Detecting Delamination in Carbon Fiber Composites Using Piezoresistive Nanocomposites

Sandeep Chava

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DETECTING DELAMINATION IN CARBON FIBER COMPOSITES USING PIEZORESISTIVE NANOCOMPOSITES

A Thesis

Submitted to the Faculty

of

Embry-Riddle Aeronautical University

by

Sandeep Chava

In Partial Fulfillment of the Requirements for the Degree of Master of Science in Aerospace Engineering

August 2016

Embry-Riddle Aeronautical University

Daytona Beach, Florida
DETECTING DELAMINATION IN CARBON FIBER COMPOSITES USING PIEZORESISTIVE NANOCOMPOSITES

by

Sandeep Chava

A Thesis prepared under the direction of the candidate’s committee chairman, Dr. Sirish Namilae, Department of Aerospace Engineering, and has been approved by the members of the thesis committee. It was submitted to the School of Graduate Studies and Research and was accepted in partial fulfillment of the requirements for the degree of Master of Science in Aerospace Engineering.

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8/1/16

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8/1/16

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8/3/16

Date
This thesis is dedicated to my father,

Venkatrao Chava

May his memory forever be a comfort and a blessing
He was the best father
a kid could have
ACKNOWLEDGMENTS

It gives me immense pleasure to acknowledge all the people that supported me and stood beside me in this journey. I would first like to thank my thesis advisor Dr. Sirish Namilae, who consistently allowed this thesis to be my own work, but steered me and motivated me in the right direction whenever he thought I needed it.

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<thead>
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<tbody>
<tr>
<td>( R )</td>
<td>resistance</td>
</tr>
<tr>
<td>( I )</td>
<td>current</td>
</tr>
<tr>
<td>( \Delta V )</td>
<td>voltage drop</td>
</tr>
<tr>
<td>( \rho )</td>
<td>resistivity</td>
</tr>
<tr>
<td>( D )</td>
<td>maximum deflection</td>
</tr>
<tr>
<td>( L )</td>
<td>length of support span</td>
</tr>
<tr>
<td>( b )</td>
<td>width of test beam</td>
</tr>
<tr>
<td>( F )</td>
<td>load at any given point</td>
</tr>
<tr>
<td>( d )</td>
<td>depth of tested beam</td>
</tr>
<tr>
<td>( w )</td>
<td>width of nanocomposites</td>
</tr>
<tr>
<td>( m )</td>
<td>slope of the secant of the force-deflection curve</td>
</tr>
<tr>
<td>( t )</td>
<td>thickness of nanocomposites</td>
</tr>
<tr>
<td>( \sigma_f )</td>
<td>maximum flexural stress</td>
</tr>
<tr>
<td>( \varepsilon_f )</td>
<td>maximum strain</td>
</tr>
<tr>
<td>( E_f )</td>
<td>flexural modulus of elasticity</td>
</tr>
<tr>
<td>( l )</td>
<td>length of nanocomposites</td>
</tr>
<tr>
<td>( V )</td>
<td>applied voltage</td>
</tr>
<tr>
<td>( R/R_o )</td>
<td>resistivity change</td>
</tr>
<tr>
<td>( \varepsilon )</td>
<td>strain</td>
</tr>
</tbody>
</table>
Carbon fiber prepreg composites are utilized successfully as structural materials for different lightweight aerospace applications. Delamination is a critical failure mode in these composite materials. As composite plies separate from each other, the composite loses some of its ability for supporting expected loads. Therefore, detection of delamination at right time is of foremost significance. This study presents a new way for detecting delamination in composite plates using piezoresistive nanocomposites. This new procedure is setup and studied through both experimental and computational investigations. In this research, nanocomposites with 5% coarse graphene platelets are fabricated for detecting delamination. 8-ply carbon fiber prepreg composite samples are fabricated through compression molding. Delaminated composite samples are fabricated by placing a Teflon film between layers of prepreg. Piezoresistive nanocomposites are attached on top of prepreg laminate samples using epoxy resin. The change in electrical resistivity of these nanocomposites due to the induced strain from flexural test (three point bend test) on delaminated and neat composite laminates are monitored to demonstrate the delamination detection method. A non-linear finite element model is developed using Abaqus software suite to compliment the mechanical testing. Virtual Crack Closure Technique (VCCT) is
used to model a delamination in the composite sample. Experimental results and the simulations in this study indicate that piezoresistive nanocomposites can be used for detecting delamination in carbon fiber composite materials.
1. Introduction

1.1 Significance

Composite materials have been widely used in for structural applications. Light weight, high specific strength, resistance to corrosion and flexibility in design, etc. are some of the properties displayed by these materials that have benefited many industries such as aerospace, automotive and marine. For example, commercial aircraft such as Boeing 787 and Airbus 380 are two of the first commercial aircraft to feature composites in fuselage and other primary structures (Garnier, Pastor, Eyma, & Lorrain, 2011).

Despite these benefits the susceptibility of composite materials to impact damage is high and creates a major concern related to structural integrity (Abrate, 2005). In aerospace structures, low-velocity impacts are often caused by tool drops during manufacturing and servicing or runway stones during landing or take off. Such impacts may result in various forms of damage such as indentation, matrix cracking, delamination or fiber fracture, leading to severe reduction in strength and also reduction in integrity of composite structures.

Although structures designed with fail-safe principles can withstand in theory, impact damage detection is an important issue in maintenance of aircraft and aerospace structures. While visible damage can be easily detected and remedial action can be taken to maintain structural integrity, a major concern to end-users is the growth of undetected, hidden damage caused by low-velocity impacts and fatigue. This internal damage is also known in aerospace applications as Barely Visible Impact Damage (BVID), and failure to detect BVIDs may result in a catastrophic collapse of the structure (Aymerich & Staszewski, 2010).
Hence, there is an increasing need for damage detection technologies on primary structural components of the aircraft (such as the fuselage or the wings) so that we can repair the damage rather than replacement of these components as a first-time solution (Hu & Soutis, 2000).

Common damage on an aircraft can arise from accidental impact, bird strike, hailstones and lightning strike or from deterioration caused by the absorption of moisture or hydraulic fluid (Cheng, Gong, Hearn, & Aivazzadeh, 2011). Because of the laminated layers in composite structures, damage often manifests as delamination between plies. Delamination is one of the major failure modes and it may cause structural failure leading to catastrophic consequences.

Development of an early damage detection method for delamination is an important requirement for maintaining the integrity and safety of composite structures. Many detection techniques have been proposed for structural health monitoring (SHM) and some of the non-destructive evaluation approaches that utilize advanced technologies, such as X-ray imaging (Tillack, Nockemann, & Bellon, 2000), ultrasonic scans (Rose, 2007), infrared thermograph (Meola & Carlomagno, 2004), and eddy current (Grimberg, Savin, & Rotundu, 2001), can identify damages.

However, most of these approaches are difficult to implement for in-service aircraft testing and in situ space structures. Almost all of the above techniques require that the vicinity of the damage is known in advance and the portion of the structure being inspected is readily accessible (Qiao, Lu, Lestari, & Wang, 2006). This thesis investigates delamination in laminates composite structures with the intention of developing a method for damage detection approach using piezoresistive nanocomposites. The nanocomposites
can be embedded in the structure facilitating potential in situ damage detection and monitoring.

The piezoresistive nanocomposites used in this work are developed based on Carbon Nanotube sheets and graphene platelets. This thesis attempts to understand the change in electrical behavior of the piezoresistive nanocomposites under applied strain when they are embedded in composite laminates with and without delamination. Additional applications are in the area of composite repair, wherein these piezoresistive nanocomposite sensors have the potential to study the effectiveness of the repair on composite laminates.

### 1.2 Motivation

Carbon fiber is generally defined as a fiber that contains at least 92 wt % carbon. On the other hand, the fiber with 99 wt % carbon is called a graphite fiber (Fitzer, 1989). Due to their excellent tensile properties and low densities, these are used in composites in the form of woven textiles, prepregs, and chopped fibers. The demand for the carbon fiber is increasing due to its use in many industries, such as aerospace, military and transport. The estimated prediction of global carbon fiber consumption is shown in Table 1 as of 2010 (Roberts, 2006).

<table>
<thead>
<tr>
<th></th>
<th>1999 (tons)</th>
<th>2004 (tons)</th>
<th>2006 (tons)</th>
<th>2008 (tons)</th>
<th>2010 (tons)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aerospace</td>
<td>4,000</td>
<td>5,600</td>
<td>6,500</td>
<td>7,500</td>
<td>9,800</td>
</tr>
</tbody>
</table>
These carbon fiber composites offer a combination of strength and modulus that are either comparable to or better than many traditional metallic materials (Harris, 1991). In addition to these properties, there are number of other differences between metallic structures and carbon fiber composites. Due to inherent heterogeneity and anisotropy, failure of composites involves many mechanisms. Whereas fatigue failure of isotropic and homogeneous materials such as metals is the result of initiation and propagation of a single dominant crack, fatigue failure of composites is characterized by initiation and multiplication of many cracks in the weak phase (Shokrieh & Taheri-Behrooz, 2009).

For example, metals exhibit plastic deformation while most fiber reinforced composites are elastic in their tensile stress-strain characteristics. Mechanisms of damage development and growth in metallic and composite structures are also quite different. While carbon fiber composites are used extensively in these many industries, there is always a need for early damage detection in composite structures. Piezoresistive nanocomposite sensor can potentially monitor the delamination in composite structures.

There are many carbon nanotube (CNT) based sensors that are currently used in fields like biomedical, automotive and food industry etc. (see Table 1.2). Sensors in Biomedical Field like CNT Implantable Nanosensor is used in detecting diseases or hazardous radiation exposure in early stages (Sinha & Yeow, 2005). In automotive
industry, sensors like antitheft sensor that prevents stealing of vehicles are also made based on CNTs (Weinberg, 2002). In food industry, sensors like pH sensors are used to determine pH value in water for survival and growth of fishes (Xu, Chen, Qu, Jia, & Dong, 2004). Sensors like CNT-based acoustic and optical sensors used for breath alcohol detection at room temperature (Penza, Cassano, Aversa, Antolini, Cusano, Cutolo, & Nicolais, 2004).

Table 1.2 CNT and Graphene based sensors

<table>
<thead>
<tr>
<th>Sensor area</th>
<th>Sensor type</th>
<th>Sensor uses</th>
<th>Ref</th>
</tr>
</thead>
<tbody>
<tr>
<td>Biomedical</td>
<td>CNT Nano biosensor</td>
<td>Detecting DNA sequences in body</td>
<td>R1</td>
</tr>
<tr>
<td></td>
<td>CNT Chemical sensor</td>
<td>Blood analysis (Detecting ‘Na’ etc.)</td>
<td>R2</td>
</tr>
<tr>
<td></td>
<td>CNT Pressure sensor</td>
<td>Eye surgery, respiratory devices etc.</td>
<td>R3</td>
</tr>
<tr>
<td>Automotive</td>
<td>CNT Force sensor</td>
<td>Determining if an air filter is bad</td>
<td>R2</td>
</tr>
<tr>
<td></td>
<td>CNT Pressure sensor</td>
<td>Controlling dampers in suspensions</td>
<td>R4</td>
</tr>
<tr>
<td></td>
<td>CNT Rollover sensor</td>
<td>Finding roll rate to prevent tipping over</td>
<td>R5</td>
</tr>
<tr>
<td>Food</td>
<td>CNT Gas sensor</td>
<td>Monitor meat freshness during shipping</td>
<td>R6</td>
</tr>
<tr>
<td></td>
<td>CNT Humidity sensor</td>
<td>Monitoring humidity changes</td>
<td>R7</td>
</tr>
<tr>
<td></td>
<td>CNT CO₂ sensor</td>
<td>Monitoring CO₂ for plants growth</td>
<td>R8</td>
</tr>
</tbody>
</table>

1.3 Problem statement and research objectives

The primary motivation is to develop a new damage detection sensor and detect delamination in carbon fiber composites using the developed sensor. In this thesis we explore and study CNT based piezoresistive nanocomposites and their application in detecting delamination through both experiments and finite element model. Experiments of tensile testing and flexural deformation of carbon fiber composites with and without delamination are used to characterize the embedded nanocomposite sensors. A finite element model is developed to further analyze the results of the experiments. The specific research objectives are identified as follows:

a) Design and fabricate nanocomposites using Buckypaper and 5 wt% coarse graphene platelets mixed with epoxy matrix. This composition has been suggested to exhibit highest piezoresistivity through prior work in our group [Li & Namilae, 2016].

b) Design and fabricate carbon fiber composite laminate samples with 8 ply layup using carbon fiber prepreg.

c) Tension test using MTS testing machine and flexural deformation test using the 3 point bend setup following ASTM standards on the carbon fiber prepreg laminate sample attached with piezoresistive nanocomposite on top of them. The results of the tests are stress-strain and resistivity-strain curves obtained through LabVIEW code developed.

d) Flexural test on a delaminated sample following ASTM standard and collecting similar results using LabVIEW code developed.

e) Developing Finite Element Model (FEM) using Abaqus CAE and doing the
same flexural test without delamination and with delamination using VCCT in Abaqus CAE.

f) Analyzing the results from experiments and finite element model and understanding the difference in test results between delaminated and baseline specimen.

g) Suggesting how the piezoresistive nanocomposite can be used to detect delamination or to identify the effectiveness of a repair in a repair patch on carbon fiber composite laminates.

By determining the change in electrical resistivity of a piezoresistive nanocomposite on a delaminated and non-delaminated sample, this thesis demonstrate the potential of nanocomposite utilization in damage/delamination sensing in aircraft structures.
2. Literature review

2.1 Composites

Composites are among the most adaptable advanced engineering materials known till date. Composites in general are heterogeneous materials and consist of two or more constituents such as fiber and a matrix (Lubin, 1982). In contrast to metallic alloys, each constituent material retains its separate chemical, physical, and mechanical properties.

The matrix of composites can be metal (Metal Matrix Composite), ceramic (Ceramic Matrix Composite) or carbon based (Carbon-Carbon or polymeric). These different matrix give composites their shape, appearance, environmental tolerance and durability while the fibers carry most of the structural load and thus, making these materials strong and stiff (Mark, Bikales, Overberger, Menges, & Kroschwitz, 1985).

Ashby (Ashby, Bush, Swindells, Bullough, Ellison, Lindblom, & Barnes, 1987) presents a chronological variation of the relative importance of each group of materials from 10,000 B.C. and extrapolates their importance through the year 2020. The information contained in Ashby’s article has been partially reproduced in Figure 2.1. From the figure it can be noticed that the importance of composites increased steadily from 1960 and is projected that it will increase through the next several decades as composites replace metallic materials in many applications.
One of the biggest benefits of composites is that the properties are adjustable per design parameters, for example, the mechanical properties, content, orientation and fiber architecture, and the properties of the matrix are all materials design parameters. Composite materials play a key role in industries like aerospace and automobile because of their outstanding strength to weight ratio and modulus to weight ratio (Figure 2.2). Some of these composites like graphite, Kevlar, boron or silicon carbide fibers in polymeric matrices have been studied extensively because of their applications in aerospace and space vehicle technology (Nielsen, 1972; Woods, 1994; Sohn, 2001; Rajeev, 2003)
Defects in carbon fiber composite structures are produced either during the manufacturing process or in the course of normal service life of the structure. There are multiple types of defects that are caused during manufacturing process. Porosity is one of the important defect in manufacturing process which is the presence of small voids in the
matrix (Kastner, Plank, Salaberger, & Sekelja, 2010). This can be caused by incorrect cure parameters such as pressure, temperature or duration of cure. Sandwich structures with honeycomb cores can suffer due to poor bonding of skin to the core. Dis-bonding can occur at these skin-to-adhesive or adhesive-to-core interactions (Fruehmann, Wang, Dulieu-Barton, & Quinn, 2011).

Service defects are mostly due to impacts. These impacts lead to matrix cracking and delamination of the ply layers. Delamination is a critical failure mode in these composite materials. In some cases the damage can be only internal referred as barely visible impact damage (BVID) (Garnier, Pastor, Eyma, & Lorrain, 2011). Sandwich structures can suffer from same matrix cracking and delamination in the skin during an impact.

### 2.2.1 Manufacturing Defects in Composite Structures

There are multiple methods in manufacturing composite structures. All of these methods aim to combine the fiber and matrix into one product. They can be separate before manufacturing or can already be combined like prepreg materials. All of these different methods selected to manufacture composites depend on the size and quality of the products required. Higher quality structures are usually used in aerospace applications to minimize weight, which are manufactures using hot pressing method or autoclaving.

During all these manufacturing processes, defects can be introduced into the structure and their effects depend on the process used to manufacture them and their applications. Multiple defect types have been identified including the following (Smith, 2009):

i. **Porosity:** Voids are created due to improper curing.
ii. Foreign bodies: Knowingly/unknowingly adding foreign bodies.

iii. Fiber vol. fraction: Incorrect fiber volume fraction due to excess or insufficient resin.


v. Fiber misalignment: This causes local changes in volume fraction by preventing ideal packing of fibers.

vi. Ply misalignment: Mistakes made in layup of plies cause this defect. This alters overall stiffness and strength of the laminate.

vii. Incomplete cure: Incompletely cured matrix during curing cycle.

viii. Wavy fibers: These are produced by in-plane kinking of the fibers and can seriously affect laminate strength.

ix. Fiber defects: The presence of defects in fibers themselves is one of the limiting factor in determining strength, these defects are considered as one of the basic material properties.

2.2.2 In Service Defects in Composite Structures

Composite structures can degrade over time in service due to many mechanisms and most of them are due to environment experienced defects. Some of these mechanisms are static overload, impact, fatigue, overheating and lightning strike etc. The main defects that are found in service are as following (Smith, 2009):

a. Delamination
b. Bond failures
c. Cracks
d. Moisture entry

e. Fracture or buckling of fibers

f. Failure of interface between the fibers and matrix

All these mechanisms vary depending on the type of loading, and mechanical properties of the constituents. Failure mechanisms are same in most composites but their mode of occurrence vary depending on the type of loading and properties of the constituents (Atiquullah, 2011). The major in service defect requiring detection in the presence of delamination. Delamination can be produced by fatigue, bearing damage, impact, etc (Xiong, 2010). Dis-bonding can also be found due to impacts. Cracks are the ones that usually lead to delamination in a critical stage.

Moisture degrades the strength properties of composites that are matrix dependent and also reduces residual strain (Smith, 2009). It may be possible to measure moisture content nondestructively. Fracture of the fibers and failure of interface between the fibers and matrix are due to impact (Kachanov, 2012). All these differ depending on application and composite type.

![Image of damage in a 4-ply laminated plate](image_url)

Figure 2.3 Showing damage in a 4-ply laminated plate (Iowa State Univ.)
Figure 2.3 is a photograph, reproduced from Iowa State University’s research on nondestructive evaluation showing damage in a 4-ply laminated plate that was subjected to thermal shock. One can see two types of cracks: cracking of the matrix material within a ply, and separation cracks (delamination) at the boundary between plies. The carbon fibers give the material the bulk of its strength, but rarely large cracks can be developed without breaking many fibers.

2.2.3 Boeing 787 Dreamliner Delamination Issue

As an example of these type of manufacturing defect one can refer to Boeing Dreamliner 787 delamination issue caused in 2012. According to Flightglobal, the structural stiffeners were found to be improperly joined to the composite skin in the aft sections of the aircraft, causing parts of the aircraft's carbon fiber to delaminate.

Boeing has found that incorrect shimming was performed on support structure on the aft fuselage on certain airplanes in their facility in Everett. Flightglobal has confirmed there are at least three affected airframes, Airplanes 56, for All Nippon Airways, where the problem was first discovered, and Airplanes 57 and 58, were the first two aircraft for Qatar Airways.

News articles (reference) mention that the stiffeners, or longerons that run along the length of the aircraft, delaminate around the rear opening of the Section 48 section above and below the cutout known as the "bird's mouth" that holds the Alenia Aeronautica-built horizontal stabilizer.

When the longerons are installed on the wound carbon fiber barrel, frames and longerons are secured to the skin of the structure to give it strength. When natural variations in the fit of parts exists, aerospace mechanics will install shims, or spacers, which
compensate for variations and wedge into structure to create a tighter fit. Without the shims, damage can be sustained to the composite when fasteners are installed by pulling the structure together, which cause damaging of the layers of carbon fiber. Over the long-term composite delamination can decrease the fatigue life of the aircraft's structure.

![Flightglobal news on 5th Feb, 2012](image)

Boeing has faced manufacturing quality issues before, most notably in the June 2010 inspection, teardown and reinstallation of many Alenia Aeronautica-built horizontal stabilizers were assembled without proper shimming, creating gaps in the structure that threatened the fatigue life of the empennage, according to Flightglobal.

### 2.3 Carbon as Graphene and Carbon Nanotubes (CNTs)

We now discuss the properties of carbon nanotubes, graphene and nanocomposites that are used in this work for damage detection application. Carbon is one of the most studied elements in the periodic table. The versatility of chemical bonds enables many
carbon allotropes. In three-dimensional form, carbon can exist as graphite and diamond, which comprise of sp2 and sp3 covalent bonds, respectively. In the 1980s and 1990s, another two types of carbon allotropes, the zero-dimensional fullerene (Kroto., 1991) and one-dimensional carbon nanotubes, were discovered.

Figure 2.5 Carbon allotropes (i) diamond; (ii) graphite; (iii) lonsdaleite; (iv) C60 (Buckminsterfullerene); (v) C540; (vi) C70; (vii) amorphous carbon; (viii) single-walled

Ever since their discovery, their contribution to development of studies in the field of physics, chemistry and material sciences is huge. There are many research studies on the structure (Dresselhaus, Dresselhaus, & Saito, 1995), properties (Mintmire & White, 1995) and their various applications (Ajayan, 1997). Graphene is composed of a honeycomb lattice of carbon atoms. Structurally, graphene is related to many carbon allotropes (Figure 2.5). For example, carbon nanotubes can be formed by rolling graphene along certain axes, and graphite can be formed by stacking graphene vertically (Geim & Novoselov, 2007).

Carbon nanotubes are basically rolled up graphene sheets (hexagonal structures) into cylindrical form and capped with half shape of fullerene structure. Many of the properties of CNTs are due to the way the graphene sheets are wrapped around. There are two types of carbon nanotubes:

(a) Single walled carbon nanotubes (SWNTs), formed by rolling a single graphene sheet into a cylinder (Figure 2.6). SWNTs with their high length to diameter ratio, atomic strength and chemical stability constitute one-dimensional molecules (Gommans, Alldredge, Tashiro, Park, Magnuson, & Rinzler, 2000)

![Figure 2.6 SWNT and MWNT](image)
Multi walled carbon nanotubes (MWNTs), can be considered as stacking of several layers of graphene in the form of cylinders with an interspacing of around 0.36nm (Figure 2.6). The length and diameter of MWNTs differ a lot from SWNTs and, of course, their properties are also very different.

Carbon nanotubes have unique electronic and mechanical properties which are achieved by chemically processing these CNTs in order to purify and get required property in them. CNTs can be metallic or semiconducting depending upon the atomic arrangements. A large number of these nanocomposites are produced in various methods such as arc evaporation method, electrolysis, laser ablation, chemical vapor deposition, etc. (Ying, Salleh, Yusoff, Rashid, & Razak, 2013). Production of carbon nanotubes in a controlled way in large quantities could potentially encounter problems that remain to be solved.

2.4 Properties of Carbon nanotubes (CNTs)

Carbon nanotubes have very unique thermal, electrical and mechanical properties. In spite of no direct methods to prove their properties, several experimental tests like SEM, AFM, TEM, nanoindentation, etc. and theoretical methods like molecular dynamics, continuum model, etc. are used to describe the mechanical properties of carbon nanotubes (Coleman, Blau, Dalton, Munoz, Collins, Kim, ... & Baughman, 2006).

Experimental studies of Georgakilas et al., found that high stiffness, high modulus and low density carbon nanotubes can be ideal material for fabrication of different composites (Georgakilas, Perman, Tucek, & Zboril, 2015). Yu et al. showed that only outer layer in a MWNT was able to withstand higher loadings while inner layers were observed to be very weak (Yu, 2000). These ultimate measurements were carried out and managed
to perform stress-strain measurements on individual arc-MWNTs inside an electron microscope and for a range of tubes the modulus ranges from 0.27 to 0.95 TPa. Fracture of MWNTs occurred at strains up to 12% and width strengths in the range 11-63 GPa (Prato & Maurizio, 2009).

Along with these mechanical properties, electronic properties of carbon nanotubes are also studied. Theoretical studies conclude that depending upon the diameter and chirality of the tube, carbon nanotubes may be either metallic or semiconducting (Terrones, 2013). In several experiments using scanning tunneling microscope (STM), it is observed that the tunneling conductance is a direct measure of local electron density of states of carbon nanotubes (Mittal & Garima, 2015).

The electrical resistivity of metallic carbon nanotubes was observed to be around $10^{-8}$ to $10^{-7}$ ohm-m (Charlier & Issi, 1996) and the electrical conductivity of individual MWNT is measured by four probe measurements using lithographic deposition of tungsten to be in the range of $10^7$ to $10^8$ S/m (Ebbesen, Lezec, Hiura, Bennett, Ghaemi, & Thio, 1996). Along with these properties, carbon nanotubes show high thermal conductivity also. New studies show that ultra-small SWNTs have shown superconductivity below 20ºK and the high value of 6000 W/mK was shown by isolated nanotubes, which is comparable to graphene monolayer and diamond (Berber, Kwon, & Tománek, 2000). The small diameter and high aspect ratio of CNTs is favorable for field emission, which results from the tunneling of electrons from metal tip into vacuum under application of strong electric field.

### 2.5 Buckypaper (CNT Sheet)

Buckypaper (BP) is an outstanding material which contains entangled networks of CNTs formed by Van der Waals interactions (Baughman, 1999), which is an effective way
of introducing carbon nanotubes into composites (Endo, Muramatsu, Hayashi, Kim, Terrones, & Dresselhaus, 2005). Buckypaper can be produced in large sizes and provides ease of handling, and improves the safety of using CNTs in industrial manufacturing facilities. Buckypaper can be fabricated by using double-walled CNTs (Gong, 2007), SWCNTs (Teague, 2007), and MWCNTs (Xu, 2008).

![Figure 2.7 Buckypaper used in this research from Nano Tech Labs](image)

Due to its high CNT concentration, buckypaper provides great advantages to enhance electrical properties (Cheng, 2010), actuation (Chen, 2010), fire retardancy (Wu, 2011), and electromagnetic interference shielding properties in composites (Gnidakouong, Kim, Park, Park, Jeong, Jung, & Park, 2013).

A number of techniques for fabricating buckypaper have been proposed. Some of which are:
i. A vacuum filtration method for fabricating large-area buckypaper less than 200 nm thick (Hennrich, Lebedkin, Malik, Tracy, Barczewski, Rösner, & Kappes, 2002).

ii. Fabrication of buckypaper by liberation of electrophoretically deposited carbon nanotubes (Rigueur, Hasan, Mahajan, & Dickerson, 2010).

iii. Fabrication of highly oriented buckypaper made of aligned carbon nanotubes (Zhang, Jiang, & Peng, 2014).

Figure 2.8 SEM micrograph of buckypaper

Due to the component material (CNTs), microstructure and properties of buckypaper in the past decade, buckypaper and buckypaper composites have been extensively studied. It is believed to be an excellent material for many engineering applications, such as electrodes, actuators, sensor, and heat conductors and as reinforcement for polymer composites (Chen, 2013). Some of the studies which
demonstrated sensors made of CNTs are:

a. Li et al. demonstrated the potential of carbon nanotube films in measuring strain at the macro scale (Li & Dharap, 2004).

b. Kang et al. developed a composite electrical resistance strain sensor based on SWNTs, and it was used to measure the strain of a structure at the macro scale (Kang, Schulz, Kim, Shanov, & Shi, 2006).

c. Li et al. have studied the possibility of using multiwall carbon nanotube (MWCNTs) films as strain sensors (Li, Levy, & Elaadil, 2008).

d. Gao et al. reported a simple approach to deposit multi-walled carbon nanotube (MWNTs) networks onto glass fiber surfaces achieving semi conductive MWNTs-glass fibers, along with application of fiber/polymer interphase as in situ multifunctional sensors (Gao, Zhuang, Zhang, Liu, & Mäder, 2010).

These features make buckypaper an excellent candidate for manufacturing large-scale composite samples with carbon fiber prepreg to achieve high CNT loading. This thesis also develops a finite element model to explain the experimental observations using Abaqus/CAE. This next section gives an introduction to finite element models for delamination and fracture.

2.6 Finite element models of Delamination

Delamination brings significant material degradations in both stiffness and strength under compression, tension and flexural loading. It occurs under any combinations of mixed Mode I, Mode II, and Mode III. Different methods have been used to model and simulate these delamination. Some of the widely used methods are Extended Finite
Element Method (XFEM), Cohesive Zone Model (CZM) and Virtual Crack Closure Technique (VCCT).

### 2.6.1 eXtended Finite Element Method (XFEM)

The extended finite element method (XFEM), also known as generalized finite element method (GFEM) or partition of unity method (PUM) has been used very successfully to model cracks because the finite element mesh can be created independent from the crack geometry, and in particular the domain does not have to be remeshed as the crack propagates (Richardson, Hegemann, Sifakis, Hellrung, & Teran, 2009). Richardson et al. used XFEM method for modelling geometrically elaborate crack propagation in brittle materials. This method was developed to reduce difficulties in solving problems with localized features that are not efficiently resolved by mesh refinement.

One of the initial applications was the modeling of fractures in a material. A key advantage of XFEM is that the finite element mesh does not need to be updated to track the crack path (Jiang, 2013). In recent years, the extended finite element method (XFEM) has emerged as a powerful numerical procedure for the analysis of fracture problems. It has been widely acknowledged that the method eases fracture growth modeling under the assumptions of linear elastic fracture mechanics (LEFM).

Since the introduction of the method in 1999, many new extensions and applications have appeared in the scientific literature (Karihaloo & Xiao, 2003). XFEM has been used in the study of composite delaminations (Motamedi, 2014; Motamedi, 2013; Hulton, 2015; Sosa, 2012).

### 2.6.2 Cohesive Zone Model (CZM)
Cohesive zone model (CZM) is one of the most versatile evolutions in the area of fracture mechanics. These partition of the surfaces involved in the cracks takes place across an extended crack tip, or cohesive zone, and is resisted by cohesive tractions (Liu, 2013). The concept of cohesive zone ahead of the crack tip, which was introduced by Dugdale (Dugdale, 1960) and Barenblatt (Barenblatt, 1962) has become a guiding idea for a class of crack propagation models. Figure 2.9 shows the schematic of cohesive zone model for various failure phenomena: damage is localized in an interface.

Figure 2.9 Schematic of cohesive zone model (CZM) (Scheider, 2006)

A cohesive model in combination with finite elements was first used for concrete (Hillerborg, Modéer, & Petersson, 1976) and, more than ten years later, also for metals (Needleman, 1990). Interface elements obeying a cohesive law are introduced between the continuum elements. New applications cover a variety of phenomena like viscoplastic (Corigliano, 2001) and viscoelastic (Rahu, 1999) separation behavior, the modelling of
fragmentation (Repetto, Radovitzky, & Ortiz, 2000) and fiber de-bonding, failure under dynamic and cyclic loading (Roe & Siegmund, 2003).

The respective cohesive law has to be chosen in dependence on the micromechanical damage mechanism leading to fracture. Commonly, two material parameters, namely a cohesive strength, and a critical separation, are chosen to characterize the cohesive behavior. Finite element simulations with cohesive elements run numerically stable up to large amounts of crack extension and yield very good results for structures with different size and constraint conditions.

CZM can be applied to both 2D (Cornec, 2003) and 3D (Gao, 2006) structures. Some studies of CZM to composite delamination include Milad saeedifar’s delamination growth prediction (Saeedifar, 2015), Qiang Ye’s cohesive strength predication for composite delamination (Ye, 2011), Libin Zhao’s simulation of delamination using cohesive elements (Zhao, 2014) and Jalal Yousefi’s CZM to simulate delamination growth (Yousefi, 2015).

2.6.3 Virtual Crack Closure Technique (VCCT)

The virtual crack closure technique (VCCT) is widely used for computing energy release rates based on results from continuum (2D) and solid (3D) finite element (FE) analyses to supply the mode separation required when using the mixed mode fracture criterion (Jimenez, 2004). Lately, an increased interest in using a fracture mechanics–based approach to assess the damage tolerance of composite structures in the design phase and during certification has also renewed the interest in the virtual crack closure technique (Tay, 2003).
The VCCT can be used to analyze delamination in laminated materials using a fracture mechanics approach. The method implements linear elastic fracture mechanics (LEFM). This LEFM method is used for delamination analysis in composite laminates which determines the total strain energy release rate \( G_T \) which is the sum of individual components \( G_I \), \( G_{II} \), and \( G_{III} \) (Mohammed, 2014).

The virtual crack closure technique (VCCT) is the most popular and powerful tool to approximately compute \( G \)’s values. It was first introduced by Rybicki and Kanninen (Rybicki, 1977) for 2D crack problems and was extended to 3D crack problems by Shivakumar (Shivakumar, Tan, & Newman, 1988).

Wang et al. made significant contributions to improve the accuracy and enhance the capability of the approach (Wang & Raju, 1996). Some applications of VCCT include the delamination of composites (Krueger & O’Brien, 2001), the de-bonding of skin-stiffeners (Krueger, Paris, O’Brien, & Minguet, 2002), and the failure of adhesively bonded joints (Xie, Chung, Waas, Shahwan, Schroeder, Boeman, & Klett, 2005). VCCT based crack-growth simulation can be created involving the following assumptions (Reeder, Song, Chunchu, & Ambur, 2002):

- a. Crack growth occurs along a pre-defined crack path.
- b. The path is defined via interface elements.
- c. The analysis is quasi-static and does not account for transient effects.
- d. The material is linearly elastic and can be one of isotropic, orthotropic or anisotropic material.

Abaqus/CAE has been used in this research to create the finite element model for delamination analysis. Delamination in aerospace structures at the Boeing Company have
been extensively investigated using VCCT implementation in Abaqus.

### 2.6.4 Abaqus software

![Figure 2.10 Abaqus/CAE main user interface](image)

The core of the Abaqus are Abaqus/Standard and Abaqus/Explicit which are the analysis modules tools integrated into Abaqus. Abaqus/Standard is a general purpose finite element module. It is used in analyzing many types of problems including nonstructural applications. On the other hand Abaqus/Explicit is an explicit dynamics finite element module in Abaqus. Abaqus/CAE (Complete Abaqus Environment) incorporates the analysis modules for modeling, managing and monitoring Abaqus analyses and results.

Abaqus/CAE can be customized to create application specific systems. It integrates modeling, analysis, job management and result evaluation seamlessly. It also provides
complete interface with Abaqus solver programs available.

Abaqus/CAE has one of the best modern graphical user interface (GUI) with icons, menus and dialog boxes. This GUI provides access to all capabilities, accelerate access to frequently used features and to select various other options.

Abaqus/CAE has various modules that are easily accessible. Each module contains a logical subset of the overall functionality. It also has a Model Tree with a graphical view of the model created, and all the objects that it contains, as shown in Figure 2.11. It acts like a convenient, centralized tool for moving between modules and for managing objects.
Figure 2.11 Model Tree with all modules and objects
3. Experimental Procedure

There were three considerations in detecting delamination in carbon fiber composites that were addressed:

(i) Fabricating piezoresistive nanocomposite sensor

(ii) Fabricating carbon fiber prepreg composite sample (with and without delamination)

(iii) Embedding nanocomposite on composite sample, and mechanical testing of the samples (Three point bend flexural test)

3.1 Fabricating nanocomposite sensor

Buckypaper (BP), which is a thin sheet of carbon nanotubes (CNTs) show a great promise in fabricating multifunctional nanocomposites. One of the serious problems to the use of CNTs in engineering applications is the inability to synthesize long nanotubes which is why buckypaper is used in this research to fabricate the sensor. Buckypaper can be considered as a composite by two ways. One by infusing with resin and the other one by incorporating into conventional fiber reinforced composites (Wang, Liang, Wang, Zhang, & Kramer, 2004). Unlike the CNTs directly added into matrix, buckypaper based composites have much higher concentration of CNTs and high conductivity.

This research uses epoxy resin as matrix material in fabricating buckypaper based composite sensor (Piezoresistive Nanocomposite). Epoxy modified by adding coarse graphene platelets, is utilized in fabricating these nanocomposites. Tensile tests and simultaneous electrical resistivity measurements are performed on these nanocomposite samples which are used for further analysis in detecting delamination in carbon fiber composites.
3.1.1 Materials

The buckypaper (multiwall CNT sheet) was procured from NanoTech Labs which consists of 100% free standing nanotubes. This buckypaper has an area density of 21.7 g/m² and surface electrical resistivity of 1.5 Ω/m². The electrical resistivity was measured independently through experiments.

The graphene sheet (6 inch x 6 inch) supplied by Graphene Supermarket has low resistance of 2.8x10⁻² Ω/m². It is used as coarse graphene platelets after finely chopping the graphene sheet in to size between 300–1000 μm. This graphene sheet is made out of multiple layers of nanoscale fine graphene platelets adhesively bonded together.

The silver epoxy resin supplied by MG chemicals has high conductivity and high adhesive properties. This epoxy has a 1:1 mix ratio of epoxy and hardener and a 4 hour working time. This conductive epoxy is used in attaching electrodes to nanocomposites.

The regular epoxy resin is a West System # 105 Epoxy Resin with West System # 206 Slow Hardener. This epoxy has a 5:1 mix ratio of epoxy and hardener, and a 20 minute working time. This epoxy is a light amber, low-viscosity liquid epoxy resin specifically formulated as functions of wetting out, bonding with fiber glass, carbon fiber and other materials.

The other materials required for the fabrication include copper plates, peelply, breather film, aluminum tooling plate, epoxy mixing cups, vacuum bag and complete vacuum set-up.
3.1.2 Procedure

The buckypaper is cut into strips of size 6.35cm x 1.27cm using a laser blade as shown in Figure 3.1 and copper plates gauging 32 with dimension 1.27cm x 1.27 cm are attached to both sides of cut buckypaper strips using the conductive silver epoxy paste as shown in Figure 3.2.

These attached copper plates are used for conductivity measurement during experiments. These buckypaper strips with copper plates are placed on a peelply which is again placed on a flat aluminum tooling plate as shown in Figure 3.3.
The coarse graphene platelets (5 wt. %) are mixed into the epoxy resin evenly without mixing hardener as shown in Figure 3.4. Hardener is mixed later before applying on the buckypaper strips. This increases the working time before the resin solidifies. The weight of graphene is calculated before to ensure right weight of graphene in the final mixture (Li & Namilae, 2016).
This epoxy mixed with 5 wt. % graphene platelets after adding hardener is applied to both sides of buckypaper strip samples on the tooling plate. The set-up is now covered with peelply followed by breather film (removes excess epoxy). The final set-up is covered with a vacuum bag of pressure 88.05 KPa. This vacuum bagging procedure helps the breather film to absorb extra epoxy.

![Figure 3.5 Vacuum bagging schematic](image)

The general vacuum bagging setup is shown in Figure 3.5 for fabricating the piezoresistive nanocomposite. These nanocomposite samples are peeled from the peelply after curing the resin for 12 hours at room temperature. The curing time and curing temperature will vary from one epoxy mixture to another.

To follow up, the buckypaper is cut into strips and copper plates are attached to the strips which act as electrodes followed by adding graphene platelets and curing them by applying vacuum which gives the required piezoresistive nanocomposite sensor. Figure 3.8 shows the SEM micrograph of coarse graphene platelets and CNTs on the nanocomposite sensor.
Figure 3.6 Showing Buckypaper strip, attached copper plate and final sensor

Figure 3.7 Peeled off nanocomposite sensors

Figure 3.7 shows some of the peeled off piezoresistive nanocomposites which are ready for further testing.
3.2 Fabricating carbon fiber prepreg composite laminate

Carbon fiber prepreg is conventional carbon fiber that has been pre-impregnated with partially cured resin during manufacture. Because the resin has already been mixed with hardener, this carbon fiber prepreg needs to be stored at very low temperatures to prevent the resin from curing before it is used. The strength of carbon fiber is its weave. The more complex the weave, the more durable the composite will be.

The angle of the weave and the type of resin used with the fiber will determine the overall strength of composite. The resin is commonly the epoxy that is applied to the carbon fiber fabric by precisely calibrated machinery with required ratio of resin to reinforcement. This material has a wide range of applications, as it can be formed at various densities in unlimited sizes and shapes.
The advantages of carbon fiber composites are their high stiffness, high strength to weight ratio, excellent fatigue endurance, corrosion resistance, impact resistance and, as discussed earlier, their flexibility in design adapt them to any design requirements.

Despite the many advantages, carbon fiber composites also have few disadvantages such as high material cost, high fabrication cost, and requirements for nondestructive inspection techniques to detect flaws and damages. This research helps in that direction by introducing a new sensor for in-situ detection of delamination in composites.

### 3.2.1 Materials

The carbon fiber prepreg (CF3327-1 EPC: Se-019K) supplied by Hankuk Carbon is based on 250°F (121°C) curing, consists of a carbon fabric impregnated with epoxy resin which includes Carbon 3K as a warp and Carbon 3K as a fill. The Fiber area weight (FAW) of this prepreg is 200 g/m². The weave of this prepreg is 2X2 twill. The fiber volume fraction and resin contents are 50% and 40%, respectively. The shelf life of this prepreg is 6 months at storage temperature of below -18°C. This material is suitable for fabricating high performance composite structures according to Hankuk Carbon.

The genesis series hydraulic compression press supplied by Wabash MPI is ideal for compression molding of rubber, plastic, composites and laminating with clamp force calculated from 15 to 150 tons of weight. This press features steel platens, programmable controller, automatic transition from closing to pressing speed, pressure relief valve with analog pressure gauge, internal hydraulic system with high efficiency motor, reservoir & water –cooled heat exchanger, digital temperature controls and many more. This hydraulic compression composite molding press is used to replicate
the pressure – temperature cycle of an autoclave cure.

Figure 3.9 Typical autoclave cure cycle (Hankuk Carbon)

The other materials required for the fabrication of carbon fiber composite laminate using Wabash hydraulic compression molding press are tooling plate, electric scissors, ruler, protractor, a roller, markers, Teflon tape and a 650X tile wet saw.

3.2.2 Fabrication Procedure

Figure 3.10 Carbon fiber prepreg strip
The carbon fiber prepreg is cut into multiple 15.24x15.24x10^{-2}m strips with 0° and 45° alignment using electric scissors as shown in Figure 3.10. A clean tooling plate is taken and eight of these strips are used to layup an 8-ply layup with (0°/45°)₄ layup.

A roller is used to roll after laying up each layer to prevent air gaps as shown in Figure 3.11. Once the layup is completed, this setup is now moved to Wabash composite compression molding press which is preheated to 250°F. The press is set to a pressure of 35 psi which is recommended by Hankuk Carbon for curing the prepreg. A small program is written in the press to cure the prepreg for 90 minutes at 250°F. This can be seen in Figures 3.12 and 3.13.
Figure 3.12 Wabash Press used in curing prepreg

Figure 3.13 Digital temperature controls on Wabash press
Once cured, an 8 ply composite carbon fiber laminate of dimensions 15.24 cm x 15.24 cm (6”X6”) is fabricated. This fabricated laminate is later cut into six pieces of 2.54 cm x 15.24 cm (1”X6”) samples using a 650XT tile wet saw. The similar procedure is followed for fabricating delaminated composite samples.

For delaminated composite laminate fabrication, after laying up four layers of prepreg on tooling plate, one layer of Teflon (PTFE) tape of dimension 7.62 cm x 15.24 cm is laid on the fourth layer from half to one end. Remaining four layers of prepreg are laid and the setup is cured as earlier. Once cured a composite laminate with known delamination fabrication is completed. Using a 650XT tile wet saw six samples of dimension 2.54 cm x 15.24 cm (1”X6”) are cut from the laminate. These sample have 2.54 cm x 7.62 cm delamination in them as shown in Figure 3.14.

Figure 3.14 Delamination in composite laminate sample (schematic)
3.3 Preparing samples for testing

After the fabrication of piezoresistive nanocomposite and making carbon fiber composite laminate samples, the next step is to attach the piezoresistive nanocomposite sensor on top of the carbon fiber composite laminate sample. Materials required for this process are primarily nanocomposite sensor and composite laminate sample, followed by the regular epoxy resin which is a West System # 105 Epoxy Resin with West System # 206 Slow Hardener.

Figure 3.15 Laminate samples and nanocomposite sensors side by side
This epoxy has a 5:1 mix ratio of epoxy and hardener, a 20 minute working time. Vacuum bagging setup is used to cure the epoxy under pressure to remove any air gaps between sensor and laminate sample.

Figure 3.16 Vacuum bagging setup for attaching nanocomposite sensor

Peelply and breather are used during vacuum bagging to peel off the final samples and to absorb excess epoxy, respectively. This setup as shown in Figure 3.16 is left for 12 hours to cure in room temperature.

Figure 3.17 Sample soldered with copper wires
Once cured, the finals samples are peeled off and copper wires are soldered to the copper tabs on nanocomposite sensor to facilitate stable resistance measurement as shown in Figure 3.17.

3.4 Electro-Mechanical measurement & Data acquisition

The resistance measurement of nanocomposite sensor is obtained by four point probe testing method to IEEE and ASTM standard testing methods (ASTM, 2004; IEEE, 2005; ASTM, 2005). This four point testing technique is specially designed to measure sheet resistance of thin films. This technique is designed to use separate pairs of current carrying and voltage sensing electrodes to make accurate measurements than two terminal testing method which is simpler and more common. Hence, this method is used in this research for resistance measurements.

This method works by forcing a current through the nanocomposite and measuring voltage using a four-wire Kelvin-connection scheme. The resistance of the sample is calculated using Ohm’s Law by passing a controlled current (0.5 Amperes) and recording a voltage drop ($\Delta V$) which is shown in Figure 3.18. The change in resistance can be monitored by the LabVIEW.

Voltage drop using Ohm’s law: $R = \frac{\Delta V}{I}$

![Figure 3.18 Schematic of voltage drop setup](image-url)
National Instruments LabVIEW system design software with a graphical programming syntax that makes it simple to visualize, create, and code engineering systems, is unmatched in helping engineers translate their ideas into reality, reduce test times, and deliver business insights based on collected data. A LabVIEW code is developed to monitor the voltage drop with a data acquisition system (DAQ) as shown in Figure 3.19.

![Figure 3.19 LabVIEW code developed for data acquisition](image)

A tensile test on the nanocomposite is performed using CS-225 Digital Force Tester. A constant head speed of 0.16 mm/sec is applied to the nanocomposite samples and the resistance change is recorded as the sample is subject to loading simultaneously. Followed by this tensile test, a three point bending test is performed on the final laminate samples.
3.5 Experimental setup of flexural test

The sample soldered with copper wires (as in Figure 3.17) is marked for flexural test according to ASTM D7264: Standard test method for Flexural Properties of Polymer Matrix Composite Materials.

![Figure 3.20 ASTM D7264: Three-point loading diagram](image)

In the three-point configuration, the maximum flexural stress is located directly under the center force application member unlike in four-point configuration the bending moment is constant between the central force application members. The resultant vertical shear force in the three-point configuration is present everywhere in the beam except right under the mid-point force application member. The equations for the three-point setup are as follows:

a. Maximum flexural stress \( (\sigma_f) = \frac{3FL}{2bd^2} \)

b. Maximum strain \( (\varepsilon_f) = \frac{6Dd}{L} \)

c. Flexural modulus of elasticity \( (E_f) = \frac{L'm}{4bd^3} \)
Where, \( L \) is the support span, \( b \) is the width of test beam, \( d \) is the depth of test beam, \( F \) is the load at any given point and \( D \) is the maximum deflection. Once marked with the appropriate dimensions from ASTM D7264 standard, the samples are ready for three-point flexural test.

![Figure 3.21 Three-point setup for MTS testing system](image1)

![Figure 3.22 MTS Testing system](image2)
A standard flexural setup for ASTM standard as shown in Figure 3.21 is used in a MTS testing system shown in Figure 3.22 for doing the test.

The three-point setup is fixed into the MTS testing machine and the sample is kept between the top and bottom fixture of the setup such that the ASTM markings are aligned with the roller pins on the three-point setup. The wires of the sample are connected to the power source as well as the data acquisition system (DAQ) and this whole setup is controlled using a MTS controller. The DAQ is connected to a computer with LabVIEW installed in it. A current of certain value is applied to the nanocomposite on the sample.

LabVIEW code developed (shown in Figure 3.19) monitors and controls the voltage on the sample and MTS testing machine respectively. LabVIEW measures the voltage drop and corresponding applied load along with the displacement on the sample. The measured voltage is voltage drop resulting from the resistance of the nanocomposite. The measured load is the load applied to bend the sample for flexural test.
The measured displacement is the deflection of the center of the sample. The voltage drop is unstable at the beginning when the current start to flow through samples and then stabilizes to a constant value.

![Sample under load, Markings made as per ASTM D7264, Nanocomposite, Sample after failure]

Figure 3.24 Sample, under load and after deformation

LabVIEW measures the drop in voltage and force-displacement-time simultaneously while the sample is deformed. The voltage drop data is used to calculate change in resistance in the nanocomposite while force-displacement data can be used to calculate stress-strain data using ASTM standard equations. Same three-point flexural setup is setup for a delaminated sample as well. The voltage drop and force-displacement data for the delaminated sample are also recorded in LabVIEW. From that data, change in resistance and stress-strain data is calculated.
4. Experimental results of Mechanical and Electrical Behavior of Composites

Results of mechanical properties and stress-strain plots of the piezoresistive nanocomposites and the composite laminate samples are presented later in this chapter.

4.1 Mechanical properties of CNTs and carbon fiber composites

4.1.1 Mechanical properties of CNTs

From the moment carbon nanotubes are discovered, it was expected that they would display good mechanical properties like graphite. Graphite had an in-plane modulus of 1.06 TPa and transverse elastic modulus is 36GPa (Palaci, 2012). CNTs are expected to display similar stiffness. It is estimated that tensile strength of graphene is as high as 130 GPa and elastic modulus of graphene is determined to be 1000 GPa (Charles & Gilmore, 2014) from the properties of C-C bonds.

The calculated Young’s modulus of SWNT using ab initio local density calculations to determine the parameters in a Keating potential was 1500 Gpa (Overney, 1993), similar to that of graphite. The first direct measurement of Young’s modulus of arc-MWNTs pinned at one end using an atomic force microscope (AFM) which gave an average value of 1.28 TPa (Wong, Sheehan, & Lieber, 1997).

Yu et al. in 2000 performed stress-strain measurements on individual arc-MWNT inside an electron microscope, for a range of tubes they obtained modulus values of 0.27 – 0.95 TPa (Yu, 2000). They also showed fracture of MWNT at strains of up to 12% and with strengths in the range of 11 to 63 GPa.

The mechanical properties of buckypaper (CNT sheet), with van der Waals bonds between CNTs, are much lower than those of single CNT. Because of the weak Van der
Waals Force in buckypaper, the stress cannot be effectively transferred between CNTs. Measured Young’s moduli of this porous fibrous material reach maximum value of 2 GPa (Yeh, 2007), which is approximately 0.2 % of the modulus of SWCNT. Compared to values reported in the literature, Young’s Modulus and tensile strength achieved in our experiments are lower because the buckypaper used in our work consists of randomly oriented multiwall nanotubes.

Table 4.1 Young’s Modulus and Tensile Strength of buckypaper/polymer nanocomposites

<table>
<thead>
<tr>
<th>Young's modulus (GPa)</th>
<th>Tensile strength (Mpa)</th>
<th>Average tube diameter (nm)</th>
<th>Average rope diameter (nm)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
<td>30</td>
<td>0.8</td>
<td>10~50</td>
<td>(Sreekumar et al., 2003)</td>
</tr>
<tr>
<td>6.9</td>
<td>57</td>
<td>0.8</td>
<td>10~50</td>
<td>(Coleman et al., 2003)</td>
</tr>
<tr>
<td>2.3</td>
<td>6.29</td>
<td>0.8</td>
<td>10~50</td>
<td>(Baughman et al., 1999)</td>
</tr>
<tr>
<td>1.1</td>
<td>17.7</td>
<td>0.8</td>
<td></td>
<td>(Pham et al., 2008a)</td>
</tr>
<tr>
<td>4</td>
<td>32.3</td>
<td>0.8</td>
<td></td>
<td>(Pham et al., 2008a)</td>
</tr>
<tr>
<td>1.5</td>
<td>13.5</td>
<td>1.36</td>
<td></td>
<td>(Pham et al., 2008a)</td>
</tr>
<tr>
<td>2.7</td>
<td>33.2</td>
<td>1.36</td>
<td></td>
<td>(Pham et al., 2008a)</td>
</tr>
</tbody>
</table>

4.1.2 Mechanical properties of carbon fiber composites

Carbon fiber composite materials in practice can be subjected to a wide variety of different loading conditions in the form of mechanical stresses and environmental effects that are related to temperature and moisture (Friedrich, 1989). Mechanical stresses occur under different types of loading, such as tension, compression, and fatigue in structural
components. The mechanical properties of carbon fiber composite material used in this research provided in seller data sheet (Cure 275 °F – Autoclave) are as follows:

Table 4.2 Mech. properties of CF lamina cured for 90min at 275 °F by Autoclave

(Hankuk carbon co. LTD, CF3327-1 EPC : SE-019K)

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Units</th>
<th>Results</th>
<th>Temp (°C)</th>
<th>Test Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile Strength 0°</td>
<td>MPa</td>
<td>716</td>
<td>23</td>
<td>25</td>
</tr>
<tr>
<td>Tensile modulus 0°</td>
<td>GPa</td>
<td>66</td>
<td>23</td>
<td>25</td>
</tr>
<tr>
<td>Poisson’s Ratio 0°</td>
<td>-</td>
<td>0.053</td>
<td>23</td>
<td>4</td>
</tr>
<tr>
<td>Comp. Strength 0°</td>
<td>MPa</td>
<td>547</td>
<td>23</td>
<td>0.20</td>
</tr>
<tr>
<td>Comp. modulus 0°</td>
<td>GPa</td>
<td>60</td>
<td>23</td>
<td>440</td>
</tr>
<tr>
<td>Flex. Strength 0°</td>
<td>MPa</td>
<td>930</td>
<td>23</td>
<td>425</td>
</tr>
<tr>
<td>Flex. modulus 0°</td>
<td>GPa</td>
<td>56</td>
<td>23</td>
<td>440</td>
</tr>
<tr>
<td>Inter laminar shear strength 0°</td>
<td>MPa</td>
<td>68</td>
<td>23</td>
<td>425</td>
</tr>
</tbody>
</table>

4.2 Experimental results of mechanical properties

Figure 4.1 Stress - Strain plot of nanocomposites with coarse graphene platelets (5 wt. %)
The stress-strain plot of piezoresistive nanocomposite with 5 wt. % coarse graphene platelets is shown in Figure 4.1. The maximum stress on the nanocomposite is observed to be 9.83 MPa and the maximum strain observed to be 0.06. The Young’s modulus is calculated to be 163.83 MPa.

Similarly, the stress-strain plot for a tension test is shown in Figure 4.2 and Flexural stress-strain plot for the non-delaminated carbon fiber composite sample calculated from the force-displacement data from LabVIEW which is plotted in Figure 4.3. The maximum flexural stress is observed to be 408.26 MPa and the maximum flexural strain is observed to be 0.0194 mm/mm. The flexural modulus is calculated to be 21.04 GPa. On the other hand, the stress-strain plot for delaminated sample is also plotted in Figure 4.4. The maximum flexural stress is observed to be 272.14 MPa and the maximum flexural strain is observed to be 0.028 mm/mm. The flexural modulus is calculated to be 9.7 GPa.

![Figure 4.2 Stress - Strain plot of composite sample in Tension](image-url)
Figure 4.3 Stress - Strain plot of non-delaminated composite sample (ASTM D7264)

Figure 4.4 Stress - Strain plot of delaminated composite sample (ASTM D7264)
4.3 Electrical properties of CNTs and carbon fiber composites

Several studies have focused over the past decade on piezoresistive polymers made by dispersing CNTs into a polymer to form a conductive matrix (Karimov, 2012; Kang, 2006; Vemuru, 2009; Thostenson, 2008; Grow, 2005; Alamusi & Hu N, 2011; Dharap, 2004; Hu N, 2008; Hu N, 2010; Hu B, 2013). The conductive polymer can be molded to any desired shape. The conductive nature of the carbon fibers allow their use in sensing. In this research, the piezoresistive nanocomposite developed by addition of coarse graphene platelets is used to detect the delamination by using the change in electrical propriety (resistance) while applying load.

CNTs have high electrical conductivities and extremely large length to diameter ratios (aspect ratio) and can improve the conductivity of the polymer matrix with only very low content (Martin, Sandler, Windle, Schwarz, Bauhofer, Schulte, & Shaffer, 2005). They are widely used in the production of conductive composites, electromagnetic shielding materials and antielectrostatic materials (Mahapatra, 2008). In addition CNT based electronics is one of the potential uses of nanotubes. The flexibility of nanoscale design and the availability of both semiconducting and metallic nanotubes enable a wide variety of device configurations, starting with an early prototypical devices utilized the surface on which a nanotube was deposited as a gate (Tans, 1998; Martel, 1998).

Moreover individual CNT have excellent conductivity of about $10^5 – 10^8$ S/m and reaches a high aspect ratio up to 100 – 1000 (Laurent, Flahaut, Peigney, & Rousset, 1998). It has been established that electrical conductivity of buckypapers and mechanical characteristics decrease with increasing molecular mass of CNTs (Boge, Sweetman, & Ralph, 2009). The results of electrical resistivity measurements of nanocomposite samples
with deformation and without deformation are shown here. The electrical resistivity of piezoresistive nanocomposite is obtained using the following expression:

\[ \rho = \frac{R(w \times t)}{l} \]

Where, \( R \) is the calculated resistance by Ohm’s Law, \( w \) and \( t \) are the width and thickness of piezoresistive nanocomposite and \( l \) is the length of composite strip.

Figure 4.5 shows the change in resistivity vs strain plot of piezoresistive nanocomposite with 5 wt. % coarse graphene platelets. All these values reported are averaged from tests on five identical nanocomposite samples. From Figure 4.4 at maximum strain of 0.06 mm/mm, the change in resistivity is observed to be 11.68x10^{-5} ohm-m.
Figure 4.6 Change in Resistivity - Strain plot of non-delaminated composite sample

Figure 4.7 Change in Resistivity - Strain plot of delaminated composite sample
Figure 4.6 shows the change in resistivity against strain plot of a non-delaminated composite sample from three-point bend flexural test. From the plot at a maximum strain of $1.94 \times 10^{-2}$, a change in resistivity of $3.68 \times 10^{-5}$ ohm-m is observed.

Similarly from Figure 4.7, which shows the change in resistivity against strain plot of a delaminated sample, at the maximum strain of $2.08 \times 10^{-2}$, a change in resistivity of $2.67 \times 10^{-5}$ ohm-m is observed and maximum change in resistivity of $2.89 \times 10^{-5}$ ohm-m is observed at a strain of $1.87 \times 10^{-2}$.

4.4 Scanning Electron Microscopy

The scanning electron microscope micrographs of the fracture specimen is shown in Figure 4.8 and Figure 4.9.

Sample after failure

Nanocomposite section

Fracture section

Figure 4.8 SEM micrograph of fracture specimen
These SEM micrographs of fracture specimen indicate that the nanocomposite is completely integrated into the composite layup and does not peel off after composite deformation.
5. Finite Element Model of Flexural Test

5.1 Finite element model of non-delaminated sample

The three-point bend flexural test (ASTM D7264) of the composite laminate is modeled in the general purpose finite element software Abaqus using the standard module for static analysis. Abaqus standard is used in analyzing many types of problems including nonstructural applications. Abaqus/CAE (Complete Abaqus Environment) incorporates the analysis modules for modeling, managing and monitoring Abaqus analyses and results.

The composite laminate is modeled using shell elements with 8-ply layup as in the experimental setup as shown in Figure 5.1

Figure 5.1 Composite 8-ply layup in Abaqus/CAE
The lamina properties (E11 = E22 = 66 GPa) are obtained from the supplier data sheet for the composite. The assembly is modeled similar to three-point bend setup from the flexural test as shown in Figure 5.2. The diameter of the rollers in the assembly is the same as the diameter of the roller pins of the three-point bend setup from the experiments.

![Figure 5.2 Assembly of three-point bend setup as experiments](image)

Shell planar elements are used to model the laminate and solid extrude elements for rollers. Contact properties with tangential and normal behaviors are given for the contact between laminate and rollers. These contact properties are given with surface-to-surface interactions. The location of rollers are as per ASTM standard as used in experiments.

After assembly, load is applied on the laminate model as in experimental setup and all the results like applied load and displacement of the center of the laminate are collected.
and plotted in Abaqus/CAE. There are a total of 15808 linear quadrilateral elements of type S4R in the laminate model with 16165 nodes. Figure 5.3 shows contour of the deformed laminate when load is applied.

Figure 5.3 Strain contour of deformed laminate sample

5.2 Finite element model of delaminated sample using VCCT

Virtual Crack Closure Technique (VCCT) for Abaqus/CAE is a capability within Abaqus that provides delaminated / debonding analysis capabilities for structures containing bonded surfaces.

VCCT for Abaqus utilizes the convergence and stabilization algorithms and the existing load incrementation capabilities. Supporting active delamination between bonded surfaces, calculating crack growth based on fracture mechanics, inclusion of mixed mode crack growth, computing intermediate crack shape, allowing VCCT to be performed during
nonlinear analysis, etc., are some of key advantages of VCCT for Abaqus/CAE. In this research, the delamination in the finite element model is incorporated using VCCT in fracture criterion in the contact property. Direction of crack growth relative to local 1 – direction is taken as maximum tangential stress direction. A tolerance of 0.2 with zero viscosity is used in VCCT. The strain energy release rate (GIC) of 600 J/m$^2$ is used as VCCT input.

Figure 5.4 shows the strain contour of deformed delaminated sample modeled in Abaqus/CAE using VCCT. The delaminated composite laminate is also modeled using shell elements (element type) in Abaqus. The results of the simulation are captured and plotted in Abaqus.
5.3 Results of Finite Element Model (Abaqus/CAE)

Force and displacements values of the three-point bend flexural test simulation are collected for both non-delaminated and delaminated (VCCT) model. Figure 6.4 shows the force vs displacement plot of a non-delaminated composite laminate model from Abaqus/CAE. This result is again plotted using the data from Abaqus.

![Figure 6.4 Force vs Displacement of non-delaminated laminate sample](image)

From the Figure 6.4, a maximum force of 255.53 N is observed for a displacement of 0.017 m. The values of this plot are obtained from Abaqus/CAE. A similar plot is plotted for a delaminated sample. Figure 6.5 shows force vs displacement plot of a delaminated composite laminate model obtained from Abaqus/CAE. The delamination is created in
Abaqus using Virtual Crack Closure Technique (VCCT) in Abaqus/CAE. A maximum force of 79.27 N is observed at a displacement of 5.39 mm.

![Force vs Displacement of delaminated laminate sample from Abaqus/CAE](image)

Figure 6.5 Force vs Displacement of delaminated laminate sample from Abaqus/CAE
6. Analysis and Discussion

The final results and discussion of experimental and finite element model are presented in this chapter. The experiments measure the electrical resistivity of the piezoresistive nanocomposite sensor when the attached composite laminate (delaminated and non-delaminated) is subject to mechanical deformation from the three-point bend flexural test. The finite element simulation measures the strain created in the nanocomposite region on the modeled composite laminate (delaminated using VCCT and non-delaminated).

6.1 Electro-mechanical properties of piezoresistive nanocomposite

![Figure 6.1 Stress – Strain and Resistivity – Strain response of a nanocomposite](image)

Figure 6.1 Stress – Strain and Resistivity – Strain response of a nanocomposite
Piezoresistive nanocomposite sensor exhibit change in resistance when subject to mechanical deformation. As the composite laminate is deformed in three-point flexural test, the strain is transferred to the nanocomposite attached on top of the laminate. This strain created on the nanocomposite results in the change in resistivity.

Figure 6.1 shows the Stress – Strain and Resistivity – Strain response of a piezoresistive nanocomposite in tension, the nanocomposite is used in this research to act as a sensor in order to detect delamination. Figure 6.2 shows the Stress – Strain response of composite laminate and Resistivity – Strain response of piezoresistive nanocomposite in three-point flexural test.
Change in resistivity of $11.68 \times 10^{-5}$ ohm-m can be observed in the piezoresistive nanocomposite from Figure 6.1 and a maximum stress of 9.83 MPa is also observed on it. Change in resistivity of $3.68 \times 10^{-5}$ ohm-m is observed in the piezoresistive nanocomposite with application of deformation on the composite laminate in Figure 6.2. Maximum flexural stress of 408.26 MPa is observed on the composite laminate sample (Non-delaminated).

![Figure 6.3 Stress – Strain and Resistivity – Strain response of nanocomposite attached to a delaminated composite laminate](image)

Figure 6.3 shows the Stress – Strain response of composite laminate and Resistivity – Strain response of piezoresistive nanocomposite. Change in resistivity of $2.89 \times 10^{-5}$ ohm-m is observed in the piezoresistive nanocomposite with application of deformation on the composite laminate. Maximum flexural stress of 272.14 MPa is observed on the composite laminate sample (Delaminated).
6.2 Comparing results of experiments and simulation

The results of experimental tests and Abaqus simulations are compared by plotting them together for both non-delaminated and delaminated laminate samples.

Figure 6.6 shows the result of experiment and simulation of a non-delaminated sample. The maximum force that is observed in this plot is 233N in experiments and 255N in simulation while the maximum displacement observed is 1.77x10^{-2} m in experiments and 1.7x10^{-2} m in the simulations.

Figure 6.7 shows the result of experiment and simulation of a delaminated sample. The maximum force that is observed in this plot is 60N in experiments and 79N in simulation while the maximum displacement observed is 5.85x10^{-3} m in experiments and 5.39x10^{-3} m in the simulations.

Both of these plots clearly show that there is only a small difference (they are very close) in the values recorded for both delaminated and non-delaminated cases. This proves that the modeling of composite laminate and the Virtual crack closure technique (VCCT) applied in Abaqus/CAE is correct. The next thing will be finding the strain on the nanocomposite region of the composite laminate model from Abaqus and compare the strain to the actual strain created on the nanocomposite during tension.
Figure 6.6 Comparing results of Simulation and Experiment in non-delaminated sample

Figure 6.7 Comparing results of Simulation and Experiment in a delaminated sample
6.3 Strain in composite region (on laminate) in Abaqus/CAE

Nanocomposite region is defined on the composite laminate modeled in Abaqus/CAE. This region helps calculating the strain created in the region when load is applied for three-point flexural test.

Assembly

Figure 6.8 Cut out region of the nanocomposite section from assembly

Quad-dominated elemental mesh

Figure 6.9 Cut out region of the nanocomposite section from mesh
Figure 6.8 and Figure 6.9 show the cut out region of the nanocomposite section separated from the assembly and mesh, respectively. After defining and solving the model for three-point bend flexural simulation, the strain values are collected from the nanocomposite region defined. Figure 6.10 shows the cut out region of nanocomposite section from visualization after simulation.

![Visualization of Abaqus/CAE](image)

Figure 6.10 Cut out region of the nanocomposite section from Abaqus visualization

The strain in the nanocomposite region of the overall sample is partitioned and calculated for three elements, one at the center and two at left and right sides of the nanocomposite region as shown in Figure 6.11. This calculation is done for both non-delaminated and delaminated laminate sample models. The calculated strain at these three points of a non-delaminated sample model are plotted against displacement in Figure 6.12.
Figure 6.11 Nanocomposite region and elements selected for strain calculation

Nanocomposite section from the non-delaminated laminate

From Figure 6.12, maximum strain is identified at the center element which is $13.97 \times 10^{-3}$. At left most and right most elements the maximum strain observed is $5.56 \times 10^{-3}$ and $6.64 \times 10^{-3}$, respectively. Similarly the strain is plotted for a delaminated laminate as well, as shown in Figure 6.13.
From Figure 6.15, maximum strain is identified at the center element which is $21.61 \times 10^{-3}$. At left most and right most elements the maximum strains observed are $7.51 \times 10^{-3}$ and $7.96 \times 10^{-3}$, respectively. Once the strain in the nanocomposite region is obtained from Abaqus/CAE, the next step is to correlate this strain to the tension test results obtained from the piezoresistive nanocomposite as shown in Figure 6.1.

### 6.4 Strain correlation of piezoresistive nanocomposite

The strain observed on the nanocomposite region in Abaqus/CAE for both non-delaminated model and delaminated model are correlated to the strain created on the piezoresistive nanocomposite during the tension test (Figure 6.1). Figures 6.14 and 6.15 show the correlation plots of strain and resistivity of a nanocomposite for non-delaminated model and delaminated model.
Figure 6.14 Correlation between the strain and resistivity of nanocomposite from three-point bend test and tension test of a non-delaminated model.

Figure 6.15 Correlation between the strain and resistivity of nanocomposite from three-point bend test and tension test of a delaminated model.
The strain correlated to the tension test of the nanocomposite and corresponding electrical resistivity is compared to that of experimentally obtained in the flexural test. A one-to-one correlation between strain in the nanocomposite and electrical resistivity in both tension and bending is observed. This proves that the simulations and the experiments in studying the piezoresistive nanocomposite are comparable.

6.5 Conclusion

Based on all the results above and plotting the experimental results of non-delaminated and delaminated samples together we have Figure 6.16
From Figure 6.16 it can be observed that, the change in resistivity at maximum strain for a non-delaminated laminate is $3.68 \times 10^{-5}$ ohm-m while in the delaminated sample it is $2.67 \times 10^{-5}$ ohm-m. This indicates that the piezoresistive nanocomposite sensor that is developed in this research can be effectively used to detect strain changes caused by delamination and other defects in composite structures.
7. Summary and Recommendations

7.1 Summary

In this research, piezoresistive nanocomposite sensors are fabricated from buckypaper with 5 % wt graphene nanoplatelets added with resin by vacuum assisting process, and cured in room temperature. Resistance of these nanocomposite sensors are measured using the four-point probe testing method under applied tensile loading. Composite laminates composed of 8 plys are also fabricated using compression press with and without delamination for testing the nanocomposite sensors.

The final sample is prepared by attaching the nanocomposite sensor on top of the composite laminate and marked with ASTM D7264 markings. A three-point bend flexural test is carried out on these samples using the MTS testing machine connected to a computer with LabVIEW code developed (Figure 3.18) in it. The sensors are subject to electrical current and LabVIEW code is used to monitor the voltage drop of the nanocomposites along with measuring the stress and strain on the laminate by flexural test. The resistance can then be calculated using Ohm’s law.

The resistance of the nanocomposite changes due to the mechanical strain created due to flexural test and is measured through these experiments. The resisitivity – strain of the nanocomposite sensor attached on top of delaminated and non-delaminated sample shows a great variation in both cases. For a non-delaminated composite laminate sample, the resistivity occurred at maximum strain is 3.68x10^{-5} \, \Omega\cdot m, while in the delaminated composite laminate sample it is 2.67x10^{-5} \, \Omega\cdot m. Seeing all these results, it is believed that the piezoresistive nanocomposite sensor can be used to detect delamination in carbon fiber composite structures.
The finite element models are used to correlate strains from the three-point flexural tests of carbon fiber composite laminates – nanocomposite sensor assembly to the strains and resistivity changes in the tension tests of standalone nanocomposites indicating applicability of these sensors under multiple loading conditions.

### 7.2 Recommendations for Future Work

The piezoresistive nanocomposite sensors developed in this research can be employed to detect the effectiveness of a composite repair patch as shown in Figure 7.1. Future work along these lines will advance the concepts developed here. Also these kinds of studies can be expanded to include more advanced structures such as stiffened panels, wing-ribs, fuselage panels etc.

Figure 7.1 Composite repair patch with nanocomposite sensor for detecting the effectiveness of the repair
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