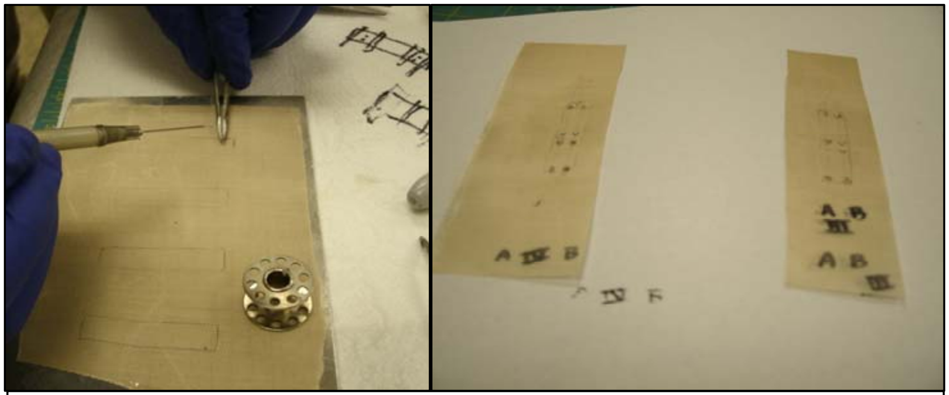

Figure 16a, b:CNT threads bonded to Teflon Fabric are sealed into a Teflon mold for casting

13.2 Electrical and Mechanical Characterization

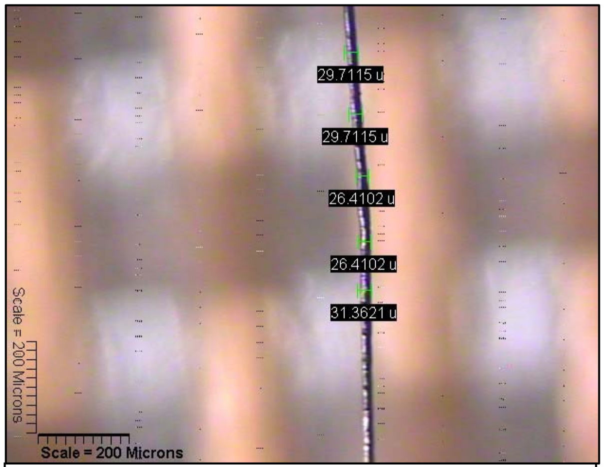

Figure 17: Thread is Viewed at 10x and the diameter is measured

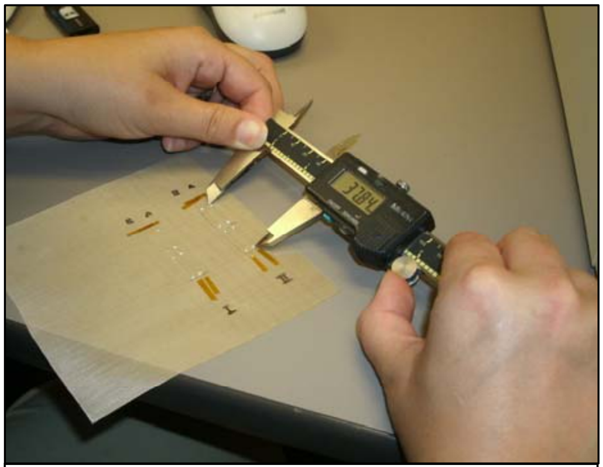

Figure 18: Thread length and resistance are measured

13.3 Polymer Epoxy Casting

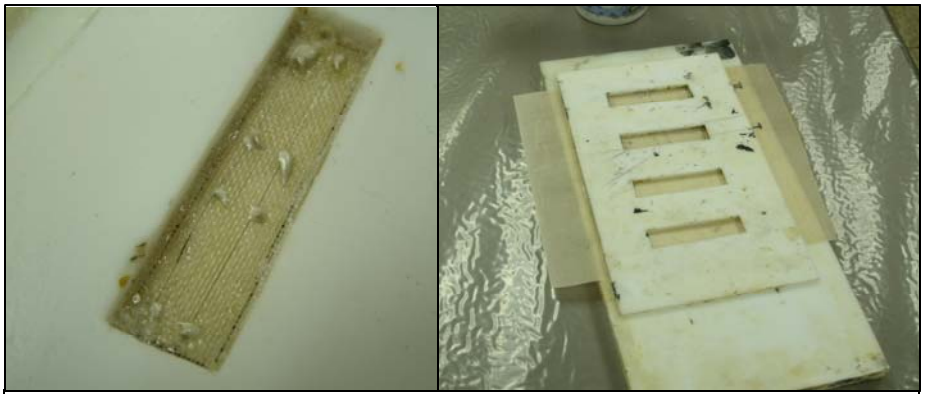

Figure 19a,b: CNT threads bonded to Teflon Fabric are sealed into a Teflon mold

Figure 20: Mold is heat cured (50°C) for 2 hours under pressure in Press Mo

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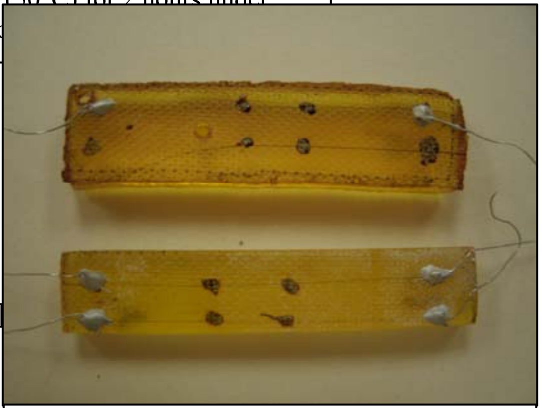

Figure 22: Heat cured composite samples are removed from molds, planed and conductive wire leads are attached with conductive