2008

Characterization of thermal and electrical properties of Fiber Reinforced Polymer (FRP) composites

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Characterization of Thermal and Electrical Properties of Fiber Reinforced Polymer (FRP) Composites

Raja Ram Tipirneni

**Thesis Submitted to the
College of Engineering and Mineral Resources
at West Virginia University
in partial fulfillment of the requirements
for the degree of**

**Master of Science
in
Industrial Engineering**

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Morgantown, West Virginia
2008**

Keywords: Thermal conductivity, Three Dimensional Thermal Conductivity, Electrical Conductivity, Thermosetting Resin, Graphite Powders, Conductive Composites, E-glass, Carbon fiber, Electrical property, ANOVA, Regression.

ABSTRACT

Characterization of Thermal and Electrical Properties of Fiber Reinforced Polymer (FRP) Composites

Raja Ram Tipirneni

Thermal conductivity of a polymer composite is a function of resin type, fiber type and architecture, fiber volume fraction, direction of heat flow, and service temperature. Recognizing the thermal responses in Fiber Reinforced Polymer (FRP) composite decks play a critical role in their performance, accurate thermal measurements of FRP decks are essential. A major focus has been placed on measurements of thermal conductivity in through-the thickness direction of a FRP composite.

Current research focus has been on characterization of thermal conductivity in the planar direction (along the direction of the fiber and transverse direction of the fiber) of a composite laminate. Thermal conductivity characterization in three dimensions has been carried out for carbon fiber reinforced vinyl ester systems. The results have revealed that the bonding between the layers of fabric depends on the type and property of the resin associated with that fabric. The characterization has been carried out using 'Guarded heat flow meter method' in accordance with ASTM E1530. Some of the composite samples were selected and exposed to thermal weathering to study the effects of multi-cycle thermal history. This enabled us to characterize thermal conductivity as a function of weathering time for a range of composite samples including carbon fiber/vinyl ester composites manufactured through pultrusion and resin infusion processes, resin infused carbon fiber/epoxy composite, and pultruded GFRP bridge deck samples. Also, the amount of thermal compound applied was found to affect the thermal conductivity.

Most neat resins, including thermoplastics and thermosets are electrical insulators. However, conductive additives or fillers can be added into resins to produce electrically conductive resins and composite materials. At a specific concentration of conductive filler, electro conductive channels are formed and the insulators are turned

into semi-conductors. This transformation process can be described within the framework of the percolation theory. Constructed Facilities Center (CFC)-West Virginia University (WVU) has developed a smart material system by adding nanomaterials to neat resins to produce conductive composite sensors. Statistical Analysis of the experimental data obtained for thermal conductivity, sensor and wearing surface have been evaluated.

ACKNOWLEDGEMENTS

The author would like to sincerely thank Dr. Ruifeng Liang and Dr. Hota V.S. GangaRao for introducing me to the field of research. They provided valuable insight into all areas of the project from conducting experiments, to analyzing the test data. Their indispensable contributions will never be forgotten.

A special thanks to Dr. Majid Jaraiedi for serving as committee chair and for his support in helping me to statistically analyze the experimental data. I extend my thanks to Dr.B.Goopalakrishnan for his valuable comments and suggestions as a committee member. I would like to express my sincere gratitude and appreciation to the Department of Industrial and Management Systems Engineering, West Virginia University for allowing me this opportunity to pursue M.S degree in the department.

I wish to thank Jim Hall, Dave Turner, Jerry Nestor and Bill Comstock for their help in preparing samples. I wish to thank Linda Rogers, Lynne Jacobs, Kasandra Kasten and the staff of Constructed Facilities Center (CFC) for their support during the course of work. Many additional students and faculty were very helpful during the course of this work, and their assistance was greatly appreciated. Among them were Dr. Robert C Creese, Liviu Magean, Shushant Agarwal, Ashish Suresh Bambal, Manab Medhi, Aneesh Reddy.

I wish to thank U. S. Department of Transportation – Federal Highway Administration (USDOT-FHWA) under the Center of Excellence program for funding this project. Anter Corporation, Pittsburgh, PA for providing latest model instrument for testing the samples for thermal conductivity.

Last but not the least, I am very much thankful to my mom, family and friends for their loving support and encouragement which made all this possible.

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Composites are engineering materials made out of two or more components. Most of the composites can be tailored to obtain properties better than each individual component. A polymer composite reinforced with fiber is called FRP composite. Considering a composite involves two or more macro constituent phases, the matrix is called continuous phase and the fibers are called reinforcing phase (Mirmira, 1999). The advantages of composites include high strength to weight ratio, non-corrosive, less maintenance, high electrical resistance, wear resistance, electromagnetic transparency, appealing appearance etc. Considering the mechanical and thermal properties of composites, they are anisotropic in nature (i.e. the properties vary with orientation), but most of the composites can be considered as orthotropic (i.e. nine different constants are required to describe an orthotropic material). This problem emphasizes the necessity of knowledge on thermal behavior of composites. As the thermal conductivity property is unique for a given composite, the thermal conductivity of a material is required in order to deal with thermal stresses of that material.

Structural health monitoring and damage detection including remaining service life have recently become major areas of interest for a large number of academic and commercial laboratories [Law, 1998]. The need to develop in-service and on-line health monitoring techniques is increasing because of structural safety consciousness of end users. Techniques using piezoelectric sensors allow systems and structures to be monitored for their structural integrity while in service, and are useful not only to improve performance reliability but also to reduce maintenance and inspection costs.

FRP composite bridges and structures are a very common site nowadays. FRP bridge decks can be pultruded in modular units and then joined to make a full-scale bridge. It is at those joints that a FRP bridge is vulnerable to crack growth and propagation. The crack can initiate at any location in the joint and has to be detected. The technique should therefore cover a broader area of the structure and also should be a real time monitoring technique that can be operated even when in service. It should also be

sensitive to deflections (strain), easy to install, operate and acquire data in the field. Finally, the production and installation costs of the developed sensor should be lower than the conventionally available techniques/sensors.

1.2 Objective

- The primary objective of this work is to develop composites with reduced thermal stresses to avoid catastrophic failures
- To develop FRP composite bridges with self-deicing capacity, thus being environment friendly and to measure the thermal conductivities of composite samples made from different fiber and matrix combinations using different manufacturing methods
- To prepare FRP composite samples with 510A vinyl ester resin and evaluate their thermal conductivity values.
- To study the effect of multi-cycle thermal weathering and the amount of thermal compound applied on the FRP samples
- Thermoset resin based conductive polymer composites have been developed and evaluated in the past work. The advantages of developing thermoset resin based conductive polymer composites include excellent compatibility with vinyl ester based composite structures, low cost, ease of manufacturing, and even in-line production through integrally embedding into part of the structures.
- Another primary objective is to develop the material to be as much sensitive to strain as the existing conventional sensors like strain gages etc.
- To develop conductive wearing surface, which can function well as fast and effective heating surface.

1.3 Scope

The structure of this report is as follows. Chapter 2 contains a brief description of published literature on prediction and determination of thermal conductivity of composite materials and conductive composites. It also addresses briefly how researchers are trying to use them for damage detection and the limitations they are facing.

Chapter 3 presents details on constituent materials and manufacturing techniques of test samples and discusses the materials used for the development of smart composites.

Fiber reinforcements include Carbon fiber while resin types are 510-A vinyl ester and Hetron 922 L25 Vinyl ester for the conductive composites. The samples were manufactured in Constructed Facilities Center (CFC) - West Virginia University (WVU) laboratories using conventional manufacturing techniques like Hand-layup, Compression molding. It also discusses the manufacturing technique that is employed to make the sensor and evaluation of the effect of wearing surface on the bridge deck panel surface.

Chapter 4 deals with the test method to determine thermal conductivity. The measurements of thermal conductivity of composite materials were carried out on a guarded heat flow meter entitled Anter system model Unitherm 2022. This model is fully computerized and equipped with a mid range flux module covering the thermal conductivity of materials in the range of 0.1 to 40 W/m-K in terms of test standard ASTM E 1530. The tests that were done on the sensors were also discussed.

Chapter 5 presents the experimental design for all the experiments that were performed on thermal conductivity, conductive composite sensor and the wearing surface. This chapter deals with the evaluation of design parameters considered for thermal property and electrical property of fiber reinforced polymer composites. Analyses of covariance, analysis of variance and regression analysis concepts have been applied to design the regression model equation for the parameters in thermal and electrical tests.

Chapter 6 deals with the thermal conductivity values obtained for a variety of composite materials and their data analysis by using the statistical methods. This chapter also includes discussions on the influence of various parameters on the thermal conductivity of FRP composites. Sensitivity of the sensors is also evaluated as a function of filler concentration and its statistical support. Tension tests were performed on the sensors to correlate mechanical property to their electrical property and obtain a relation between them. The mechanical properties of the sensor were found to vary with graphite powder's concentration and their size. The conductive resin system was further evaluated as conductive wearing surface (heating element).

Chapter 7 describes the conclusions obtained from the experimental results and recommendations for future research.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Increasing use of composites for various applications emphasizes its importance/significance in the thermal property analysis of an engineering system. Thermal conductivity of a composite (combination of two or more constituents) can be measured by experimental methods. Analytical equations are essential to predict thermal conductivities of a composite material. Information on the thermal properties of composite materials would facilitate the design of an engineering system made of FRPs.

2.2 Theory of thermal conductivity

The theory of thermal conductivity was proposed by Fourier in 1822 (Source: Electronic Properties of Materials by Rolf E. Hummel, Published by Springer, 2001). According to Fourier, the fundamental heat conduction equation can be stated as “*For a homogeneous solid, the local heat flux is proportional to the negative local temperature gradient*”. For one dimensional steady state heat transfer, this statement can be represented by Equation 2.1:

$$q'' = -K \frac{dT}{dx} \quad 2.1$$

Where q is the heat flux, K is the thermal conductivity of the material, which is a positive 2nd order tensor quantity, $\frac{dT}{dx}$ represents change in temperature across the thickness and negative sign indicates the temperature reduction from hotter surface to cooler surface. According to Equation 2.1, conductivity can be given as (under the assumption, that heat is not lost in its plane)

$$K = (Q/A) / (\Delta T / \Delta L) \quad 2.2$$

where K is the thermal conductivity (W /m-K), Q is the Heat Flux (W), A is the cross sectional area of the specimen (m²), ΔT is the Temperature difference (K), ΔL is the over all distance (m). Thus, the thermal conductivity of a material can be defined as a rate at which heat is transferred by conduction through a given unit area of a given material,

when the temperature gradient is normal to the cross sectional area. The thermal conductivity of a composite material depends on the fiber, resin materials, fiber volume fraction, orientation of the fiber, direction of heat flow and operating temperature.

2.3 Factors affecting thermal conductivity of composite materials

Thermal conductivity of composites is anisotropic in nature. The knowledge of thermal conductivity of composites is needed for accurate design. Data about thermal conductivity of resin helps reduce stresses related to shrinkage of composites during cure and mismatch in thermal expansion coefficients. Before conducting experiments to determine thermal conductivity of various composites, knowledge about the effect of different parameters influencing thermal conductivity is essential.

2.3.1 Fibers

Fiber is the reinforcing phase of a composite material. Thermal conductivity of a composite depends upon the thermal conductive nature of the fiber and matrix. Commonly used fibers for composites include Glass, Carbon, and Aramid etc.

Glass fibers are commonly used for engineering composites. Their uses include the manufacturing of automotive parts, pipes, structural members etc. Glass fiber is available economically in abundance with good mechanical properties; thus widely used in composite structures (Barbero, 1998). Basing upon different applications glass fiber (silica–oxygen network) is classified into E glass, C glass, and S glass fibers (Barbero, 1998). E glass is used as an insulator and mostly used in electrical industry, hence got the name “E” before the word glass. E-glass also has good mechanical properties in addition to low cost and ease of usability. The letter “S” in S-glass stands for structural applications. S-glass got different chemical formulation and it has higher strength to weight ratio, and higher elongation strain percentage. S glass is a bit expensive and mostly used in structural application, and C-glass fibers are advantageous in resisting chemical corrosion. Glass fibers are available in different forms like continuous, chopped and woven fabrics.

The microstructure of any fiber plays vital role in carrying heat. The glass fiber has an amorphous structure. It consists of SiO_2 molecules and forms a three dimensional

silica polyhedral network along the length of the fiber. It behaves nearly isotropic, resulting in nearly same conductivity properties in any direction of the fiber.

Carbon fibers are manufactured using precursor materials like rayon, petroleum or coal tar pitches and polyacrylonitrile (PAN) (ASM Handbook, 2001). The conversion of pitch or PAN precursor to carbon fibers involves manufacturing steps like fiber formation by spinning, stabilization to thermoset the fiber, carbonization, graphitization, surface treatment and sizing (Mirmira, 1999). During graphitization stage at higher temperatures the crystallites are properly ordered and oriented along the axis direction of the fiber. In PAN based carbon fibers, during the graphitization stage the linear structure of carbon atoms transforms into a planar structure called as basal planes and are oriented or stacked along the axis of the fiber. These basal planes are closely packed and are responsible for the high modulus and higher electrical and thermal conductivities along the axis of the fiber.

Natural fibers are becoming potential alternatives for glass fiber reinforced composites in many applications (Joshi et al 2004). Usage of natural fibers like hemp, jute, flax, cotton etc. instead of synthetic fibers leads to increase in specific properties like impact strength, crash behavior, sound absorption, thermal insulation and reduction in component's weight, pollutants, and greenhouse gas emissions making the composite more environmental friendly. Natural fiber composites have many applications including automobile parts like door trim panels, headliners or back panels. Natural fiber is filled with cellulose material, which acts as an insulator, thus a natural fiber composite shows much lesser thermal conductivity when compared to a glass fiber reinforced polymer (GFRP) composite.

2.3.2 Resins

Matrix materials are of different types like metal matrix, ceramic matrix and polymer matrix. Polymer matrices are most commonly used because of cost efficiency, ease of fabricating complex parts with less tooling cost and they also have excellent room temperature properties when compared to metal, ceramic matrices.

Polymer matrices can be either thermoplastic or thermoset. Thermoplastic materials are formed by addition polymerization. Thermoplastics soften or fuse when heated, harden and become rigid after cooling. Unlike thermosets, thermoplastics can be

modified or reused upon the need. Thermoplastics have longer shelf life and higher fracture toughness than thermoset resins. Thermoplastic resins have high viscosity and less creep resistance when compared to thermosets (Barbero, 1998).

Thermoset matrices are formed due to an irreversible chemical transformation of a resin into an amorphous cross-linked polymer matrix. Due to huge molecular structures, thermoset resins provide good electrical and thermal insulation. Thermosets have low viscosity, which allow proper fiber wet out, excellent thermal stability and better creep resistance. The most commonly used thermoset resins are epoxy, polyester, vinyl ester and phenolics. Mostly thermoset resins can be formulated to give a wide range of properties upon the requirement.

Epoxy resin has excellent adhesion property compared to other resins. In addition to that it has low shrinkage upon curing, good chemical resistance, excellent mechanical properties. Epoxies have been used for advanced composites due to their adhesion to wide variety of fibers, with superior mechanical and electrical properties, good performance at elevated temperatures. Epoxies are expensive compared to polyester and less resistant to moisture. Polyester has the advantages of low cost, ease of handling, good chemical resistance with reasonable mechanical properties. Polyester and epoxy makes approximately 85% of the fiber reinforced polymer composites.

Vinyl ester has the chemical backbone of epoxy and curing mechanism of polyester, so it got improved resistance to chemical attack and ease of fabrication. Vinyl ester is stronger than polyester and less expensive than epoxy. Vinyl ester has better resistance to moisture absorption than polyester and the bonding capability is not good when compared to epoxy to all kinds of fibers. Vinyl ester offers good mechanical properties and excellent corrosion resistance. The bonding ability of vinyl ester is good to glass fiber but less efficient with carbon or Kevlar. Phenolic resin cures through condensation reaction, which produces water during the reaction. It has excellent properties like high temperature and creep resistance, good thermal insulation and sound damping properties in addition to first-rate fire properties.

2.3.3 Fillers

The primary advantage of using filler material in composites is to reduce the overall cost of the composite. In addition to reduction of cost, filler materials also serve

as major ingredient, which improves the performance of the composite by enhancing crack resistance, reducing shrinkage, influencing mechanical strength, improving fire resistance etc. Major filler materials used in composite manufacturing are calcium carbonate, kaolin (clay), Aluminum-trihydrate etc.

2.3.4 Additives

Additive materials are primarily used to modify and tailor material properties of the composite. By introducing additives into resin system it enhances the processability or durability of the composite. Additives help in increasing the performance or a specific property as well it increases the overall cost of the product. Various additives widely used to boost the thermal and electrical conductivity of the resin are graphite powder, chopped carbon fiber, carbon nano-tubes etc.

2.3.5 Manufacturing Methods

As the thermal conductivity of a polymer composite is based upon the conductivity of fiber and resin, being a polymer, resins are usually insulating and the conductivity is dominated by fiber material. The compactness of fibers per unit area influences the conductivity of the composite. Fiber packing in a composite depends on the method of manufacturing. The various composites manufacturing techniques are Hand lay-up, Compression molding, Resin Transfer Molding, Pultrusion etc (Barbero, 1998).

2.3.5.1 Hand lay-up

Hand lay-up is the oldest and simplest method of manufacturing composites. The tools required for the process are a mold to accommodate dry manufacturing according to the desired shape and a roller to facilitate uniform distribution of resin. Virtually any sized composites can be manufactured using this method. This method is the cheapest method of manufacturing but it has some disadvantages such as long curing time, low production rate, and further the quality of the composite depends on the skill of the worker.

2.3.5.2 Compression molding process

This method is commonly used to manufacture sheet molding or bulk molding. Compression molding machine consists of a male and female dies or platens to form the mold. The reinforcement combined with resin is placed in the mold and a hydraulic press is used to apply high pressure by closing male and female halves of the mold. After the material is cured, the pressure is released and the part is removed from the mold. Exterior body panels for structural members such as automobile bumpers are widely manufactured using this method.

2.3.5.3 Resin Transfer Molding (RTM)

This manufacturing method uses a mold with an inlet to introduce resin/catalyst mixture and vent ports to allow air to escape. In this method, resin and catalysts are mixed proportionally in an injection head and then pumped into the mold. Dry reinforcement is placed inside the mold and is closed. Resin is pumped into the mold till the mold is full. After the resin is cured, the part is removed from the mold. RTM has been further modified by using vacuum to suck resin catalyst mixture inside. In this case, vacuum bags are used along with molds and hence this method is called as Vacuum Assisted Resin Transfer Molding (VARTM). VARTM process enhances resin flow and reduces void fraction. RTM process is cleaner with less emissions of volatiles. The applications of this process includes manufacturing of auto body panels, swim pool panels, sandwich panels etc. A schematic of Resin Transfer Molding is shown in Figure 2.1

Figure 2.1 Schematic of Resin Transfer Molding

(Photo Courtesy: <http://www.esi-group.com>)

2.3.5.4 Pultrusion

Pultrusion is a continuous and highly automated molding process used in fabrication of composite parts that have a constant cross section. Reinforcement materials are arranged in such a pattern so that they match the profile of the die. The fibers are pulled through a resin bath and then to the heated metal pultrusion die. The die is maintained at a precise temperature so that it can transfer heat to the fiber and liquid resin. The heat energy is used to carry on the polymerization of the resin to matrix. The cooled solid part is then pulled from the die and cut to desired length. Pultrusion is a low cost process for a large volume production. Applications of this process are in the fields like construction, transportation, electrical etc. A schematic of pultrusion process is shown in Figure 2.2

Figure 2.2 Schematic of Pultrusion Process

(Photo Courtesy: www.pulwellpultrusions.com)

2.4 Experimental approach for thermal conductivity measurements

There are various test methods available for thermal conductivity measurement and each method depends mainly on configuration of a material and job requirement. One principal experimental method for determination of thermal conductivity is discussed below:

2.4.1 Guarded heat flow meter

This method is based on two dimensional steady state techniques and is used to measure and compare thermal properties of materials under controlled conditions and their ability to maintain required thermal conductance levels (ANTER, 2003).

The specimen and a heat flux transducer (HFT) are sandwiched between two flat plates controlled at different temperatures, to produce a heat flow through the stack. A cylindrical guard surrounds the test stack and is maintained at a uniform mean temperature of the two plates, in order to minimize the lateral leak of heat. At steady state, the difference in temperature between the surfaces contacting the specimen is measured with temperature sensors embedded in the surfaces, together with the electrical output of the HFT. The output voltage is proportional to the heat flow through the specimen, HFT and the interfaces between the specimen and the apparatus. The coefficient of thermal conductivity can be obtained by prior calibration of system with the specimens of know thermal conductivity.

At equilibrium, the thermal conductivity of material can be given as

$$K = (\Delta x) / R_s \quad 2.3$$

$$R_s = (N(\Delta T)/Q) - R_o \quad 2.4$$

where K is the thermal conductivity (W/ m-K), R_s is the resistance of unknown specimen ($m^2 \cdot K/W$), N is the HFT calibration constant, ΔT is the temperature difference between one surface of the specimen and the other surface (K), Δx is the specimen thickness (m), and R_o is the contact thermal resistance.

Other methods also based on two dimensional steady state techniques for wide range of operating temperatures include Heat Flow Meter, Guarded Hotplate, Hot Wire, and Laser Flash.

2.5 Conductive composites

Electrically conductive plastics are commonly made by adding conductive fillers such as metallic powders or carbon black into neat resins. For example, when particulate additives are mechanically mixed with molten polymers, particle chains may be formed by chance as a result of the random positions of individual particles. Such chains, or networks, provide conducting pathways. High shear rate flows, which characterize

ordinary compounding methods, such as extrusion or batch mixing, lead to random and nearly uniform particle distributions. The likelihood for extended conducting pathways to form is accordingly low at small particle concentrations. If the particle concentration is higher than a certain value called the percolation threshold, these associations among particles yield a network that spans the material and the solidified composite becomes electrically conductive [Danescu, 2002].

The concept of conductive composites is not new at all. There have been a huge number of references on conductive composites and their applications. However, they are mostly concerned about thermoplastic resin based conductive polymer composites instead of thermosetting resin based conductive composites as covered in the present study. Conducting polymers also have a wide variety of applications in microelectronics [Angelopoulos, 2001]. Conducting polymers are effective discharge layers in electron beam lithography. They also find applications in metallization of plated through-holes for printed circuit board technology, provide excellent electrostatic discharge protection for packages and housings of electronic equipment, provide excellent corrosion protection for metals, and may have applications in electromagnetic interference shielding.

Conductive composites using carbon fiber are being proposed as tools for health and usage monitoring of structures. Since this is an area, which is being investigated, there are very few references and publications. Researchers have explored the conductivity of carbon fiber reinforced plastics (CFRP) and are considering them a self monitoring material without any need for additional sensing elements [Schueler, 1997]. However for this to become a reality the conductivity map of the entire structure need to be constructed and relationship between the conductivity and various usage and damage related variables need to be established. The method requires lot of skill for extracting and interpreting the data. The results of their experiments are based on the fact that internal damage, such as fiber fracture and delamination, decreases the conductivity of the composite laminates.

Figure 2.3 Experimental setup of EIT applied to a quadratic specimen using the adjacent data collection strategy [Ruediger 1997]

A possible experimental setup is shown in Figure 2.1. An electric current is injected via the two electrodes and the potential difference between all other neighboring electrodes is measured. By taking various combinations of current injecting electrodes and repeating the potential difference measurements, the resistivity distribution inside the sample can be obtained. The change in the resistivity profile can be attributed to the damage in the specimen. Technological and manufacturing hurdles are barriers in the implementation of the technique in addition to those mentioned in the preceding paragraphs.

CHAPTER 3

MATERIALS AND SAMPLE PREPARATION

3.1 Introduction

The objective of this investigation is to determine conductivity properties of FRP composites in three different directions (i.e. longitudinal, transverse, and through-the-thickness). The properties are function of composition parameters and process parameters. Herein, the thermal conductivity measurement in the transverse direction means that a sample is prepared in such a way that heat flow direction is transverse to primary fiber orientation of the sample; similarly, the thermal conductivity measurement in the longitudinal direction means heat flowing along the fiber direction; and the thermal conductivity measurement in the thickness direction means heat flowing through the thickness direction. The thermal conductivity of an anisotropic composite material depends on the resin nature, fiber type and architecture, fiber volume fraction, manufacturing technique, direction of heat flow and operating temperature, leading to a high degree of complexity.

Most polymers or plastics after curing are insulators of heat and electricity. They can be made to conduct both heat and electricity by adding conductive fillers into the resin systems before curing. These fillers have to be added in controlled amounts so they do not affect adversely the mechanical properties of the polymer. In this research, we dealt with vinyl ester thermosets as our polymer resin and the conductive filler was graphite powder. Description of the material, manufacturing of coupons and testing procedure are provided in the following sections.

3.2 Thermal conductivity test matrix

The research proceeded as outlined in Table 4.1, Thermal Conductivity Test Matrix. The test matrix has included various material parameters and process parameters, leading to a spectrum of composite samples for their thermal conductivities along 0° and 90° fiber orientations and through the thickness direction. The resins used for the study were 510-A Vinyl ester. It should be noted that sample preparation was the most

challenging part of the entire process. Many tools were used specially for sample preparation such as carbide grit, carbide tipped hole-saw, and diamond tipped hole-saw. In addition, attention to environmental and health safety issues were also needed.

The test samples were prepared in the form of two-inch diameter circular discs using compression molding and hand lay-up methods. Ideally all samples for three-dimensional measurements should be cut from the same block. Since one layer of fabric of density 27 oz/yd² approximately gives a laminate of thickness 0.04", 100 layers of fabric would be required to manufacture a three-inch thick block. Technically this poses a great difficulty in manufacturing such a block in terms of proper alignment of fabric and wet out.

3.3 Thermal Conductivity of Carbon Composite Samples

3.3.1 Description of samples

The carbon composite samples tested include: 1) pultruded carbon/510A vinyl ester composite laminate. This sample was cut as face sheet from a composite sandwich panel consisting of two 1/4" thick carbon FRP face sheets sandwiching 3" thick balsa wood core. The panel was mass-produced through the pultrusion process by Bedford Reinforced Plastics Inc. for potential naval structural applications; 2) resin infused carbon/510A vinyl ester composite laminate; and 3) resin infused carbon/ FR-7 epoxy composite laminate. Both items 2 and 3 were cut from 2' x 2' carbon composite sheets that were manufactured by Fiber-Tech Industries Inc.

The Dow Derakane 510A-40 vinyl ester resin is a U.S. Navy certified standard resin for naval applications. It is a grade of brominated epoxy- vinyl ester resin that offers the maximum degree of fire retardancy combined with enhanced chemical resistance and toughness, while FR-7 epoxy resin has been recently developed by Applied Poleramic Inc. (API) also for naval applications. FR-7 has low viscosity, high Tg and toughness and is curable at 160-180 F.

The same batch of Toray T700SC /12K / FOE carbon fabric was used in the above samples. T700S is a grade of carbon fiber of highest strength (711 ksi) and standard modulus (33.4 msi). The selected code represents a never twisted carbon fiber of 12000 filaments per tow, with a sizing type designated for vinyl ester and surface-treated

at a sizing amount 0.7%. The fabric configuration was based on 6 layers of 28 oz per sq yd quad axial distributed carbon fabric. Each layer has 6 oz (21.4%) carbon in 0 direction, 8 oz (28.6%) carbon in -45 direction, 6 oz (21.4%) carbon in 90 direction, and 8 oz (28.6%) carbon in +45 direction.

3.4 Three Dimensional Thermal Conductivity of Carbon Composite Block

3.4.1 Sample preparation

A carbon fiber/510A vinyl ester composite block was prepared to characterize its three-dimensional thermal conductivity. The block was made in the shape of cuboids of dimensions 6"x 6" x 3", consisting of 100 layers of 28 oz per sq yd quad axial distributed carbon fabric. A rectangular wooden mold was used to help hold carbon fabric in place. Each layer of fabric was applied/wet-out with 510A resin on both sides before stacking together and then compression molded into the block. In order to ensure the uniform pressure to be applied across the entire block, 100 layers of fabric were assembled together in 3 batches. In other words, first 33 layers were wet-out, placed into the mold, and then compressed, followed by 2nd 33 layers and remaining 34 layers.

Figure 3.1 Fabrication of the composite block with Compression molding unit

Two plates of a quarter inch in thickness were cut from the block from each direction and a two-inch diameter disk was cut from each plate using 2-inch diameter

carbide tipped hole saw. Thus two disks for each direction were prepared giving a total of six specimens. These specimens along with the block are shown in Figure 3.2.

Figure 3.2 Carbon fiber/vinyl ester composite block and specimens through the thickness direction (X), in transverse direction (Y), and along 0 fiber direction (Z) for thermal conductivity measurements

Figure 3.3 Debonding between layers of fabric of the through-the-thickness direction samples cut from carbon/vinyl ester composite block, due to starved impregnation and poor wet-out

Note that as shown in Figure 3.2, y-direction disks have some cracks while x-direction disks have decomposed into layers of fabric. The debonding between layers of fabric of the through-the-thickness direction samples is more closely shown in Figure 3.3. These defects occurred during hole-saw cutting and can be attributed to starved impregnation and poor wet-out during block preparation. Another carbon/vinyl ester block was made using a higher resin/fiber content ratio.

The disc samples cut from the previously made carbon/510A vinyl ester composite block had shown debonding between the layers of fabric in the through-the-thickness direction. So, another composite block was constructed with precaution, to eliminate the above problem. For example, a higher resin/fiber content ratio (60% resin and 40% fabric) was used in order to overcome the poor wet-out of fabric, which most likely existed in the first carbon composite block.

This carbon/510A vinyl ester composite block was prepared to further characterize its three-dimensional thermal conductivity and verify our observations reported earlier. More specifically, each layer has 6 oz (21.4%) carbon in 0 direction, 8 oz (28.6%) carbon in -45 direction, 6 oz (21.4%) carbon in 90 direction, and 8 oz (28.6%) carbon in +45 direction. In order to ensure the uniform pressure to be applied across the entire block, 90 layers of fabric were assembled together in 3 batches. In other words, first 35 layers were wet-out, placed into the mold, and then compressed, followed by 2nd 35 layers and then remaining 20 layers.

Three plates of a quarter inch in thickness were cut from the block in each direction and a two-inch diameter disk was cut from each plate using 2-inch diameter carbide tipped hole saw. Thus three disks for each direction were prepared giving a total of nine specimens. These specimens along with the block are shown in Figure 3.4 and no debonding was observed with this batch of samples.

Figure 3.4 Carbon fiber/510A vinyl ester composite block II and specimens through the thickness direction (X), in transverse direction (Y), and along 0 fiber direction (Z) for thermal conductivity measurements

3.5 Thermal History Effect on the Thermal Conductivity of FRP Bridge Deck Samples

The thermal history effect on the thermal conductivity of FRP composite materials is being carried out in order to better understand the long term performance of FRP composite structures such as FRP bridge decks in terms of their thermal fatigue responses.

Five FRP bridge deck samples were selected for this study. These bridge deck samples were made with E-glass fiber and vinyl ester resin by pultrusion process by BRP Inc. The fabric configuration is based on multiple layers of 0/+45° tri axial fabrics TV 4000 and 60-glass fiber roving. The bridge deck plates were first made to required thickness by mill-grinding. Then the plates were cut using a hole-saw into two-inch diameter disks ready for thermal conductivity measurements. These bridge deck disks are also shown in Figure 3.5.

The carbon composite samples tested include: 1) pultruded carbon/510A vinyl ester composite laminate. This sample was cut as face sheet from a composite sandwich panel consisting of two 1/4" thick carbon FRP face sheets sandwiching 3" thick balsa wood core. The panel was mass-produced through the pultrusion process by Bedford

Reinforced Plastics Inc. for potential naval structural applications; 2) resin infused carbon/510A vinyl ester composite laminate; and 3) resin infused carbon/ FR-7 epoxy composite laminate. Both items 2 and 3 were cut from 2' x 2' carbon composite sheets that were manufactured by Fiber-Tech Industries Inc.

The Dow Derakane 510A-40 vinyl ester resin is a U.S. Navy certified standard resin for naval applications. It is a grade of brominated epoxy- vinyl ester resin that offers the maximum degree of fire retardancy combined with enhanced chemical resistance and toughness, while FR-7 epoxy resin has been recently developed by Applied Poleramic Inc. (API) also for naval applications. FR-7 has low viscosity, high Tg and toughness and is curable at 160-180 F.

The same batch of Toray T700SC /12K / FOE carbon fabric was used in the above samples. T700S is a grade of carbon fiber of highest strength (711 ksi) and standard modulus (33.4 msi). The selected code represents a never twisted carbon fiber of 12000 filaments per tow, with a sizing type designated for vinyl ester and surface-treated at a sizing amount 0.7%. The fabric configuration was based on 6 layers of 28 oz per sq yd quad axial distributed carbon fabric. Each layer has 6 oz (21.4%) carbon in 0 direction, 8 oz (28.6%) carbon in -45 direction, 6 oz (21.4%) carbon in 90 direction, and 8 oz (28.6%) carbon in +45 direction.

Figure 3.5 Disk samples for thermal conductivity measurements, including bridge deck samples (top row); resin infused carbon composite laminates (middle row); and pultruded carbon composite face sheet of sandwich panel (bottom row)

Figure 3.6 Bridge deck samples along with carbon/510A and carbon/epoxy samples subjected to climatic conditions to study thermal history effect on thermal conductivity

These samples along with resin infused carbon/510A and carbon/epoxy, and pultruded carbon/510A samples were placed into a frame box as shown in Figure 3.6. The frame box with total 10 samples has been placed outdoor and subjected to weathering effect. All the samples will be tested for their thermal conductivity properties every 4 weeks (app) for a period of 24 months.

3.6 Materials for Conductive Composite Sensor

3.6.1 Conductive fillers

For casting the conductive composite samples we need the polymer as well as the conductive filler. Four kinds of graphite powders of different sizes supplied by UCAR Carbon Company, Clarksburg, WV, were used in the previous study and a different kind of powder GP44R-B has been used in this work. These graphite powders were used to establish how both the conductivity, sensitivity (ratio of the increase in resistance due to stress to the original resistance when no stress is applied) & mechanical properties of the sensor change with particle size of the conductive filler.

Table 3.1 Properties of the graphite powder

Properties	Graphite powder
	GP44R-B
Ash	0.2% max
Moisture	0.5% max
Density	2.25 g/cc
Surface Area	8.0 sq.m/g
Particle Size Distribution	98.5% < 75 microns 90% < 41 microns 50% < 18 microns
Average Particle Size	40 microns

3.6.2 Polymers

A new resin system 510-A Vinyl ester and a promoted RTM grade resin Hetron 922 L25 vinyl ester supplied by Ashland Company have been used. Both the resins were

catalyzed using MEKP peroxide at a concentration of 2%. Hetron 922 L25 has a viscosity of 275 cps and an elongation to failure of ~5%.

3.6.3 Molds

All the samples were prepared by hand cast-molding. There had been several improvements with different batches of composite samples. The mold used was made of 0.125 inch PTFE sheet supplied by McMaster Carr. Since the samples are to be tested for tension, the molding had dog bone shapes. This PTFE mold has 10 cavities.

3.7 Manufacturing of coupon samples

3.7.1 Casting of conducting composites using vinyl ester resin

All the samples were prepared by hand cast-molding. First, all samples cast had perfect dog bone shapes, which eliminates or reduces the high stress concentration points. Secondly, line electrodes were used instead of point contact inside the moldings, which significantly improve conducting stability and give consistent reliable resistance readings. Thirdly, nonconductive end tabs were bonded onto test specimens, which not only provide insulation from conducting through the testing machine but also make test samples to fail absolutely in gauge section.

The mold was first placed on a flat surface, which was covered with a thin sheet of aluminium foil. Electric contacts were accomplished by embedding two bared copper rods, at each end of the mold cavity, which were connected to extended fine copper wires. These contacts were placed inside the mold prior to pouring of the mixture into the mold. Care is taken to make sure the copper rod gets completely immersed/insulated in the mixture while curing. Then polymer matrix and the conductive filler were weighed respectively and mixed thoroughly and manually in a container for 5 minutes at a particular weight ratio ranging from 0-40% wt.

Figure 3.7 Samples being cured with the set up of mold clamped to the flat surface covered with aluminum foil, and with electric contacts in place

It was easy to cast the samples till 25% of graphite concentration, above 25% the mixture was very viscous and the ease of casting was difficult. Proper care was taken to remove any sort of air bubbles. The mixture of the polymer resin with the conductive filler was poured into the mold and was allowed to set for 24 hours and then de-molded. The samples made of 510A Vinyl ester resin had lots of air bubbles, which can be observed from figure 3.8

Figure 3.8 Samples made of 510A vinyl ester

So, all the samples further casted are made of Hetron 922 L vinyl ester, which had a good surface without any air bubbles as shown in the figure 3.9.

Figure 3.9 Tension specimens made of graphite in Hetron 922 L vinyl ester

In order to know the variation of different parameters effecting the sample preparation, a batch of neat resin samples were made first and then with the variation in the amount of graphite powder. The graphite powder was made to dry in the oven at 100°C for about three hours in order to ensure that it is free from moisture. Some of the samples were shown in figure 3.9. In past four types of graphite powders GP44-B, GP55-B, GS75-E, GS150-E all of which have different particle size have been used. In this work GP44R-B has been used as the conductive filler.

3.8 Evaluation of Conductive Composites as heating elements

In the past, work on conductive composites was evaluated and demonstrated that they function well as heating elements by connecting the two ends of conductive composite samples to a supply of AC voltage, and the electric current passing through the sample generated heat. As one of the potential applications for this function is to heat a highway bridge deck for de-icing in winter, the conductive resin has been thought of being applied as a wearing surface on the bridge deck panel to study how the deck

surface temperature varies with different parameters for feasibility of the conductive resin.

3.8.1 Sample preparation

The conductive wearing surface had Hetron 922 L vinyl ester as polymer and graphite powder as conductive filler catalyzed with 2% of MEKP. A sample bridge deck panel of 12"x 24" long has been selected and the surface of the panel is sanded to get a good surface contact with the conductive resin. A schematic picture of the sample geometry can be seen in the figure 3.10. Initially small holes of diameter equal to that of the copper rod are drilled on the panel surface to make the rod pass through them in such a way that they are in contact with the panel surface. The span length between the copper rods is varied from 2", 4" and 6" respectively.

Figure 3.10 A schematic of the bridge deck panel with copper rods as electrodes

The polymer matrix and the conductive filler were mixed in specific weight ratio for about 5 min. Thus prepared conductive resin is applied uniformly in between the specified span length of copper rods, and care is taken to make sure that the copper rods are covered with the layer of wearing surface. The panel is set for 24 hours to cure the wearing surface.

CHAPTER 4

TESTING METHODS

4.1 Introduction

This chapter presents the development of an experimental method for thermal conductivity measurements of composite materials in transverse, longitudinal and through the thickness directions. After a brief description on the test equipment, the operation principle of the unit is discussed and followed by sample requirement and testing procedure.

4.2 Experimental setup for thermal conductivity

A guarded heat flow meter method has been developed for thermal conductivity measurements. This is achieved by using a thermal conductivity testing system Unitherm model 2022 from ANTER Corp., Pittsburgh, PA. The experimental set up of this instrument in CFC-WVU laboratory is shown in Figure 3.1.

Figure 4.1 Experimental set up of Unitherm 2022

This unit is supplied with a mid range flux module covering a thermal resistance range from 0.002 to 0.02 m² K/W and is able to measure the thermal conductivity of

materials in the range of 0.1 to 40 W/m-K in terms of test standard ASTM E 1530. The materials that can be tested include metals, ceramics, polymers, composites, glass etc. The test samples need to be prepared in a form of two-inch diameter circular discs with their thickness depending on the materials' thermal conductivity. The thermal conductivity machine was supplied with three sets of calibration samples span the R_s range from 0.0005 to 0.05 m²K/W. These samples were tested for conductivity values and compared with the given values by the manufacturer for calibration purpose. The relationship among the thermal conductivity of a material, its thermal resistance and sample thickness is discussed in detail under Section 3.3.

This equipment requires compressed air to raise and lower the upper stack assembly. It also needs either city water or a chiller to cool the heat sink, giving an operation temperature range from 20⁰C to 300⁰C. ANTER model Unitherm 2022 is a computerized system. The computer automatically controls the equipment for testing and data processing through a latest version of electronics and operating software once a test program is designed and initiated.

Figure 4.2 Schematic model showing the system arrangement in Unitherm 2022

A schematic picture shown in Figure 4.2 represents the system functioning in detail. The assembly is a stack of parts with different functionalities. The heater on top

and bottom helps to maintain steady state heat transfer through the sample, two polished surfaces on top and bottom of the sample transfer heat from top and bottom heaters with reduced thermal resistance through surface. A reference calorimeter is placed under the lower plate, which acts as a heat flux transducer. The heat sink at the bottom avoids excessive temperature from the system. The sample is compressed in between the polished surfaces, each controlled at different temperatures, using pneumatic load. The pressure is maintained at 10 psi using pressurized air supply. As an option, coolant water is circulated through heat sink. A circular low temperature heat insulation ring is wrapped around the lower stack of the assembly to restrain heat flow to outside atmosphere. The entire system is maintained in a thermally insulated glass chamber. Unitherm is completely automatic while testing as the apparatus is completely controlled by a computer. The test system is hooked to the computer by means of an USB cable.

Considering the operation of the system, the sample to be tested is prepared into a flat surface on both sides and thermal compound is applied on the sample to reduce thermal resistance caused due to surface roughness. Then the prepared sample is placed in between two polished surfaces and a pneumatic pressure of 10 psi is applied on the top portion of the stack. The sample can be tested in the temperature range from 20⁰ C to 300⁰ C. For steady state heat transfer, the user can divide the testing into different zones, called as set point temperatures. In this thesis the testing was divided into three temperature zones i.e. 60, 80, 100⁰ C. For higher thermal conductivity materials, a difference of 50 degrees is recommended for the machine. At every set point temperature, the system checks for steady state heat flow through the sample and thermal conductivity is measured.

4.3 Operation principle of Unitherm 2022

By definition thermal conductivity means “*The material property that describes the rate at which heat flows with in a body for a given temperature change.*” For one-dimensional heat conduction the formula can be given as Equation 3.1

$$Q = KA \frac{T_1 - T_2}{x} \quad 4.1$$

Where Q is the heat flux (W), K is the thermal conductivity (W/m-K), A is the cross-sectional area (m^2), T_1-T_2 is the difference in temperature (K), x is the thickness of the sample (m).

The thermal resistance of a sample can be given as Equation 4.2

$$R = \frac{T_1 - T_2}{Q / A} \quad 4.2$$

Where R is the resistance of the sample between hot and cold surfaces (m^2 -K/W).

From Equations 4.1 and 4.2 we can derive that

$$K = \frac{x}{R} \quad 4.3$$

In Unitherm 2022 the heat flux transducer measures the Q value and the temperature difference can be obtained between the upper plate and lower plate. Thus the thermal resistance can be calculated between the upper and lower surfaces. Giving the input value of thickness and taking the known cross sectional area, the thermal conductivity of the samples can be calculated using Equation 4.3.

4.4 Sample requirement and test procedure

Preparation of test sample is a challenging task. One accurate measurement requires a sample of dimensional precision of ± 0.005 inch both in diameter and thickness. A core drill is employed to cut a two-inch diameter disk and then the disk is machine-milled to appropriate thickness.

A sample thickness is determined by the relationship between thickness, resistance and thermal conductivity as defined by Equation 4.3. By assigning an estimated thermal conductivity value to the testing material specimen with a given thickness, the calculated thermal resistance should fall into the working window of the equipment, which is 0.002-0.02 m^2 K/W for ANTER Unitherm model 2022. The thickness usually can be considered any value between 50* K to 0.5* K (mm) with 25mm being the maximum thickness for Unitherm model 2022.

A test begins from sample preparation. Major operation procedures include: 1) application of thermal compound onto sample surface to reduce the interfacial resistance;

2) proper loading of sample into the stack assembly so that it completely occupies the gap between upper and lower stacks; 3) closing the furnace chamber and mounting on protective glass shield; 4) switch on air and water supply; 5) designing of a test program that need input of sample thickness and temperature segments; 6) switching on heaters before starting a test; 7) after testing, allowing the unit to cool down to room temperature before removing the protective shield; 8) cleaning the upper and lower stacks; and 9) data processing. Typically a three-segment test lasts for three hours.

4.5 Electrical resistance measurement

Electrical resistance is a measure of the degree to which a material opposes the passage of an electric current. The SI unit of electrical is the Ohm. Polymers generally have a very huge resistance. They are very good insulators of heat and electricity and their resistivity are of the order of $\sim 10^{10}$ Ohm-Meters. Conductive polymer composites can be made by adding conductive fillers to polymers in their uncured states and then using the catalysts.

We have measured resistance of the samples using the two-wire measurement. The electric resistance of the samples was measured using a Radio shack 46 Range LCD digital multimeter as shown in figure 4. This multimeter has an RS 232 port through which it can be interfaced to a PC for monitoring the resistance of the samples at periodic intervals of time.

The samples were prepared in such a way that the wires were put in the mold even before pouring the uncured resin system into the mold. After the resin with fillers and conductive material in the mold was cured and the samples were de-molded the wires were a part of the test samples. A multi-meter was then connected to the wires across the sample as shown in figure 4.3. It gave the resistance of the sample. Instead of using the point contacts, the wires were soldered to a copper rod, which is of 0.125 inches in diameter. Then this copper rod was placed in the mold perpendicular to the axial

Figure 4.3 Meter used for resistance measurement. RS-46

direction (length). This cylindrical type contact gave a stable and non-fluctuating resistance.

4.6 Tension test

4.6.1 Test specimen

For correlating the mechanical properties to the electrical properties of the sensor we need to perform several mechanical tests. Tension test is a method for determining behavior of materials under axial loading. Dimensions of the tension test specimens are listed in Table 4.1 with the nomenclature identified in Figure 4.4.

Table 4.1 Dimensions for tension test specimen

Width of narrow section (W)	0.5"
Width overall (Wo)	0.75"
Length Overall (Lo)	4.5"
Thickness (T)	0.125"
Gauge Length (L)	2.0"
Center section length (D)	2.5"

Dog-bone shaped tension test specimen

4.6.2 Tension test setup and procedure

Tension test was carried out first on an Instron machine model 8501. This Instron model is a dynamic testing system designed to test mechanical properties of a wide range of materials. Using a hydraulic actuator, tensile load was applied to the test specimen to monitor tensile strain or deflection of the specimen by observing the position of the hydraulic actuator in response to the applied load. The load is also monitored under the applied strain.

Tension test was carried out with a position control mode at a stretching rate of 0.05inch/minute ASTM standard D-638. During the test, the electrical resistance was measured along the variation in applied stress. A computer through RS 232C interface recorded the measured resistance values. Two cables were used for connecting the sample to the multimeter. The electric resistance was monitored with a Radio Shack 24-range LCD digital multimeters. Figure 4.6 shows a close-up view of a vinyl ester composite specimen under tension. Instron machine 8501 was not very sensitive for small loads, since our samples failed at a maximum load of 1500N. Therefore, tests were done with Instron model 5567, which is tabletop load frame model. It evaluates the materials under investigation by applying a tensile or compressive load on the specimen between the rigid frame and the moving crosshead and carries a 5KN load cell.

Figure 4.4 Tension test set-up using Instron 5567

Figure 4.5 A vinyl ester conductive composite sample under tension

The entire setup is shown in Figure 4.5. The test was conducted with a position control mode at a stretching rate of 1mm/min. During the test electrical resistance was measured along with applied load and strain. This was accomplished using Radio Shack 46 Range LCD digital multimeters. Two extension cables were used to connect the sample to the multimeters. The measured resistance values were automatically recorded on a computer through an RS 232C interface.

4.7 Evaluation of Conductive Composites as heating elements

In the past, work on conductive composites was evaluated and demonstrated that they function well as heating elements by connecting the two ends of conductive composite samples to a supply of AC voltage, and the electric current passing through the sample generated heat. As one of the potential applications for this function is to heat a highway bridge deck for de-icing in winter, the conductive resin has been thought of being applied as a wearing surface on the bridge deck panel to study how the deck surface temperature varies with different parameters for feasibility of the conductive resin.

4.7.1 Basic Theory

Upon connection to a supply voltage, a resistor is to generate heat because of the voltage drop between its terminals. If the resistor has a resistance R and is subjected to AC voltage V , the current passing through the resistor, I , can be determined using the formula:

$$I \text{ (Amp)} = V \text{ (Volts)} / R \text{ (Ohms)} \quad (1)$$

Then, the power P is

$$P \text{ (Watts)} = V \text{ (Volts)} * I \text{ (Amps)} = V^2 / R \quad (2)$$

The heat generated, Q for a period of time (t) is

$$Q \text{ (Joules)} = P \text{ (Watts)} * t \text{ (sec)} = (V^2 / R) * t \quad (3)$$

For an AC voltage, $V = V_0 / \sqrt{2}$, giving the amount of heat generated as

$$Q = 0.5 * (V_0^2 / R) * t \quad (4)$$

For a conductive composite sample under investigation, if the material has a specific heat capacity C_p and the sample has a mass M , the heat required, Q to create a temperature increment ΔT will be

$$Q \text{ (Joules)} = C_p \text{ (J/ g. K)} * \Delta T \text{ (K)} * M \text{ (g)} \quad (5)$$

Combining equations 4 and 5 gives a relationship between the resistance and temperature difference:

$$\Delta T = 0.5 * (V_0^2 / R) * t * (1 / C_p * M) \quad (6)$$

Thus, the temperature change is inversely proportional to the resistance.

4.7.2 Experiment

For a conductive composite sample that has a resistance R , when the terminals of the sample are connected to a voltage, the sample will generate heat just like any other resistor. In the experiment, the bridge deck panel with Hetron 922L and graphite powder as wearing surface is subjected to AC voltage (110V-regular). This is accomplished by connecting the ends of the copper rod at each end of the span length to the AC voltage using a two-pin connector as shown in figure 4.7. The amount of time the voltage supplied to the panel was kept constant at 60 minutes during all measurements. The temperature profile of the panel under investigation was observed using an infrared camera (ThermaCAM S60) and the temperature is recorded as a function of time. The entire experimental set up is shown in the figure 4.8.

Figure 4.6 A bridge deck panel with conductive wearing surface plug into outlet power

Figure 4.7 Experimental setup for evaluation of conductive wearing surface on the bridge deck panel using an infrared camera model ThermoCAM

CHAPTER 5

EXPERIMENTAL DESIGN

5.1 Introduction

An experiment can be defined as a test or series of tests in which purposeful changes are made to the input variables of a process or system so that we may observe and identify the reasons for changes that may be observed in the output response. This chapter deals with the evaluation of design parameters considered for thermal property and electrical property of fiber reinforced polymer composites. Analyses of covariance, analysis of variance and regression analysis concepts have been applied to design the regression model equation for the parameters in thermal and electrical tests.

In statistics, analysis of variance (ANOVA) is a collection of statistical models, and their associated procedures, in which the observed variance is partitioned into components due to different explanatory variables. Linear regression investigates and models the linear relationship between a response(Y) and predictor(s) (X). In particular, linear regression analysis is often used to predict the value of the response variable for any value of the predictor variable or combination of values of the predictor variables. Analysis of covariance (ANCOVA) is a general linear model with one continuous explanatory variable and one or more factors. ANCOVA is a merger of ANOVA and regression for continuous variables. ANCOVA tests whether certain factors have an effect after removing the variance for which quantitative predictors (covariates) account. The inclusion of covariates can increase statistical power because it accounts for some of the variability.

Source: Design and Analysis of Experiments, Douglas C. Montgomery, 6th Edition.

All the models designed for analyzing the data were taken to follow a linear model (GLM). The General Linear Model (GLM) underlies most of the statistical analysis that are used in applied and social research. It is the foundation for the t-test, Analysis of Variance (ANOVA), Analysis of Covariance (ANCOVA) and the regression analysis. The model analysis was done using the MINITAB statistical package. Minitab's Stat

Guide provided statistical guidance for interpreting statistical tables and graphs in an easy-to-understand way. The type I error probability i.e., α -level of 0.05 was chosen for all statistical tests and the confidence is $1-\alpha$. The general model for the regression analysis is as shown below,

$$Y = \beta_0 + \beta_1(x_1) + \beta_2(x_2) + \dots + \beta_n(x_n) + \varepsilon$$

Where:

- Y is the dependent variable.
- $x_1 \dots x_n$ are the independent variables.
- β_0 is the intercept.
- $\beta_1 \dots \beta_n$ are the coefficients of interest.
- ε is the error.

In this model, Y represents the variable of interest in the analysis, while $x_1 \dots x_n$ represent the independent variables. The coefficients on each of the independent variables ($\beta_1 \dots \beta_n$) represent the effect of a change in the independent variable ($x_1 \dots x_n$) on the dependent variable (Y). The sign on the coefficient reflects the direction of the relationship. Regression analysis, is used to model numerical data obtained from observations by adjusting the parameters of a model so as to get an optimal fit of the data.

Regression and ANOVA procedures make these assumptions about the errors

1. The errors are normally distributed with mean zero
2. The error variance does not change for different levels of a factor or according to the values of the predicted response.
3. Each error is independent of all other errors. In a designed experiment, the best way to obtain independent errors is to randomize the run order of the experimental trials.

Guidelines for Experimental Design:

- Recognition and statement of the problem
- Selection of the response variable
- Choice of factors, levels and ranges
- Choice of experimental design
- Performing the experiment
- Statistical analysis of the data

Source: Design and Analysis of Experiments, Douglas C, Montgomery, 6th Edition.

5.2. Thermal Property

5.2.1 Thermal conductivity of Carbon/510A Vinyl ester Composite

The Carbon/510A vinyl ester composite samples manufactured with pultrusion and resin-infusion process are considered for the experiment. Thermal conductivity measurements were obtained using the guarded heat flow meter method to analyze the three dimensional thermal conductivity at three different set point temperatures. This is achieved by using a thermal conductivity testing system unitherm model 2022. A general linear model (GLM) is used to perform the analysis of variance (ANOVA). The data obtained for the conductivity have been thought of being analyzed by applying a statistical model for the parameters in the thermal conductivity measurement.

$$Y_{ijkl} = \mu + \alpha_i + \beta_j + \alpha\beta_{ij} + \gamma_k + \alpha\gamma_{ik} + \beta\gamma_{jk} + \alpha\beta\gamma_{ijk} + \varepsilon_{ijkl}$$

Where,

Y_{ijkl} - Thermal Conductivity (W/mk-response)

μ - Overall mean

α - Effect due to Thermal Resistance, m²k/W

β_j - Effect due to Set point temperature with j = 1, 2, 3

γ_k - Effect due to the type of manufacturing method k = 1, 2

$\alpha\beta_{ij}, \alpha\gamma_{ik}, \beta\gamma_{jk}, \alpha\beta\gamma_{ijk}$ - Two factor and three factor interactions

ε_{ijk} - random error term

In the above model, the set point temperature, manufacturing method of the composite and the thermal resistance of the sample were considered as the factors to be studied. The three set point temperatures considered for the conductivity test are 60 deg, 80 deg and 100 deg. Two types of manufacturing methods (1) pultrusion and (2) resin-infusion were considered. Three replications for the response were collected from the conductivity experiment for data analysis using ANOVA and Regression procedures.

5.2.2 Thermal conductivity of the Composite Block

The three dimensional composite block discussed in the previous chapter is considered. The data obtained for the conductivity have been thought of being analyzed by applying a statistical model for the parameters in the thermal conductivity measurement.

$$Y_{ijkl} = \mu + \alpha_i + \beta_j + \alpha\beta_{ij} + \gamma_k + \alpha\gamma_{ik} + \beta\gamma_{jk} + \alpha\beta\gamma_{ijk} + \varepsilon_{ijkl}$$

Where,

Y_{ijkl} - Thermal Conductivity (W/mK-response, $l = 4$ replications)

μ - Overall mean

α - Effect due to Thermal Resistance, m^2k/W

β_j - Effect due to Heat flow direction at $j = 1, 2, 3$ levels

γ_k - Effect due to Set point temperature at $k = 1, 2, 3, 4$ levels

$\alpha\beta_{ij}, \alpha\gamma_{ik}, \beta\gamma_{jk}, \alpha\beta\gamma_{ijk}$ - Two factor and three factor interactions

ε_{ijk} - Random error term

In the above model, the heat flow direction, the set point temperature and thermal resistance of the sample are considered as the factor effects. Three levels of heat flow direction 1-Through the thickness(Z), 2-Transverse(Y) and 3-Longitudinal(X) are considered. The three set point temperatures considered for the conductivity test are 60 deg, 80 deg and 100 deg. Four replications of response were taken from the experimental data for analysis using ANOVA and regression procedures.

5.3 Electrical property

The electrical property tests were evaluated with two scenarios.

5.3.1 Conductive composite sensor

One scenario is with conductive composite sensors, where conductive fillers were added to the thermoset polymer resin to make it conductive. Mechanical tests have been performed on the developed sensor to evaluate its sensitivity in terms of change in their electrical resistance as a function of deformation, which is the key factor in the performance of sensor. Analysis of variance concept has been applied to model the composite sensor. The parameters considered for the evaluation are designed with the model given below.

$$Y_{ij} = \mu + \alpha_i + \varepsilon_{ij}$$

Where,

Y_{ij} - Resistance change of the composite sensor (response, j= 4 replications)

μ - Overall mean

α_i - Effect due to amount of graphite concentration with i = 1, 2, 3, 4, 5, 6

The response variable resistance change in the model is the change in resistance of the sample R_1 and the resistance of the test sample at 0.5% strain which is R_2 . With the change in amount of graphite concentration the sensitivity of the sensor varied. Six levels of graphite concentration i.e., 13%, 14%, 15%, 17.5%, 20% and 22.5% were considered for evaluation. Four replications of response were taken from the experimental data for analysis.

5.3.2 Conductive wearing surface

The conductive resin has been applied as a wearing surface on the bridge deck panel to study, how the deck surface temperature varies with different parameters for feasibility of the conductive resin. The temperature profiles for the bridge deck panel samples with wearing surface have been studied for different parameters. A typical infrared camera and infrared temperature gun were used to study the property. Analysis

of variance concept has been used to model the parameters of conductive wearing surface.

$$Y_{ijk} = \mu + \alpha_i + \beta_j + \alpha\beta_{ij} + \varepsilon_{ijk}$$

Where,

Y_{ijk} - Temperature of the wearing surface (response, k=3 replications)

μ - Overall mean

α_i - Effect due to Span length of copper rods with i = 1, 2, 3

β_j - Effect due to Amount of graphite concentration with j = 1, 2, 3, 4

$\alpha\beta_{ij}$ - Effect due to interaction between span length and graphite concentration

The temperature profile for the wearing surface has been studied at three span lengths i.e., 2", 4" and 6" respectively. Four different graphite concentrations 10%, 15%, 20% and 22.5% have been used. Three replications of response were taken from the experiments for statistical analysis.

CHAPTER 6

RESULTS AND DISCUSSION

6.1 Introduction

The objective of the experimental investigation is to determine longitudinal, transverse and through-the-thickness thermal conductivity of composite materials with known material compositions and fiber configurations. In addition the thermal conductivity variation was examined as a function of thermal compound and thermal weathering. This chapter presents the results and discusses thermal conductivity variations as a function of composite material parameters. Results from the test procedure as described in Chapter 4 on conductive composite samples and conductive wearing surface prepared as per the description in Chapter 3 are presented and analyzed in this chapter.

6.2 Thermal Conductivity of Carbon Composite Samples

Thermal conductivity was determined for the carbon composite laminates mentioned in Chapter 3 using the guarded heat flow meter model Unitherm 2022, two replications for each type of laminate and two runs for each sample. The two-inch diameter disk samples are shown in Figure 5.1. The through-the-thickness thermal conductivity values of the carbon composite laminates are listed in Table 6.1.

The result indicates that for samples prepared from resin infusion process, epoxy based composite has slightly higher thermal conductivity (~ 0.413 W/mK) than 510A vinyl ester based composite (~ 0.328 W/mK). However, it appears that the manufacturing method has some effect on the thermal conducting behavior of the product. The pultruded carbon/510A composite has the highest thermal conductivity, i.e. ~ 0.432 W/mK. These values are not significantly different from those of E-glass composites (0.340-0.380 W/mK) when the heat flow direction is through the thickness of a sample.

Table 6.1 Through-the-thickness thermal conductivity of carbon composite samples

Sample Description	Sample No		Thickness mm (inch)	Thermal conductivity, W/mK		
				Segment 1 at 140°F (60°C)	Segment 2 at 176°F (80°C)	Segment 3 at 212°F (100°C)
Pultruded Carbon Composite Laminates (Navy sandwich panel face sheet): Carbon/510A Vinyl Ester	1	Run 1	6.660 (0.262)	0.419	0.445	0.463
		Run 2	6.660 (0.262)	0.418	0.440	0.466
		Average		0.419	0.443	0.465
	2	Run 1	6.699 (0.264)	0.399	0.420	0.440
		Run 2	6.699 (0.264)	0.396	0.420	0.443
		Average		0.398	0.420	0.442
Resin Infused Carbon Composite Laminates: Carbon/510A Vinyl Ester Resin	1	Run 1	8.080 (0.318)	0.319	0.336	0.349
		Run 2	8.080 (0.318)	0.325	0.336	0.353
		Average		0.322	0.336	0.351
	2	Run 1	8.730 (0.344)	0.302	0.319	0.338
		Run 2	8.730 (0.344)	0.309	0.319	0.339
		Average		0.306	0.319	0.339
Resin Infused Carbon Composite Laminates: Carbon /FR-7 Epoxy Resin	1	Run 1	6.932 (0.273)	0.378	0.404	0.408
		Run 2	6.932 (0.273)	0.388	0.401	0.419
		Average		0.383	0.403	0.414
	2	Run 1	6.566 (0.259)	0.398	0.421	0.429
		Run 2	6.566 (0.259)	0.416	0.425	0.441
		Average		0.407	0.423	0.435

6.2.1 Statistical Analysis

The conductivity of carbon/510A Vinyl ester composite samples manufactured with the two methods was analyzed. The ANOVA output for this model from MINITAB showed high R-Sq and R-Sq (adj) values and the complete variance is taken by the thermal resistance factor, giving least weight to other factors in the model. So, general linear regression analysis is applied to the model to evaluate the linear relation.

The REG Procedure					
Model: MODEL1					
Dependent Variable: conductivity					
Number of Observations Read			18		
Number of Observations Used			18		
Analysis of Variance					
Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	4	0.05449	0.01362	10346.8	<.0001
Error	13	0.00001712	0.00000132		
Corrected Total	17	0.05451			
Root MSE		0.00115	R-Square	0.9997	
Dependent Mean		0.38911	Adj R-Sq	0.9996	
Coeff Var		0.29489			
Parameter Estimates					
Variable	DF	Parameter Estimate	Standard Error	t Value	Pr > t
Intercept	1	0.52330	0.02093	25.01	<.0001
resistance	1	-12.59971	2.46155	-5.12	0.0002
Manufacturing	1	0.05565	0.02470	2.25	0.0421
temp	1	0.00127	0.00008494	14.91	<.0001
Mxt	1	-0.00060126	0.00004549	-13.22	<.0001

The regression analysis for the carbon composite conductivity model is run in SAS (Statistical Analysis Software) to study the factors and interaction effect. The regression output from SAS is as shown above. The regression equation is estimated to be

$$E(\text{Conductivity}) = 0.523 - 12.59 \cdot \text{Thermal Resistance} + 0.055 \cdot \text{Manufacturing Method} + 0.0013 \cdot \text{temp} - 0.0006 \cdot \text{Manufacturing} \cdot \text{temp}$$

The constant (intercept) value (**bo = 0.523**) is the predicted value of conductivity when each predictor (manufacturing method, Set point temp, thermal resistance) is zero. The slope (**b₁ = -12.59**) is the change in conductivity when thermal resistance increases by 1. It shows the inverse relationship between the conductivity and thermal resistance. The slopes (**b₂ = 0.055, b₃ = 0.0013**) are the change in conductivity for unit increase of manufacturing method and set point temperature and the slope (**b₄ = - 0.0006**) is the change in conductivity with unit increase of the interaction between manufacturing method and temperature. The relationship between the response, conductivity, and the predictors, manufacturing method (p = 0.0421), Set point temp (p<0.0001) and thermal resistance (p=0.0002) and the interaction (p<0.0001) are significant, i.e., all the predictors and the interaction term have influence on the conductivity of the carbon/510A Vinyl ester composite.

Example: (Conductivity) = $0.523 - 12.59 \times 0.01590 + 0.055 \times 1 + 0.0013 \times 60 - 0.0006 \times 1 \times 60$
 $= 0.41874$

E (Conductivity) ~ 0.419 W/mK

The expected conductivity value for carbon composite sample manufactured by pultrusion process (1) at 60 degC set point temperature is 0.419 W/mK, which is slightly higher than the original value i.e. 0.418 W/mK.

Least Squares Means for conductivity		
Manufacturing	Mean	SE Mean
1	0.4418	0.000671
2	0.3364	0.000671
Setpoint temp		
60 degC	0.3703	0.000822
80 degC	0.3888	0.000822
100 degC	0.4082	0.000822

Figure 6.1 Variation of conductivity with manufacturing method and set point temperatures

The plots are done using the obtained data. The least square means for composite block model and the main effects plot tells that the conductivity of the sample is highest for the carbon/510A vinyl ester made from pultrusion process and at higher set point temperature. The conductivity value increases with increase in set point temperature as shown in figure 6.1.

Figure 6.2 Normal probability plot of residuals for carbon/510A Vinyl ester

Figure 6.2 plots the residuals versus their expected values when the distribution is normal. For the carbon/510A vinyl ester data, the residuals appear to follow a straight line with a small deviation. No evidence of non-normality or outliers. In order to test the normality, Kolmogorov-Smirnov test is applied on the residuals.

Figure 6.3 Kolmogorov-Smirnov test on normality of residuals for carbon/510A Vinyl ester

H_0 : Data follows a normal distribution

H_A : Data do not follow a normal distribution

If the p-value of the test is greater than the desired α , then H_0 is accepted. In the above test from figure 6.3, the p-value (> 0.150) is greater than 0.05, which leads to the conclusion that the data follows a normal distribution. So the model for carbon/510A Vinyl ester is significant.

Interactions plot is used to visualize the interaction effect of the factors on the response. The variation of conductivity for the three levels of set point temps differs slightly from the variation of conductivity for the two manufacturing methods. The lines

in the plot appear to be close to parallel but not at the same rate as shown in figures 6.4 and 6.5. That is the effect of one of these factors on the response, conductivity is influenced by the other factor, which is an interaction between the two factors.

Figure 6.4 Interaction plot between set point temperature and manufacturing method

Figure 6.5 Interaction plot between manufacturing method and set point temperature

6.3 Three Dimensional thermal conductivity of composite block

6.3.1 Carbon fiber/ Vinyl ester composite block I

All specimens cut from carbon fabric/vinyl ester composite block were tested for their thermal conductivities using the guarded heat flow meter model Unitherm 2022. After-test disk samples are shown in Figure 5.5. The three dimensional thermal conductivity values of carbon fabric /vinyl ester composite materials in longitudinal, transverse and through-the-thickness directions are listed in Table 5.2.

The following observations can be made from Table 5.2 for the carbon fabric/vinyl ester composite block prepared: 1) The through-the-thickness thermal conductivity of carbon composite block is about 0.185 W/mK, much lower than the values shown in Table 5.1 (~0.328 W/mK for carbon/510A vinyl ester composite). This property is very much under-measured because of poor wet-out of fabric, leading to debonding and higher thermal resistance; 2) For the samples of same thickness, the transverse thermal conductivity of carbon composite block is about the same as that of longitudinal direction (0.954 W/mK in transverse versus 0.956 W/mK in longitudinal).

This is in agreement with the fabric configuration that fiber density and percentage share is identical in 0 degree orientation and 90 degree orientation 3) The carbon composite block has a typical orthotropic thermal conducting behavior and the heat conducting capacity is three times higher along the fiber direction than perpendicular to the fiber direction. 4) It is further verified that carbon composites have significantly different thermal conductivity values in three different directions. This is distinctive from E-glass composite materials for which the thermal conductivity in three directions is almost the same.

**Table 6.2 Three-dimensional thermal conductivity values of carbon/510A
vinyl ester composite block I samples prepared using compression molding**

Heat flow direction	Sample No		Thickness mm (inch)	Thermal conductivity, W/mK		
				Segment 1 at 140°F (60°C)	Segment 2 at 176°F (80°C)	Segment 3 at 212°F (100°C)
Through-thickness ($\perp$ 0 & 90 fiber)	1	Run 1	1.980 (0.078)	0.173	0.197	0.233
		Run 2	1.950 (0.077)	0.150	0.172	0.211
		Average		0.162	0.185	0.222
	2	Run 1	6.375 (0.251)	0.170	0.178	0.209
		Run 2	6.375 (0.251)	0.177	0.191	0.214
		Average		0.174	0.185	0.212
Transverse (// 90 & $\perp$ 0 fiber)	1	Run 1	5.296 (0.208)	0.851	0.938	1.065
		Run 2	5.296 (0.208)	0.875	0.969	1.075
		Average		0.863	0.954	1.070
	2	Run 1	8.600 (0.338)	1.108	1.232	1.398
		Run 2	8.600 (0.338)	1.109	1.232	1.405
		Average		1.109	1.232	1.402
Longitudinal (// 0 & $\perp$ 90 fiber)	1	Run 1	5.578 (0.219)	0.744	0.843	1.037
		Run 2	5.578 (0.219)	0.833	0.917	1.027
		Average		0.789	0.880	1.032
	2	Run 1	5.922 (0.233)	0.919	1.006	1.127
		Run 2	5.922 (0.233)	0.954	1.059	1.217
		Average		0.937	1.033	1.172

It is noted that some disks are not perfectly flat. This may result in variations in thermal conductivity values obtained. Proper application of thermal compound onto disk surface is also essential, leading to representative and reproducible thermal conductivity values.

Figure 6.6 After-test disk samples of carbon fabric/vinyl ester composite block I

6.3.2 Carbon fiber /vinyl ester composite block II

The disc samples cut from the previously made carbon/510A vinyl ester composite block had shown debonding between the layers of fabric in the through-the-thickness direction. So, another composite block was constructed with precaution to eliminate the above problem. For example, a higher resin/fiber content ratio (60% resin and 40% fabric) was used in order to overcome the poor wet-out of fabric, which most likely existed in the first carbon composite block.

Two out of the three specimens cut from carbon/vinyl ester composite block II in each direction (shown in Figure 3.4) were selected in terms of surface flatness and uniform thickness and tested for their thermal conductivities using the guarded heat flow meter model Unitherm 2022. After-test disk samples are shown in Figure 6.7 The representative thermal conductivity values of carbon fabric/vinyl ester composite materials in longitudinal, transverse and through-the-thickness directions can be extracted from Table 6.1 and are listed in Table 6.2 again for discussion purpose.

The following observations can be made from Table 6.2 for the carbon fabric/vinyl ester composite block studied: 1) The average through-the-thickness thermal conductivity of carbon composite block is 0.328 W/mK, identical to that of resin infused carbon/510A vinyl ester composite laminates but slightly lower than that of pultruded samples (0.431 W/mK); 2) For the samples of similar thickness, the transverse thermal

Figure 6.7 After-test disk samples of carbon fabric/vinyl ester composite block II

conductivity of carbon composite block is about the same as that of longitudinal direction (1.385 W/mK in transverse versus 1.308 W/mK in longitudinal). This is in agreement with the fabric configuration where fiber density and percentage share is identical in 0 degree orientation and 90 degree orientation; 3) The carbon composite block has a typical orthotropic thermal conducting behavior and the heat conducting capacity is four times higher along the fiber direction than perpendicular to the fiber direction 4) The data reported here are more representative than those of the previous for carbon/vinyl ester composite block that had poor wet-out of fabric, leading to debonding and higher thermal resistance. For comparison, the poorly wet-out block had a thermal conductivity 0.185 W/mK in the through-the-thickness direction, 0.954 W/mK in transverse and 0.956 W/mK in longitudinal direction; 5) Note that for the unidirectional carbon fiber composite block reported earlier, its 3D thermal conductivities are 1.089 W/m K along the fiber direction, 0.504 W/m K in the transverse direction and 0.263 W/m K through the

thickness direction, the current carbon fabric composite block has different 3D thermal conducting behavior; and 6) It is further verified that carbon composites having significantly different thermal conductivity values in three directions is distinctive from E-glass composite materials for which the thermal conductivity in three directions is almost the same.

**Table 6.3 Three-dimensional thermal conductivity values of carbon/510A
vinyl ester composite block II samples prepared using compression molding**

Heat flow direction	Sample No		Thickness mm (inch)	Thermal Conductivity, W/mK		
				Segment 1 at 140°F (60°C)	Segment 2 at 176°F (80°C)	Segment 3 at 212°F (100°C)
Through - thickness ($\perp$ 0 & 90 fiber)	1	Run 1	7.353 (0.289)	0.277	0.286	0.299
		Run 2	7.353 (0.289)	0.277	0.285	0.302
		Average		0.277	0.286	0.301
	2	Run 1	7.448 (0.293)	0.351	0.362	0.383
		Run 2	7.448 (0.293)	0.352	0.368	0.386
		Average		0.352	0.365	0.385
Transverse (// 90 & $\perp$ 0 fiber)	1	Run 1	6.99 (0.275)	1.253	1.392	1.526
		Run 2	6.99 (0.275)	1.235	1.374	1.529
		Average		1.244	1.383	1.528
	2	Run 1	6.905 (0.272)	1.210	1.352	1.544
		Run 2	6.905 (0.272)	1.239	1.402	1.561
		Average		1.225	1.377	1.553
Longitudinal (// 0 & $\perp$ 90 fiber)	1	Run 1	6.468 (0.255)	1.178	1.323	1.502
		Run 2	6.468 (0.255)	1.237	1.391	1.566
		Average		1.208	1.357	1.534
	2	Run 1	6.453 (0.254)	1.130	1.248	1.364
		Run 2	6.453 (0.254)	1.125	1.252	1.377
		Average		1.128	1.250	1.371

6.3.3 Statistical Analysis

The ANOVA output for the three-dimensional conductivity of composite block from MINITAB is shown below. R-Sq and R-Sq(adj) values for the model are very high and the complete variance is taken by the thermal resistance factor, giving least weight to other factors in the model. So, general linear regression analysis is applied to the model to evaluate the linear relation.

The REG Procedure					
Model: MODEL1					
Dependent Variable: conductivity					
Number of Observations Read				36	
Number of Observations Used				36	
Analysis of Variance					
Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	4	8.13243	2.03311	1018.04	<.0001
Error	31	0.06191	0.00200		
Corrected Total	35	8.19434			
Root MSE		0.04469	R-Square	0.9924	
Dependent Mean		1.02106	Adj R-Sq	0.9915	
Coeff Var		4.37671			
Parameter Estimates					
Variable	DF	Parameter Estimate	Standard Error	t Value	Pr > t
Intercept	1	2.32200	0.11604	20.01	<.0001
resistance	1	-71.86593	2.05081	-35.04	<.0001
heatflow	1	-0.38747	0.04887	-7.93	<.0001
temp	1	-0.00499	0.00121	-4.12	0.0003
Hxt	1	0.00379	0.00055897	6.77	<.0001

The regression analysis for the three dimensional conductivity model is run in SAS (Statistical Analysis Software) to study the factor and interaction effects. The regression output from SAS is as shown above. The regression equation is estimated to be

$$E(\text{Conductivity}) = 2.31 - 71.86 \cdot \text{resistance} - 0.382 \cdot \text{heat flow} - 0.005 \cdot \text{temp} + 0.004 \cdot \text{heat flow} \cdot \text{temp}$$

The constant (intercept) value (**bo** = 2.32) is the predicted value of conductivity when each predictor (heat flow, Set point temp, thermal resistance) is zero. The slope (**b₁** = -71.865) is the change in conductivity when thermal resistance increases by 1. That is

when the thermal resistance increases by 1 unit conductivity decreases by 71.61 units. The slopes ($b_2 = -0.387$, $b_3 = -0.005$) are the change in conductivity with unit increase of heat flow and set point temperature and the slope ($b_4 = -0.004$) is the change in conductivity with unit increase of the interaction between heat flow and temperature. The relationship between the response, conductivity, and the predictors, heat flow ($p < 0.0001$), Set point temp ($p = 0.0003$) and thermal resistance ($p < 0.0001$) and the interaction ($p < 0.0001$) are significant, i.e., all the predictors and the interaction term has influence on the conductivity.

Example:
$$E(\text{Conductivity}) = 2.31 - 71.61 \times 0.00468 - 0.382 \times 3 - 0.005 \times 100 + 0.004 \times 3 \times 100$$
$$= 1.461$$

$E(\text{Conductivity}) \sim 1.46 \text{ W/mK}$

The expected conductivity value for the 3-D Carbon composite block in the heat flow direction Z- (Longitudinal) at 100degC set point temperature is 1.46 W/mK, which is higher than the original value i.e. 1.382 W/mK. There is 0.079W/mK difference between the expected and original value, which is because of the proportion of variance (R-square) in the model.

Least Squares Means for conductivity		
Heat flow	Mean	SE Mean
1	0.3630	0.01233
2	1.3896	0.01233
3	1.3106	0.01233
Setpoint temp		
60 degC	0.9252	0.01233
80 degC	1.0163	0.01233
100 degC	1.1217	0.01233

The least square means for composite block model and the main effects plot tells that the conductivity of the sample is highest in 2- transverse direction (Y) and at higher set point temperature. The conductivity value increases with increase in set point temperature as shown in figure 6.8.

Figure 6.8 Variation of conductivity with heat flow and set point temperatures

Figure 6.9 Normal probability plot of residuals for 3-D Composite Block

Figure 6.9 plots the residuals versus their expected values when the distribution is normal. For the composite block data, the residuals appear to follow a straight line with a small deviation. The presence of small outliers in the conductivity data are attributed to the improper application of thermal compound on the sample while testing for conductivity. In order to test the normality, Kolmogorov-Smirnov test is applied on the residuals.

Figure 6.10 Kolmogorov-Smirnov test on normality of residuals for 3-D Composite Block data

From figure 6.10, the p-value (<0.010) less than desired α (0.05), so the null hypothesis gets rejected, which tells that the data does not follow a normal distribution. The usual approach to deal with nonconstant variance when it occurs for the above reasons is to apply a **variance-stabilizing transformation** and then to run the analysis of variance on the transformed data. Logarithmic transformation is applied on the original data.

The regression analysis for the three dimensional conductivity model for the transformed data is run in SAS (Statistical Analysis Software) to study the factor and interaction effects. The regression output from SAS is as shown below.

The REG Procedure					
Model: MODEL1					
Dependent Variable: Conductivity					
Number of Observations Read				18	
Number of Observations Used				18	
Analysis of Variance					
Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	4	1.31349	0.32837	2651.19	<.0001
Error	13	0.00161	0.00012386		
Corrected Total	17	1.31510			
Root MSE		0.01113	R-Square	0.9988	
Dependent Mean		-0.06340	Adj R-Sq	0.9984	
Coeff Var		-17.55362			
Parameter Estimates					
Variable	DF	Parameter Estimate	Standard Error	t Value	Pr > t
Intercept	1	0.54772	0.04094	13.38	<.0001
Resistance	1	-39.70536	0.71264	-55.72	<.0001
Heatflow	1	-0.12196	0.01723	-7.08	<.0001
Temp	1	-0.00172	0.00042705	-4.04	0.0014
HxT	1	0.00111	0.00019693	5.63	<.0001

The regression equation for the transformed data from the output is estimated to be

$$E(\text{Conductivity}) = 0.55 - 39.71 \cdot \text{Resistance} - 0.122 \cdot \text{Heat flow} - 0.002 \cdot \text{Temp} + 0.001 \cdot \text{Heat flow} \cdot \text{Temp}$$

Example:
$$E(\text{Conductivity}) = 0.55 - 39.71 \cdot 0.00468 - 0.122 \cdot 3 - 0.002 \cdot 100 + 0.001 \cdot 3 \cdot 100$$

$$= 0.157$$

$$E(\text{Conductivity}) = 0.157 \text{ W/mK}$$

The expected conductivity value for the 3-D Carbon composite block in the heat flow direction Z (Longitudinal) at 60degC set point temperature is 0.157 W/mK, which is slightly larger than the original value i.e. 0.141 W/mK. The difference between the expected value and original value is 0.016 W/mK, which is better after the data transformation.

Figure 6.11 Kolmogorov-Smirnov test on normality of residuals for transformed data on 3-D Composite Block

From figure 6.11 the p-value (0.037) is slightly less than the desired α , which is negligible for rejection of null hypothesis and leads to the conclusion that the data does follows a normal distribution

Figure 6.12 Interaction plot between heat flow and set point temperature

Figure 6.13 Interaction plot between set point temperature and heat flow

It can be observed from Figure 6.12 that the lines representing heat flow levels 2, 3 (i.e. heat flow in transverse and longitudinal direction) appear to be close to parallel so there is no evidence of an interaction between them, but the heat flow in through the thickness direction (1) has variation from the other two directions indicating its interaction for heat flow and set point temperature. That is the change in response differs across the levels of heat flow depending on the level of set point temperature, the two factors i.e., heat flow direction and set point temperature appear to interact.

6.4 Thermal History Effect on the Thermal Conductivity of FRP Bridge Deck Samples

The baseline thermal conductivity values of the bridge deck samples, resin infused carbon/510A and carbon/epoxy, and pultruded carbon/510A mentioned in chapter 3 were measured and listed in Table 5.4 along with the values for each cycle. These samples were placed into a frame box as shown in Figure 3.6. The frame box with total 10 samples is being placed outdoor and subjected to weathering effect (conditions like ice-snow, rain, sun and cloud). All the samples were tested for their thermal conductivity

properties in eight cycles. All the samples were tested for their thermal conductivity properties every 4 weeks for a period of 24 months (~1 year).

The selected FRP Thermal samples have been tested for eight cycles. The first cycle was for 30 days, the second for 25 days, the third cycle for 31 days, the fourth for 27 days, the fifth round for 51 days and the sixth round for 44 days, the seventh round for 42 days, and the eight round for a long time i.e., 136 days making the entire thermal history cycle for 368 days, which is about an year.

Thermal conductivity was determined for the above bridge deck samples and carbon composite samples using the guarded heat flow meter model Unitherm 2022. The baseline thermal conductivity values for these samples are indicated as 0 days in the Table 5.5.

The results show that the conductivity values have small changes for each cycle. But even though there may not be significant improvement, it makes to know the thermal effect of the samples when exposed to climatic conditions. After the third and fourth cycle the conductivity values for all the samples i.e. Bridge deck, Resin infused carbon composite laminates, Pultruded carbon composite laminates slightly improved and mostly significant with those of second cycle. For the fifth cycle and sixth cycle the conductivity values are more or less the same as of before and the slight variation in the conductivity values is attributed to the amount of thermal compound applied on the surface of the sample. The samples were let to weather for more cycle time after the seventh cycle for 136 days which made the total cycle time for 365 days app. one year, to see if there is variation in thermal conductivity. But there was hardly any difference in the thermal conductivities of the samples.

**Table 6.4 Through-the-thickness thermal conductivity values of bridge deck
glass composite samples**

Sample Description	Sample No		Thickness mm (inch)	Thermal conductivity, W/mK		
				Segment 1 at 140°F (60°C)	Segment 2 at 176°F (80°C)	Segment 3 at 212°F (100°C)
Pultruded Bridge Deck Samples: E-Glass/Vinyl Ester (to be used for studies of thermal history effect)	1	Run 1	4.740 (0.187)	0.337	0.359	0.378
		Run 2	4.740 (0.187)	0.337	0.361	0.379
		Average		0.337	0.360	0.379
	2	Run 1	5.118 (0.201)	0.221	0.233	0.249
		Run 2	5.118 (0.201)	0.221	0.233	0.252
		Average		0.221	0.233	0.251
	3	Run 1	5.000 (0.197)	0.347	0.371	0.394
		Run 2	5.000 (0.197)	0.351	0.374	0.395
		Average		0.349	0.373	0.395
	4	Run 1	4.796 (0.189)	0.322	0.334	0.353
		Run 2	4.796 (0.189)	0.316	0.334	0.349
		Average		0.319	0.334	0.351
	5	Run 1	5.100 (0.201)	0.345	0.367	0.379
		Run 2	5.100 (0.201)	0.346	0.367	0.388
		Average		0.346	0.367	0.384

**Table 6.5 Multi-Cycle Thermal history effect on Bridge deck samples and
carbon composite samples**

Pultruded Bridge deck glass composite samples						
Sample Description	Sample No	Cycle (no of days)	Segment 1 at 140°F (60°C)	Segment 2 at 176°F (80°C)	Segment 3 at 212°F (100°C)	Average K (W/mk)
E-glass/Vinyl Ester	1	0 Days	0.337	0.361	0.379	0.359
		30 Days	0.361	0.374	0.394	0.376
		25 Days	0.366	0.384	0.404	0.385
		31 Days	0.371	0.385	0.396	0.384
		27 Days	0.357	0.381	0.396	0.378
		51 Days	0.360	0.371	0.390	0.374
		44 Days	0.349	0.369	0.388	0.369
		42 Days	0.347	0.368	0.377	0.364
		136 Days	0.346	0.368	0.380	0.365
	2	0 Days	0.221	0.233	0.249	0.234
		30 Days	0.224	0.233	0.250	0.236
		25 Days	0.227	0.239	0.258	0.241
		31 Days	0.224	0.237	0.251	0.237
		27 Days	0.227	0.242	0.258	0.242
		51 Days	0.228	0.240	0.252	0.240
		44 Days	0.227	0.236	0.250	0.238
		42 Days	0.274	0.297	0.319	0.242
		136 Days	0.291	0.305	0.325	0.245
	3	0 Days	0.351	0.374	0.395	0.373
		30 Days	0.360	0.382	0.399	0.380
		25 Days	0.372	0.392	0.408	0.391
		31 Days	0.372	0.391	0.409	0.391
		27 Days	0.371	0.395	0.409	0.392
		51 Days	0.365	0.380	0.397	0.381
		44 Days	0.351	0.374	0.391	0.372
		42 Days	0.361	0.385	0.402	0.383
		136 Days	0.357	0.374	0.398	0.376
	4	0 Days	0.316	0.334	0.349	0.333
		30 Days	0.327	0.341	0.349	0.339
		25 Days	0.327	0.343	0.361	0.344
		31 Days	0.320	0.337	0.350	0.336
		27 Days	0.325	0.346	0.358	0.343
		51 Days	0.311	0.329	0.346	0.329
		44 Days	0.307	0.327	0.344	0.326
		42 Days	0.318	0.332	0.342	0.331
		136 Days	0.317	0.329	0.344	0.330

Resin Infused Carbon Composite Laminates						
Carbon/7FR-EPOXY	1	0 Days	0.388	0.401	0.419	0.403
		30 Days	0.397	0.408	0.419	0.408
		25 Days	0.390	0.410	0.424	0.408
		31 Days	0.391	0.405	0.417	0.404
		27 Days	0.398	0.407	0.424	0.410
		51 Days	0.390	0.401	0.414	0.402
		44 Days	0.394	0.400	0.412	0.402
		42 Days	0.385	0.399	0.407	0.397
		136 Days	0.380	0.392	0.408	0.393
	2	0 Days	0.416	0.425	0.441	0.427
		30 Days	0.417	0.428	0.446	0.430
		25 Days	0.415	0.431	0.450	0.432
		31 Days	0.414	0.434	0.442	0.430
		27 Days	0.416	0.437	0.445	0.433
		51 Days	0.416	0.429	0.437	0.427
		44 Days	0.410	0.424	0.436	0.423
		42 Days	0.413	0.423	0.434	0.423
		136 Days	0.410	0.427	0.437	0.425
Carbon/510A Vinyl Ester	3	0 Days	0.325	0.336	0.353	0.338
		30 Days	0.327	0.346	0.358	0.344
		25 Days	0.330	0.338	0.360	0.343
		31 Days	0.332	0.346	0.363	0.347
		27 Days	0.327	0.345	0.363	0.345
		51 Days	0.329	0.328	0.358	0.338
		44 Days	0.327	0.340	0.355	0.341
		42 Days	0.326	0.340	0.349	0.338
		136 Days	0.324	0.334	0.351	0.336
	4	0 Days	0.309	0.319	0.339	0.322
		30 Days	0.310	0.328	0.341	0.326
		25 Days	0.314	0.326	0.346	0.329
		31 Days	0.317	0.330	0.363	0.337
		27 Days	0.312	0.329	0.339	0.327
		51 Days	0.314	0.322	0.333	0.323
		44 Days	0.310	0.325	0.335	0.323
		42 Days	0.310	0.323	0.334	0.322
		136 Days	0.315	0.320	0.337	0.324

Pultruded Carbon Composite Laminates (Navy sandwich panel face sheet)						
Carbon/510A Vinyl Ester	1	0 Days	0.419	0.445	0.463	0.442
		30 Days	0.419	0.447	0.469	0.445
		25 Days	0.439	0.454	0.479	0.457
		31 Days	0.435	0.456	0.479	0.457
		27 Days	0.434	0.462	0.480	0.459
		51 Days	0.403	0.450	0.475	0.443
		44 Days	0.423	0.446	0.468	0.446
		42 Days	0.401	0.425	0.441	0.422
		136 Days	0.420	0.439	0.468	0.442
	2	0 Days	0.399	0.420	0.440	0.420
		30 Days	0.406	0.427	0.454	0.429
		25 Days	0.412	0.432	0.453	0.432
		31 Days	0.412	0.435	0.459	0.435
		27 Days	0.408	0.433	0.458	0.433
		51 Days	0.401	0.421	0.441	0.421
		44 Days	0.395	0.420	0.437	0.417
		42 Days	0.397	0.418	0.436	0.417
		136 Days	0.398	0.414	0.443	0.418

6.5 Effect of the Amount of Thermal Compound on Thermal Conductivity

Thermal compound (also called thermal grease) is a substance that increases thermal conductivity between the surfaces of two objects. During the thermal conductivity measurement, standard silicone-based thermal grease has been used that has a thermal conductivity of 0.73 W/mK to mediate small surface imperfections between test sample and heat sink. When applied in appropriate quantities, it fills the tiny pits and grooves on both surfaces of the sample, helping surface-to-surface contact. During the course of our study, it has been observed that it is important to use no more than is necessary to exclude any air gaps in order to obtain representative thermal conductivity value of the material.

The effect of the amount of thermal compound on thermal conducting behavior was studied by measuring the thermal conductivity with varying the number of minor drops applied on both surfaces of a composite sample. Tests were made on the Carbon/Vinyl ester composite block samples. Six samples, two from each direction (i.e. X- through-the-thickness, Y- longitudinal and Z- transverse directions), were first tested with the number of drops ranging from 2 to 5 and then samples X1 and Y2 were further

examined with the number of drops ranging from 2 to 15 for a better trend and the results are listed in Table 6.6.

Data in Table 6.6 were represented in Figures 6.14-6.16, where thermal conductivity was plotted as a function of the number of minor drops of thermal compound. It appears that the amount of applied thermal compound affects the resulting thermal conductivity in a limited manner. The following observations can be made: 1) without application of thermal compound, the poor sample-surface-to-heat-sink-surface contact results in lower thermal conductivity than that with appropriate application of thermal compound; 2) within a certain amount (2–5 drops), the applied thermal compound almost has no significant effect on the material's thermal conductivity; 3) only when it is over-applied, the thermal compound has a slight to moderate effect on the material's thermal conductivity, leading to higher values; 4) this effect even becomes weaker for low thermal conductivity samples (X-direction samples) than high thermal conductivity samples (Y-direction samples), 5) the enhancement on thermal conducting behavior with the use of the thermal compound will become saturated at a point above which further increasing the amount of thermal compound will not result in better thermal conducting; 6) a maximum of about 10% increase in thermal conductivity is observed for the X-direction sample studied while about 40% for the Y-direction sample studied; and 7) the Z-direction samples are expected to behave the same as Y-direction samples because of their identical fabric configuration. Hereafter all the thermal conductivity data are generated with application of a couple of minor drops of the thermal compound on both surfaces of the samples.

Table 6.6 Effect of the amount of thermal compound on the thermal conductivities of Carbon/Vinyl ester composite block samples

X – direction samples						
Sample No	Thickness (mm)	Amount of compound No. of drops	Segment 1 at 140°F (60°C)	Segment 2 at 176°F (80°C)	Segment 3 at 212°F (100°C)	Average conductivity K (W/m.K)
1	7.353	14	0.307	0.319	0.334	0.320
		13	0.309	0.323	0.334	0.322
		12	0.303	0.324	0.334	0.320
		12	0.306	0.318	0.334	0.319
		11	0.307	0.323	0.331	0.320
		10	0.298	0.310	0.326	0.311
		9	0.288	0.304	0.310	0.301
		8	0.291	0.303	0.319	0.304
		7	0.281	0.292	0.304	0.292
		6	0.277	0.288	0.311	0.292
		5	0.279	0.289	0.307	0.292
		4	0.277	0.286	0.299	0.287
		3	0.285	0.295	0.303	0.294
		2	0.276	0.288	0.297	0.287
		2	0.277	0.285	0.302	0.288
		1	0.276	0.291	0.305	0.291
		0	0.269	0.283	0.298	0.283
2	7.448	4	0.351	0.362	0.383	0.365
		3	0.345	0.368	0.387	0.367
		2	0.352	0.368	0.386	0.369
		0	0.329	0.349	0.373	0.350
Y- direction samples						
1	6.990	3	1.253	1.392	1.526	1.390
		2	1.235	1.374	1.529	1.379

		2	1.286	1.420	1.585	1.430
		0	0.857	0.977	1.150	0.995
2	6.905	15	1.854	2.019	2.172	2.015
		14	1.946	2.102	2.246	2.098
		14	2.023	2.185	2.350	2.186
		13	1.919	2.093	2.246	2.086
		12	1.860	2.027	2.192	2.026
		11	1.694	1.856	2.031	1.860
		10	1.686	1.835	2.017	1.846
		9	1.647	1.807	2.002	1.819
		8	1.646	1.806	1.970	1.807
		7	1.435	1.590	1.778	1.601
		5	1.186	1.414	1.731	1.444
		4	1.313	1.485	1.67	1.489
		4	1.239	1.402	1.561	1.401
		3	1.126	1.291	1.469	1.295
		2	1.210	1.352	1.544	1.369
Z - direction samples						
1	6.468	5	1.392	1.518	1.670	1.527
		4	1.278	1.426	1.589	1.431
		3	1.178	1.323	1.502	1.334
		2	1.087	1.247	1.455	1.263
		2	1.237	1.391	1.566	1.398
		0	0.943	1.046	1.181	1.057
2	6.453	5	1.296	1.433	1.551	1.427
		4	1.125	1.252	1.377	1.251
		3	1.123	1.250	1.392	1.255
		2	1.095	1.208	1.319	1.207
		2	1.130	1.248	1.364	1.247

Figure 6.14 Effect of the thermal compound on thermal conductivity for X-direction samples

Figure 6.15 Effect of the thermal compound on thermal conductivity for Y-direction samples

Figure 6.16 Effect of the thermal compound on thermal conductivity for Z-direction samples

6.6 Hetron 922 vinyl ester resin based conductive composite samples

The manufacturing of Hetron 922 L25 vinyl ester resin based samples with graphite powder GP44R-B is described in Chapter 3. Vinyl ester is an engineering resin, which finds many applications in structural and civil engineering. The reason to choose this resin is due to the fact that most of the FRP bridge decks are made of this polymer reinforced with glass fiber, thereby giving a higher degree of compatibility. After demolding and post curing, the samples were tested for their conductivity as described in Chapter 4. Their electrical resistance data were plotted as a function of graphite concentration ranging from 0-30% wt in Figure 6.17.

Figure 6.17 Conducting behaviors of graphite filled vinyl ester samples

Neat vinyl ester resin is conductive and thus has very high resistance. Above Figure shows that as more and more graphite powder is needed into the resin, the sample is becoming conductive. Since the resistance data of the samples appeared to have an exponential relationship with graphite concentration, the data were replotted in a log-log scale and presented in figure 6.18. As seen from the figure, the relationship between resistance and graphite powder concentration becomes linear in a log-log chart. Data fitting demonstrates that the resistance is reciprocal of graphite concentration with a power index of approximately 3.

Samples of graphite powder dispersed in Hetron 922 vinyl ester were tested under tension using an Instron machine model 5567 as shown in Figure 4.6. The stress and resistance data as a function of tensile strain are plotted in Figures 6.27 to 6.33 for all Hetron 922 L vinyl ester samples with graphite concentration ranging from 12% to 22.5% by weight except 8% and 10% graphite samples that shows infinitely large resistance.

Figure 6.18 A power data fit with logarithmic axes

As seen from those figures, a general trend is that the resistance and the stress increase with strain until failure.

It has also been found that the variation of resistance with strain can be described well by fitting the data into a polynomial model. The fitting model can be expressed as follows:

$$R = R_0 + R_1 (\epsilon) + R_2 (\epsilon)^2 \quad (6.1)$$

Where R is the resistance, R_0 is initial resistance, R_1 and R_2 are coefficients, and ϵ is strain. Sensitivity (S_A), i.e. identical to Gauge factor (GF) for a strain gauge, is defined as the ratio of fractional change in electrical resistance to the fractional change in length (strain), i.e.

$$S_A = \frac{\Delta R / R}{\Delta L / L} = \frac{\Delta R / R}{\epsilon} \quad (6.2)$$

We are only interested in operating our sensor in the linear range (stress vs strain linear) instead of operating it in the entire range till failure. This is because we want the sensor to be in the elastic region so that it would come back to its original state after releasing the load on the sensor. We have taken 0-0.5% of strain as the linear region in the case of Hetron 922 based samples. We therefore model the resistance as a linear

equation instead of modeling it as a polynomial with an order 3 as shown in Equation 6.2.

Therefore the new equation for the resistance is as follows:

$$R = R_0 + R_1(\varepsilon\%) \quad (6.3)$$

Where, Z is the slope of the resistance vs. strain curve. Z tells us the rate of change of resistance with respect to strain. The stress vs. strain is linear in the region of 0-0.5% strain. The following equations were established:

$$R - R_0 = R_1 (\varepsilon\%) \quad (6.4)$$

$$R - R_0 = \Delta R \quad (6.5)$$

From equations 5.4 & 5.5, we have

$$\frac{\Delta R}{R_0} = \frac{R_1 (\varepsilon\%)}{R_0} \quad (6.6)$$

Therefore

$$\frac{\Delta R / R_0}{\varepsilon} = 100 \frac{R_1}{R_0} \quad (6.7)$$

We know that the left hand side of equation 5.7 is the sensitivity (gauge factor) of the sensor. So,

$$\text{Gauge Factor} = \text{Sensitivity} = 100 \frac{R_1}{R_0}$$

We can therefore conclude that the sensitivity of a conducting composite sensor in the linear range is directly proportional to the rate of change of resistance with strain and inversely proportional to the initial resistance.

6.7 Scanning Electron Microscopy

The behavior and performance of polymer composite cannot be understood solely on the basis of the specific properties of its principal components (the fibers and the matrix). The interface that exists between fibers and matrix is an essential element of the composite. Adequate adhesion between the fibers and matrix is a precondition for an optimized stress transfer in fiber reinforced polymer. Some of the observations cited in literature show,

- Surface treatment improves the fiber-matrix adhesion by modifying the structure and the chemical composition of the fiber surface,
- When sizing is used, stress transfer from the matrix to the fibers depends both on the fiber-resin adhesion and the quality of the sizing-matrix junction.

Electron microscopy Figure 6.19, in a high resolution mode is necessary to obtain useful information about the fine structure of the fiber-matrix interface. For the microstructure study by SEM, small sample dimension of cubical shape of size 0.25” was selected.

Figure 6.19 Hitachi S-4700 field emission scanning electron microscope (FE-SEM)

6.7.1 SEM for GP44R-B Samples

It is surprising to observe that graphite powder dispersed in vinyl ester makes a conductive composite but graphite powder dispersed in epoxy makes an insulator. This fact implies that a conducting graphite network is formed in graphite/ vinyl ester system, but not formed in graphite / epoxy system. In order to provide an understanding, both the cured samples were studied under a scanning electron microscope (SEM) so as to observe their morphology. Typical micrographs were shown in Figures 6.20- 6.26.

As clearly revealed in Figures 6.20 - 6.22, graphite powder is very well dispersed in epoxy resin and epoxy resin acts as an insulating coating on graphite particle surfaces so that no conducting graphite channel or network is formed. Instead, Figures 6.23- 6.26 indicates that graphite powder is poorly dispersed in vinyl ester resin in a microscopic

scale and graphite particles remain in the form of aggregates. There are many conducting graphite channels and network in the composite. The distinction in morphology between graphite/ epoxy and graphite/vinyl ester is attributed to graphite's compatibility with epoxy and incompatibility with vinyl ester.

Figure 6.20 SEM micrograph of graphite GP44-B in E-Bond at 10wt%

Figure 6.21 SEM of graphite GP44-B in E-Bond at 25wt%

Figure 6.22 SEM of graphite GP44-B in E-Bond at 25wt%

Figure 6.23 SEM of graphite GP44R-B in Vinyl ester at 20wt%

Figure 6.24 SEM of graphite GP44R-B in Vinyl ester at 20wt%

Figure 6.25 SEM of graphite GP44R-B in Vinyl ester at 20wt%

Figure 6.26 SEM of graphite GP44R-B in Vinyl ester at 20wt%

Therefore, SEM morphological micrographs provide direct information for understanding the conducting behavior of the composites.

From the Figures 6.27 to 6.33 and the other tension test results it has been clearly observed that there is a linear relationship between stress and strain up to a strain value of 0.5%. So the sensitivity of the sensor is computed in this region taking the strain to be 0.5% instead of taking the final strain to failure. In order to use as a sensor, the material needs to demonstrate as high sensitivity as possible. The sensitivity ratio or else known as gauge factor is also listed in Table 6.7 for all the samples.

Figure 6.27 Stress and resistance w.r.t strain for 12% graphite/vinyl ester samples

Figure 6.28 Stress and resistance w.r.t strain for 13% graphite/vinyl ester samples

Figure 6.29 Stress and resistance w.r.t strain for 14% graphite/vinyl ester samples

Figure 6.30 Stress and resistance w.r.t strain for 15% graphite/vinyl ester samples

Figure 6.31 Stress and resistance w.r.t strain for 17.5% graphite/vinyl ester samples

Figure 6.32 Stress and resistance w.r.t strain for 20% graphite/vinyl ester samples

Figure 6.33 Stress and resistance w.r.t strain for 22.5% graphite/vinyl ester samples

Figure 6.34 Stress and resistance w.r.t strain for 17.5% graphite/vinyl ester samples

From the figure 6.34 it is observed that the stress versus strain curves from replication samples overlap each other and show good consistency and reproducibility while the consistency and reproducibility for the resistance versus strain curves from replication samples improves as the graphite powder concentration increases.

The results indicate that 13% graphite in vinyl ester samples have lower sensitivity ratios when compared to other graphite powder concentrations. A general trend appears to be that the change in resistance increases with decreasing in the powder concentration as seen in Table 6.7. It is because of the fact that, if there are more graphite powder particles in the matrix, the particles will be more closely packed. Increase in resistance can be attributed to the separation of graphite particles from each other. If the particles are loosely packed at lower graphite concentrations, it will be easier to separate them, leading to a larger change in resistance. However, if the graphite concentration is too low as in the case of 12%, the initial resistance becomes large which also depresses the sensitivity. The variation of gauge factor with concentration of graphite powder is shown in Figure 6.35.

Figure 6.35 Variation of gauge factor w.r.t graphite powder concentration

Table 6.7 Change in resistance under tension of graphite/vinyl ester samples

Graphite Concentration (%)	Sample No	Initial resistance (KOhms)	Resistance at 0.5% Strain	Change in Resistance, ΔR (KOhms)	$\Delta R/R$	Sensitivity (gauge factor) $\Delta R/R/\text{strain}$
22.5	1	2.96	3.06	0.1084	0.36650	7.33
	2	2.89	2.96	0.0694	0.02396	4.80
	3	2.91	2.98	0.0781	0.02686	5.37
	4	4.08	4.18	0.0965	0.02365	4.73
	5	3.22	3.31	0.0900	0.02795	5.59
	Average	3.21	3.30	0.09	0.09	5.56
20	1	4.47	4.89	0.4200	0.09396	18.79
	2	4.39	4.79	0.4000	0.09112	18.22
	3	4.16	4.73	0.5700	0.13702	27.40
	4	3.95	4.34	0.3900	0.09873	19.75
	5	4.28	4.83	0.5500	0.12850	25.70
	6	4.17	4.69	0.5200	0.12470	24.94
	Average	4.24	4.71	0.48	0.11	22.47
17.5	1	7.90	8.62	0.7200	0.09114	18.23
	2	9.03	10.38	1.3500	0.14950	29.90
	3	8.20	9.15	0.9500	0.11585	23.17
	4	6.55	7.24	0.6900	0.10534	21.07
	5	7.61	8.34	0.7300	0.09593	19.19
	6	7.36	8.03	0.6700	0.09103	18.21
	Average	7.78	8.63	0.85	0.11	21.63
15	1	19.98	21.05	1.0700	0.05355	10.71
	2	22.42	24.28	1.8600	0.08296	16.59
	3	22.56	26.31	3.7500	0.16622	33.24
	4	21.10	22.97	1.8700	0.08863	17.73
	5	28.10	31.28	3.1800	0.11317	22.63
	Average	22.83	25.18	2.35	0.10	20.18
14	1	31.33	34.07	2.7400	0.08746	17.49
	2	35.73	38.34	2.6100	0.07305	14.61
	3	32.01	33.90	1.8900	0.05904	11.81
	4	25.92	28.46	2.5400	0.09799	19.60
	5	23.18	26.12	2.9400	0.12683	25.37
	6	28.56	31.49	2.9300	0.10259	20.52
	Average	29.46	32.06	2.61	0.09	18.23
13	1	47.65	52.25	4.6000	0.09654	19.31
	2	41.54	45.37	3.8300	0.09220	18.44
	3	46.75	51.84	5.0900	0.10888	21.78
	4	46.22	50.35	4.1300	0.08936	17.87
	5	69.85	73.85	4.0000	0.05727	11.45
	Average	50.40	54.73	4.33	0.09	17.77

It can be clearly observed from the Figure 6.35 that the sensitivity of the sensor is maximum at ~15% by weight of graphite. The gage factor of the sensor is a function of change in resistance as well as the initial resistance (resistance of the sample with no applied strain). The sensitivity ratio also depends on the particle size from the previous work. If the polymer used is more ductile, the strain to failure will be more and thus higher the resin's elongation to failure, the greater the change in resistance will be, leading to a larger sensitivity ratio. Hence the sensitivity of the conductive composite sensor has been greatly affected by:

- The insulation of end electrodes (copper rods) in the composite sensor
- Diameter of the copper rod used
- Type of polymer and conductive filler
- Particle size of the conductive filler

6.6.1 Statistical Analysis

The ANOVA output for the conductive composite sensor from MINITAB is shown below. The most important statistic in the analysis of variance table is the p-value (P). The p-value for the graphite concentration factor is 0.000, which means that the

General Linear Model: Resistance change versus Graphite %

Factor	Type	Levels	Values
Graphite %	fixed	6	13.0, 14.0, 15.0, 17.5, 20.0, 22.5

Analysis of Variance for Resistance change, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Graphite %	5	49.5352	49.5352	9.9070	33.33	0.000
Error	18	5.3496	5.3496	0.2972		
Total	23	54.8848				

S = 0.545160

R-Sq = 90.25%

R-Sq(adj) = 87.55%

actual p-value is less than 0.0005. Since this is less than the chosen α -level of 0.05, the effect of graphite concentration on the resistance change is significant. The predictor, graphite concentration explains 90.25% the amount of variation in the observed response

i.e., resistance change ($R^2 = 90.25$). The regression equation for the composite sensor data obtained from MINITAB is shown in the below table.

Regression Analysis: Resistance change versus Graphite %					
The regression equation is					
Resistance change = 8.26 - 0.387 Graphite %					
Predictor	Coef	SE Coef	T	P	
Constant	8.2620	0.8330	9.92	0.000	
Graphite %	-0.38665	0.04806	-8.05	0.000	
S = 0.795486 R-Sq = 74.6% R-Sq(adj) = 73.5%					
Analysis of Variance					
Source	DF	SS	MS	F	P
Regression	1	40.963	40.963	64.73	0.000
Residual Error	22	13.922	0.633		
Total	23	54.885			

The regression equation for the conductive composite sensor from MINITAB is given below

$$E(\text{Resistance Change}) = 8.26 - 0.387 * \text{Graphite\%}$$

The slope ($b_1 = -0.387$) gives inverse relationship between the resistance change and graphite concentration. The resistance change is different for different graphite concentrations and it decreased with increase in graphite concentration as shown in figure 6.36. Resistance change is high at less graphite concentration and is vice versa. This is because the initial resistance of the sample is very high at less graphite concentration making a high resistance change.

Example: $E(\text{Resistance Change}) = 8.26 - 0.387 * 13.0$
 $= 3.229$

$$E(\text{Resistance Change}) \sim 3.23$$

The expected value for resistance change at 13% of graphite concentration is 3.23 KOHms which is smaller than the original value which is 3.83 KOHms.

Figure 6.36 Variation of Resistance Change versus Graphite%

Unusual Observations for Resistance change					
Obs	Resistance change	Fit	SE Fit	Residual	St Resid
13	1.07000	2.13750	0.27258	-1.06750	-2.26 R
15	3.75000	2.13750	0.27258	1.61250	3.42 R

The unusual observation table presents, if there are observations with standardized residuals (St. Resid) that differ from zero by more than 2.00. Such observations may be outliers. The resistance change analysis output indicates that observations 13 and 15 have standardized residuals of -2.26 and 3.42 (R denotes an observation with a large standardized residual).

Least Squares Means for Resistance change		
Graphite %	Mean	SE Mean
13.0	4.26250	0.2726
14.0	2.44500	0.2726
15.0	2.13750	0.2726
17.5	0.76000	0.2726
20.0	0.44500	0.2726
22.5	0.08350	0.2726

The least squares means table displays the least squares means (Mean) and standard error of the mean (SE Mean) for each level of the graphite concentration factor. The results indicate that graphite concentration of 13% has the highest resistance change and 22.5% graphite concentration has the lowest resistance change as shown in figure 6.37.

Figure 6.37 Main effect plot of Resistance change with graphite %

Figure 6.38 Normal Probability plot of residuals for composite sensor

For the conductive composite sensor data , the residuals appear to follow a straight line as shown in figure 6.38, although the negative and positive tail fall slightly away from the line. No evidence of non-normality or evidence of unidentified variable exists. In order to verify the normality, Komogorov-Smirnov test is performed on the residuals.

Figure 6.39 Kolmogorov-Smirnov test on the normality of residuals for composite sensor data

From figure 6.39, the p-value (<0.010) is less than the desired α (0.05), so the null hypothesis gets rejected, which tells that the data does not follow a normal distribution. Apply a **variance-stabilizing transformation** and then to run the analysis of variance on the transformed data. Logarithmic transformation is applied on the original data.

The regression analysis for the three dimensional conductivity model for the transformed data is run in MINITAB to study the factor and interaction effects. The ANOVA and regression output from MINITAB is as shown below.

General Linear Model: Resistance Change versus Graphite %						
Factor	Type	Levels	Values			
Graphite %	fixed	6	13.0, 14.0, 15.0, 17.5, 20.0, 22.5			
Analysis of Variance for Resistance Change, using Adjusted SS for Tests						
Source	DF	Seq SS	Adj SS	Adj MS	F	P
Graphite %	5	7.6946	7.6946	1.5389	126.84	0.000
Error	18	0.2184	0.2184	0.0121		
Total	23	7.9130				
S = 0.110151 R-Sq = 97.24% R-Sq(adj) = 96.47%						

Regression Analysis: Resistance Change versus Graphite %

The regression equation is

Resistance Change = 2.76 - 0.165 Graphite %

Predictor	Coef	SE Coef	T	P
Constant	2.7638	0.1476	18.72	0.000
Graphite %	-0.165176	0.008518	-19.39	0.000

S = 0.141004 R-Sq = 94.5% R-Sq(adj) = 94.2%

Analysis of Variance

Source	DF	SS	MS	F	P
Regression	1	7.4756	7.4756	375.99	0.000
Residual Error	22	0.4374	0.0199		
Total	23	7.9130			

The regression equation for the conductive composite sensor from MINITAB is given below

$$E(\text{Resistance Change}) = 2.76 - 0.165 * \text{Graphite\%}$$

Example: $E(\text{Resistance Change}) = 2.76 - 0.165 \cdot 13.0$
 $= 0.615$

$E(\text{Resistance Change}) = 0.615 \text{ KOhms}$

The expected value for resistance change at 13% of graphite concentration is 0.615 KOhms which is slightly different from that of the original value 0.6159 KOhms. The normal probability plot for the residuals after transformation is as shown below.

Figure 6.40 Kolmogorov-Smirnov test on the normality of residuals for transformed composite sensor data

From figure 6.40, the p-value (0.067) less than the desired α (0.05), so rejection of null-hypothesis fails and thus the transformed data follows normal distribution.

6.7 Application of conductive composites as heating elements

As one of the potential applications is to heat a highway bridge deck for de-icing in winter, the conductive resin has been thought of being applied as a wearing surface on the bridge deck panel to study how the deck surface temperature varies with different parameters for feasibility of the conductive resin. A typical infrared camera is shown in Figure 4.8 to study the wearing surface on bridge deck panel. The image provides information on global temperature profiles over the panel surface.

The temperature of the wearing surface was observed to increase till the optimum level in about 60 to 80 minutes. The Snapshots / thermal images for the bridge deck panel with 4-inch and 6-inch span length wearing surface on it generated by the infrared camera were taken at periodic intervals of time till it reached the maximum temp. Some of those thermal images for the bridge panel with heated wearing surface from time to time are shown in figure 6.39.

Image 1. Typical thermal image generated by infrared camera for 25% graphite/922L composite sample at the beginning of the test

Image 2. Typical thermal image generated by infrared camera for 25% graphite/922L composite sample after 10 minutes of the test

Image 3. Typical thermal image generated by infrared camera for 25% graphite/922L composite sample after 20 minutes of the test

Image 4. Typical thermal image generated by infrared camera for 25% graphite/922L composite sample after 30 minutes of the test

Image 5. Typical thermal image generated by infrared camera for 25% graphite/922L composite sample after 40 minutes of the test

Image 6. Typical thermal image generated by infrared camera for 25% graphite/922L composite sample after 50 minutes of the test

Figure 6.41 Typical thermal images generated by an infrared camera for 25% graphite/Hetron 922 L wearing surface

It can be observed from these images that the surface with 2-inch span is brighter than the surface with 4-inch span, which shows that the conductive wearing surface with 2-inch span showed high conduction. But the thermal camera/image is not able to give the global temperature profile over the entire wearing surface, since the lens of the camera was not enough to focus on the bridge deck panel surface area. In order to get the temperature profile of the complete wearing surface area, a portable infrared temperature gun has been used. The laser ray from the gun is made to focus at different points on the wearing surface which is connected to an output AC voltage and the temperatures were recorded as shown in the figure 6.41.

Figure 6.42 Bridge deck panel with 2-inch span length wearing surface at four different concentrations

Initially the span length between the copper rods was taken as 2 inches and four different layers of wearing surface each with different graphite concentrations were prepared on a bridge deck panel as shown in the figure. The temperature on the surface was taken for every five minutes with the temperature gun and is plotted as a function of time as shown in the figures 6.43 to 6.45. The surface with 10% graphite concentration did not show any temperature increment. It has been observed that the temperature increased with time till it reached an optimum level for the surfaces with 15%, 20% and 25% graphite concentration. Higher the graphite concentration higher is the temperature of the wearing surface.

The general trend of the behavior for wearing surface with regards to the graphite concentration and the span length between the electrodes has been summarized as shown in Table 6.8.

Table 6.8 Electrical conductivity behavior of the wearing surface

Distance between the electrodes (inch)	Graphite powder concentration by weight	Electrical conductivity behavior of the wearing surface
2"	10%	No conduction observed
	15%	Conduction is observed
	20%	Conduction is observed
	25%	Conduction is high
4"	20%	Conduction is good
	25%	Conduction is good
6"	20%	Conduction is low
	25%	Conduction is low

Figure 6.43 Variation of temperature at the top end of the wearing surface at four different concentrations

Figure 6.44 Variation of temperature at the center of the wearing surface at four different concentrations

Figure 6.45 Variation of temperature at the bottom end of the wearing surface at four different concentrations

The temperature profiles are summarized in Table 6.9 for all concentrations. The data shows that the final temperature on the wearing surface increases with graphite concentration. According to Equation 6 the temperature increment is inversely proportional to the resistance. This relation is verified in Figure 6.46.

Table 6.9 Temperature profiles of Hetron 922L based conductive wearing surface subjected to an AC voltage for 60 minutes

% Of Graphite	Resistance	Min. temp at two ends F	Max. temp at the center F	Average temperature F
10	0.876 Mohms	81	81	81
15	51.9 KOhms	92	98	95
20	1.217 Kohms	190	216	203
25	0.585 Kohms	214	254	234

Figure 6.46 Maximum temperature and temperature difference at the center versus reciprocal of resistance for graphite/922L conductive wearing surface

In order to evaluate the feasibility of the span length with temperature variation, span length has been increased to four inches and six inches. The samples with 4-inch and 6-inch span length were also successful in heating the surface. The variation of temperature with time for these span lengths is shown in figures 6.47 and 6.49.

Figure 6.47 Variation of temperature with time for 4-inch span length bridge deck panel

Figure 6.48 Variation of temperature with time for 6-inch span length bridge deck panel

The temperature range for the wearing surface decreased with increase in the span length. A general trend showing the variation of temperature profile for three different span lengths is shown in figure 5.36.

Figure 6.49 Variation of maximum temperatures on the wearing surface at 2-inch, 4-inch and 6-inch span lengths

Table 6.10 Temperature variation of the wearing surface at different span lengths

Distance between the copper rods (inch)	Min temp at the ends F	Max temp at the center F	Average temperature F
2	200	222	211
4	162	163	162.5
6	113	121	117

The temperature range between the span lengths had consistent increment at an average value of 40 deg F and is high for the lowest span length and vice versa. Thus from the different temperature profiles, the conductive wearing surface is attributed to

- The area of the wearing surface on the panel
- The amount of graphite concentration in the wearing surface
- The distance between the copper rods/Span length
- Type of wearing surface (with/without aggregates)

The results have demonstrated that the developed conductive wearing surface function well as fast and effective heating surface.

5.7.1 Statistical Analysis

The temperature profile for the wearing surface has been studied at three span lengths i.e., 2", 4" and 6" respectively. Four different graphite concentrations 10%, 15%, 20% and 22.5% have been used. The ANOVA output for the conductive wearing surface from MINITAB is shown below.

General Linear Model: Temperature versus Span Length, Graphite %							
Factor	Type	Levels	Values				
Span Length	fixed	3	2, 4, 6				
Graphite %	fixed	4	0.100, 0.150, 0.200, 0.225				
Analysis of Variance for Temperature, using Adjusted SS for Tests							
Source	DF	Seq SS	Adj SS	Adj MS	F	P	
Span Length	2	19188	19188	9594	11141.26	0.000	
Graphite %	3	92663	92663	30888	35869.54	0.000	
Span Length*Graphite %	6	10840	10840	1807	2097.99	0.000	
Error	24	21	21	1			
Total	35	122711					
S = 0.927961 R-Sq = 99.98% R-Sq(adj) = 99.98%							

R-Sq and R-Sq (adj) values for the model are very high making the model inappropriate. So, general linear regression analysis is applied to the model to evaluate the factors. The regression equation for the conductive wearing surface with the factor effects and interaction term obtained from SAS is given below.

The REG Procedure					
Model: MODEL1					
Dependent Variable: Temperature					
Number of Observations Read				36	
Number of Observations Used				36	
Analysis of Variance					
Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	3	119087	39696	350.56	<.0001
Error	32	3623.48870	113.23402		
Corrected Total	35	122711			
Root MSE					
Dependent Mean		10.64115	R-Square	0.9705	
Coeff Var		142.97222	Adj R-Sq	0.9677	
		7.44281			
Parameter Estimates					
Variable	DF	Parameter Estimate	Standard Error	t Value	Pr > t
Intercept	1	-117.09981	17.14839	-6.83	<.0001
Spanlength	1	20.93644	3.96908	5.27	<.0001
AmtGraphite	1	18.74501	0.97742	19.18	<.0001
SxG	1	-2.07401	0.22623	-9.17	<.0001

The regression equation obtained from SAS output is given below,

$$E(\text{Temperature}) = -117.1 + 20.94 * \text{Span length} + 18.75 * \text{Graphite \%} - 2.07 * \text{Span length} * \text{Graphite \%}$$

The two factor effects (Span length, Graphite concentration) and the interaction between span length and graphite concentration have significant effect on temperature of the wearing surface because the P-value for all of them are less than the confidence level.

Example: $E(\text{Temperature}) = -117.1 + 20.94 * 2 + 18.75 * 22.5 - 2.07 * 2 * 22.5$
 $= 253.2$

$$E(\text{Temperature}) \sim 253 \text{ F}$$

The expected temperature for the wearing surface with 2 inch span length between the copper rods and 22.5% of graphite concentration is 253 F which is acceptable with the original data values obtained at three levels i.e. 252, 254, 254.

Unusual Observations for Temperature

Obs	Temperature	Fit	SE Fit	Residual	St Resid
1	81.000	79.000	0.536	2.000	2.64 R
29	100.000	98.333	0.536	1.667	2.20 R

Least Squares Means for Temperature

Span Length	Mean	SE Mean
2	169.42	0.2679
4	146.33	0.2679
6	113.17	0.2679
Graphite %		
0.100	77.00	0.3093
0.150	116.22	0.3093
0.200	167.78	0.3093
0.225	210.89	0.3093

Figure 6.50 Variation of span length and graphite% with temperature

The temperature of the wearing surface is high at 2-inch span length and is high for 22.5% graphite concentration. The temperature of the wearing surface decreases with increase in span length and increases with increase in graphite concentration as shown in figure 6.50.

Figure 6.51 Normal Probability plot of residuals for conductive wearing

The residuals appear to follow a straight line as shown in figure 6.51 and thus the error is normally distributed. No evidence of non-normality, outliers or unidentified variable exists. In order to verify the normality, Kolmogorov-Smirnov test is performed on the residuals.

Figure 6.52 Kolmogorov-Smirnov test on the normality of residuals for
conductive wearing surface data

From figure 6.52, the p-value (0.079) greater than the desired α (0.05), so null hypothesis is accepted and hence the data follows a normal distribution.

Figure 6.53 Span length by Graphite % interaction plot

Figure 6.54 Graphite% by Span Length interaction plot

The temperatures for the three levels of span length are plotted with respect to the graphite concentration in figure 6.53. All the three lines increase with increase in graphite concentration, but not at the same rate. The line for 2-inch span length increases the most, followed by 4-inch. The line with 6-inch span length shows least increase. Figure 6.54 shows the interaction plot for graphite concentration by span length. All the four lines decrease with increase in span length but not at the same rate. The lines with 10% and 15% graphite concentration have the highest decrease whereas the line with 22.5% of graphite concentration has the least decrease. Because graphite concentration affects the response depending on the level of span length and vice versa, the two factors appear to interact.

CHAPTER 7

CONCLUSIONS AND FUTURE WORK

7.1 Introduction

In this chapter conclusions are drawn from the experimental data obtained and the statistical analyses performed on the conductivity, sensor and the wearing surface. For each of these characteristics, we have also made some recommendations for future research.

7.2 Thermal Conductivity

- The pultruded carbon/510A composite has the highest thermal conductivity, i.e. ~ 0.432 W/mK.
- The transverse thermal conductivity of the quad axial distributed carbon fabric composite block is high and about the same as that of longitudinal direction (1.385 W/mK in transverse versus 1.308 W/mK in longitudinal). This is in agreement with the fabric configuration where fiber density and percentage share is identical in 0 degree orientation and 90 degree orientation.
- The average through-the-thickness thermal conductivity of carbon composite block is 0.328 W/mK.
- The statistical analysis of the data shows that the heat flow direction and the set point temperatures for the composite have significant effect on thermal conductivity. The effect of manufacturing method on the conductivity is also slightly significant.
- Thermal weathering of the bridge deck samples and other composite samples for approximately one year did not show much significant effect on the conductivity.
- The application of thermal compound on the test sample has shown significant effect on conductivity. It is important to use no more than what is necessary to exclude any air gaps in order to obtain representative thermal conductivity value of the material.

Future Work:

- Verify the effect of fiber orientation by testing composites with different orientations along with uni-directional and cross-ply composites.
- Control the manufacturing methods to reduce anomaly like fiber distortion, void content.

7.3 Conductive Composite Sensor

- The objective of our study was to develop conductive polymer composites as sensors in terms of change in electrical resistance as a function of deformation for health monitoring of civil structures and in particular, fiber reinforced polymer (FRP) composite structures. A different kind of powder GP44R-B was used as the conductive filler. The sensitivity (gauge factor) that is defined as an index of change in resistance in response to strain over the sensor's original resistance was calculated for each sample.
- The three main parameters involved in the preparation of conducting composite samples/sensors are the filler particle concentration, filler particle size and the polymeric resin.
- Gauge factor of 27 is achieved for the GP44R-B conductive filler which is more than that is obtained from the previous work i.e., 16 for other graphite powders (GP44B, GP55-B, GS75-E, and GS150-E).
- This sensitivity achieved is larger than existing strain gauges. The typical sensitivity of a metallic type strain gauge is around 2.

Future Work:

- Establish solid results for the variation of resistance with moisture and temperature.
- Examine the feasibility of embedding the conductive composite sensor in-line with production process of the structure.

7.4 Conductive Wearing Surface

- The conductive resin was applied on the bridge deck panels and the surface temperature profile was studied.
- The deck panel surface with 10 % graphite concentration did not show any increment of surface temperature.
- Surface with graphite concentration of 22.5 % and 2-inch span between the copper rods showed highest temperature.
- The temperature of the wearing surface increased with increase in the filler concentration and decreased in the span length of copper rods.
- The inverse relation between the resistance and the temperature was studied as per the basic theory.
- The statistical analysis of the results showed that the interaction between the span length and the amount of graphite concentration has significant effect on the temperature of the wearing surface.

Future Work:

- Study the wearing surface on bridge deck panel exposed to thermal weathering.
- Maintain uniform application of wearing surface on the deck panel.
- Evaluate the effectiveness of heating the wearing surface with the current method and that of conventional methods.

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