

INVESTIGATING THE ELECTRICAL BEHAVIOR OF NANOPARTICLE-INFUSED
HOLES ON CARBON FIBER-REINFORCED COMPOSITES
DURING FATIGUE LOADINGS

A Thesis by

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Bachelor of Engineering, Punjab Technical University, India, 2014

Submitted to the Department of Mechanical Engineering
and the faculty of the Graduate School of
Wichita State University
in partial fulfillment of
the requirements for the degree of
Master of Science

July 2018

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The following faculty members have examined the final copy of this thesis for form and content, and recommend that it be accepted in partial fulfillment of the requirement for the degree of Master of Science with a major in Mechanical Engineering.

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DEDICATION

To my respected parents, my dear friends, and my inspiration—Dr. Ramazan Asmatulu

ACKNOWLEDGMENTS

I would like to express my honest appreciation to Dr. Ramazan Asmatulu for his guidance and continuous support throughout my academic studies. Without his guidance and help, I would not have been able to succeed in this research. His dedication and leadership during the project were incredible. Conducting presentations every week, meeting with students, and providing valuable ideas on research are some of his assets that I greatly appreciate.

A huge respect and credit goes to Mr. Upul R. Palliyaguru and Mr. Nalinda Warnakulasooriya Waas of the National Institute for Aviation Research (NIAR) at Wichita State University (WSU) for continuously supporting and helping me to accomplish the necessary tests for this research. Without their permission to use the cyclic tensile machine at NIAR, testing would not have been successful.

I express my deep respect to my parents for encouraging me during my studies and always believing in me. Also, I feel blessed to have a brother, Rajan Bhatta, who has always been a support to me during my career.

In addition, I am so thankful to my other committee members, Dr. Davood Askari and Dr. Deepak Gupta, who set their busy schedule aside to be part of my thesis defense and supported me throughout my presentation. I am so thankful to my friends Rajan Shrestha, Kiran Nepal, Ankit Khadka, and other students as well as undergraduate and graduate research assistants currently working under Dr. Asmatulu who played an important part in my research.

ABSTRACT

One of the major problems and engineering issues in aircraft and automotive industries is galvanic corrosion on metal-metal and metal-composite interfaces. This occurs when two dissimilar metals or alloys are connected to each other at a common interface. Two metals, such as a carbon fiber-reinforced composite (CFRC) and a steel alloy, when joined together, experience galvanic corrosion at the joined interfaces because the difference in their electrical potentials and the presence of electrolytes or moisture causes the generation of galvanic cells. Carbon fiber being more noble than other metals and having excellent electric conductivity corrodes slowly. Therefore, the metals or alloys attached to a CFRC on an aircraft, automobile, or other structure corrode faster, and the resulting corrosion weakens the structural integrity of the composites. This thesis provides a detailed study of the mitigation of galvanic corrosion and improvement of corrosion resistance on a composite-metal joined structure. The experiments executed here were based on the application of 2, 4, and 8 weight percentages of nanoparticles (nanoclay and nanotalc) on a carbon fiber-reinforced composite hole having a baseline composition of epoxy resin (LOCTITE EA 9394). The composite specimens were treated with the nanoparticles and subjected to cyclic tensile loads on a MTS 810 test machine to investigate the variation of electrical resistance with respect to applied loads and time. The plots of resistance vs load and plots of resistance vs time mostly show an upward trend, indicating that with the application of nanoparticles as a sealant between two composite structures, the resistance to corrosion and deformation is increased, thereby decreasing the corrosive current throughout the composite surface. Nanoclay particles displayed a better performance, with the highest resistance measured at 43.9 ohms with the application of 8% wt% nanoclay particles.

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ABBREVIATIONS

CFRC	Carbon Fiber-Reinforced Composite
FTIR	Fourier Transform Infrared Analysis
NIAR	National Institute for Aviation Research
PVC	Polyvinylchloride
RTM	Resin Transfer Molding
VARTM	Vacuum-Assisted Resin Transfer Molding

CHAPTER 1

INTRODUCTION

1.1 Background

Carbon fiber-reinforced composite (CFRC) materials are the most preferred and widely used engineering materials in almost every structural use in today's engineering technology. Since CFRCs are very light in weight and high in strength, their popularity and usage has grown tremendously. Aircraft structures, automobiles, and wind turbines are the main application areas of CFRCs because the reduction in weight makes these structures perform better. It is always desirable to join two materials with similar physical properties by using composites, but occasionally a different material, such as aluminum, is also required to join two different materials with different interfaces. For example, in an aircraft structure, aluminum is used in the ground path because aluminum has a higher electrical conductivity than that of carbon composites [1]. Figure 1 shows a crack that occurs when a composite panel is joined to a removable steel fastener, and corrosion occurs as a result.

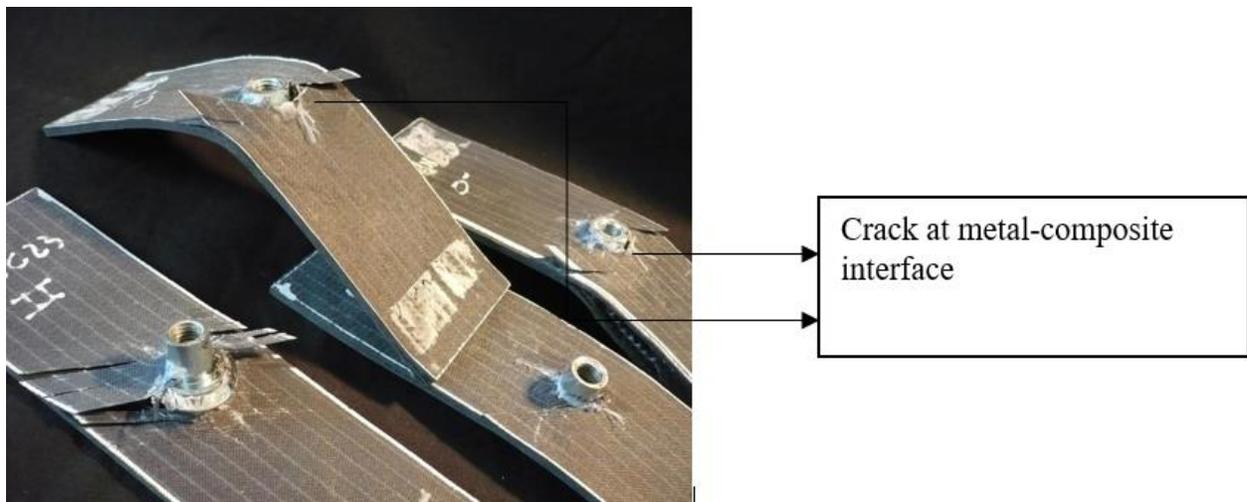


Figure 1. Crack at composite-steel joint.

Depending upon the application and the structure, two types of joints are used in most applications when it comes to attaching carbon fiber-reinforced composites to another structure such as an aluminum substrate. The first method of attachment is a permanent joint, and the second is a removable joint.

1.1.1 Permanent Joint of Composite Structures

In the case of a permanent joint, two materials with the same properties or two materials with different properties are joined together at a common interface by using a non-removable fastener. This method of attaching a material with a carbon fiber-reinforced composite material is mainly used in aircraft structures, such as joining two aircrafts skins with a fastener. Another example is composite decking material attached to one another with the help of a nail in residential structures. Therefore, in the case of a permanent joint, the fastener material must be chosen in such a way that it is most compatible with the composite's material properties. Figure 2 shows that the galvanic corrosion is much less when a permanent fastener is used in a composite structure.

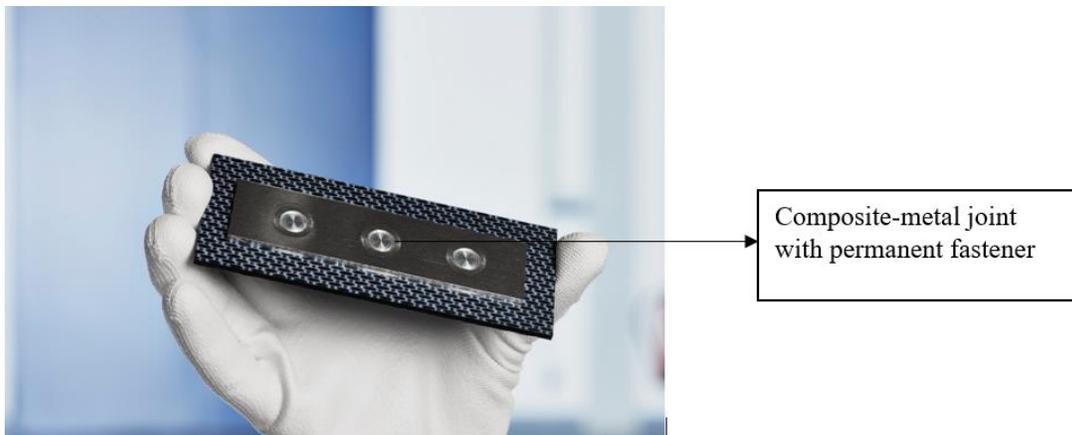


Figure 2. Composite-metal permanent joint.

1.1.2 Removable Joint of Composite Structures

Another very common method of joining materials with a carbon fiber-reinforced composite is a removable joint, whereby an easily removable bolt connects two materials together. Aircraft structures having an access panel in which a carbon composite is attached to an aluminum structure using a bolt is an example of this. Here, the bolt is attached with two materials, one a carbon fiber composite and the other an aluminum substrate, both having two different electric potentials as well as physical properties [2]. For this reason, a high percentage of corrosion occurs, and the nature of corrosion would be galvanic corrosion. Another very dominant factor in galvanic corrosion in this kind of joint would be the presence of moisture and dissimilarity in the physical properties. As shown in Figure 3, galvanic corrosion is likely to occur at a composite and fastener interface.

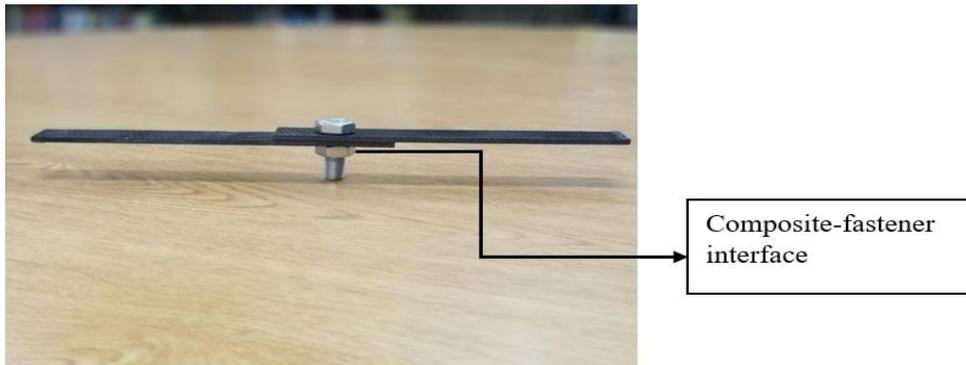


Figure 3. Carbon-steel fastener removable joint.

Fasteners that are used to join two different materials, including carbon fiber composites in a removable joint, are most likely to corrode over a period of time as it is exposed to moisture. This is a topic of great concern in aircraft and automobile companies because carbon fiber composites are continuously being used in these structures, and galvanic corrosion is the main issue when it comes to joining these structures together.

In the case of using a removable joint to attach two different structures a bolt is normally used, and a hole is drilled into the carbon fiber composite material. During this process, the outer most protective layer of the carbon composite is removed, leaving the fibers to attract moisture because they are left exposed to nature. When the bolt is attached between a carbon fiber composite and aluminum substrate, galvanic corrosion occurs. Galvanic cells are created where the carbon fiber composite acts as a cathode and the aluminum acts as an anode. Since carbon fiber is less noble than aluminum, aluminum will corrode faster, resulting in inhomogeneous structural integrity of the aircraft or other product. This is a serious issue in most structural companies that produce aircraft, automobiles, and wind turbine structures.

Many companies and researchers are working on several methods and processes to mitigate galvanic corrosion in the carbon-aluminum substrate. Depending upon the type of industry and the product being produced, various methods are used to mitigate such corrosion issues. An important factor is the type of fastener material, which should be similar to the substrate in order to minimize the galvanic impact. Another factor to be considered during the design process is the addition of glass plies as well as some sealants or paints, which greatly eliminates the galvanic effects [3]. Another option would be treating the holes of carbon fiber composites with sealant to mitigate corrosion. The work in this thesis is based upon a theory that in order to eliminate the galvanic effects and improve the barrier between two dissimilar materials, a very practical and long-term method is needed.

1.2 Motivation for Study

The motivation for this work comes from a very long-term issue that most structural industries have experienced. In general, carbon fibers are more electrochemically noble than most metals found in nature, which obviously conduct electricity. When carbon fiber composites

are used to join metals, they act like a cathode in an electrochemical cell when exposed to moisture. This phenomenon results in galvanic corrosion. When a hole is drilled in a carbon fiber composite skin to join metals using a bolt, the surface of the composite is exposed and has a greater tendency to absorb moisture. During this process, galvanic cells are created, and thereby galvanic corrosion occurs, easily corroding the metal. To overcome this issue, a special treatment on the composite structures is needed. The motivation for this research is based on this issue and results in applying sealant to the holes of carbon fiber composites, which would disable the galvanic bridge between the metals and the bolt, and thereby solve this long-term issue which has plagued the aviation industry.

1.3 Research Objectives

One objective of this research is to eliminate the galvanic effects occurring in metals that are joined together with carbon fiber-reinforced composite with a removable bolt. This research aims to mitigate the galvanic corrosion by treating the holes of CFRCs with nanoparticle-infused sealant in order to increase the corrosion resistance of the metal substrate. Another objective of this study is to evaluate and test the durability of infused nanoparticles compared with the baseline configuration in order to reduce crack propagation and increase corrosion resistance on the composite structures. This research is more focused on coating the composite specimen with different weight percentages of nanoparticles to ensure there is an increase in resistance as well as decrease in corrosive current with the applied load.

CHAPTER 2
LITERATURE REVIEW

2.1 Introduction to Composites

A composite material is defined as a material made up of two or more constituents having significantly different physical and chemical properties. Since the reaction between different combined constituents is more physical, the constituents retain their mechanical properties, which results in a final product with different characteristics than the original constituents. As shown in Table 1, carbon composite has physical properties that are distinctly excellent, with greater maximum tensile and specific strength than any other metals and alloys.

TABLE 1

COMPARISON OF PHYSICAL PROPERTIES OF METALS AND CARBON COMPOSITE

Materials	Tensile Strength (Mpa)	Specific Strength (Kn.m/kg)	Mass Density (kg/m³)
Aluminum	600	214	2.8
Magnesium Alloy (AZ91D)	230	135	1.7
Carbon Steel (0.45%C)	850	108	7.8
Titanium	950	216	4.4
Carbon Composite	1240	785	1.6

A composite material is distinct from any other material because it is composed of two different constituents—matrix and fibers—whose combination plays an important role in making a new material that is completely different from its parent material. A very good example demonstrating the unique nature of a composite material is that of glass fiber, which has a very high tensile strength but is also susceptible to damage [4], and a polymer resin, which has a very

low tensile strength but is malleable and very tough. When these two materials combine, a new, completely different material is formed, irrespective of its original characteristics, which is stronger and has higher stiffness.

Composite materials are primarily used in every structure in today's world, from airplanes to automobiles, clothing, wind turbines, electronics, and many more. Since carbon fiber-reinforced composites are light in weight and high in strength, they are one of the most desired structural elements and have gained considerable research value. Every composite, such as the carbon fiber composite, has basically two constituents: one is fiber, which is embedded in a matrix form and the other is a polymer matrix. The fiber acts as a load-carrying member, which carries and provides strength, the while polymer matrix transfers load to the fiber and acts as a binder, connecting the matrix together and resulting in greater stiffness of the composite [5].

Carbon fiber-reinforced composites are widely used in structural applications and they have many desirable properties, including the following [6]:

- Good fatigue resistance
- Good creep resistance
- Stronger than steel and stiffer than titanium
- Excellent wear and chemical resistance
- Low friction resistance
- Lower density than aluminum

Composite materials are resistant to fatigue, corrosion, and wear, and they provide a design flexibility and are easy to manufacture. These assets are important in the manufacturing industry because they help to reduce the overall lifetime of the material and production costs. Therefore, composite material is the future of engineering and manufacturing industries. The properties of

CFRCs mean that they are widely used in many structural applications such as automotive, aerospace, energy, electronics, biomedical, construction, household equipment and many other civil structures including residential homes and others. There are mainly two types of composites being used today: natural composites and artificial composites.

2.2 Natural Composites

Natural composites are those found in nature. Wood is one of the commonly occurring natural composite materials. It is referred to as a composite material because wood is fibrous, and these cellulose fibers are embedded in a lignin matrix. One of the main reasons behind wood's stiffness and strength is that the lignin matrix binds the cellulose fibers together strongly, whereas in cotton, for example, with the the same cellulose fibers, there is no lignin matrix, thus making it weaker and soft, and the fibers are left unbounded. To be stronger and stiffer, a composite always contains two constituents: fiber and polymer matrix [8].

Human bone is another example of a naturally occurring composite material. Human bone is composed of short collagen fibers bound together by a mineral matrix called apatite, which holds the fiber together and makes it harder, stronger, and able to support the weight of the body. Collagen, which combines with the brittle material calcium phosphate, makes the bone much harder; otherwise, collagen fibers alone would not be that strong. Cellulose fibers are aligned together in the direction of principal stress, along the branch, and a lignin matrix binds the fibers together. Hemicellulose, which is also present in wood, is a less crystalline form of cellulose [9].

The surrounding natural environment is comprised of various fibers that are embedded in a matrix form and act as a composite. Some of these are leaves, water plants, woody plants, fruits, grasses, wild plants, animal feathers and skins, and many more. As an example, Figure 4

shows the spatial arrangement of hemicellulose and lignin, which together act as a composite material.

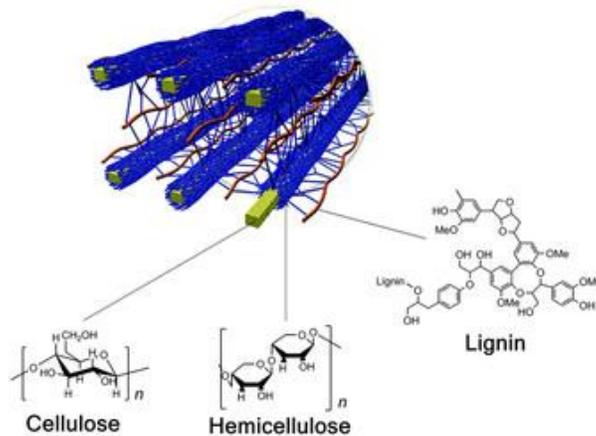


Figure 4. Wood as a natural composite [9].

2.3 Artificial Composites

Depending upon their usage and applications, various composites are currently being manufactured and fabricated, and their popularity is tremendously increasing. However, artificial composites have been manufactured and used in very ancient times. In 3400 B.C., composite materials were used in ancient Egypt by Jewish workers. Also, ancient Mesopotamians used composite materials to make plywood, which is considered to be one of the earliest usages of composite [10]. Also, some workers found a way to make clay bricks made stiffer and stronger by embedding them with straw, which greatly prevented crack propagation and provide more reinforcement. In the 1800s, people discovered the process of polymerization, which resulted in a cross-linked structure, thereby inventing synthetic resins. Also, during this time, other plastics, polyesters, polystyrene, and vinyl were being produced, all of which needed reinforcement to make them stronger and more rigid.

The very first fiber composite, known as a glass fiber polymer matrix, was developed in the early 1940s with the help of a hand layup technique. During the process, glass fiber and was

mixed with an unsaturated resin called polyester, and the materials were later used in various structures. The main application areas of glass fiber polymer matrix were military and aircraft structures such as fuel tanks and boats. Glass fiber was the greatest invention at that time and was widely used in various structures. But the disadvantages of glass fiber composites were low stiffness and the same density as aluminum, which directly affected the long-term use and durability of the fiber.

With the issues of low stiffness and durability on glass fiber composites, researchers continued working on inventing another composite with ideal mechanical and physical characteristics, and in doing so developed the idea of carbon fiber composites. In 1961, researchers, found that a composite reinforced with carbon fibers had excellent mechanical properties such as light weight and high strength. Since then carbon fiber-reinforced composites have been widely used in several structural application areas such as aircraft, automotive, construction, sports, and others. Carbon fiber composites have a much high strength than aluminum and glass fibers, and these properties made it an excellent alternative to glass fiber composites. Other artificial composites are used in some structures, but carbon fibers have proven to be more ideal and popular [11].

During the 1940s, after World War II, the fiber-reinforced polymer industry, after considerable research, started actually producing composites. They found that glass-fiber composites had high strength-to-weight properties and were transparent to radio frequencies, so they were used in electronic and radar equipment. Researchers in the fiber-reinforced polymer industry kept researching, and eventually produced an automotive design with the car body made up of composite; testing on this was done in 1947. Also, various methods of fabrication and molding of composites, such as sheet molding compound and bulk molding compound

techniques, were developed during that time. The 1970s was the time when high-molecular-weight polyethylene was used to make fibers. The research and advancement of manufacturing techniques of composites made it much easier for the aviation industry to produce stronger materials and structures, and caused a breakthrough in biomedical devices and many other applications. Figure 5 shows a typical carbon fiber-reinforced composite sheet composed of carbon fibers and epoxy resins, which is used primarily in aircraft and automobiles.

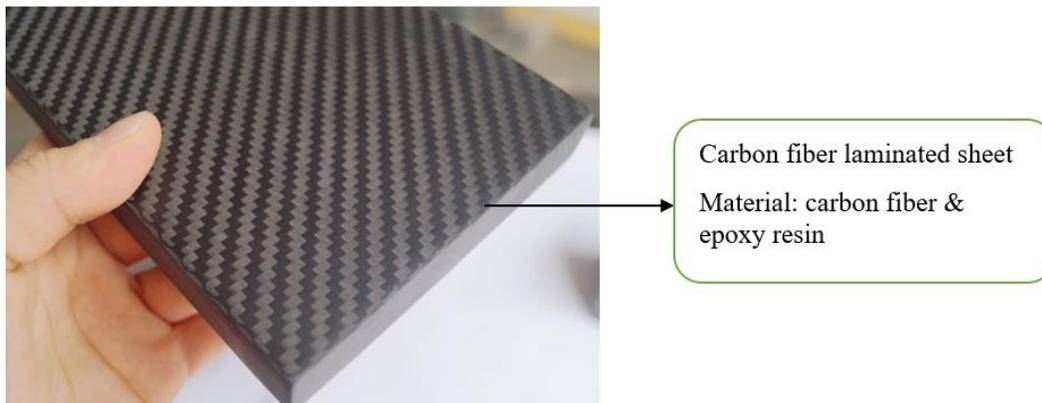


Figure 5. Carbon fiber composite representing an artificial composite.

2.4 Components of Carbon Fiber-Reinforced Composite Material

A carbon fiber-reinforced composite material basically consists of two very important constituents: a load-carrying fiber and a matrix. The matrix is generally in the form of a resin, such as epoxy resin. The most important constituent of a carbon composite is the fiber because it carries the load and is capable of providing strength and stiffness [12].

2.4.1 Constituent I: Carbon Fiber

Carbon fiber is known as the primary and most important load-carrying member of a composite material, capable of carrying a load that is almost 70–90% of the entire structure. A fiber provides structural properties such as strength, thermal and dimensional stability, and stiffness, and these properties depend on how the plies of composite laminates are stacked together. The fiber orientation is one of the important parameters that can directly affect the load-

carrying capacity of a carbon fiber. A composite material becomes stronger and stiffer with respect to the fiber orientation. For example, an anisotropic composite is unidirectional and has very strong mechanical properties [13]. In addition, carbon fibers play a vital role in providing structural damping. Carbon fibers in a composite material can have various fiber orientations and configurations such as continuous fibers, chopped fibers, unidirectional carbon fibers, roving carbon fibers, mat carbon fibers, and many more. The structural and physical stability as well as stiffness of a composite material is greatly influenced by the fiber reinforcement. In general, almost all aircraft structures, military structural equipment, sporting materials, turbines, and industrial parts, which are considered commercial composites, are made up of randomly oriented fibers. In summary, all structural properties of a commercial high-performance composite are directly affected by the fiber orientation and reinforcement, which triggers the thermal stability of the composite material.

In general, carbon fibers can be categorized into two types based on method of production: pan-based fiber and pitch-based fiber. Pitch-based carbon fibers are primarily used and popular in most structural applications, such as aircraft and automobiles. The resin-impregnated fiber, generally known as prepreg, is produced from pitch-based fiber, and is one of the easiest and cost-effective fibers because it is already impregnated with epoxy resin. The manufacturing process of a prepreg is quite easy because it comes in a roll and can be cut in desired shapes and sizes. Basket, twill, and plain weave fibers are some examples of prepreg carbon fibers that are commonly used in many structural applications [14].

The orientation and configurations of the fiber as well as the sequence of the fibers strongly affect the structural stability and stiffness of a composite material. The stiffness, strength, thermal stability, and susceptibility to damage are all directly linked with the fiber

orientation. For example, one should anticipate the formation of cracks and blisters on the composite laminate before stacking the plies in a random manner. A proper selection of fiber orientation during the manufacturing and curing of composite laminate can greatly influence the characteristics of carbon fiber composites. During the design of a composite structure, the parameters that should be considered consistently and accurately are fiber sequence, orientation, and applied load. These are all connected to each other and can affect the design efficiency. For example, an axial load applied to the composite should have a 0-degree orientation. Similarly, in the case of shear loads, the composite laminate should have a fiber orientation of 45 degrees. Basically what this means is that the stiffness and strength of a carbon fiber composite panel is the function of orientation and sequence of fibers as well as the way the plies are stacked and the degree of orientation. Therefore, the stiffness and strength of the composite acts in the same direction as the fiber orientation. Basically, a unidirectional material has its fiber oriented in one direction, and its strength acts in the same direction. For example, in a prepreg resin used to make a composite material, fibers have a unidirectional sequence and the stiffness will be in the same direction as the fiber. Figure 6 shows carbon fiber and the different types of fiber patterns [15].

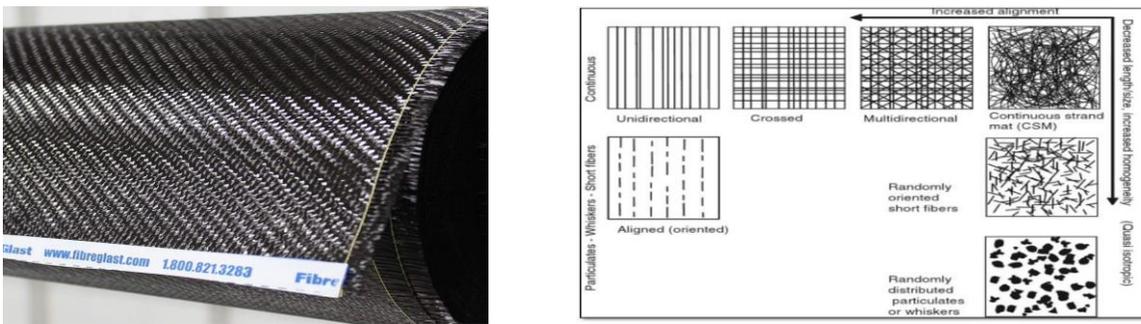


Figure 6. Carbon fiber (left) and types of fiber patterns (right) [15].

2.4.2 Constituent II: Matrix

A composite material has two constituents: fiber and matrix. Both components play a crucial role in the material's strength and durability—one carries the load and the other transfers the load and binds the fibers. The matrix is the component of a composite on which the whole reinforcement of the material depends [16]. Basically, a matrix binds the fibers together to make a composite stiffer and stronger. Also, the matrix transfers load from one fiber to another and prevents the composite from delamination and crack formation. When a crack is formed in a fiber due to environmental effects or any other external agents, the matrix isolates the fiber and transfers the load to another fiber, thus preventing the composite from failing. The matrix basically protects the structure of the composite from possible failure and can be manufactured using various types of resin. A special kind of matrix, a self-healing matrix, tends to slow down crack formation and can also stop cracks from propagating along the fibers [17].

An epoxy resin is considered to be one of the most popular matrix components because it is very easy to produce and is also cost effective. An epoxy resin has all the desired mechanical characteristics required in a composite material. Some of the desired properties include self-healing properties, impact and fatigue resistance, and compatibility with other fibers, such as glass and carbon fibers. Excellent chemical and electrical properties, thermal stability during the cure cycle, and resistance to fatigue and shrinkage are other characteristics of an epoxy resin. Today, the use of epoxy resin is very extensive, and it is generally used to make the surface of any composite material stronger [18]. It is also used to improve the adhesive properties of a composite laminate. In addition, it is less expensive, and the risk of damage to human skin by epoxy resin is less than any other curing agent. As shown in Figure 7, a matrix is an adjacent part of a composite material which transfers the load carried out by fibers.

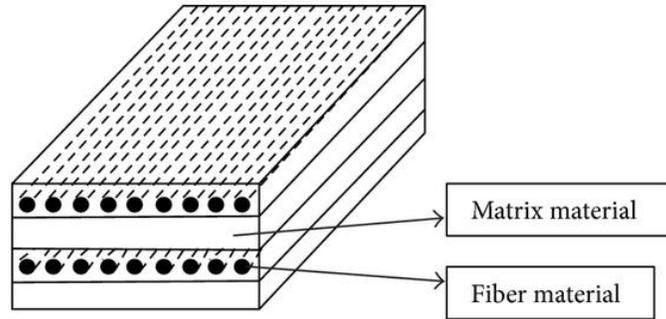


Figure 7. Matrix and fiber materials.

This experiment performed in this thesis only focused on the use of an epoxy resin system to produce a strong carbon composite material. Basically, an epoxy resin is categorized into two types: thermoplastic and thermoset. These two distinct epoxy resin systems differ in their physical and chemical characteristics, even though the composition of both resin systems consists of long polymer chains [19]. A weak van der Waals force holds those long polymer chains together. The significant difference between thermoplastic and thermoset resin systems is that thermoset polymer has a permanent solid structure, whereas thermoplastic polymer can change its state from solid to liquid when heated and then back to its original state after reheating.

2.5 Thermoplastic Polymers

In general, a thermoplastic resin has its constituents as polymers with very minimal cross linking and high molecular weight. Due to the small amount of cross linking, when the temperature is increased the polymeric chains weaken quickly, thus turning the polymer into a liquid state. The curing cycle of a thermoplastic polymer is a reversible process [20], which makes the polymer stronger and reusable, even after multiple curing cycles, without damaging the polymer chain. The desired properties of a thermoplastic polymer resin are generally high strength, resistance to shrinkage, and use in many materials from low-stress plastic bags to materials with a high-stress concentration. Thermoplastic resins are basically impact resistant as

well as chemical resistant. They can be recycled multiple times and have a tendency to remold into their original shape and structure. The manufacturing process of a thermoplastic polymer resin is more environmental friendly, and the surface finishing is more aesthetic. There are many advantages of thermoplastic polymer resins, but they also have some disadvantages, which is mainly its cost effectiveness. Thermoplastic resin is more expensive than thermoset resin, and because they are a function of heat, this basically means it can change its structure and be melted if is heated. Polyvinylchloride (PVC), polystyrene, polyethylene, Teflon, acrylic, polycarbonate, and polyamide are some of the widely used thermoplastic polymers in use today [21]. As shown in Figure 8, a thermoplastic resin is comprised of linear long-chain polymers.

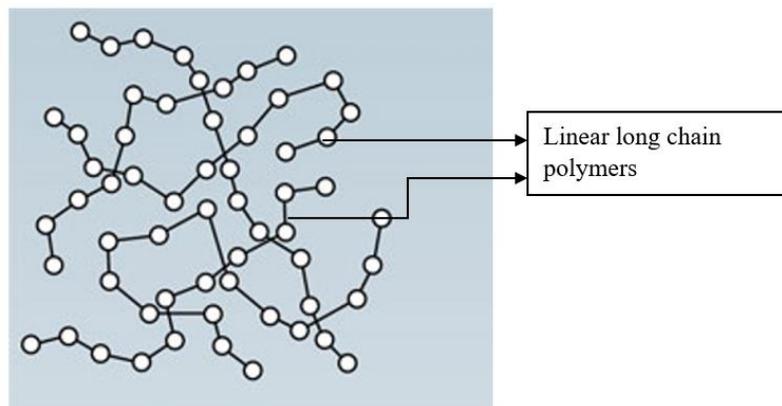


Figure 8. Thermoplastic resin [22].

The following are advantages of thermoplastic resins:

- Easy recycling
- Can be remolded and reshaped anytime
- Impact and chemical resistance greater than thermoset polymers
- Very hard crystalline surface finish that is superior to thermoset polymer
- Flexible design
- Use in low-stress applications

2.6 Thermoset Polymers

Thermoset polymers are resins that are composed of a densely cross-linked polymer structure and are considered to be very strong and can withstand extreme load and stress concentrations. Thermoset polymers are connected by chemical bonds that make it more durable and distinct from thermoplastic polymers. In contrast to thermoplastic polymers, thermoset polymers are almost impossible to remold, and heating does not change its structure because of the highly cross-linked polymeric chain. Their main advantage over thermoplastic polymers is that because of the irreversible bonding and curing process, the chance of product damage and decay is almost negligible. Due to high-heat and chemical resistance, design flexibility, and cost effectiveness, these polymers are widely used in aircraft, automobiles, and other electronic products [23].

Thermoset polymers are more often used to make products that are not likely to deform. Therefore, they are best used for solid products, and these polymers are ideal for use in electronic packaging. Having tremendous desired characteristics and mechanical properties, thermosets have some disadvantages too. One of the main drawbacks of thermoset polymer is its irreversible bonding nature, which means that recycling and remolding cannot be done with these polymers, and once the polymers are heated, it is impossible to bring them back to the solid state again. In addition, thermoset polymers have a very weak elastic nature [24].

Epoxy resins, polyester resins, and phenolic resins are some examples of thermoset polymers that are being widely used in many industries like automotive, aircrafts and other fiberglass industries. Figure 9 shows a schematic representation of the cross-link polymer chains of a typical thermosetting resin.

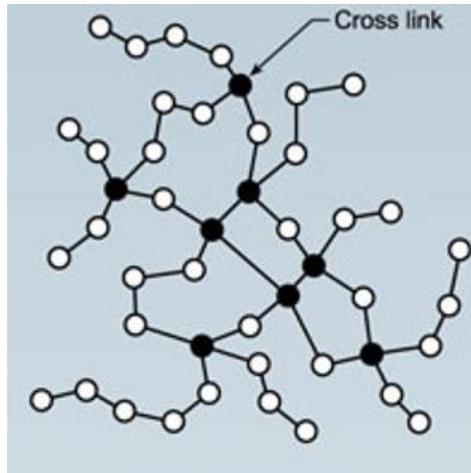


Figure 9. Thermosetting resin [25].

The following are some of the advantages of thermoset polymers:

- Environmentally friendly and cost effective
- Suitable for high-temperature conditions
- Design flexibility
- Chemical and heat resistant
- Structural stability and dimensionally very strong and difficult to remold

2.7 Applications of Carbon Composites in Aerospace

Carbon fiber-reinforced composites are the most dominant and widely used material in modern aircraft because of its light weight and excellent mechanical properties. The Boeing 787 is an example of a carbon fiber application in the aerospace industry. Here, the airframe and main structures are primarily composed of composites. Because CFRCs are extensively used in aircraft, they have a very large potential difference with the aluminum alloys; therefore, galvanic corrosion is one of the major issues in this industry [26]. The tolerance of galvanic corrosion in the aircraft industry is very minimum because it directly affects aircraft performance. As shown in Figure 10, 50% of advanced composites are being used in the Boeing 787 aircraft.

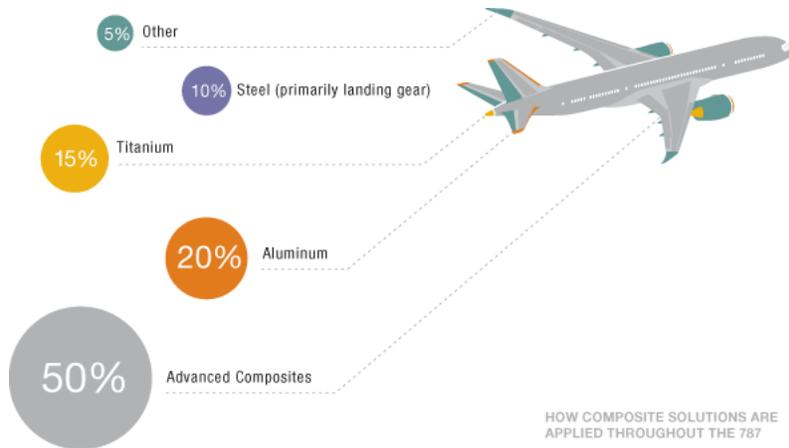


Figure 10. Composite composition of Boeing 787 [27].

The traditional distribution of materials in aircraft was totally different than how it is used today. Previously, almost 81% of aircraft structures was comprised of aluminum. Figure 11 shows the differences in weight percentages of composite uses in the traditional Boeing 747 and today's Boeing 777 [28].

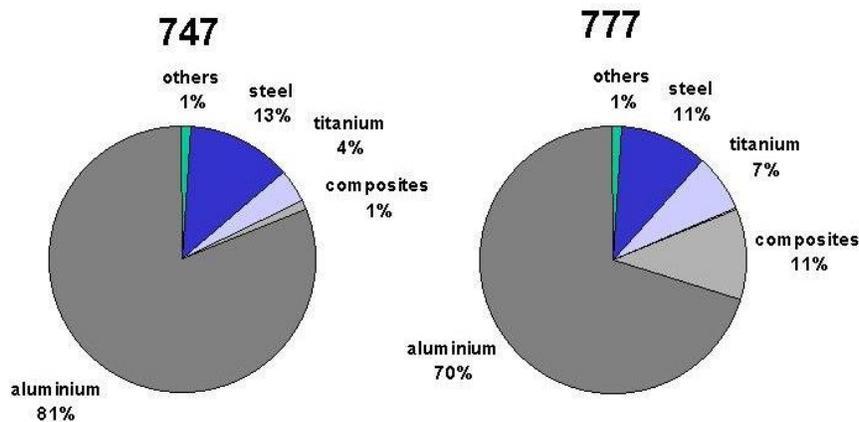


Figure 11. Percentage of structural composition of traditional Boeing 747 (left) vs current Boeing 777 (right).

2.8 Manufacturing of Composites

The manufacturing process of composite material can be different depending upon the size of the product desired and its applications. Various methods of manufacturing of composites are in use in many industries. Some industries that use composite materials in large volume on

their structures are the aircraft, automotive, and marine industries, as well as sports companies. They implement various techniques to produce a composite material, but almost all industry and manufacturing techniques require a mold to manufacture a composite material. The mold holds the composite material during manufacturing and gives the desired shape before applying any kind of heat or curing process.

In general, a composite manufacturing technique requires two main ingredients before starting the process: fibers and matrix. The fiber constituents and matrix polymers make the composite material stronger and resistant. Fibers carry the load, and the matrix holds the fibers together and transfer the load to neighboring fibers. The manufacturing of a composite material depends upon the following parameters, which should be taken into consideration before the manufacturing process begins:

- Fiber orientation
- Amount of fiber volume fractions used
- Proper curing of composite material
- Proper bagging and layup
- Proper bonding of fiber and matrix within the material
- Sequence of fibers and their coatings
- Control over possible defects and delamination
- Proper heat treatment
- Cost
- Reinforcement materials

The shape and size of the components, the structural characteristics of the product requirements, and the resin material are the main factors to be considered during composite

manufacturing. The technique used for manufacturing is categorized based on the applications, type of industry, and molding process [29]. Techniques can be grouped by the curing and consolidation processes for the polymer resins. To have a composite material in a desired shape and to have proper bonding of carbon fibers and matrix constituents, manufacturing processes can be divided into open-forming and closed-forming methods.

2.9 Open-Forming Methods

The open-molding method is widely used in composite manufacturing because it is easy to handle and is cost effective. This method is used to produce large but uniform composite structures by employing a simple hand layup technique or other spray techniques. Some of the techniques that fall under this open-forming method are as follows:

2.9.1 Hand Layup

The hand layup technique is one of the cost-effective methods of manufacturing a composite material. Here, a laminator impregnates the resin into the fibers using his/her hands. The fibers employed in this method generally come in a roll so they can be cut into various dimensions and geometry. Generally, a mold is used to give the geometrical shape of the material, and the fibers impregnated with the resins are laid on the mold. The mold is chosen according to the shape of the desired final product. The viscosity of the resin obviously should be low in order to be used by the laminator. Following the layup, the orientation of the fibers is determined, the specimen is bagged, and the composite is left to cure in an oven or autoclave, or under standard conditions [30].

In this technique, rollers or brushes are used to apply the resin throughout the fibers and to remove the voids. Consistent impregnation and quality of the product depends upon the accuracy of the user. This is a common and simple technique and can be repeated until the

desired number of layers is built up. This cost-effective method is not very accurate and is engineered for a better surface finish of the final product. It is completely dependent upon the manual layup by the user and is sometimes not very accurate; therefore, this process is mainly applicable for non-uniform products. Figure 12 shows a typical hand layup technique used for composite fabrication.

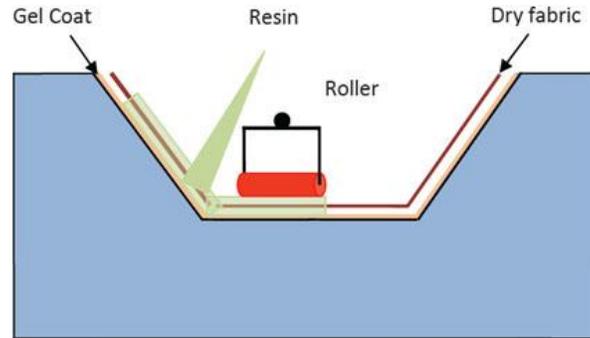


Figure 12. Hand layup technique

2.9.2 Wet Layup

The wet layup technique is another composite manufacturing technique where the fibers are allowed to dry and hold together. The mixing of resin before applying it to the fiber is very tricky and messy, and can cause non-uniform fiber orientation. Once the resin is mixed completely, a roller or brush is used to apply the resin to the dry fiber, ensuring that the resin application is uniform throughout the fibers. Then the fibers are left to saturate in a proper heat and environmental condition [31], and once saturated, the fibers are cured within a certain temperature in an autoclave or oven. This technique is similar to that of the prepreg hand layup, but the process is quite messy and needs more attention and skill while applying and mixing the resins. Also, this process is not repeatable. Figure 13 shows the method for resin impregnation by hand using the wet layup technique for composite manufacturing.

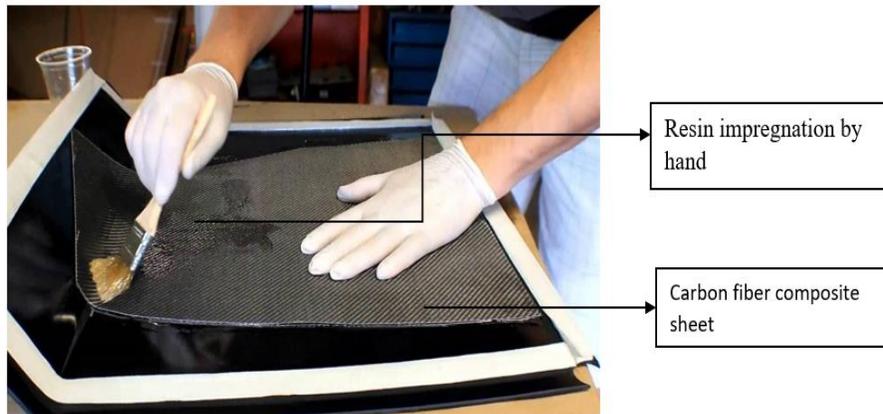


Figure 13. Wet layup technique.

2.9.3 Spray-Up

The spray-up technique is almost as ideal as the hand layup technique for manufacturing a composite material. The main difference between the two is that the hand layup technique is only applicable for a uniform composite structure, whereas the spray-up technique is useful for complex non-uniform shapes and structures. In this method, under standard temperature and atmospheric conditions, the fibers are sprayed into a mold that has been made for the desired shape and size. Once the required thickness spraying is done, curing is done at room temperature or at desired atmospheric conditions [32]. The time of curing is completely a function of type of polymer. A combination chopper or a spray gun is used in this method to directly deposit the chopped fibers and catalyzed resins into the mold. Sometimes, a roller is used to remove the air and make the reinforcement work, and sometimes woven fabric is used to strengthen the fiber-matrix bonding. This method is also inexpensive, but it has some drawbacks.

The spray-up technique can sometimes be harmful and hazardous to the health of the user because high styrene resins are used on the fiber, and the spray gun can only be fed with short fibers and low viscous resins, which ultimately reduce the mechanical characteristics of the composite materials. Also, the fiber-matrix combination loaded into the spray gun tends to be

heavy because the fibers are heavily impregnated with resin. This process is typically useful for producing fiberglass boat hulls, caravan bodies, shower trays, bathtubs, and other lower-load-carrying panels, but it can also be hazardous and difficult to control the emission of pollutants during spraying. To manage and reduce the possible hazard and harmful compounds, this method can be expensive compared to the hand layup technique. As shown in Figure 14, a high-pressure air spray gun is used to spray resin over the fibers during composite manufacturing.

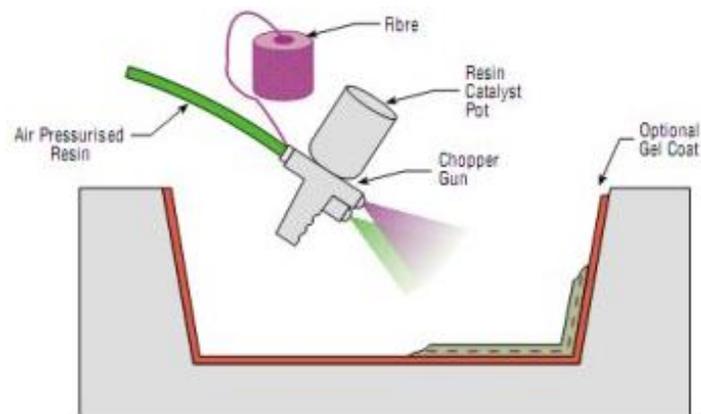


Figure 14. Spray-up technique

2.9.4 Filament Winding

Filament winding is another composite manufacturing technique that produces circular or hollow composite structures, such as cylindrical pipes, tanks, and other oval-shaped structures. This method is categorized by two winding processes: wet and dry. The manufacture of hollow-shaped composite structures can be performed by two methods. In the wet-winding process, a special kind of resin bath is made in order to have a proper impregnation of fiber into the resin, whereas in the dry-winding process, the fibers that are impregnated in a resin bath rotate in a mandrel. Therefore, the feed rate and orientation of the fibers depend on the speed of the mandrel. The filament winding technique is an efficient and very fast method of manufacturing such structural composites. The total cost of this one-directional process that does not include further processes is minimum [32].

After processing, a filament-wound composite can have very good structural characteristics. This technique can produce storage tanks, cylinders, pipelines, drive shafts, axles, struts etc. The main drawback of this process is that this method is only applicable for oval sectioned structures and convex structures. Also, the rotating compound mandrel is expensive, and the surface finish of the composite looks unaesthetic. In addition to the mandrel itself, the structure for this application includes a carriage drive, cross-feed housing, and a mandrel drive, as shown in Figure 15.

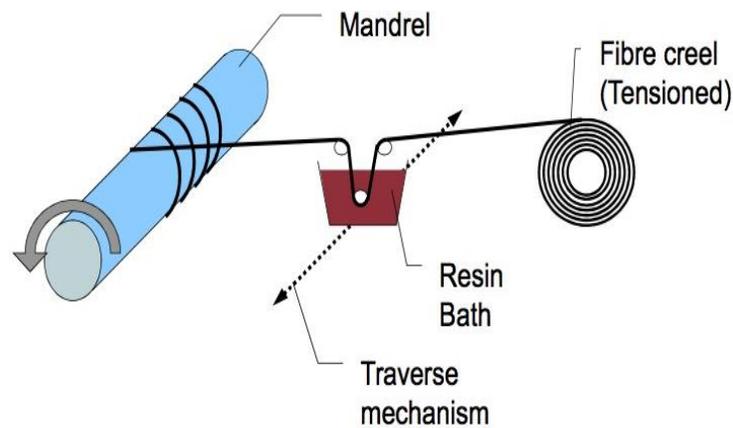


Figure 15. Filament winding technique.

2.9.5 Pultrusion

Pultrusion is another open-forming method that produces a very aesthetic and smooth composite laminate. This technique consists of a heated dye that is used to pull the material, and then fibers are impregnated through it. Then the material is clamped into a molding, and curing starts. Pultrusion and metal extrusion are similar methods, but they are based on two different principles. In pultrusion, process the fibers are pulled through a resin bath, whereas in extrusion, metal is pushed into the dye.

During this technique, fibers are pulled into the resin bath and feed into the heated dye. The resin-impregnation process begins as the fibers pass through the dye. Curing of the

composite laminate starts after the fiber is completely impregnated with the resin. A cutoff saw is used to cut the cured composite into the desired length. Pultrusion is a process of manufacturing uniform cross-sectioned structures such as beams, ladders, and frameworks. The resins used in this process are epoxy, polyesters, phenolics, etc. Pultrusion is a cost-effective, very-accurate method of manufacturing a composite, but its main disadvantage is that production is limited to only uniform cross-sectional structures. A schematic view of the pultrusion technique used to manufacture a carbon fiber composite is illustrated in Figure 16.

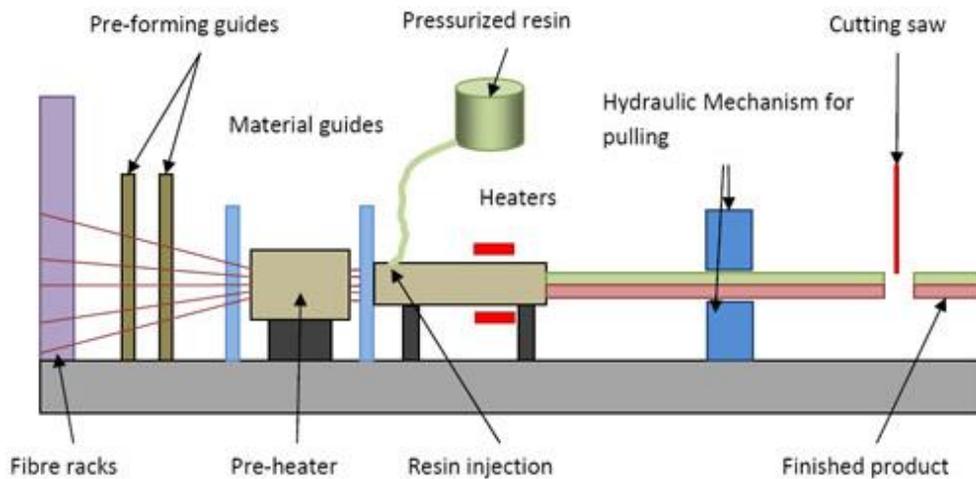


Figure 16. Pultrusion technique [34].

2.10 Closed-Forming Methods

2.10.1 Injection Molding

Injection molding is a commonly used and cost-effective manufacturing processes that uses fibers impregnated with thermoplastic resins. This technique basically consists of a hopper where the fibers are fed along with the resin enforcement. The hopper is then connected to a barrel that is heated continuously with rotating screws. During the process, the fibers and resin are mixed and put into a mold, and then the mixture becomes solid after a sufficient period of cooling. The final composite laminate product of the process is ejected from the mold cavity after curing.

This technique has a very short production cycle and is easy to operate. Automotive parts such as seats and dashboards are the main parts that can be manufactured with this process. Although this technique is applicable for large-volume composite production, the initial cost of the production and the molds involved are much higher. Figure 17 shows an injection molding setup for manufacturing complex composite structures.

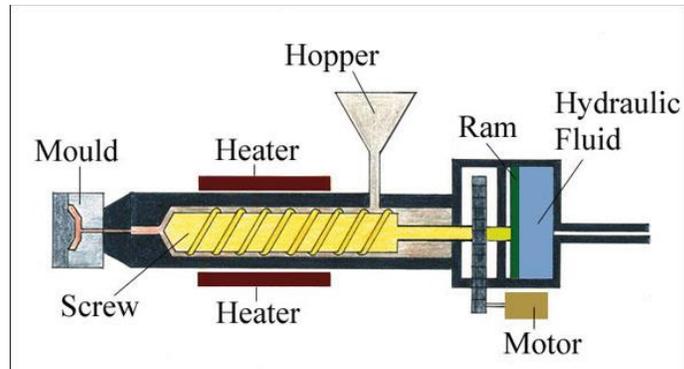


Figure 17. Injection molding setup [35].

2.10.2 Resin Transfer Molding

Resin transfer molding (RTM) is another closed-forming method of composite fabrication. Here a mold is prepared in such a way that the desired shape of the composite is exactly the shape of the mold. The main applications for this technique are aircraft and automotive parts, mainly the seats. In this technique, dry fiber sheets are laid up in a desired sequence and the mold cavity is already made into a desired shape needed for the composite. Once the dry stack of fibers is laid up in the mold cavity, a pressure is applied to the fibers and held together by a binder. After pre-stressing the fibers into the cavity and applying pressure, it is then clamped over by the top portion of the mold, and fibers are stacked and compressed better within the cavity. Now the resin is injected into the mold and given enough time to make the fiber wet with the resin. After injecting the resin into the mold cavity and allowing the fibers to saturate with resin, the curing process begins. Curing of the fabrics is generally performed by

applying enough heat at an elevated temperature condition. After the curing process, the last step is to remove the composite from the mold.

Resin transfer molding is one of the commonly used and very accurate composite manufacturing techniques because the composite laminates produced from this process have very limited voids, and the process is environmentally friendly and not harmful for users. Also, because both up and down portions of the laminates are covered by the mold, the surface finish is very smooth with minimum defects. The only disadvantage would be the high cost of the tooling and molds. An engineering drawing of RTM with all parts is shown in Figure 18.

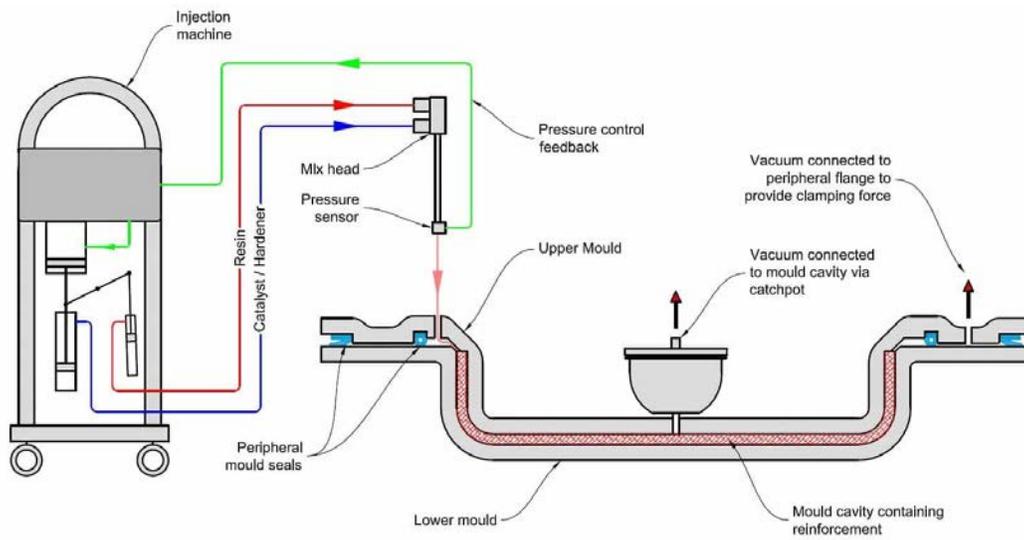


Figure 18. Resin transfer molding setup.

2.10.3 Vacuum-Assisted Resin Transfer Molding

Vacuum-assisted resin transfer molding (VARTM) is based on the same principle and fabrication procedures as RTM; however, the resin is applied into the mold using a vacuum. This process is a slight modification to the RTM process, whereby the upper portion of the mold involves vacuum bagging instead of the mold. Since this process is using only one mold surface, the overall tooling and manufacturing costs are reduced, but one surface of the composite laminate is not finished as smoothly as in the RTM technique. In VARTM, fibers are laid up in a

desired sequence in the lower portion of the mold cavity, and the top portion of the mold cavity is vacuum bagged. Once the fibers are laid over the mold and a bagging plastic is put over the top, the resin is applied through a manifold that is placed on top to make the resin flow efficiently over the entire mold. The fibers are reinforced with resin using a vacuum inserted into the mold from a port made at the top of the cavity, as illustrated in Figure 19.

This technique is cost effective, and less labor and force are required to perform the task because the upper tooling of the mold cavity is simply a vacuum bagging. With this technique, very complex and large and core composite structures can be manufactured, but the disadvantage is the complexity of the handling process. Also, low-viscous resins are required, which can limit the composite characteristics [36].

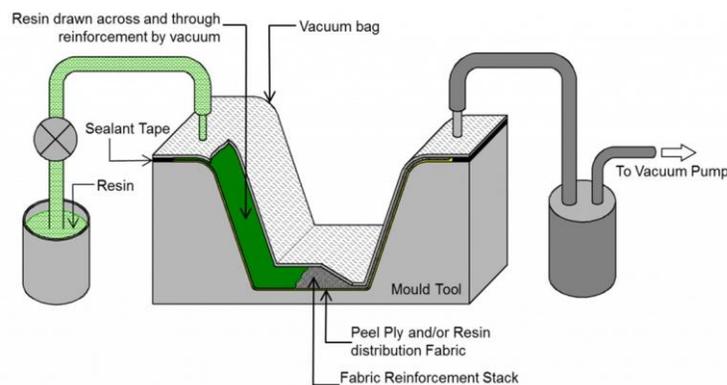


Figure 19. Vacuum-assisted resin transfer molding setup.

2.11 Introduction to Galvanic Corrosion

When different metals with different physical characteristics or metals with the same characteristics are connected to each other at a common interface, corrosion occurs. Corrosion is an important factor that must be addressed in various engineering industrial works. Corrosion may be of several different kinds:

- Localized, such as pitting, crevice, and filiform
- Galvanic

- Environmental cracking, such as corrosion fatigue, stress corrosion cracking, and hydrogen-induced cracking
- Flow-assisted, such as impingement, cavitation, and impingement
- De-alloying
- Intergranular
- High-temperature
- Fretting

Since this paper is mainly focused on the type of corrosion that occurs in a composite structure, galvanic corrosion is discussed in detail. Galvanic corrosion, also referred to as dissimilar metal corrosion, usually occurs in the presence of electrolytes between two metals, one being nobler than the other metal. These two metals act as anode and a cathode. The metal that acts as an anode is more active in the process of corrosion and deteriorates quicker than the metal that acts as a cathode. As a cathode, the metal corrodes slowly. Galvanic corrosion in a metal acts like a battery in which an electrolyte is present in the corrosive environment. Galvanic corrosion in metals occurs when one of these following conditions are present in the metals.

- An electrical contact should be present when the two dissimilar metals are connected to each other (composite and aluminum)
- The metals that are joined together should be exposed to an electrolyte.
- The two metals should be electrochemically dissimilar.
- An actively corroding metal should be present.
- Salt water, oxygen, or any other electrolyte should be present.

Aircraft and automotive industries are the two dominant engineering domains where a massive amount of aluminum, copper, and, recently, carbon fiber-reinforced composite

structures are used for making many parts and systems. The structures where two different metals need to be coupled together are most likely to be corroded faster with the presence of moisture. Therefore, galvanic corrosion occurs at the interface between two dissimilar metals, or composite structures, when a corrosive environment is present. Also, in aluminum structures, the aluminum has thermodynamic characteristics that are considerably more active than any other metals, and they corrode faster.

Galvanic corrosion is one of the main issues that must be fixed and considered while developing structures that include composite-composite and composite-aluminum interfaces. For example, in the aircraft industry, when a carbon fiber composite is attached to an aluminum panel at the airframe, the chances of galvanic corrosion occurring at the metal interface is extremely high because carbon and aluminum are distinctly opposite, and they acquire two opposite ends in the galvanic series. Because the aluminum is more electrochemically active, it corrodes much faster than carbon. This corrosion then becomes the most serious issue and decrease the overall performance of the aircraft. Therefore, galvanic corrosion is the top most major issue in aircraft structures, and there are several ways to reduce it. One important solution is to infuse nanoparticles at the interface between the carbon fiber composite and the aluminum. Also, the plies of glass fibers can be used as a barrier to reduce the galvanic current. The rate and intensity of corrosion depends upon the surface area of the anode and cathode. For example, if the aluminum has more area than that of the carbon fibers, then the galvanic corrosion is less.

2.11.1 Mechanism of Galvanic Corrosion

Basically, when an electrolyte is present in a metal that is connected to another dissimilar metal, a corrosive current is generated. An electrolyte can be of any form such as water or any conductive material. Due to the potential difference between two dissimilar metals, a galvanic

current is produced, and the ions flow from the anode, which is thermodynamically more active, to the cathode. This process is generally referred to as a metallic couple. The corrosive current produced due to the flow of negatively charged ions from anode to cathode corrodes the anodic metal faster than cathodic metal.

Generally, galvanic corrosion occurs in metals when the metals are coupled and an electrolyte is present, which makes the galvanic cell complete. The basic function of an electrolyte in a galvanic cell is to allow the ions to flow from anode to cathode. A more conductive electrolyte in a corrosive environment allows the metals to corrode in a very rapid propagation because the ions flow faster between the pathway of the more-active metals and the less-active metals. Seawater is one of the best electrolytes for galvanic corrosion because of its high electrical conductivity. With the anode being less noble than the cathode, it forms an oxide layer on its surface and corrodes faster. Basically, the galvanic potentials and rate of corrosion may vary depending upon the nature of the environment and type of electrolyte used because of their conductivity differences. Figure 20 shows the galvanic reaction and galvanic corrosion on a fastener during the process of corrosion on a composite structure.

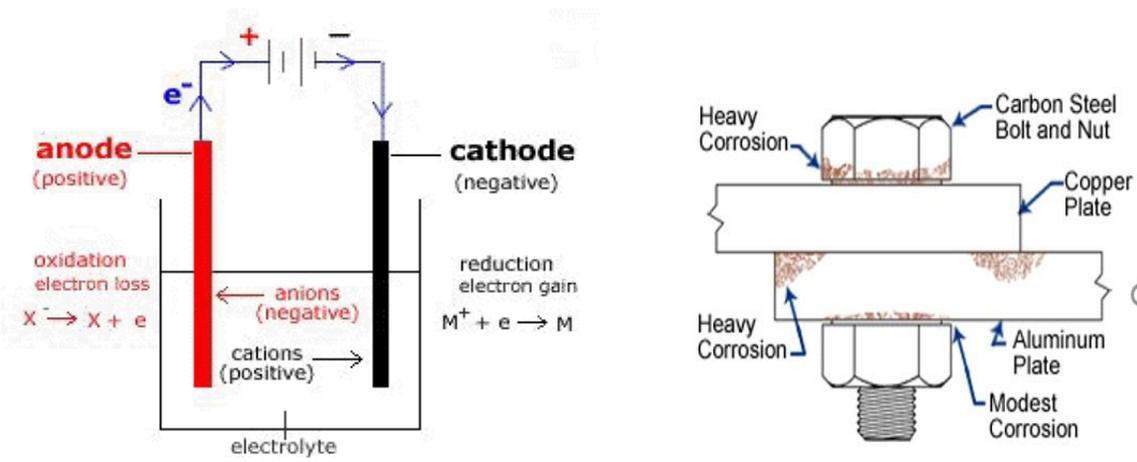


Figure 20. Galvanic reaction (left) and galvanic corrosion on fastener (right).

2.11.2 Factors Affecting Galvanic Corrosion

The severity and corrosion rate of galvanic corrosion depend upon various factors:

- Electrode efficiency
- Electrolyte
- Variable potential
- Ratio of anodic and cathodic areas
- Flow rate
- Electrode potential

2.11.3 Thermodynamics of Galvanic Corrosion

In galvanic corrosion, oxidation and reduction reactions occur one after the other. Due to these simultaneous reactions, the galvanic corrosion undergoes a redox reaction. With an anode and cathode on each end of the reaction, oxidation occurs at the anode and reduction occurs at the cathode, whereby the entire mechanism of galvanic corrosion acts like a battery. For galvanic corrosion to occur at the interface of a composite, the materials should be in a metallic connection, and there should be a bridge of electrolytes. Basically, when two metals are attached to each other at a common interface and moisture is present and has a different potential, galvanic corrosion occurs. The rate of galvanic corrosion in a galvanic cell depends upon the electrode efficiency. The total galvanic corrosion rate can be determined by the carbon composite area that is exposed to nature and is under cathodic and diffusion control. In Figure 21, a composite and steel fastener joint has experienced a severe crack at the interface due to galvanic corrosion.

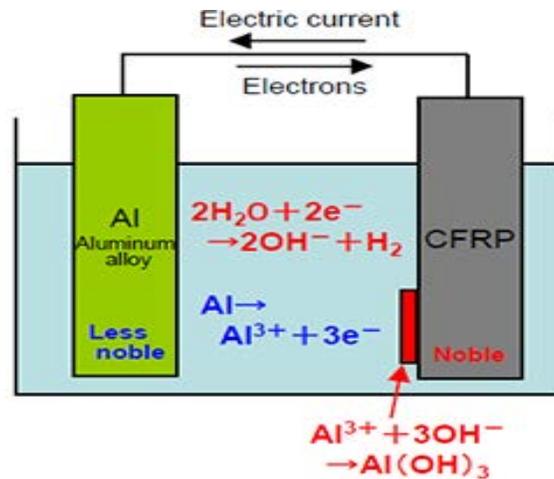


Figure 21. Galvanic reaction on aluminum and carbon fiber-reinforced composite

2.12 Galvanic Corrosion in Carbon Fiber Composites

Galvanic corrosion is generally a process in which two metals are connected electrically in the presence of an electrolyte sharing a common interface. Those metals have different electrical potentials. This is an electrochemical process and is one of the major issues in every industry that uses carbon composites in relation to another metal. For example, when a carbon fiber composite is attached with aluminum in an aircraft panel and airframe, galvanic corrosion could occur. Carbon and aluminum alloy having significant differences in their electrical potentials, where the aluminum alloy corrodes at a higher speed. This is one of the serious issues that most of industries are facing. In a carbon fiber composite, galvanic corrosion is a serious problem because even though carbon fiber is excellent in electrical resistance, it acts as a cathode in a galvanic cell. Also, carbon fiber is nobler than aluminum or any other alloy. So when a carbon fiber composite is attached to aluminum or any other alloy, and moisture is present, a galvanic cell is created at the interface between the carbon fiber and any other metals. This galvanic has two ends, carbon fiber acting as a cathode and aluminum acting as an anode, which results in a higher rate of corrosion on the aluminum.

The major problem arises when a composite panel needs to be attached with another metal at an interface such as on a ground path in an aircraft. The composite structure must be coupled with aluminum by making a hole at its surface in order to use a temporary fastener to attach another metal. Therefore, when a composite is drilled, the outermost fibers are exposed to the corrosive environment and can easily absorb the moisture. This results in galvanic corrosion at the interface between the carbon fiber and the aluminum structure and fastener. Also, the aluminum corrodes at a much higher speed. The outermost surface that is exposed to the environment absorbs moisture, thereby creating a galvanic cell at the composite surface. This is a major issue for the temporary fastening of a composite with another metal in aircraft as well as in automotive structures.

The rate of galvanic corrosion highly depends upon the cathode surface area ratio. When a very large carbon fiber-reinforced composite structure needs to be attached with a small bolt or a fastener, galvanic corrosion becomes a serious issue. Because the ratio of carbon fiber composite (cathode) surface area to fastener (anode) area becomes very high, this results in massive galvanic corrosion on its surface. Since carbon fiber composites are being widely used in various areas such as aircraft, automobiles, turbines, and other energy devices, fastening the composite to metal with a bolt is a common phenomenon, but due to the temporary fastening of carbon fiber and other metal, the isolation of metals is extremely difficult, and the chances of galvanic corrosion on the fastener are extremely high. Due to this corrosion phenomenon on the fastener, the overall performance of the aircraft and automobile are decreased.

2.13 Major Effects of Galvanic Corrosion on Carbon Fiber-Reinforced Composites

Generally galvanic corrosion occurs at a common interface between a carbon fiber composite and aluminum or any other attached metal. When galvanic corrosion occurs, the

metals being an anode corrode faster and at a higher speed than the carbon fiber composite. But the composite itself gets corroded to a certain limit and the structural integrity of the carbon fiber composite is greatly affected. Since the carbon fiber composite has an open area on the hole and the fibers are exposed in that area, the tendency to absorb moisture is greater, resulting in corrosion. Once corrosion occurs at the surface of the composite, evolution of hydrogen gas occurs that may result in the formation of cracks and voids on the surface of the composite. Galvanic corrosion on a carbon-metal interface is shown in Figure 22.

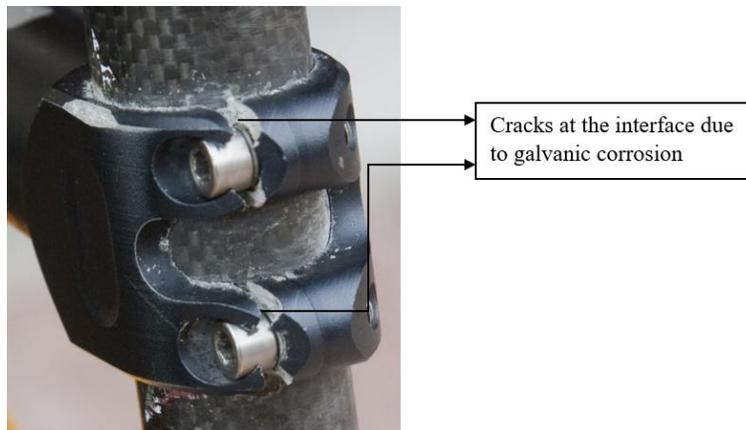


Figure 22. Galvanic corrosion on carbon-metal interface.

Blisters are generally formed when the voids on the surface of the composite are elongated, and the crack speed is increased. The crack is formed at one point, and with the increase in load, it initiates and propagates throughout the surface. The galvanic coupling of carbon fiber composite to aluminum or any other metals with the help of a fastener results in metallic corrosion as well as degrading the carbon fiber composite itself.

Also, when aluminum or magnesium is connected to the surface of a carbon fiber composite with a removable fastener, an excessive amount of calcareous deposits occurs with a maximum hydrogen gas emission and cathodic reactions. The very first step in blister formation due to galvanic corrosion is the diffusion of oxygen and water into the matrix. Ions are also

diffused, which causes the polymers to be the solid electrolyte. After the diffusion of ions, debonding of the polymers takes place, resulting in the formation of a Helmholtz water layer on the surface of the composite. After that, the reduction of oxygen takes place and the osmotic pressure builds up. Then, the phenomenon of creep occurs on the polymer, which results in the formation of blisters. Figure 23 shows voids and micro blisters on a composite surface observed through scanning electron microscopy.

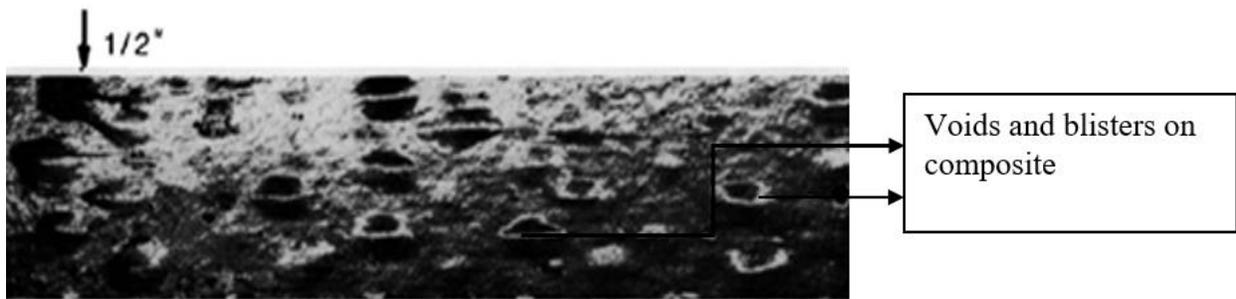


Figure 23. Micro blisters in carbon fiber-reinforced composites.

2.14 Mitigation of Galvanic Corrosion on Composite-Metal Surface

The materials used in engineering applications are made up of different materials that have different physical and chemical properties. For example, carbon fiber-reinforced composites are used extensively in aerospace, automotive, and other areas, and depending upon the applications here, composites need to be attached with a metal. A removable or permanent fastener is used to couple composites and metals. To reduce the weight and increase the strength at the same time, the considerable mass of an automotive structure is typically comprised of different alloys, components, metals, and composites. Composites being lighter and strong, they are used in a larger part of the surface area, and due to the large variety of metals and components used in the structure, the chances of corrosion are higher. Metals or alloys joined with carbon composites are prone to galvanic corrosion. Hence, different techniques are used to

reduce these corrosion problems. Some of the mitigation techniques used in carbon fiber-metal galvanic corrosion are as follows:

2.14.1 Insulating E-Glass-Reinforced Polymer Matrix Composites

Insulating e-glass-reinforced polymer matrix composites is one of the techniques of mitigating the possible galvanic corrosion on the surface of metal-carbon composite surface during fastening. When an aluminum and CFRC are electrically in contact with each other, the chances of galvanic corrosion are greater because aluminum and composites are coupled with a fastener galvanically. But inserting G-10 fiberglass between these two-metal interfaces helps to separate the composite and the aluminum, thereby reducing the direct contact. Figure 24 shows the galvanic corrosion on a metal surface through a salt bridge and the addition of fiberglass on the matrix.

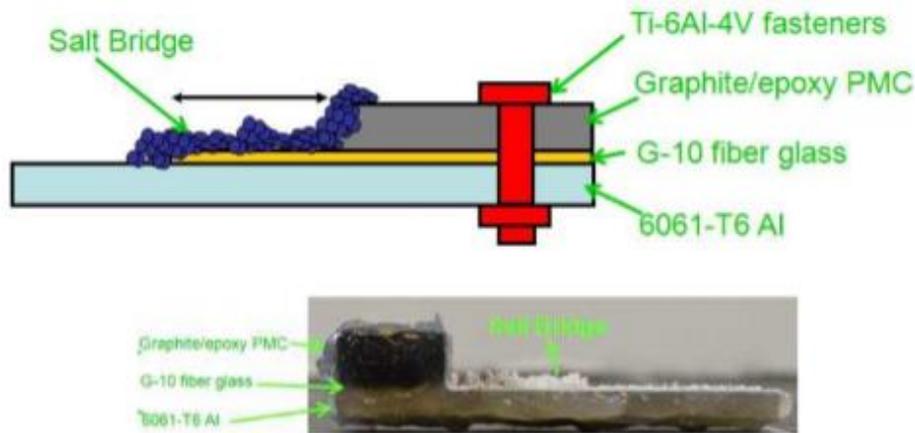


Figure 24. Galvanic corrosion in dissimilar materials through salt bridge

2.14.2 Zinc-Vanadium Oxide Coatings on Composite Surface

Another solution for mitigating galvanic corrosion to some extent would be a coating on the surface of the composite and the metal while joining the two at a common interface. Deposited zinc-vanadium oxide (Zn-VO) acts as a barrier between two metals and composites,

and prevents the interface from corroding. With the electrodeposition of Zn-VO, the corrosion resistance of the composite increases at a higher rate, thus decreasing the current value.

2.14.3 Plasma Electrolytic Oxidation

Plasma electrolytic oxidation is another technique to reduce the corrosion mainly on aluminum attached with steel. Generally, when an aluminum alloy or steel is attached to the surface of carbon fiber composites with a fastener, either permanent or removable, the surface of the attached metal area corrodes very quickly. With plasma electrolytic oxidation, the reduction occurs at a very high speed because an oxide coating is synthesized onto the surface of the composite.

2.14.4 Use of Nanoparticles for Hole Sealing Composites to Metals

The use of nanoparticle coatings on the surface of composites while CFRCs with any metal has been a very useful method to reduce possible galvanic corrosion. Nanoparticles enhance the overall performance of the metals and composites at an interface between metals and composites. Most metals that are used in engineering structures are treated with organic coatings, which are not particularly corrosion resistant because the chances of electrolyte formation in the presence of moisture is maximum with such coatings. However, nanoparticle coatings have excellent barrier characteristics, which minimize the corrosion by isolating the metals and composites, and restricting the formation of electrolytes on the surface. Also, an epoxy resin can absorb water very easily, thereby acting as an electrolyte in the corrosion process. But when a certain weight percentage of nanoparticles is added to the baseline coating, galvanic corrosion would greatly be affected, and the overall performance of the system would increase at a tremendous speed. Figure 25 illustrates nanoclay and nanotalc nanoparticles being used in this research.



Figure 25. Nanoclay (left) and nanotalc nanoparticles.

The use of nanoparticles on an epoxy resin coating not only increases the corrosion resistance but also enhances the coatings' mechanical behavior, increases the thermal stability, and then boosts the coatings' isolation properties. This research is based on the application of nanoparticles, especially nanoclay and nanotalc, in different weight percentages and mixed with regular epoxy resin and coated on a surface.

CHAPTER 3

EXPERIMENTAL SETUP

3.1 Materials, Methodology, and Equipment

This research is based on using a nanoparticle coating to reduce the galvanic corrosion between metals and composites. Test specimens were prepared in a laboratory at Wichita State University (WSU), and all the instruments were calibrated before the experiments were implemented. This chapter explains the materials and equipment used in the research. The preparation of the materials and test specimens are also explained here in detail.

3.1.1 Carbon Composite Laminate/Carbon Prepreg

A typical prepreg PMC resin was used in this research. A prepreg is generally referred to a composite fiber that is already been impregnated with resin and therefore easy to use. This prepreg carbon fiber is designed in such a way that it gives an aesthetic surface finish as well as clarity when it comes out of the autoclave after the curing process. The fiber orientation of this prepreg fiber provides maximum strength and has a desirable load-carrying capacity. Figure 26 shows a typical prepreg carbon fiber composite used in this research. Table 2 shows the physical characteristics of this carbon fiber-reinforced prepreg chosen in accordance with the load and displacement of the cyclic tensile machine.



Figure 26. Prepreg carbon fiber composite.

TABLE 2
PHYSICAL CHARACTERISTICS OF CFRP PREPREG

Weight	416 g/m ²
Carbon Type	2×2 twill, 6k
Epoxy Resin	38% by weight
Cure Temperature	> 85°C
Storage Life	-18°C
Roll Width	1250 mm

A carbon composite is generally the combination of fiber and matrix constituents. Fiber is the load-carrying member, while the matrix binds the fiber together and provides strength. The resin impregnation on the fibers is done by various methods, the most reliable of which is vacuum bagging, whereby a mold is prepared in a desired shape, the resins are vacuumed into the mold, and finally they are cured at the required temperature cycle. After the curing cycle, The composite has an excellent surface finish as well as a great load-withstanding physical characteristic. As shown in Table 3, the unique range of the physical properties of the particulate composite makes it suitable for extensive used in aircraft and other industries.

TABLE 3
RANGE OF PHYSICAL PROPERTIES OF PARTICULATE CARBON COMPOSITE

Electrical Resistivity	10 ⁻⁶ to 10 ⁻⁴ Ωm
Flexural Modulus	Up to 14 GPa
Thermal Conductivity	0.05–0.4 Wm ⁻¹ k ⁻¹
Flexural Strength	10–120 MPa

3.1.2 Steel Bolts

Both threaded and unthreaded bolts are used to join composite specimens. When two composite structures in an aircraft, automobile, or any other application are joined temporarily with a steel fastener or bolts, corrosion occurs at the interface where the hole is created and the fastener is used. Threaded and shafted bolts are used to calculate the corrosion resistance when they are used in both aircraft and manufacturing parts. In this thesis work, the composite test specimens had a diameter of 0.5 in. Figure 27 shows the shafted and threaded bolts used to connect the composite coupons in this experiment, and bolt specifications are listed in Table 4.

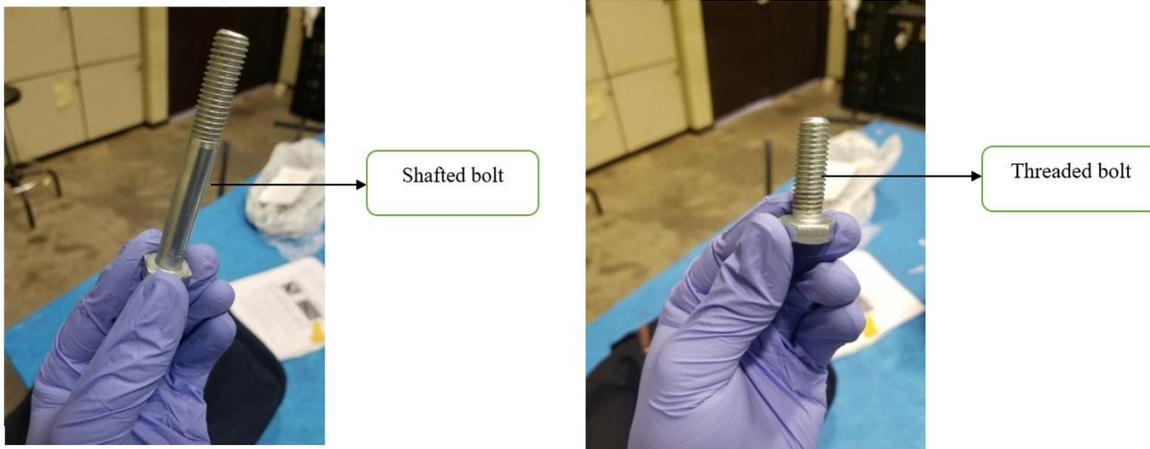


Figure 27. Shafted bolt (left) and threaded bolt (right).

TABLE 4

SPECIFICATIONS OF BOLT USED ON SPECIMENS

Specimen Hole Diameter	0.5"
Bolt Diameter	0.3937"
Specimen-to-Bolt Clearance	0.1437"

3.1.3 Drill Bit

A high-speed unique type of drill bit was purchased from Amazon. Carbon fiber composite has a very smooth surface finish with excellent load-bearing capacity; therefore, the drill bit was chosen so that the fibers on the outer surface would not delaminate while drilling. The carbide drill bit used to make holes in the composite coupons in this research is shown in Figure 28. Its characteristics and specifications are listed in Table 5.



Figure 28. Carbide drill bit.

TABLE 5

SPECIFICATIONS OF CARBIDE DRILL BIT

Length	4"
Cutting Angle	118°
Shank Type	Round
Point Type	Conventional
Diameter	0.5"
Total Flute Length	3"
Finish Type	Uncoated
Material	Carbide

3.1.4 Epoxy

LOCTITE EA 9394 is a common two-part structural adhesive epoxy paste of resin and hardener, which is used extensively in aerospace industries. It is also referred to as Hysol EA

9394 and has a high strength capacity of 350°F and even higher. This epoxy has two parts: hardener (part A) and resin (part B), as shown in Figure 29. This epoxy is mainly used for filling and other adhesion applications such as in potting, joining, hole sealing, and liquid shim areas.



Figure 29. Epoxy resin part A (left) and hardener part B (right)

The main physical characteristics of LOCTITE EA 9394 are as follows.

- Excellent filling characteristics
- High mechanical properties
- Low toxicity
- Easy to cure at room temperature
- Can be stored at room temperature
- Excellent adhesion capacity

As shown in Table 6, LOCTITE EA 9394 possesses various properties before and after mixing the hardener and resin.

TABLE 6

PROPERTIES OF LOCTITE EA 9394 BEFORE AND AFTER MIXING

Properties	Part A	Part B	Mixed
Color	Gray	Black	Gray
Viscosity (Pa.S)	400–800	20–70	160
Density(g/ml)	1.50	1.00	1.36
Shelf Life	1 year	1 year	1 year

3.1.5 Release Agent Spray

A mold release spray was used during the experiment to ensure the proper release of the bolt from the hole in the composite. When a bolt is inserted in the specimen hole and sealant is applied, there would a chance of the bolt sticking to the sealant at the interface. Therefore, with release spray, the bolt can be easily removed from the specimen hole. A typical mold release spray used in the experiment is shown in Figure 30.



Figure 30. Mold release spray

3.1.6 Magnetic Stirrer

A Microyn digital laboratory magnetic stirrer hotplate was used to mix the resin and hardener thoroughly before applying it to the specimen. The stirrer setup, provided by a WSU laboratory, contains a sensor and a supporting clamp, as shown in Figure 31. The magnetic stirrer was also used to mix nanoparticles with the epoxy LOCTITE 9394 during the experiments.



Figure 31. Microyn magnetic stirrer

3.1.7 Laboratory Oven

An oven located in the WSU Nanotechnology Laboratory was used in this research to cure the coating after applying nanoparticle sealant inside the hole of the carbon fiber-reinforced composite test specimen. This laboratory oven has a maximum temperature set point of 200°C and a temperature scale that can monitor digitally. The built-in timer can be set with the requirements of the actual and required temperature simultaneously by the user. The curing of the test specimen after the coating is described later on in Section 3.2, Experimental Method. Figure 32 shows the oven used for curing the nanoparticle coating.



Figure 32. Oven used for curing nanoparticle coatings.

3.1.8 Nanoparticles

Nanoparticles are generally referred to as ultra-fine particles having a geometric length of 1–100 nm and excellent physical and chemical properties. The nanoparticles used in this research had one main purpose—to reduce the galvanic corrosion produced on the interface of the carbon fiber composite and the steel bolt. Two types of nanoparticles were used in this research, and tests were performed separately to compare the behavior and effects of the nanoparticles.

3.1.8.1 Nanoclay

One of the nanoparticles, nanoclay, has a thickness of 1 nanometer and width of 70–150 nanometers and is extensively used in nanoparticle research. Nanoclays are layers of silicates that have an excellent barrier property and enhance the mechanical and chemical characteristics of experimental samples.

Nanoclays have a unique crystal structure in which the silicon atoms are stacked in a layer on a tetrahedron coordination, and the layers of silicon are bonded by van der Waals forces and referred to as a gallery. Nanoclay was used in this research to enhance the sealing of the carbon composite hole in order to have better corrosion resistance at the interface. Some of the

main advantages of nanoclay are its excellent performance, corrosion resistance, easy processibility, and cost effectiveness. Figure 33 shows a typical nanoclay used in this research to increase the resistance of the test specimen to galvanic corrosion.



Figure 33. Nanoclay.

3.1.8.2 Nanotalc

Nanotalc is a special type of nanoparticles with a high impact strength, heat and corrosion resistance, high tensile strength, and the ability to increase the thermal and chemical properties of metals or fiber composites. Nanotalc has excellent filler properties and greatly affects the overall performance of the composite structure after its application. The nanotalc powder used in this research was provided by WSU and is illustrated in Figure 34.



Figure 34. Nanotalc.

3.1.9 Multimeter

After the application of nanoparticle-infused sealant on the joint between the carbon fiber composite and the steel bolts, a resistance is produced and was measured by a digital multimeter provided by WSU. The resistance built up at the interfaces was then plotted against the load, which is described later in detail in Section 3.2, Experimental Method. Figure 35 shows a digital multimeter setup with probes, which was connected to the composite coupons to measure resistance.



Figure 35. Multimeter.

3.1.10 Tensile Machine

After nanoparticle coatings were applied to the test specimens of carbon fiber-reinforced composites, they were tested on an MTS Criterion® 810 tensile testing machine at the National Institute for Aviation Research (NIAR) Composites Laboratory at WSU. This system can be used for testing a variety of composite materials under various static-hydraulic, electromechanical, and cyclic load conditions. This easy-to-use machine is provided with TestSuite™ TW software. Resistance was measured using the multimeter, and load was measured with the tensile machine. The reaction graph between load and resistance is explained later on in Results and Discussion, Chapter 4. The tensile machine setup for conducting the experiment is shown in Figure 36.



Figure 36. MTS Criterion® 810 tensile testing machine.

3.2 Experimental Method

3.2.1 Preparation of Carbon Fiber-Reinforced Composite from Prepreg Epoxy Laminate

The composite panels used in this research for the application of nanoparticles were made from prepreg plies of fiber matrix laminates that were already pre-impregnated with the resin.

Thirty-two plies of these prepregs panels were stacked in -45 and 45 degrees fiber sequences. Figure 37 shows the prepreg laminate stacking process before the debulking of the composite.



Figure 37. Carbon prepreg setup on aluminum plate.

Steps used in the preparation of the composites are as follows:

- The 32 plies of carbon fiber prepregs with dimensions of 15×15 inches were cut and stacked in a fiber orientation of -45 and 45 degrees. A hand roller was used to apply pressure during the stacking of the plies by placing it on a smooth aluminum square panel.
- A tacky tape was used to seal all corners of the aluminum sheet in order to completely block out the air.
- Once the plies of prepregs were stacked, a berating film was used on the top, and a cold sheet was used to apply pressure from the top and ensure no air leakage.
- After sealing, the sample was put through a debulking process, using a vacuum pressure of 27 Hg for 16 hours in order to completely suction out the air.
- Following the debulking process, curing was carried out on the debulked carbon fiber prepregs.

The experimental setup for debulking the composite laminate with the high-pressure suction system and the laboratory oven used to cure the debulked composite are shown in Figure 38.

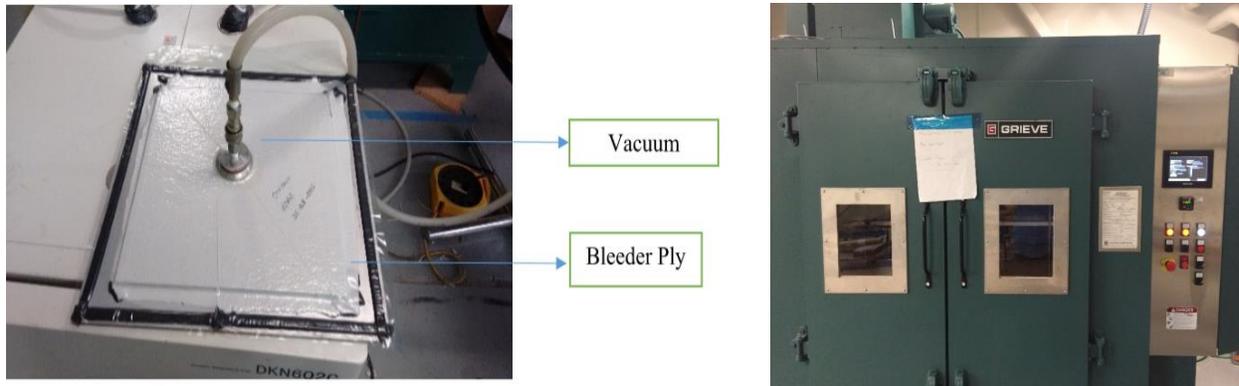


Figure 38. Setup for debulking carbon composite (left) and laboratory oven for composite curing (right).

Curing the debulked composite was carried out as follows:

- The composite was heated at 50°C for 30 minutes.
- After heating for 30 minutes, the carbon fiber composite was heated again for 1 hour at 120°C.
- Then, heating was done at 180°C for 2 hours.
- Last, the composite was heated at 50°C for 30 minutes.

As shown in Figure 39, the curing cycle for the composite consisted of three main processes: oxidation, carbonization, and purging organ.

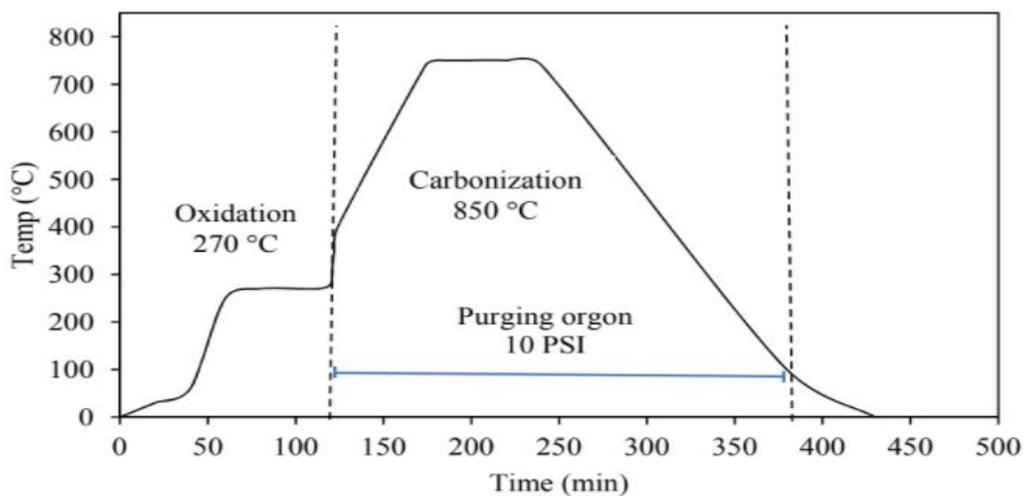


Figure 39. Curing cycle of composite.

Once the composite was entirely fabricated, the test specimens were prepared as follows:

- After the curing process, composite panels were measured into smaller units and cut into the desired length and width.
- Samples where the coating was applied had dimensions of 6 inches by 1.5 inches.
- The 1.5-inch width of the specimen was chosen in such a way that it would fit in the jaw of the tensile machine
- After cutting the specimens to the required dimensions, samples were drilled with a CNC drill machine at a very high speed.
- The diameter of the specimen holes was 0.5 inch, and the steel bolt had a diameter of 0.3937 inch, thus leaving a gap of 0.1437 inch between the two.
- The gap was then coated with the nanoclay and nanotalc with a uniform thickness of 250 microns.

3.2.2 Nanoparticle Coating Procedures

The experiments were performed with different weight percentages of the nanoparticles. The LOCTITE EA 9394 epoxy was the baseline test configuration. The overall performance of different percentages of nanoparticles was compared with the test specimen that had 0 wt% of nanoparticles infused. Basically 2 wt%, 4 wt%, and 8 wt% of nanoclay and nanotalc were mixed separately with the LOCTITE EA 9394 epoxy, and the individual tests were performed.

3.2.3 Mixing of Resin and Hardener

The proper mixing of part A resin and Part B hardener was done thoroughly just prior to the application on the composite samples. The mixing of LOCTITE EA epoxy part A resin and part B hardener was done very carefully because of possible heat build-up after the mixing. Mixing smaller quantities of epoxy is always better because the chance of heat formation is less.

The temperature prior to mixing should not be critical. An average room temperature of 77°F is preferable. The volumes and proper process of mixing the resin and hardener is illustrated in Figure 40.

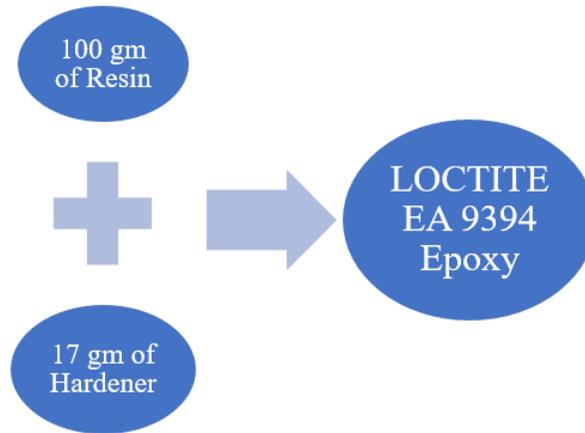


Figure 40. Mixing proportions of parts A and B epoxy.

3.2.4 Application Procedure of Nanoparticle Sealants

The procedure used for apply the nanoparticle sealant is as follows:

- Once the LOCTITE EA 9394 epoxy was ready after proper mixing of the resin and hardener, different weight percentages of nanoclay and nanotalc were added to it and stirred thoroughly.
- Nanoclay in 2 wt%, 4 wt% and 8 wt% was mixed separately with LOCTITE EA 9394.
- Similarly, 2 wt%, 4 wt% and 8 wt% of the nanotalc was mixed separately with LOCTITE EA 9394.
- Test specimens had a hole diameter of 0.5 inch, so nanoparticles were infused on the entire inner surface at a uniform thickness of 250 microns.

As shown in Figure 41, the proportion of nanoparticles coatings on the interface of the composite-bolt joints was chosen in such a way that it would not affect the homogeneity of the coating and the structural integrity of the composite surface.

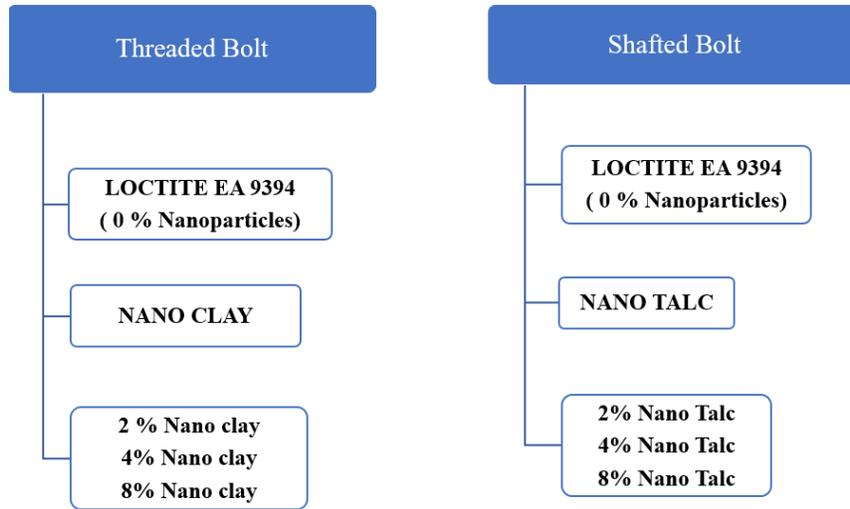


Figure 41. Schematic representation of proportions of nanoparticle mixture.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Fourier Transform Infrared Spectroscopy Analysis

Fourier transform infrared spectroscopy (FTIR) is a spectrum absorption mechanism used for liquids, solids, and gases. It records information based on either the percentage of absorbance or the percentage of reflectance with respect to wavenumbers. This technique is useful for collecting information over a wide range of spectra and at very small wavelengths.

FTIR is an analytical method that basically measures the absorption or reflection of infrared radiation and identifies the materials properties when a sample is placed on a machine. This technique also examines the molecular structure of a sample and its components. The FTIR setup consists of an interferometer that modulates the range of wavelength and a detector that records the intensity of light when transmitted or absorbed as a form of wavelength. FTIR was used to measure nanoparticles and resins—nanoclay, nanotalc, epoxy and 8% nanoclay and resin, and epoxy and 8% nanotalc and resin—in this experiment. Results obtained from the FTIR analysis of nanoparticles are expressed in terms of plots with absorbance on the Y-axis and wavenumbers on the X-axis. Figure 42 shows the FTIR spectrometer located in a WSU chemistry laboratory used for infrared spectroscopy analysis of the nanoparticles and resins.



Figure 42. FTIR setup in WSU chemistry laboratory.

4.1.1 FTIR Absorption Spectra of Nanoclay Particles

FTIR analysis of nanoclay particles used as a sealant in specimens in order to reduce galvanic corrosion are plotted in Figure 43 as absorbance on the Y-axis and wavenumber on the X-axis. It can be seen that the peak intensities of the wavelengths are at 1469.4 and 2925.24 cm^{-1} . These peak intensities are the function of vibration of modes of the C-H groups.

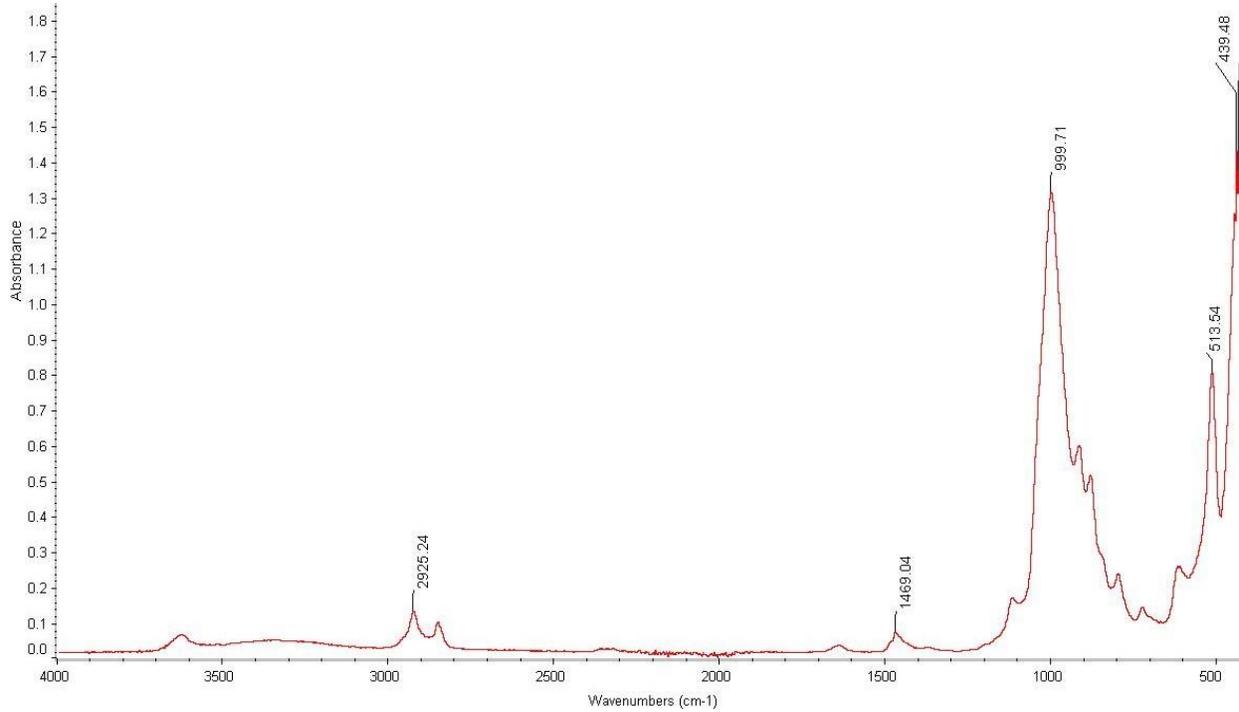


Figure 43. FTIR absorption spectra of nanoclay particles.

4.1.2 FTIR Absorption Spectra of Nanotalc Particles

The spectra of the wavelengths of nanotalc particles are almost like those of nanoclay. The vibration characteristics of both nanoparticles are of the C-H group. Peak intensities at 2925.49 and 988.91 cm^{-1} are assigned to the C-H group vibration of different modes in C-C and C-F. Basically the functional groups in nanotalc particles are better than those in nanoclay particles. Figure 44 shows the absorption spectra of nanotalc particles obtained using FTIR.

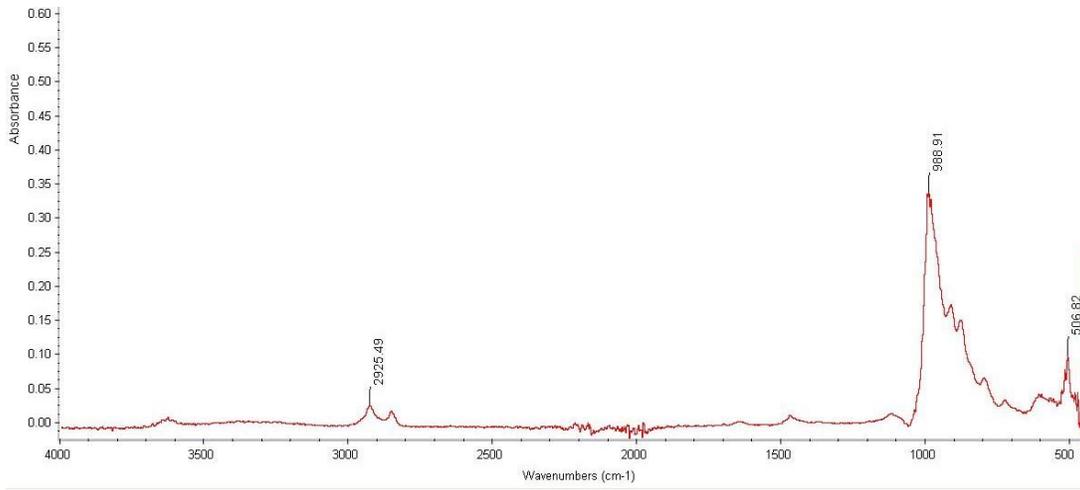


Figure 44. FTIR absorption spectra of nanotalc particles.

4.1.3 FTIR Absorption Spectra of LOCTITE EA 9394 Epoxy Resin

The FTIR spectra of the hardener and resin constituents without adding any nanoparticles indicate major wavelength intensities at 1467.85, 2851.69, and 2924.00 cm⁻¹, as shown in Figure 45. The variation of spectra is due to the nature of the epoxy resin, which is mainly used for sealing and surface adhesion on carbon fiber composites and other aircraft structures.

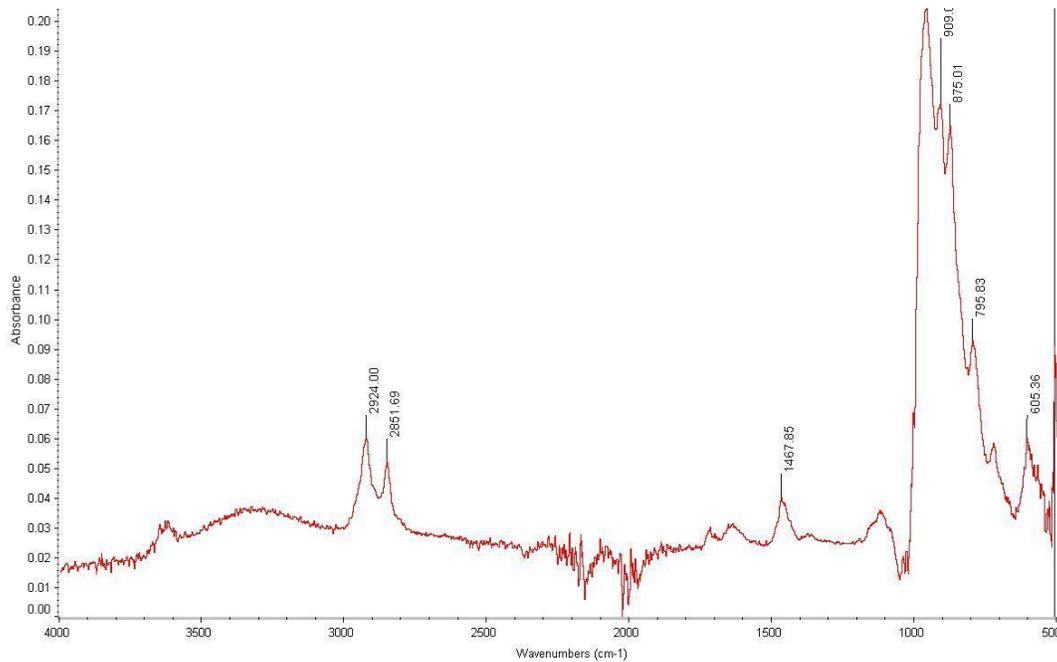


Figure 45. FTIR absorption spectra of LOCTITE epoxy resin.

4.1.4 FTIR Absorption Spectra of Resin and 8% Nanoclay Inclusions

FTIR absorption spectra of a mixture of resin and 8% nanoclay used to make a sealant more adhesive on a hole surface of specimens is shown in Figure 46. The spectra pattern looks different than that of resin without any inclusions. The highest peak intensities were found at a wavenumber of 2925.74 cm^{-1} . After the addition of nanoclay particles to the resin, the solution became stronger, with the molecular structure being more compact and resistive to corrosion when applied to the specimen surface.

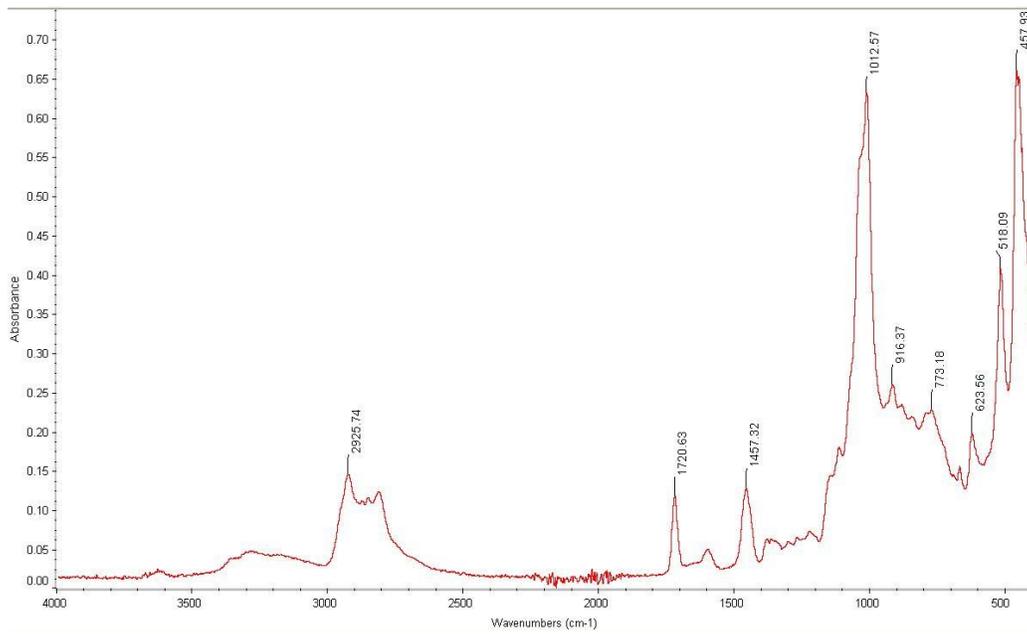


Figure 46. FTIR absorption spectra of resin and 8% nanoclay inclusions.

4.1.5 FTIR Absorption Spectra of Resin and 8% Nanotalc Inclusions

The FTIR absorption spectra of the mixture of resin and 8% nanotalc is quite different than that of resin without any inclusions. Wave intensities are at peak level at the first 500–100 wavenumbers, and then the spectra pattern changes, with slight peaks throughout the plot, thereby indicating that the resistivity of the solution increases with the addition of nanotalc

particles. Figure 47 illustrates the insight of functional groups and the absorption spectra of the resin and nanotalc mixture as well as peak intensities at different wavelength numbers.

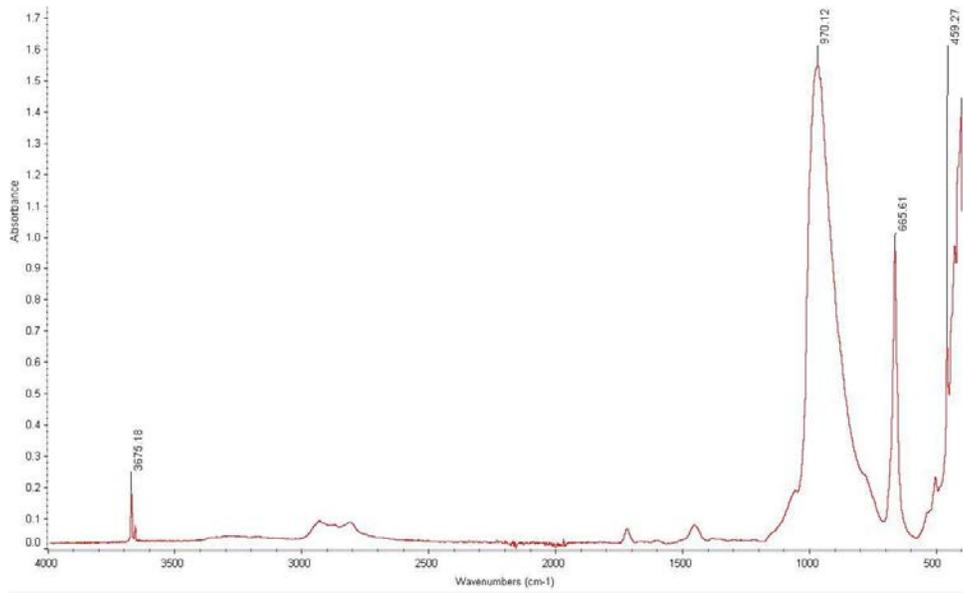


Figure 47. FTIR absorption spectra of resin and 8% nanotalc inclusions.

The results of this study are represented by two different graphical methods. Different weight percentages of nanoclay and nanotalc particles were used as a sealant for the hole treatment of carbon fiber-reinforced composites. Composite specimens with different weight percentages of nanoparticles and specimens without any nanoparticle inclusions were then tested on a cyclic fatigue machine in the NIAR Research and Development Composites Laboratory at WSU. The purpose of this research was to find the characteristics of the electrical resistance with respect to the varying cyclic loads and to determine the resistance changes. Resistance is measured in Ohms, and load is represented by newtons.

These graphs are plotted with applied loads on the X-axis and resistance on the Y-axis. Also, resistance versus time is plotted with electrical resistance on Y-axis and time on the X-axis. The graphical representations of the outcome of this study are shown in the following sections.

4.2 Graphical Representations of Specimen Resistance with and without Nanotalc Inclusions

Carbon fiber-reinforced composite specimens were treated with nanotalc inclusions on the surface of holes made for joining specimens to each other. Two types of bolts were used—threaded and shafted—to compare the behavior of specimens while applying loads.

4.2.1 Threaded Bolt with and without Nanotalc Inclusions

The graphical representation of specimen behavior plotted with resistance on the Y-axis and applied load on X-axis for a threaded bolt without nanotalc inclusions is shown in Figure 48. This resistance vs load graph for the baseline configuration with 0% nanotalc inclusions is almost a straight line, without any significant variations, because with increasing load, the specimen has a resistance with a yield point at almost 11 Ohms. After reaching the yield point, the resistance value remains the same until specimen failure. This graph suggests that without any nanoparticle inclusions, the specimen is more likely to experience galvanic corrosion. As shown in Figure 48, due to the inhomogeneous composition of the matrix, the coupons failed at a loading condition of 5000 N, and resistance dropped after the failure. Another reason behind the resistance drop in this experiment is the low barrier properties of the resin and hardener.

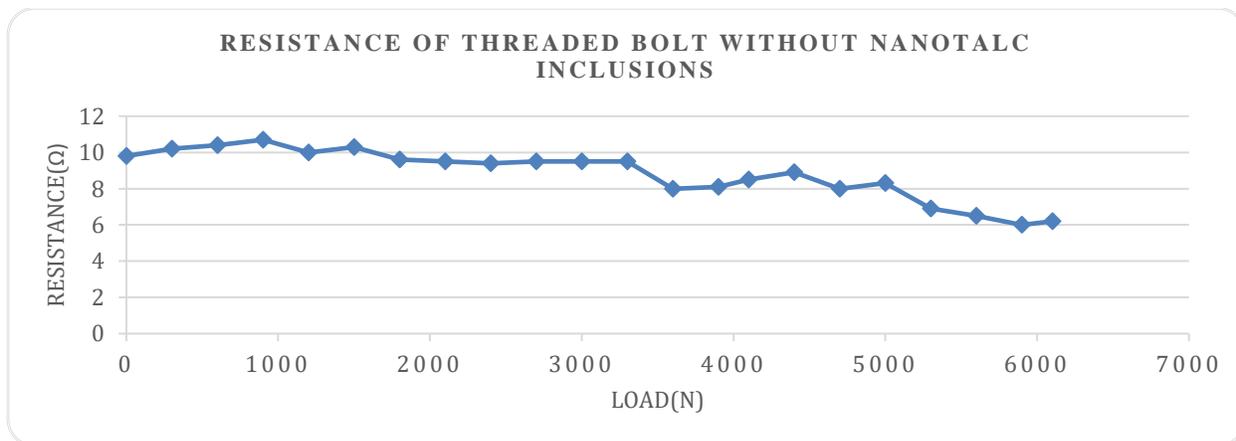


Figure 48. Resistance vs load plot for threaded bolt specimen without nanotalc inclusions.

After a 2 wt% coating of nanotalc was applied to the surface of the specimen hole and using the threaded bolt, there was a slight difference in the resistance value on the curve, as shown in Figure 49. Coatings with 2 wt% nanotalc on the hole of specimen made it go through the same elastic region, but the resistance value increased, which prompted the use of a greater weight percentage of nanotalc and a comparison of the results. Nanotalc inclusions of 2 wt% nanotalc had better result than without any inclusions on the sample, but the behavior of the graph was not as sufficient as expected. The highest resistance value measured during this experiment was 17.9 Ohms at a load of 2900 N. After the addition of 2 wt% nanotalc with the threaded bolt as a fastener, the coupons had better corrosion control over the coupons without any coating. Figure 48 shows the resistance vs load graph for a composite joint with 2 wt% nanotalc coating on its surface.

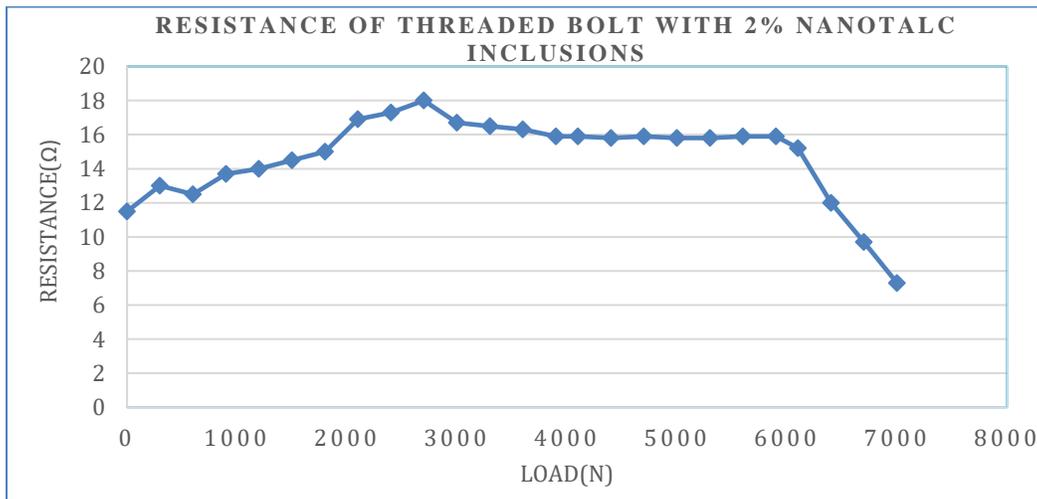


Figure 49. Resistance vs load plot for threaded bolt specimen with 2% nanotalc inclusions.

After coating the samples with 4 wt% of nanotalc and changing the loading conditions, there was a significant increase in the resistance values over the 2 wt% samples, as shown in Figure 50. The purpose of adding a greater percentage of nanoparticle coating on the composite-bolt joint was to evaluate the behavior of composite coupons over the increased load and

understand how the addition of nanotalc can act as a barrier between the bolt and a composite surface and reduce the chances of corrosion on the bolt.

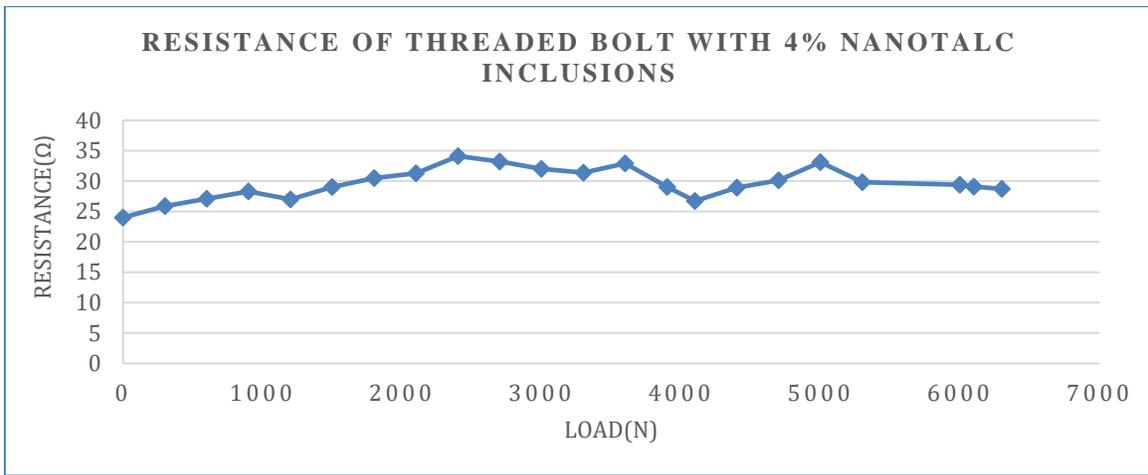


Figure 50. Resistance vs load plot for threaded bolt specimen with 4% nanotalc inclusions.

When the percentage of nanotalc coating was increased to 8%, the resistance vs load plot, shown in Figure 51, indicates a very steep slope, which is not favorable. What can be predicted from this result is that in aircraft or other structures, when two metals are joined with a threaded fastener, there are chances that the structure will become corroded and galvanic cells will be generated. The resistance to corrosion decreases at a very high rate due to fracturing on the composite surface, which may be the result of anisotropic behavior of the composite.

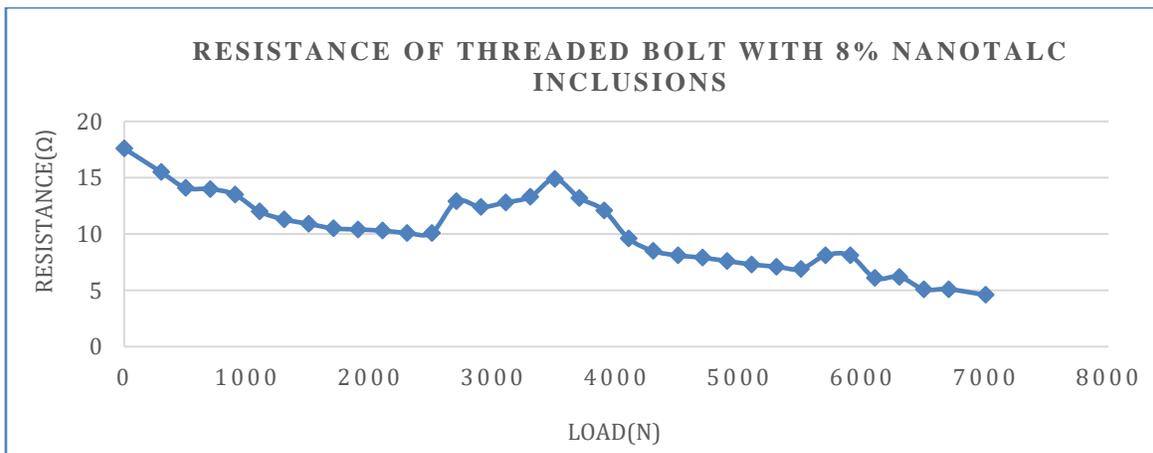


Figure 51. Resistance vs load plot for threaded bolt specimen with 8% nanotalc inclusions.

A summary of the plots showing resistance vs load for a composite joint with epoxy coatings as well as 2 wt%, 4 wt% and 8 wt% nanotalc coatings on its interface is shown in Figure 52. Even though nanotalc particles have excellent barrier properties, better structural integrity, and high performances, the resistance changes shown in this experiment when adding nanoparticles were not absorbed, as expected. The main purpose of this experiment was to join two composite coupons together either with a threaded bolt or shafted bolt, and evaluate the resistance variation with respect to load while coating the specimen hole with nanoparticles. As shown in Figure 51, a composite joint with 4% nanotalc has better resistance to corrosion and a tendency to eliminate galvanic corrosion.

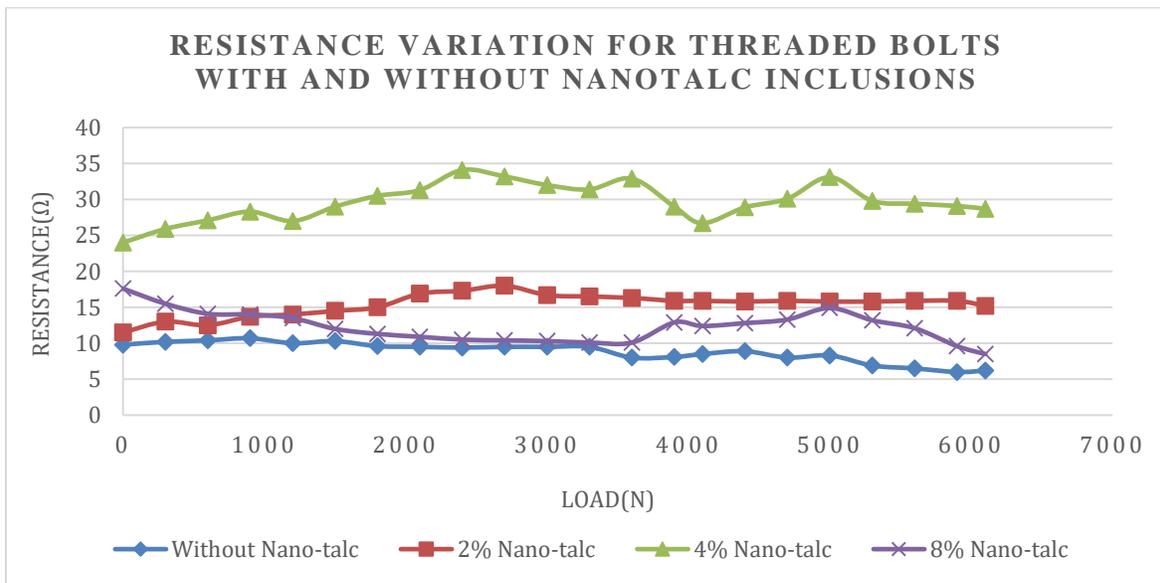


Figure 52. Summary of resistance plots for threaded bolts with and without nanotalc inclusions.

The carbon fiber-reinforced composite coupons after the fatigue test are shown in Figure 53. As can be seen, the coupons cracked at the interface, causing the delamination of fibers on the composite surface. The tensile failure that occurred on the joint surface of the composite specimens resulted in blistering. The results shown here from this experiment are an average, after three individual tests were performed on each sample.



Figure 53. Specimens before testing (left) and cracks at interfaces after testing (right).

4.2.2 Shafted Bolt with and without Nanotalc Inclusions

In this experiment, a shafted bolt was used to connect two specimens, and the same weight percentages of nanotalc coatings were used to compare resistance with respect to the baseline configuration, which was the coating without any weight percentage of nanotalc. First, the specimen was coated with epoxy resin and put into the fatigue machine to determine how resistance changes with increasing load. Resistance of the epoxy coating on the specimens gradually increased throughout the experiment. In this case with the shafted bolt, the baseline configuration had a great resistance value and was sufficient for the specimen to resist galvanic cells from developing on its surface. The electrical resistance of the composite specimen was 13.7 Ohms with the first 1000 N loads. When the load was increased, the resistance remained constant up to 3300 N loads because of the durability and resistivity of the epoxy. Figure 54 shows the resistance plot for the shafted bolt specimen without nanotalc inclusions.

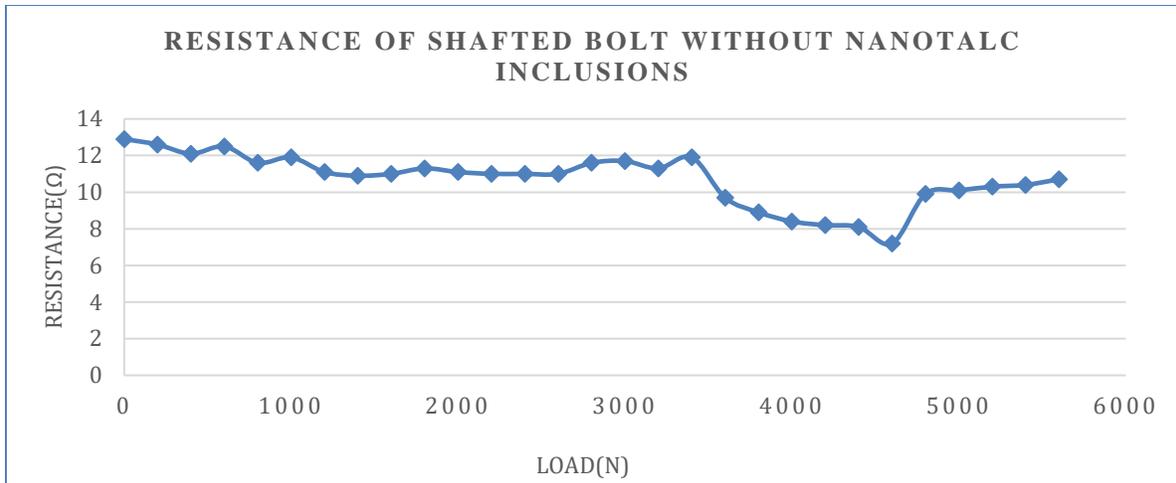


Figure 54. Resistance vs load plot for shafted bolt specimen without nanotalc inclusions.

Even though the resistance vs load curve for the epoxy coating was good compared to previous test results for the threaded bolt with nanoclay particles, the increase in resistance was the ultimate purpose of this research. Therefore, for the shafted bolt, 2 wt%, 4 wt%, and 8 wt% of nanotalc were again added to the epoxy and coated on the hole surface of specimens and compared to the baseline coating results. The change in resistance during the loadings of the 2% inclusions was much lower than the baseline. The initial resistance measured was 35 Ohms, which was the function of the 2 wt% nanotalc inclusions. But when the load was increased from 1000 N to 3000 N during the first 5 minutes of testing, the resistance dropped at a very fast rate. The reason behind this drop in resistance is the tensile effect of the shafted bolt on the surface of the fibers. The specimen cracked at 7800 N load with a resistance of 23 Ohms. Figure 55 shows the graph of resistance vs load for a 2% nanotalc coating on specimens joined together by a shafted bolt.

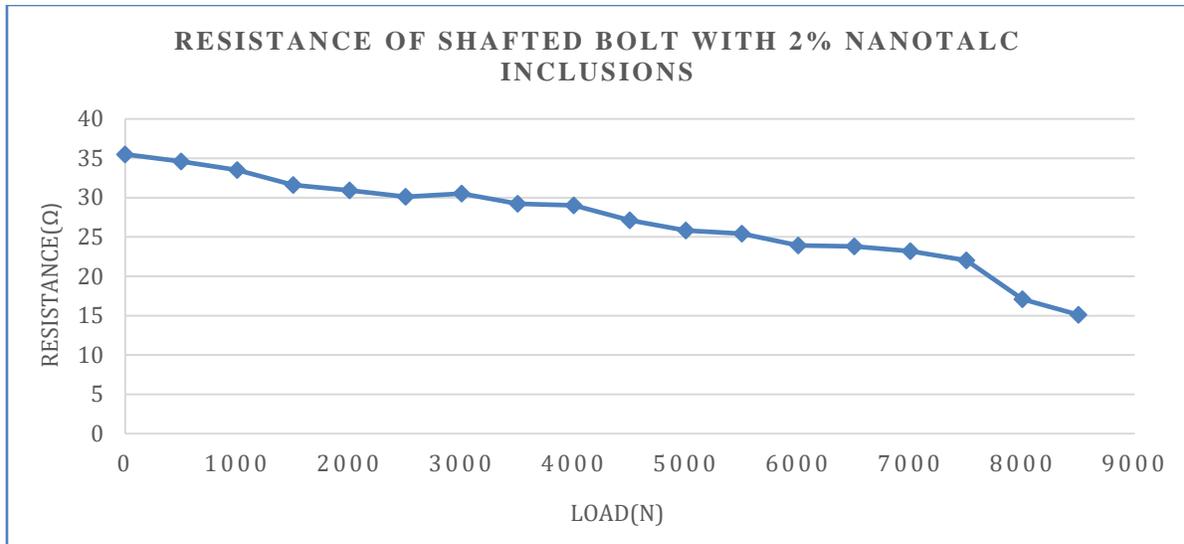


Figure 55. Resistance vs load plot for shafted bolt specimen with 2% nanotalc inclusions.

Additional tests were performed for the 4 wt% and 8 wt% nanotalc inclusions, and the average is plotted after three tests results for both inclusions on a single shafted bolt composite joint. After the tests, the results were as expected, which makes the perspective of the experiment more viable. The resistance vs load curve was very favorable with a high resistance value. As the load was increased, the corrosion resistance also increased. The shafted bolt specimen with 8% nanotalc coatings had a higher resistance value, meaning that galvanic corrosion can be minimized when two composite structures are joined together with a shafted bolt or fastener, if the higher weight percentages of nanotalc mixed with epoxy on the hole surface are used during the sealing of composites at a common interface. Figures 56 and 57 show resistance vs load plots for a single shafted bolt specimen joint with 4 wt% nanotalc and 8 wt% nanotalc coatings, respectively.

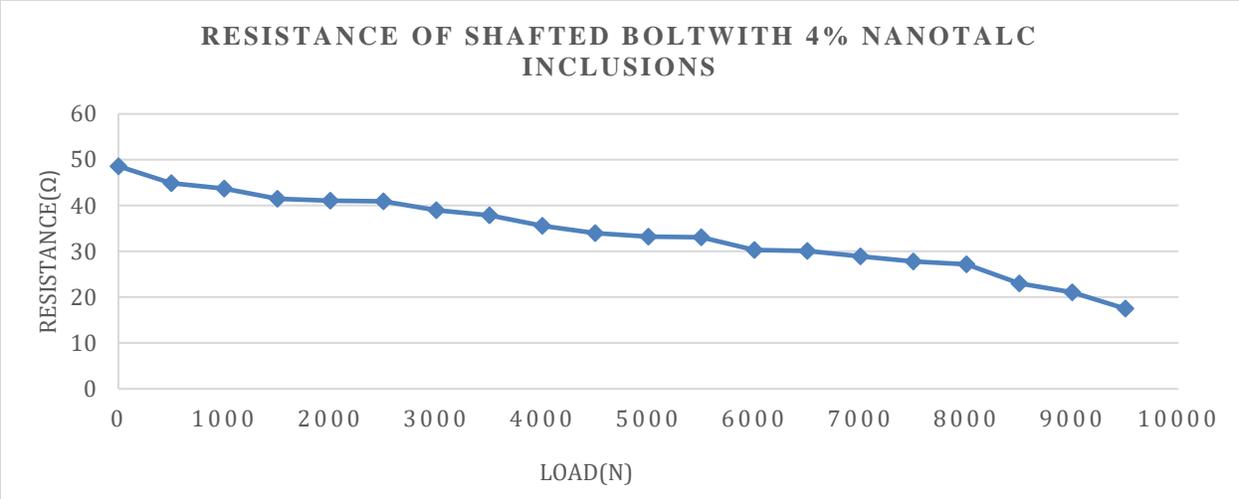


Figure 56. Resistance vs load plot for shafted bolt specimen with 4% nanotalc inclusions.

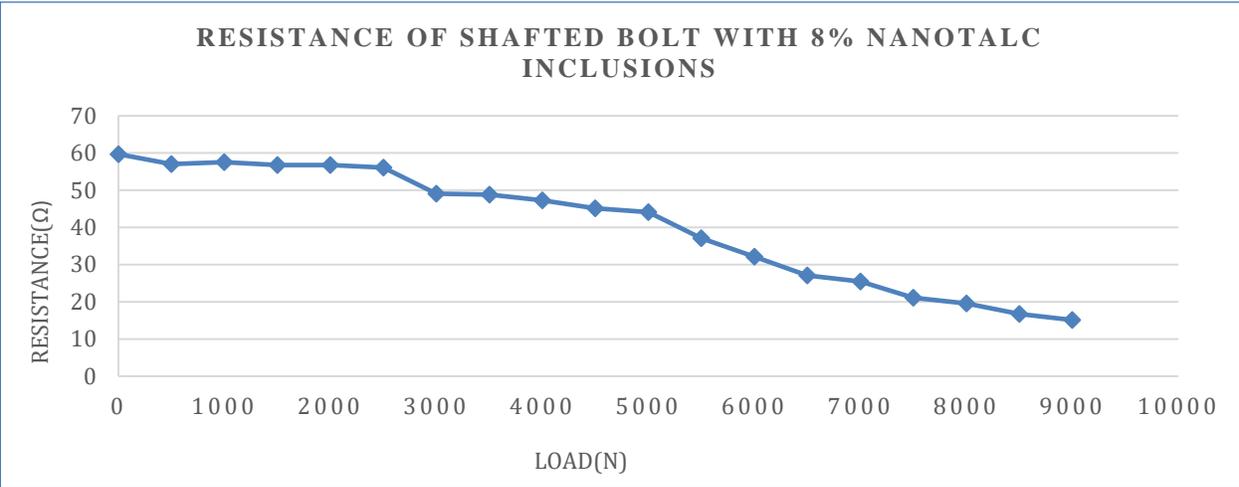


Figure 57. Resistance vs load plot for shafted bolt specimen with 8% nanotalc inclusions.

After the results for resistance variation with load for a single shafted bolt composite joint, a summary of the graphs was plotted for all four nanocoating iterations, as shown in Figure 58. The electrical resistance of an epoxy coating without nanoclay inclusions was found to have the lowest resistance value of 10 Ohms. During testing, the loading conditions were increased up to 5800 N and the resistance value did not change for the baseline coating, which means that the specimen can withstand loads for a long period of time without corrosion but fails after 6000 N

loads. Similarly, the 8 wt% nanotalc inclusion displayed the maximum resistance, but this dropped steeply because of the material failure at an early stage of cyclic loading.

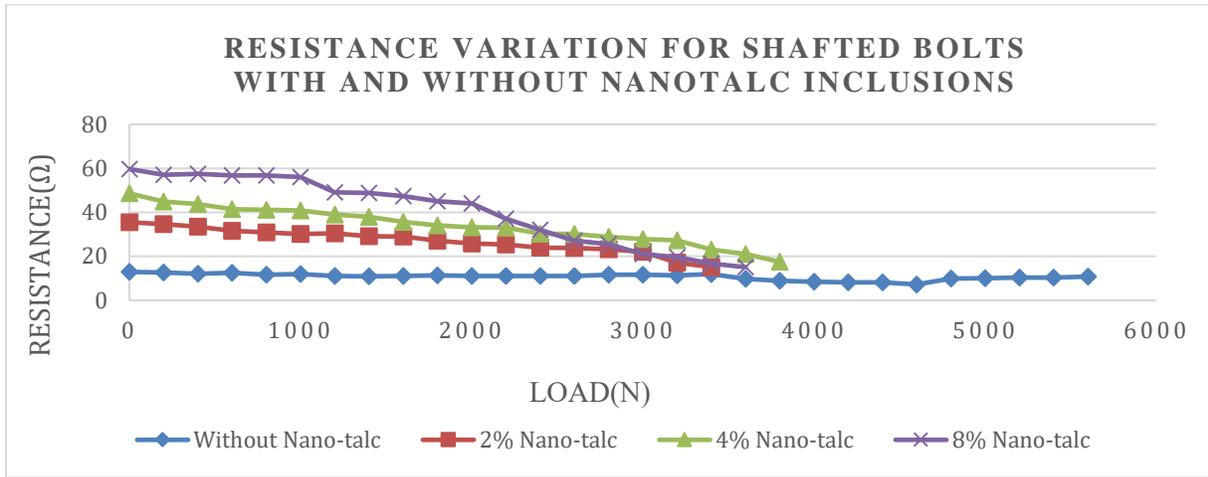


Figure 58. Summary of resistance plots for shafted bolts with and without nanotalc inclusions.

4.2.3 Threaded Bolt with and without Nanoclay Inclusions

Figure 59 shows the change in resistance with increasing load for a specimen joined together with a threaded bolt without any nanoclay used in the inclusion. The resistance vs load plot for this experiment has a slightly different pattern. The electrical resistance of the specimen is almost consistent until the load is 5 KN and then decreases because the specimen experiences a fracture on its hole surface by the threaded bolt.

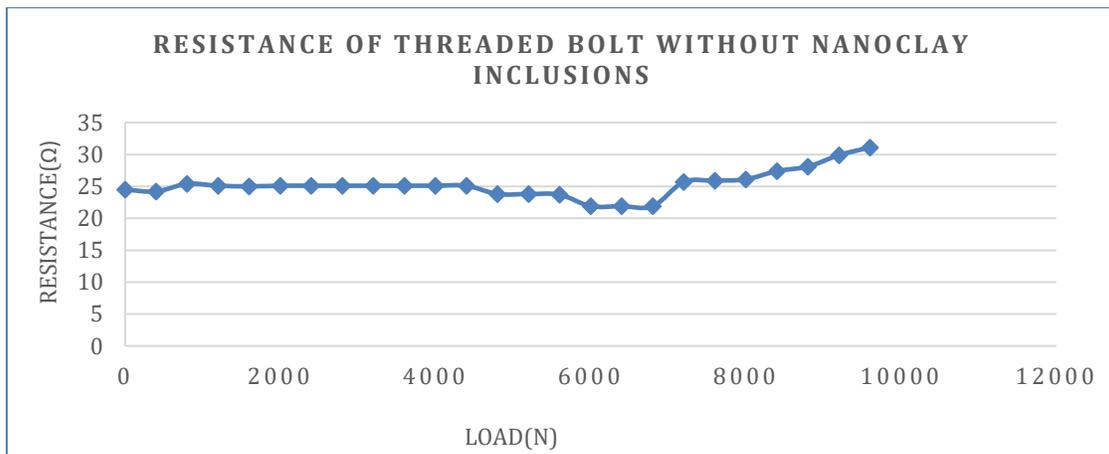


Figure 59. Resistance vs load plot for threaded bolt specimen without nanoclay inclusions.

Figure 60 demonstrates the change in resistance of a specimen as a function of load when 2 wt% of nanoclay particles is used to coat the hole surface of the specimen after mixing with the epoxy resin. It was found that this resistance plot has a very significant increase throughout the loading because of the nanoclay coatings on the hole surface. This result was expected and is favorable with the objective of this experiment.

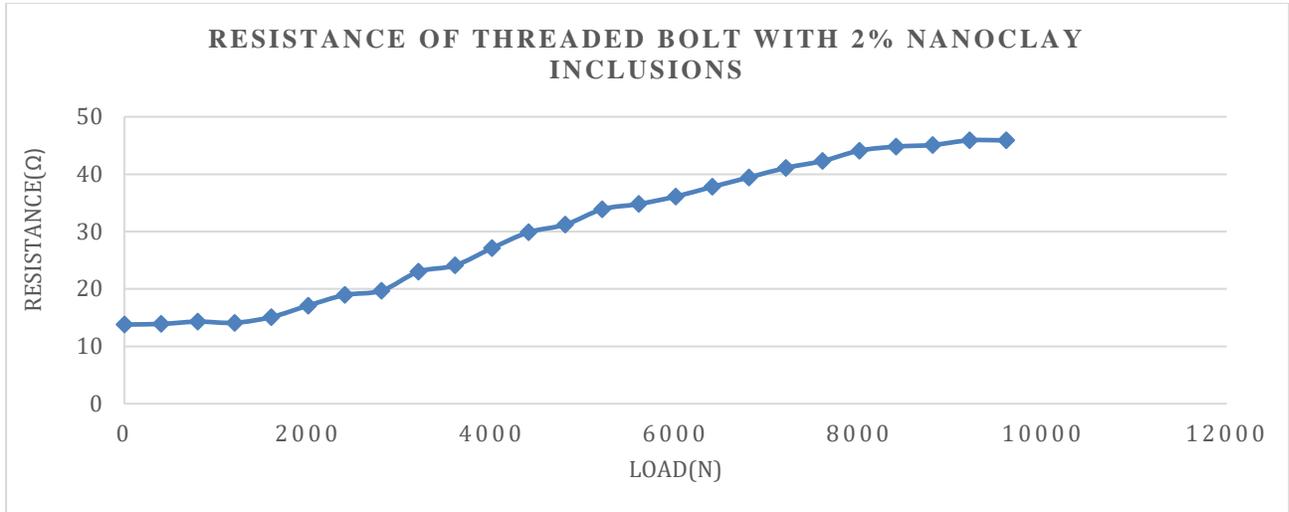


Figure 60. Resistance vs load plot for threaded bolt specimen with 2% nanoclay inclusions.

The plot of resistance vs increasing load followed a different trend for the carbon fiber-reinforced composite with a threaded bolt and 4 wt% nanotalc inclusions than that of the 2 wt% nanotalc inclusions, as shown in Figure 61. Resistance dropped from 40 Ohms to 22 Ohms on the first 5800 N loads. Because of the nanotalc inclusions, resistance increased again and reached 35 Ohms, and then failure of the specimen occurred, which caused the resistance to drop steeply again to 25 Ohms. This plot is also favorable and upholds the objective of this research, which is to increase the resistance to galvanic corrosion. The pattern of resistance curve shows a downward trend, which indicates that with increasing load, the resistance of the specimen decreases because the threads of the bolts touched the outermost fibers of composite thus forming the cracks on the surface of composite.

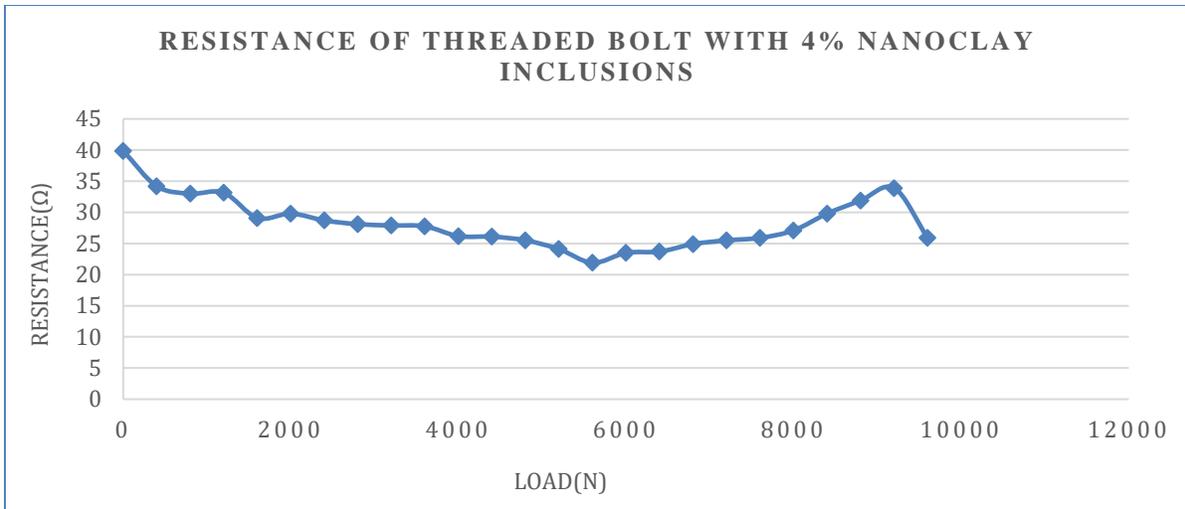


Figure 61. Resistance vs load plot for threaded bolt specimen with 4% nanoclay inclusions.

Figure 62 shows the resistance vs load plot for the carbon fiber-reinforced composite specimen with 8% nanoclay inclusions when a threaded bolt is used as a removable fastener. The nanoclay coating on the hole surface of composite acts as a filler to reduce the voids, thereby increasing resistance. This plot indicates that with the increase in weight percentage addition of nanoclay particles on the epoxy and applied on the surface, the resistance to deformation increases thereby making the specimen withstand more loads for a longer period of time.

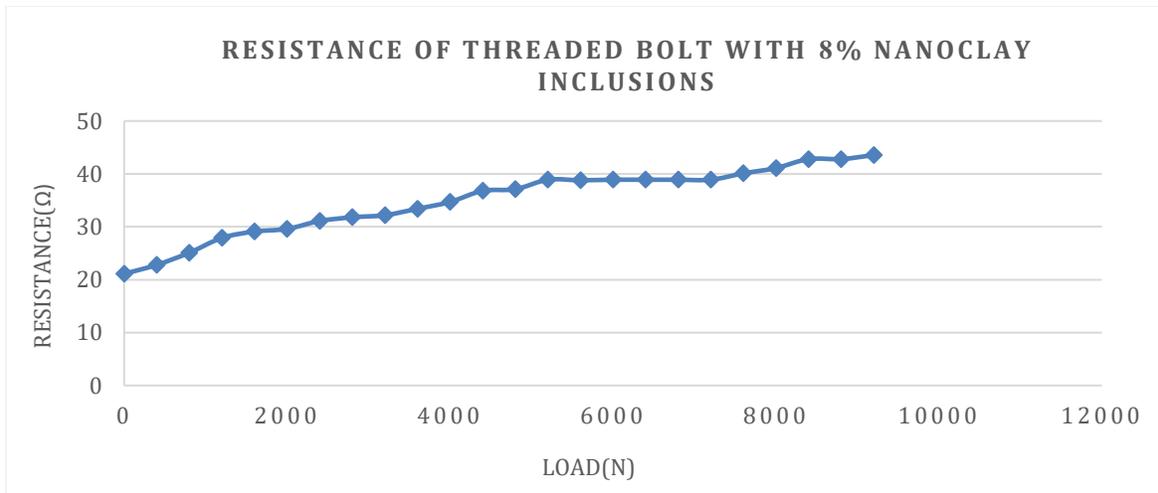


Figure 62. Resistance vs load plot for threaded bolt specimen with 8% nanoclay inclusions.

A summary of the graphs showing resistance change vs load on a tensile machine with four different iterations of coating are shown in Figure 63. The electrical resistance is the reciprocal of the corrosive current, which means the higher the resistance observed on the interface, the lower the corrosion current on the surface. In this experiment, the maximum corrosion resistance value was observed when the composite joint was sealed by 2% nanoclay particles. The resistance vs load curve for the 2 wt% iteration is very satisfactorily increasing. Due to the better barrier properties and excellent physical properties, the nanoclay particles when coated on the hole surface of the composite isolates the bolt from the fibers, thereby not allowing the bolt to corrode and reduce the delamination of matrix. For this reason, the specimen withstands maximum load under the cyclic loading without any fracture or crack formation.

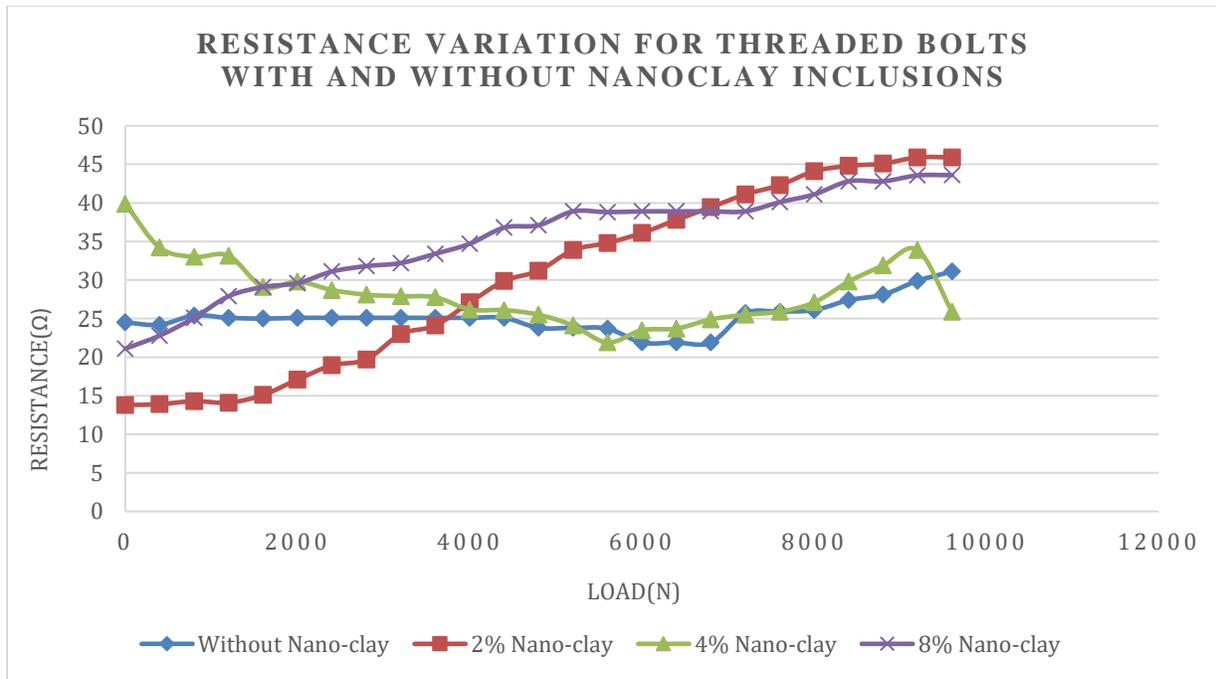


Figure 63. Summary of resistance plots for threaded bolts with and without nanoclay inclusions.

4.2.4 Shafted Bolt with and without Nanoclay Inclusions

As shown in Figure 64, the plot of resistance vs load for the carbon fiber composite specimen without any nanoclay coating on the hole surface indicates a downward trend. Without

the influence of nanoclay inclusions on the hole surface of the composite, there is a direct contact between the fibers and the shafted bolt, which acts as a bridge for the fibers; therefore, galvanic corrosion occurs on the surface of the specimen and fastener. The resistance to corrosion in this case is very minimal, and the chances of galvanic cell generation is high. For this reason, the resistance with increase in load decreases and reaches almost 12 Ohms at a loading condition of 5800 N.

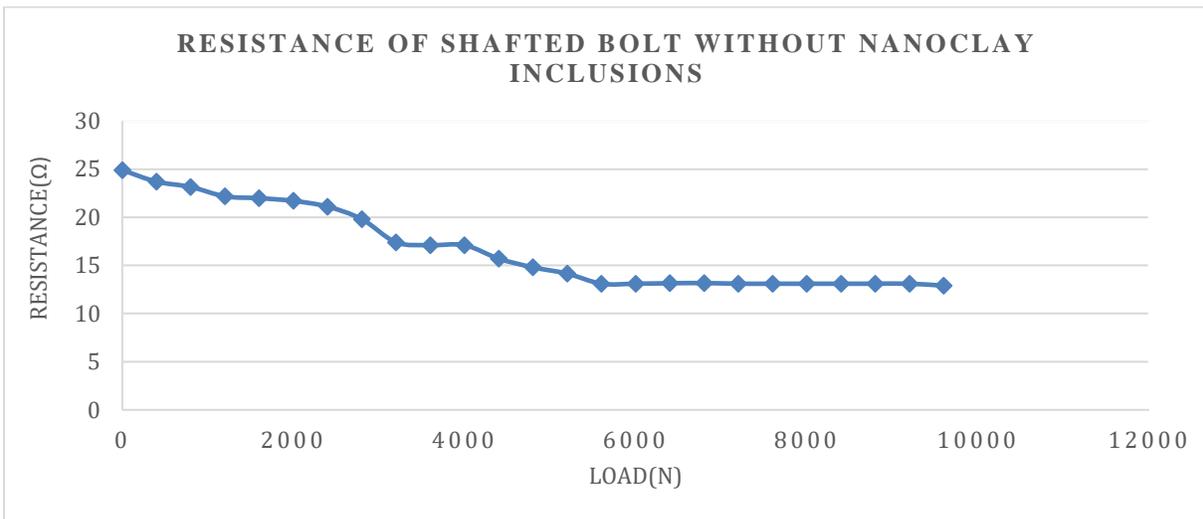


Figure 64. Resistance vs load plot for shafted bolt specimen without nanoclay inclusions.

Figure 65 shows the change in resistance to deformation of the specimen when using a shaft bolt to join two specimens together and coating with 2 wt% nanoclay. The reaction of 2 wt% nanoclay on the hole surface became very useful for reducing corrosion on the fastener. This plot of resistance shows that at first loading conditions, the resistance of the specimen drops, but when the load is gradually increased on the testing machine, the resistance increases satisfactorily.

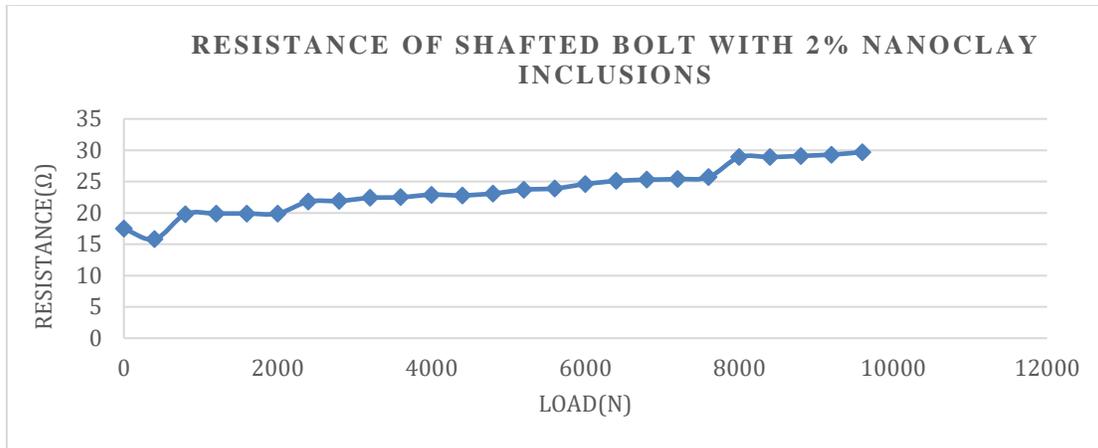


Figure 65. Resistance vs load plot for shafted bolt specimen with 2% nanoclay inclusions.

After the application of 2% of nanoclay on the hole surface of specimen, the resistance increased with less deformation on the sample. This experiment provided the idea that when the proportion of nanosealant was increase by some percentage, that it would have good impact on crack resistance, thereby increasing the resistance to corrosion. After the addition of 4 wt% nanoclay on the sample with the epoxy resin, and testing the resistance behavior, the sealant really worked better to minimize the corrosion current, thereby gradually increasing the resistance with respect to increasing load. The resistance vs load graph for the composite joint with 4 wt% nanoclay coated on the surface is shown in Figure 66.

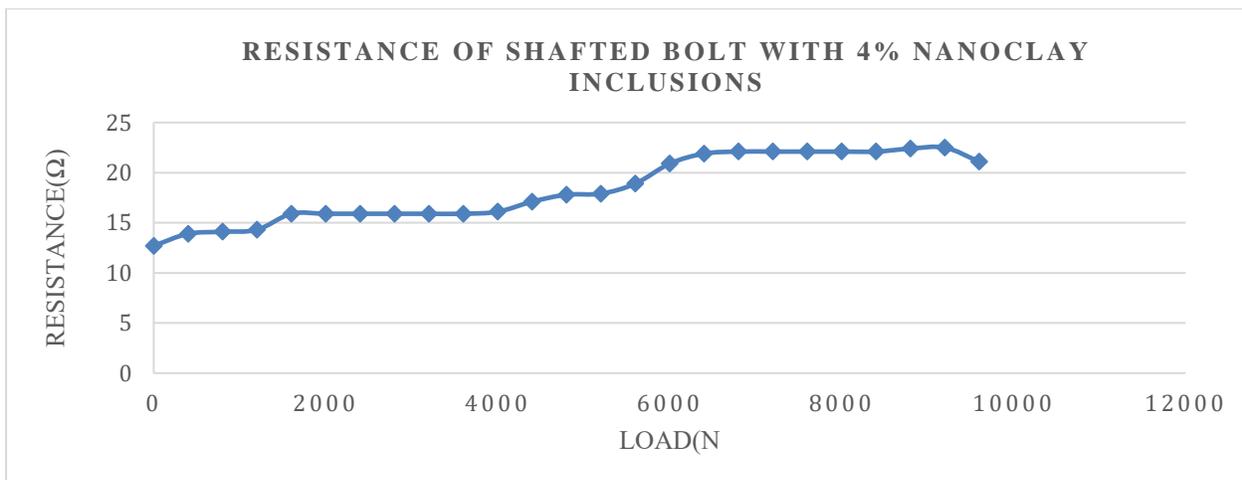


Figure 66. Resistance vs load plot shafted bolt for specimen with 4% nanoclay inclusions.

In addition to 4 wt% nanoclay inclusions, another experiment was conducted by adding more nanoclay particles with the resin and coating the sample. The 8 wt% nanoclay had a very good response to resistance change, which directly proved the objective of the experiment as true because the sealant increased the resistance while decreasing the corrosive current. The lifetime of epoxy is limited, so after the addition of nanoclay particle, the resistance change was found to trend upward trend. Figure 67 is the graphical representation of resistance vs load for a composite joint a shafted bolt and 8% nanoclay coating.

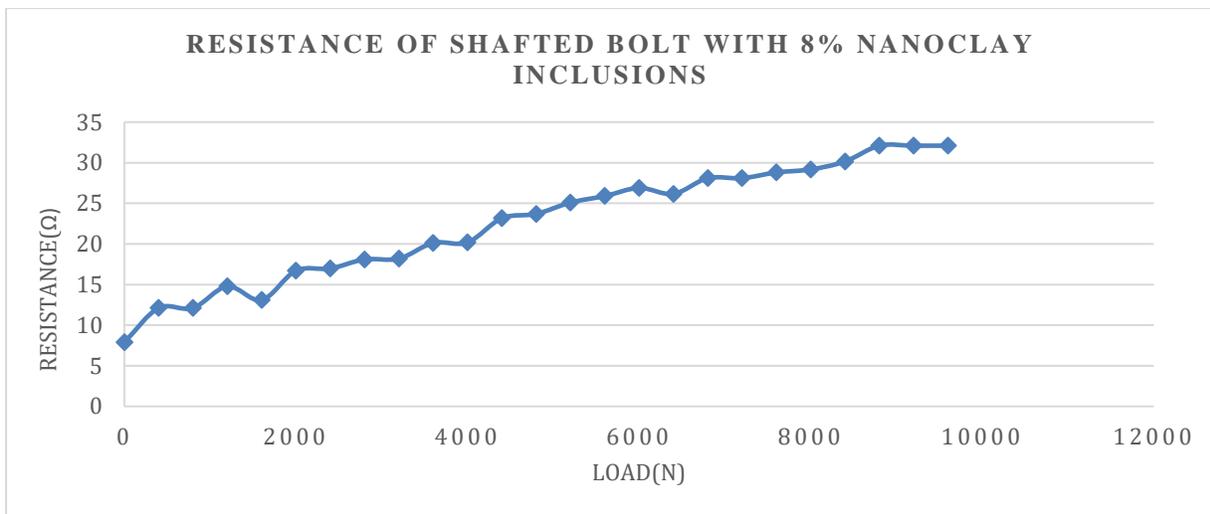


Figure 67. Resistance vs load plot for shafted bolt specimen with 8% nanoclay inclusions.

Following the application of multiple nanoclay coatings on the composite joint with 2 wt%, 4 wt%, and 8 wt% iterations, the average results were combined in a single plot. The maximum electrical resistance measured during this test was 33 Ohms. The conclusive result for this test observed from the fatigue test was involved the 8 wt% nanoclay coating, the resistance value of which was gradually increased with increasing load without any damage and delamination on the composite. Figure 68 shows the combined plots for change in resistance with load for a single shafted bolt joint sealed with nanoclay particles.

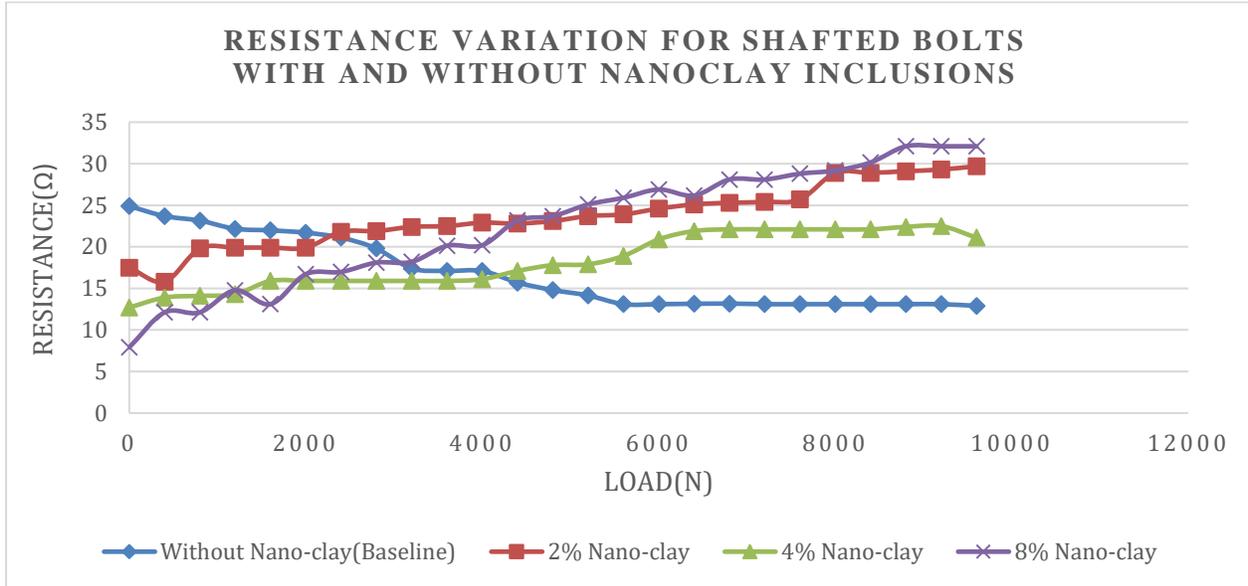


Figure 68. Summary of resistance plots for shafted bolts with and without nanoclay inclusions.

4.3 Graphical Representations of Specimen Resistance with Respect to Time Change

After conducting the experiment on the tensile fatigue machine and plotting the graphs resistance vs load, the next experiment was to evaluate the change in resistance with respect to time. The plots in this section describe the effects of nanoclay and nanotalc particle coatings on the hole surface of carbon fiber composite specimens and their direct influence on the corrosion control and resistance to sample deformation.

4.3.1 Resistance vs Time Plot for Specimens with and without Nanotalc Coating

Figure 69 provides a graph of resistance versus time of the carbon fiber composite sample after the fatigue test. As can be seen, resistance of the sample decreased at a noticeable rate when the sample was tested with the epoxy coating alone on the interface between the two sample joints. The idea behind this experiment was to evaluate the resistance change during the fatigue loading to determine and reduce the galvanic corrosion that might affect the lifetime of the composite. Therefore, 2 wt% of nanotalc nanoparticles were added to the epoxy, mixed very

well, and applied on the surface of the joints. The nanotalc coated on the surface acts as a sealant, which works as a barrier for the fastener or bolt, preventing contact with the fiber constituents of the sample. After applying 2 wt%, 4 wt%, and 8 wt% nanotalc coatings on the interfaces, the resistance changed, which shows an upward trend in the plots. This indicates that with nanotalc coatings, the resistance increases, thereby decreasing the corrosive current on the composite surface.

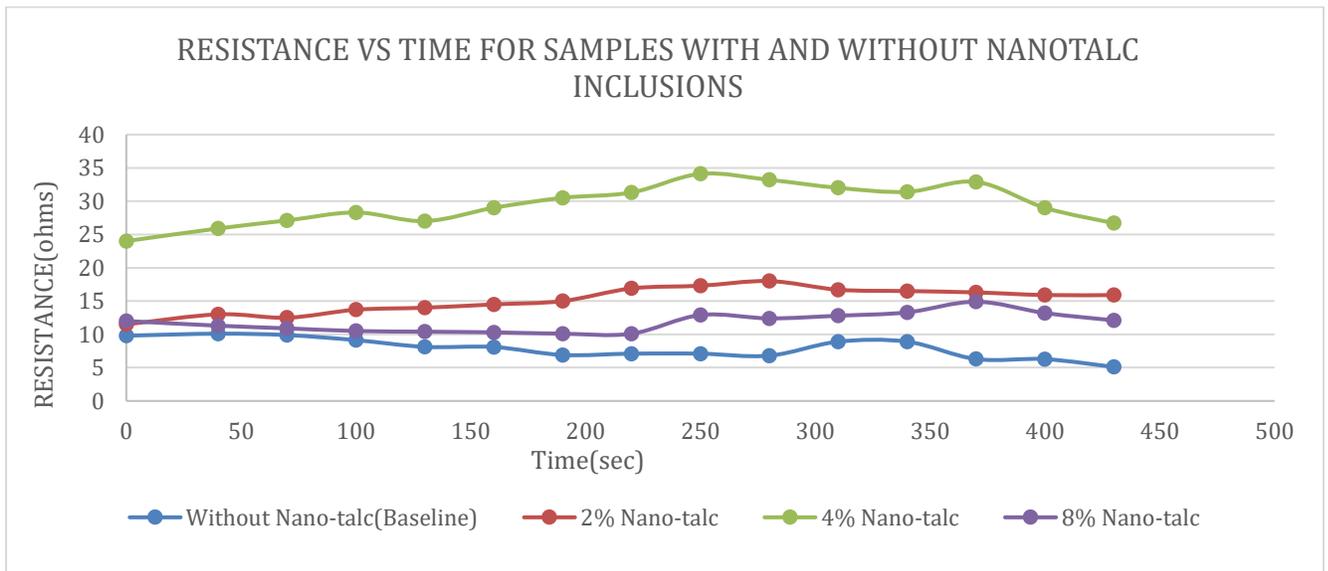


Figure 69. Resistance vs time plot for samples with and without nanotalc inclusions.

4.3.2 Resistance vs Time Plot for Specimens with and without Nanoclay Coating

After evaluating the resistance change with respect to time with a nanotalc coating on the composite sample, another nanoparticle was used to measure the resistance change and influence of nanoparticle. This time, nanoclay was used in the same proportions and weight percentages as that of the nanotalc, and the results were compared. The resistance vs time graph, when a composite specimen was tested without nanoclay coating was in decreasing order, which means that over time, the resistance of a composite decreases because the lifetime of the epoxy resin alone and its resistance to corrosion is very low. With the application of different percentages of

nanoclay particles on the specimen interfaces, the fastener was not able to touch the fibers, and the chance of galvanic cell generation was minimum. The resistance change during the experiment with the nanoclay coating was almost at a minimum rate most of the time, and the resistance remained constant without any significance changes. The plot of resistance vs time for samples with and without nanoclay coatings is shown in Figure 70.

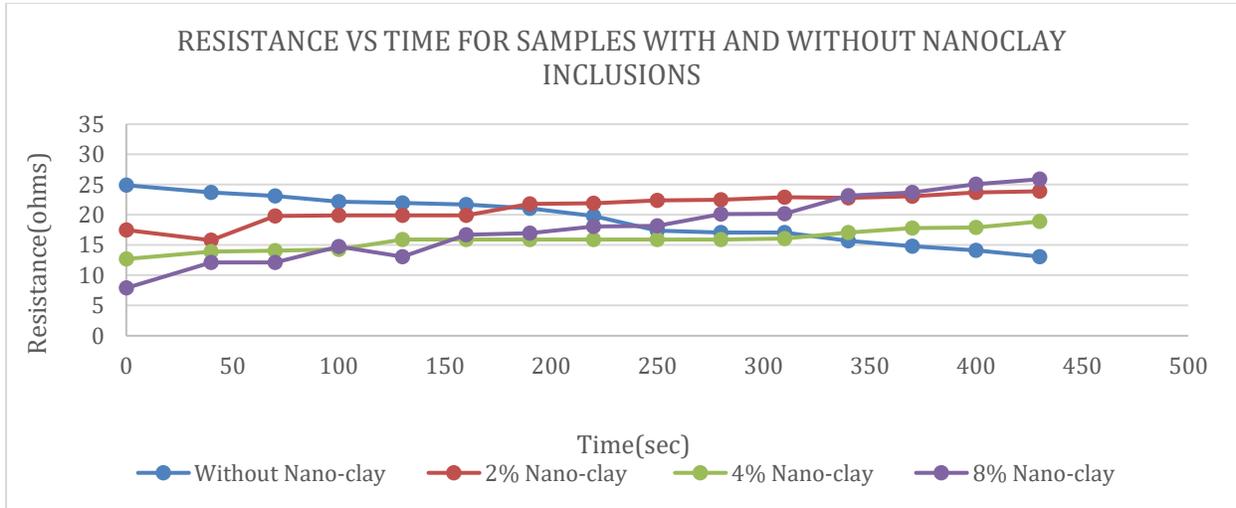


Figure 70. Resistance vs time plot for samples with and without nanoclay inclusions.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE WORK

5.1 Conclusions

The main purpose of this experiment was to evaluate the change in resistance during the fatigue loading of a composite. When two composite samples are joined together using a removable fastener, the chance of galvanic corrosion on the surface of the fastener is very high. Also, deformation of the composite due to corrosion is maximum. This issue is a major drawback in most aircraft and mechanical industries that use composite structures; therefore, an alternative way of minimizing galvanic corrosion is needed. In this research, two different nanoparticles—nanoclay and nanotalc—were mixed with a common epoxy resin to act as a sealant. The treatment of holes in a composite structure by applying nanoparticles proved to be feasible, with experimental results from a comparison of resistance changes with respect to time and load being as expected.

The plots of resistance vs time and resistance vs load mostly showed an upward trend, indicating that with the application of nanoparticles as a sealant between two composite structures, the resistance to corrosion and deformation is increased, thereby decreasing the corrosive current throughout the composite surface. The application of 8% of nanoclay particles exhibited a better performance, with the highest resistance measure during the experiment at 43.9 Ohms.

5.2 Recommendations for Future Work

The following is a list of recommendations for future work:

- A sample could be attached to a metal substrate and placed in a salt chamber to see how the sealant behaves under extreme corrosive conditions.

- More variation of weight percentages of the nanoparticle coatings could be done to obtain more efficient results.
- The geometry of the specimen could be changed to understand how the nanoparticle coating works for larger composite coupons.
- Human error could be minimized by recording the resistance values through inbuilt software.
- The four-probe method could be used to measure the resistance of the fiber-reinforced composite.
- A method that includes four equally spaced electrodes—the two outer electrodes for current and two inner electrodes for voltage—could be employed.
- Other nanoparticles could be used to compare the best results.

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