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ELASTOMERIC EVOLUTION: A NEW LOOK AT CARBON NANOTUBE REINFORCED ELASTOMERS

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ABSTRACT

The use of elastomers in the rotating reference frame of helicopters is pivotal in the dissipation of vibration as a means of mitigating fatigue damage. Three of the largest cost drivers for elastomers from the perspective of design, maintenance and warranty are dampers, bearings, and hydraulic systems. One of the primary modes of failure of these components is degradation of elastomeric materials. To address these failures, elastomer compounds have been improved in conjunction with carbon nanotubes (CNTs) and structural health monitoring (SHM) technologies. Early work[1],[2] on embedding CNTs in elastomers focused on strength and electrical effects including modulus, conductivity and electro-magnetic interference (EMI) shielding properties. In some of the most recent work,[3],[4] CNTs have been dispersed into viscous materials or spun into threads and used to measure strain in composite materials to functionalize CNTs embedded in elastomers. The CNTs used to form CNT thread originate in sheet form. This configuration or a variant of it would be ideal to use in hyperelastic materials such as elastomers due to its ability to undergo large deformations and maintain contact upon return to its original geometry. CNTs, when used as electrical sensors, also can enable material state awareness through SHM with the added benefits of improved strength properties and EMI shielding. The ability to sense strain using CNTs in the form of CNT thread is demonstrated on composites, the need to experimentally determine mechanical material properties of CNT elastomers is established through large variations among existing homogenization methods and the ability to detect damage in specific directions and monitor load with conductive elastomers is exhibited.

NOMENCLATURE

α	Material phase (matrix or inclusion)
ε_{kl}	Strain in kl direction
σ_{ij}	Stress in ij direction
c_α	Volume fraction of the particular phase
c_i	Volume fraction of the inclusion phase
c_m	Volume fraction of the matrix phase
CNT	Carbon Nanotube
C_α	Elasticity tensor of material phase
C_I	Elasticity tensor of inclusion
C_m	Elasticity tensor of matrix
C_{ijkl}	Fourth order elasticity tensor
$C^*_{(MT)}$	Mori-Tanaka effective elasticity tensor
$C^*_{(Reuss)}$	Reuss effective elasticity tensor
$C^*_{(Voigt)}$	Voigt effective elasticity tensor
DIC	Digital Image Correlation
E	Elastic Modulus
EMI	Electromagnetic Interference
R_o	Original Resistance
R_1	Resistance measurement in compression
R_2	Resistance measurement in tension
TRL	Technology Readiness Level

1 INTRODUCTION

Elastomers are used extensively in rotorcraft, especially in the rotating reference frame, where their applications are most critical. Rotorcraft and these elastomers experience one of the harshest environments imaginable including extreme weather, vibration and combat hazards in military applications. Billions of dollars are spent annually on detection and prevention of failure of critical rotorcraft components; among the most frequent failures are those involving bearings,[5] bushings, dampers and seals, all of which utilize elastomers.

Current detection and prevention methods rely primarily on maintenance crews and daily inspections, which are costly and time consuming. Even the most advanced decision support systems such as Health and Usage Monitoring Systems (HUMS) rely on vibration data and post processing and tend to be reactive rather than proactive, requiring clues provided by previous component failures to predict impending failures across a fleet.

One current area of active research is Integrated Structural Health Management, which utilizes structural health monitoring systems to detect and even predict component failures before they occur so that preventive maintenance actions can be taken to reduce unscheduled maintenance and improve aircraft availability.[6] Technologies being developed to address these needs include energy harvesting, wireless sensing and data analytics.

Carbon nanotubes (CNTs) are simultaneously being developed for use in other parts of the airframe, e.g., fuzzy fibers to improve the matrix/fiber interface. Additionally, CNTs are being used to improve the interfacial strength between plies of composites. CNT thread has also been developed to measure strain on composite materials.[7] Despite all of these advancements and active areas of research very few of these technologies have been considered or directly applied to elastomers. While elastomers are often times considered secondary structure, their failure often can and does lead to failure of primary structures.[8]

A concept that is currently in development that pulls from aforementioned technology development efforts is the CNT reinforced elastomer. The development of a CNT reinforced elastomer stems from the design of an advanced strain energy accumulator.[9] The advantages of CNT reinforced rubber include improved strength properties, EMI shielding, strain sensing capability, and the potential for functionally graded elastomers.

1.1 Early CNT Elastomer Work

In the early 2000's preliminary work was done to embed CNTs in various rubber materials at various volume fractions and weight percentages. The focus of these studies was to investigate the general effect on material properties including strength, conductivity, and electromagnetic shielding ability.[1],[2] At this time, carbon nanotubes were new, at a low technology readiness level (TRL), costly and were not produced in large quantities; it was also not yet possible to functionalize CNTs.

1.2 Current Measurement Techniques

Several different measurement and sensing techniques exist for obtaining material properties for elastomers but each has drawbacks. The standard load frame used in determining strength properties is accurate but lacks the ability off-the-shelf to monitor the material in situ. Standard foil strain gages are a reliable measurement technology but are subject to disbonding and also fail under the hyperelastic deformations experienced by some elastomers.

Additionally, strain gages are typically surface mounted and difficult, if not impossible in most applications, to mount inside structures. Digital Image Correlation (DIC) is another commonly used measurement technique that uses a speckle pattern to track displacements of the material. DIC has its own challenges and drawbacks including in situ monitoring, the required speckle paint pattern that would not be feasible in most military rotorcraft applications, and the tendency of the paint specs to disbond from the elastomeric materials in high strain or hyperelastic deformation situations.[10]

2 CARBON NANOTUBE ELASTOMERS

A material that has the potential to address these challenges and provide in situ material data for structural health monitoring of elastomers is CNT embedded elastomers. Kang and Schulz et al. first reported on the use of CNT in polymers for strain sensing but were limited at the time by the length of the CNTs.[4] One of the first reported uses of a flexible CNT sensor was by Yamada et al. in 2011 for human-motion sensing.[11] Since then Song et al. successfully utilized long, vertically aligned CNTs as shown in Figure 1 to create CNT thread for strain sensing in composites.[7]

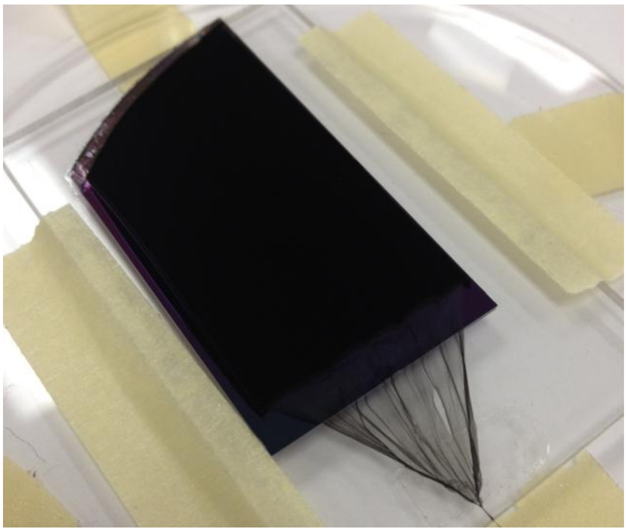

Figure 1: Vertically aligned spinnable CNTs.

The key findings of each of these prior works suggest the concept of a CNT embedded elastomer utilizing vertically aligned spinnable CNTs for in situ structural health monitoring of rotorcraft parts.

2.1 Carbon Nanotube Embedded Elastomers

Early investigations of CNT elastomers were focused on the mechanical and electrical properties and not on functionalizing the CNTs within the material due to the purity and size limitations of early CNTs. Figure 2 shows the effect that the addition of CNTs has on the material properties of natural rubber, which is an elastomer.

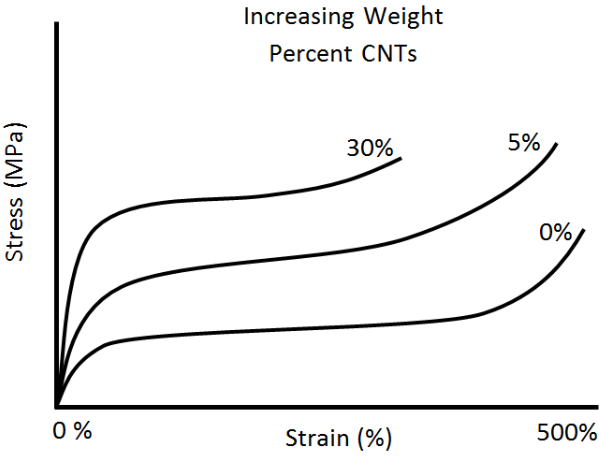

Figure 2: Effect of increasing weight percent of CNTs on rubber modulus. Adapted from [1].

It is observed that as the CNT percent increases, both the strength and elastic modulus increase as one would expect while the maximum strain percent obtainable decreases along with the toughness. In addition to improved strength properties, a number of other material property advantages exist for CNT embedded elastomers. Figure 3 shows the Electro Magnetic Interference (EMI) shielding capability of CNTs embedded in rubber that is of particular interest in military and shipboard rotorcraft applications.

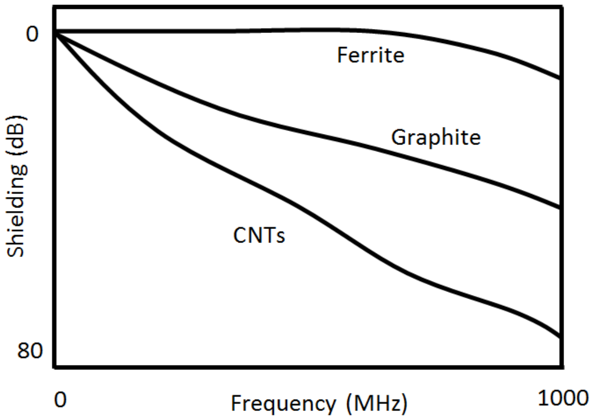

Figure 3: EMI shielding ability of carbon nanotubes in rubber at 30 wt%. Adapted from [1].

The ability for CNT embedded elastomers to be used for strain sensing and functionally graded materials is considered here in the context of rotorcraft elastomeric components.

2.2 CNT Embedded Elastomer Components

Degradation of elastomeric components can lead to sudden and catastrophic failure in the vibration intensive environment in which rotorcraft operate. Early detection of the degradation of elastomeric components is difficult in operation. In a ground test environment, detection is aided in the visual clues left behind in the form of pieces or residues from the degrading component. It is more challenging to detect early degradation in a flight test as the visual clues from the material that are shed are lost to the operating environment. With additional sensors and manpower available in flight tests, degradation can still be detected.

In daily operations, when maintainers perform inspections during regular intervals, and are in charge of a portion of a fleet of aircraft, early detection is challenging and early indicators of degradation can be missed. With the addition of vertically aligned CNTs in elastomers to enable strain sensing capability, early detection is possible and reductions in unscheduled maintenance or worst case scenario mishaps can be avoided.

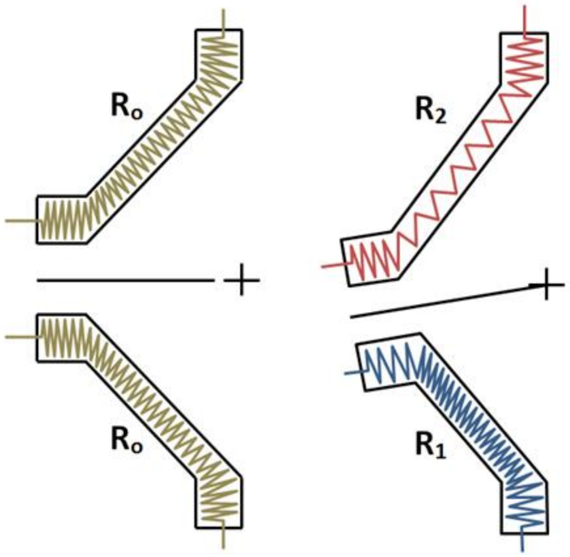

Figure 4: Cross sectional view of focal bearing elastomer with vertically aligned CNT embedded elastomer (a) initial/unstressed (b) final/stressed

Figure 4 shows a representative elastomeric component, i.e. a focal bearing, bushing, or bearing seal, with a vertically aligned CNT sheet embedded with the ends spun to form CNT thread coming out of the component serving as leads and having initial resistance, R_o. As a load is applied, the component deflects and the resistance changes to experience a compressive load/resistance, R_1, and a tensile load/resistance, R_2. As the component degrades the connections between the CNTs may be interrupted and result in a change in the initial, unstressed resistance of the material. This functionalization of the CNTs within the rubber could add the capability to monitor loads in the component and detect early signs of failure.

Similarly for functional grading of materials, vertically aligned CNT sheets can be replaced with high purity CNT powder. Again using Figure 4 as an example, as the component is loaded CNTs can be dispersed to certain regions of the material depending on the load profile and where an increase, or decrease, in stiffness would be of benefit to the component.

3 DISPERSION OF CARBON NANOTUBES IN VISCOUS SUBSTANCES

Dispersion of CNTs has been an area of research for some time and has focused on polymers, elastomers, and concrete. Lessons have been learned and new techniques developed to achieve more uniform dispersion enabling improved functionality of CNTs

3.1 Carbon Nanotube Dispersion in Polymers

Early work performed by the University of Cincinnati[4] focused on dispersion of CNTs in polymers for sensing capability, which utilized ultrasound and shear force mixing methods. One of the issues faced at the time was the inability to grow long CNTs, thereby limiting the ability to utilize them for measuring mechanical and electrical properties. While shear force mixing is the simplest, it is limited to primarily coarse dispersion where ultrasound can improve dispersion on a nanoscale. A drawback to ultrasound is that it can damage the nanotubes during the dispersion process.

3.2 Carbon Nanotube Dispersion in Concrete

The conventional approach to incorporate CNTs in concrete is to first disperse the CNTs in the mix water using a combination of superplasticizer and ultrasonication and then mix the resulting dispersion with the cement powder. Work performed at Vanderbilt University[12] has shown that while the use of polycarboxylate-based high range water reducer can help in the dispersion of CNTs in concrete, there is still evidence of the formation of sub-micron and micro-scale CNT clusters and non-uniform arrangement of individual CNTs within the cement paste leading to CNT rich and CNT poor regions. It has been demonstrated that the quantification of the agglomeration state of the CNTs by optical microscopy observation and image analysis is a useful tool to understand the structure-property relationship of concrete containing CNTs.

3.3 Magnetic Nanoparticle Dispersion

A new method that has been developed for dispersing nanoparticles in materials uses magnetic fields.[13] One of the key findings of Stuyven et al. is that magnetic fields can impact laminar flow profiles and produce velocity gradients that result in varying levels of dispersion. Given the electromagnetic properties of CNTs, magnetic fields should be able to achieve enhanced dispersion enabling functionally graded materials.

4 CARBON NANOTUBE STRAIN SENSING

Carbon nanotube materials are piezoresistive, which means the resistance of the carbon nanotube thread/ribbon changes with strain. The piezoresistive behavior can be used to sense strain and potentially damage in a host structure because local strain fields are influenced by mechanical damage. The sensitivity of strain sensing depends on the length of the sensor because average strain is measured over the length of the sensor. Carbon nanotube sensor threads are barely visible and do not add significant weight to the host composite structure.

4.1 Carbon Nanotube Thread Strain Sensing

A carbon nanotube forest was synthesized and spun into carbon nanotube sensor thread with a diameter of 20μm in the Nanoworld Laboratory at the University of Cincinnati. The sensitivity of the sensor thread was compared to the strain measured from a regular strain gage. Both sensors were bonded onto a 4-ply symmetric cross-ply laminated IM7 composite coupon for strain measurement. The tensile stress versus strain and resistance change versus strain were plotted. The gage factor of the carbon nanotube sensor thread was 0.91. The relative strain measurement error between the carbon nanotube sensor thread and regular strain gage was 5.9%.[7]

Unlike strain gages, the carbon nanotube sensor thread could be integrated into the inner plies of a laminated composite and will not affect the integrity of the host composite structure. Therefore, carbon nanotube sensor thread provides a simple and reliable way for strain measurement and damage detection on composite structures on a near real time basis. The ability of CNT thread to measure strain on carbon fiber composites near real time can be extended to strain measurement in elastomers utilizing vertically aligned CNTs embedded in elastomers.

5 MODELING OF CNT ELASTOMERS

Homogenization models are used to extrapolate macroscale properties from their individual molecular scale material properties in this section. A brief overview of the homogenization methods is provided because it indicates a need for experimentally determined material property data. A detailed analysis is given in Cummins et al.[9]

5.1 Homogenization Techniques Estimating the Elastic Modulus of a CNT Embedded Elastomer

All homogenization methods begin with the stress-strain relations given in Eq. (1):

$$\sigma_{ij} = C_{ijkl}\varepsilon_{kl} \tag{1}$$

The homogenized modulus is determined by the effective elasticity tensor, C^*. In the upper limit Voigt homogenization method, the effective elasticity tensor is defined as:

$$C^*_{(Voigt)} = \sum_{\alpha=1}^{n} c_\alpha C_\alpha \tag{2}$$

Where α is the phase (matrix or inclusion), c_α is the volume fraction or the ratio of the volume of the particular phase to the total volume, and C_α is the stiffness tensor of the respective phase. In the CNT reinforced rubber there are only two phases, the CNTs and the rubber, where C_i and C_m are the stiffness tensors for the inclusion and matrix material respectively. The lower limit Reuss homogenization method is:

$$C^*_{(\mathrm{Re}uss)} = \left(\sum_{\alpha=1}^{n} c_\alpha C_\alpha^{-1}\right)^{-1} \tag{3}$$

The homogenized modulus in the longitudinal direction for these two methods and the Mori-Tanaka homogenization method is the C_{11} element of the effective elasticity tensor. The Mori-Tanaka effective elasticity tensor is defined[14] as:

$$C^*_{(MT)} = C_m + \sum_i c_i (C_I - C_m) A_{I(MT),i} \tag{4}$$

Where C_I is the elasticity tensor of the inclusion, C_m the elasticity tensor of the matrix, c_i the volume fraction of the inclusion and $A_{I(MT),i}$ as defined by Klusemann and Svendsen.[14]

5.2 Summary of Homogenization Methods

The estimates of homogenized modulus for CNT rubber vary greatly depending on the method employed.[9] Table 1 shows the large variation in estimated homogenized modulus values for a soft rubber material. The homogenized elastic modulus values in Table 1 are normalized by the elastic modulus of the rubber material. Even at small volume fractions the methods are orders of magnitude apart. The discrepancy between the homogenization methods only increases as the volume fraction of the CNTs increases.

Table 1: Normalized Homogenized Modulus Values

Volume Fraction %	Homogenization Method		
	Reuss	Mori-Tanaka	Voigt
5	1.05	2.42	$1.45*10^3$
50	2.00	7.36	$14.47*10^3$
95	19.99	104.24	$27.50*10^3$

Such large discrepancies among the methods, especially with their dependence on the material properties of CNTs which can vary greatly, point to a need to obtain experimental data on the material properties of CNT elastomer samples.

6 PRELIMINARY TESTING ON METAL RUBBER

A first step in determining material properties of CNT elastomers, while the final formulation and construction is being finalized, is to study those of the most comparable commercially available product which in this case is a material called Metal

Rubber which was used for the test specimens shown in Figure 5.

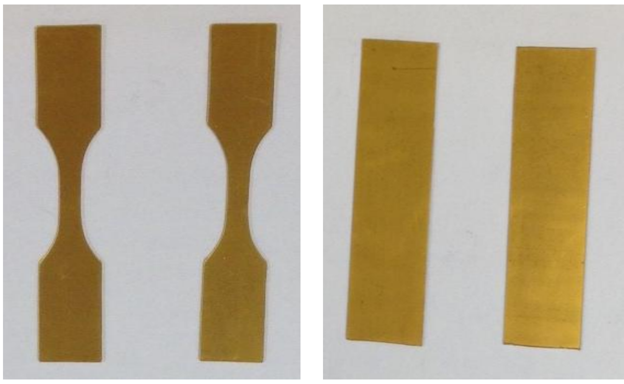

Figure 5: Metal Rubber test specimens: dogbone (left) and rectangular (right)

Metal Rubber consists of rubber and gold nanoclusters and exhibits the material properties of both metal and rubber. Early experiments were performed by Lalli et al.[15] measuring the resistance of the material at varying stress-strain levels. In their work they found that they were able to accurately measure strain levels up to 20% strain but, beyond that, the metal nanoclusters lost conductance paths and resistance saturated at 75% strain. It is believed the vertically aligned CNT elastomers, specifically rubbers in large strain deformation applications, will be able to maintain conductivity due to the large aspect ratio and van der Waals forces between the nanotubes.

One application envisioned for the CNT elastomers that can be tested on the Metal Rubber is the use on bearings and hydraulic seals to test for damage and leakage. In the current body of work, the Metal Rubber rectangular test specimens were measured for resistance values using a Fluke IV Ohmmeter. Three different damage modes were introduced to the metal rubber specimen and are labeled one through three in Figure 6. Damage mode one was a vertical snip using wire cutters at approximately half the vertical dimension of the Metal Rubber rectangle. The second damage mode introduced was a horizontal surface scratch and the third was a vertical surface scratch the entire vertical length of the specimen.

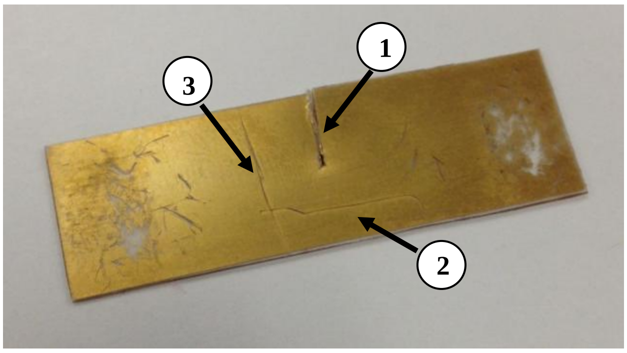

Figure 6: Three damage modes on Metal Rubber

Alligator clips from the Ohmmeter were vertically fixed to a bench to properly hold the specimen and reduce fluctuation in resistance due to unnecessary movement as shown in Figure 7.

Figure 7: Damaged Metal Rubber rectangular test specimen experimental test setup

Table 2 gives the Metal Rubber's change in resistance when physical damage is introduced to the test specimen.

Table 2: Change in resistance with damage

	R_1	R_2	ΔR
Half Snip	18.0	31.3	13.3
Horizontal Scratch	31.3	35.0	3.7
Vertical Scratch	35.0	Infinite*	Infinite*

***Saturation of ohmmeter resistance reached**

It is important to note that the Metal Rubber is not very durable and prone to scratching when the alligator clips are connected. The material's gold nano cluster layer is easily rubbed off by the metal alligator clips, revealing a clear non-conductive rubber layer. Once the thin layer of gold nano clusters was rubbed off, that portion of the material is no longer conductive and loses its sensing capabilities. This significantly changes the base resistance measurements throughout the experiment. Therefore, it is critical to notice the changes in resistance when the damage is induced rather than the resistance value itself.

Data from Table 2 indicates that vertical abrasions will significantly alter the material’s conductive properties with the possibility of complete loss of conductivity. The “half-snip” and the horizontal scratches increase the resistance, but alternate conductive pathways are easily utilized by the material resulting in an increase in resistance while maintaining conductivity. The substantial decrease, or complete loss, of conductivity due to damage in a certain direction allows for directional sensing capabilities. This directional sensing capability could enable elastomers that are used for sealing applications to sense damage across the seal, severing electrical connection, and indicating a possible leak initiation site.

Subsequently, resistance changes in the Metal Rubber due to increasing load were examined through tensile testing. Vise Grip pliers were used to grip both ends of a dogbone Metal Rubber specimen while resistance changes were monitored by an Ohmmeter as weights were added to the lower Vise Grip incrementally. Figure 8 shows the experimental test setup used for the simple tensile test.

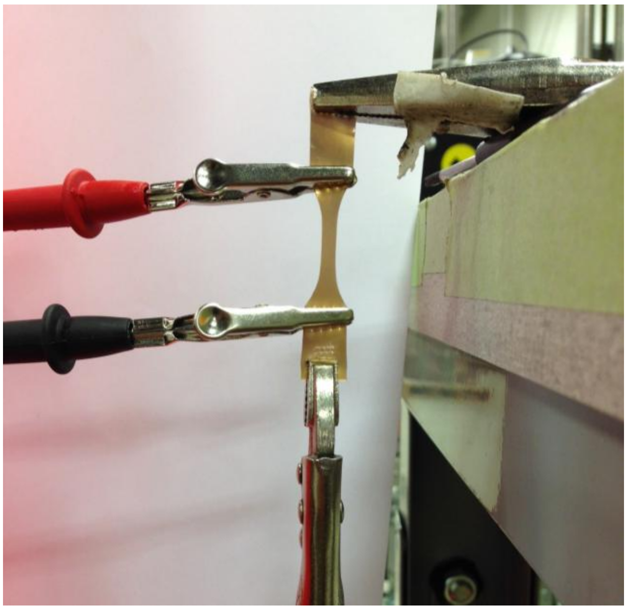

Figure 8: Metal Rubber dogbone specimen experimental setup for tensile test

The results of the tensile test are shown in Figure 9. An apparent non-linear relationship is observed between load and resistance.

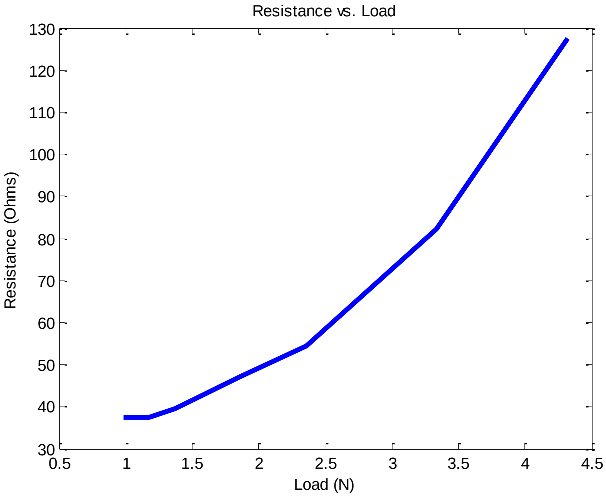

Figure 9: Plot of Resistance vs Load for Metal Rubber

The non-linear relationship between resistance and mass is best explained in correlation to a segmented stress-strain curve of rubber in Figure 10. As small amounts of weight are added, there is no significant change in resistance because the elastic modulus or slope of the line in Region 1 is large and linear. As more weight is added to the specimen, the stress-strain curve of rubber in Region 2 starts to transition into the hyperelastic regime. In this transition region, the elastic modulus starts to decreases resulting in larger deformations and therefore greater measured resistances as the gold nanoclusters in the Metal Rubber start to loose contact.

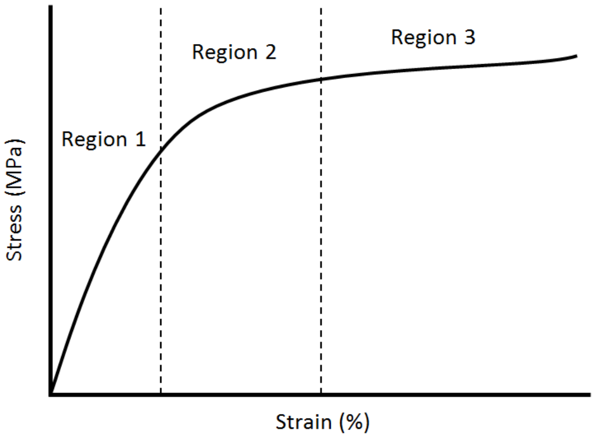

Figure 10: Stress-Strain Curve of Rubber

Finally, as the transition into the hyperelastic region is complete, the elastic modulus reaches a local minimum in Region 3 of Figure 10. This new lower elastic modulus gives way to even larger elastic deformations and measured resistance values as more electrical pathways are severed, thus resulting in the non-linear behavior that is observed. One last point worth noting is that the range of load applied herein is only 3 N. Such large changes in resistance with relatively small changes in load bode well for a sensor with great sensitivity to changes in load.

7 CONCLUSIONS AND FUTURE WORK

The current work looks at the evolution of CNTs embedded in elastomers; from the early years when a simple study to investigate the impact of CNTs on mechanical and electrical properties was performed to recent years when great strides have been made to functionalize CNTs within elastomers.

Much has been learned in the area of dispersion of CNTs within materials including polymers and concrete, new methods are being investigated to achieve uniform and repeatable dispersion including the use of magnetic fields. Such methods have the potential to lead to achieving functionally graded elastomers. Realizing that the material properties and functionality of CNTs dispersed into elastomers may differ from vertically aligned CNTs embedded in elastomers, parallel paths and their corresponding results are being pursued. The functionality of each as applied to elastomeric rotorcraft components will continue to be investigated and reported.

The ability of CNT thread to measure strain has proven successful and the results of which are currently being developed and extended to vertically aligned CNTs embedded in elastomers. Preliminary multiscale modeling efforts have resulted in a wide variation of predicted material properties indicating a need to further investigate existing homogenization techniques and both verify and validate experimentally.

The nearest commercially available product to vertically aligned CNT embedded rubber is that of Metal Rubber which exhibits both hyperelastic behavior and electrical conductivity at mid-range strains. While the Metal Rubber samples tested experienced durability issues, they did allow proof of concept to both detect damage and measure load in rubber materials. Once constructed, the vertically aligned CNT embedded rubber will be compared to the results of Metal Rubber presented in the current study and extended to representative rotorcraft applications in the lab utilizing both the RC helicopter in Figure 11 and the in-house whirl test stand in Figure 12.

Figure 11: RC helicopter used for load/strain data acquisition testing

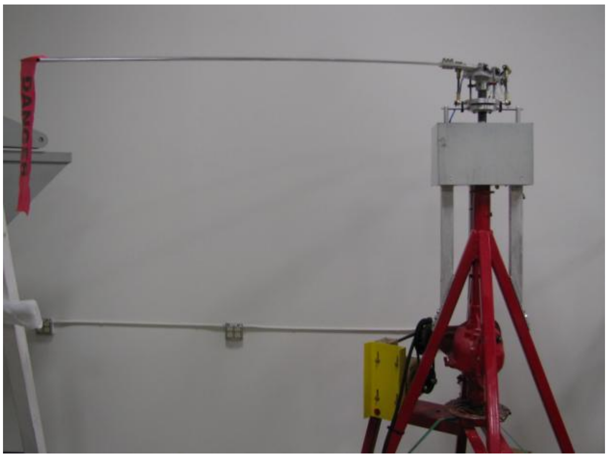

Figure 12: Whirl Test Rig

ACKNOWLEDGMENTS

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