5-2011

Influence of Material Selection and Fabrication Process Repeatability on Mechanical Properties of Glass-Polymer Matrix Composite Structures

Charles Edwards
Clemson University, credwar@clemson.edu

INFLUENCE OF MATERIAL SELECTION AND FABRICATION PROCESS REPEATABILITY ON MECHANICAL PROPERTIES OF GLASS-POLYMER MATRIX COMPOSITE STRUCTRES

A Thesis
Presented to
The Graduate School of
Clemson University

In Partial Fulfillment
Of the Requirements of the Degree
Master of Science
Polymer and Fiber Science

---

by
Charles Edwards
May 2011

Accepted by:
Dr. Kathleen Richardson, Committee Chair
Dr. Michael Ellison, Committee Co-Chair
Dr. Philip Brown
Dr. Chris Norfolk

Abstract

This study has aimed to evaluate property uniformity from data obtained utilizing one design of a single layup composite plaque, three sources of glass fibers and a single, industry accepted resin to produce a repeatable fabrication process. This thesis has investigated the following:

1. Whether the type of glass (E-Glass, S-Glass, and R-Glass) influences the property values of individually tested samples compared between glass types.
2. Whether the type of glass influences the property uniformity throughout the set of tested samples.
3. Whether the composite plaque design and resulting performance, as defined by ASTM Standards or industry accepted parameters, is adequate for use in the defined military application or wind specific application.

The resulting data showed trends that established the relationship between the mechanical properties of the materials used in constructing the composites and the properties of fabricated composite test plaques. The S-glass resulted in the highest ultimate fracture strength and modulus, yet had the highest properties per cost value. The E-glass demonstrated the worst mechanical properties of the three grades, however had the highest value comparing properties to cost. All of the composites were fabricated at <2% void content and considered a quality test sample.

Dedication

I dedicate this report to both of my parents, my brother, my advisors, and all my friends that have been with me through this process. I appreciate all the advice and support that all of you have given me and the encouragement to complete this degree.

Acknowledgments

First of all I would like to personally thank all my advisors for everything done that has motivated me and assisted me in completing this degree. I wish to recognize both of my Committee Chairs, Dr. Kathleen Richardson and Dr. Michael Ellison. Without your advising and direction this project would not have been possible. I would also like to thank Dr. Philip Brown for finding me the opportunity and funding that was aided greatly to this project. Last but not least, I would like to thank Dr. Chris Norfolk for all of his assistance and guidance in lab plus the knowledge of mastering the VARTM process.

Table of Contents

List of Figures

List of Tables

Chapter 1 – Introduction

1.1 Motivation

Composite materials, herein defined as the combination of two interfacial bonded materials consisting of a matrix component and a reinforcement component, are finding applications in a range of commercial products due to their essential attribute of achieving properties that exceed those of the individual components. These properties include being lighter-weight, while having high specific strength (strength to density ratio); possessing high impact and good fatigue resistance, with high toughness; and being of a lower density than metals. In application to vehicles, composite materials enable fuel savings; these applications include motorsports and consumer automotive, aerospace, airplanes and military (Navy ship structures, military armored vehicles).[30]

Composite materials can be broken down into several different categories, which include ceramic, metal and polymer matrices with reinforcing fibers of the same or different materials, each having advantages and limitations, as material performance is dictated by application environment as well as material-specific properties.

The present study has focused on the use of glass-reinforced polymer matrix composites, and specifically, their mechanical properties, with potential for use in a defined military or wind power application. In the investigation, we have optimized a composite plaque manufacturing protocol to obtain 14'' x 36'' specimens, which have been characterized for their physical properties. The objective of this work was to quantify the repeatability of this optimized manufacturing process in creating high quality

composite materials, defined as samples which showed low standard deviations of property variation, within a (single) type of glass fabric used. Additionally, the study evaluated what differences, if any, resulted from varying the type of glass fabric and keeping the resin material constant to all samples. The resulting material properties were compared to standards routinely used to quantify performance in the two targeted application areas: load bearing structural parts for wind turbine blades and as light-weight components for armored military vehicles. For purposes of comparison in the description used here, these applications will be referred to as ‘wind’ and ‘military’. The background and details of the research effort are discussed later in this thesis.

1.2 Objective

The primary goal of this thesis was to use a repeatable layup and infusion process to obtain consistent specimens for a standard set of material testing experiments. With the high level objective of establishing a reproducible fabrication process, the physical property uniformity of the resulting plaques would translate into excellent repeatability of the composite fabrication method applied. Specifically, this work has aimed to investigate:

1. Fabrication of a simple design utilizing glass-resin composites
2. The influence of glass fiber type on within-type and across type plaque morphology and property uniformity; and,
3. Composite mechanical property performance and uniformity.

The objective for the fabrication process was to realize a time efficient, low void content, uniform property composite sample that was suitable for testing and possible

use in wind and/or military applications. Once the repeatable process had been developed for the single layup design chosen, multiple plaque samples (12 plaques for each fabric type) were produced. With high quality test samples produced (as defined by low void content of typically less than 1-2% by volume[i]), statistical analysis of physical property testing results could be used to establish trends between process variables and resulting composite sample mechanical properties. ASTM testing standards have been used to acquire and assess property and performance data that are acceptable for high performance application to current wind turbine blade properties and ballistic military shielding.

In this chapter, the rationale and background associated with the materials used, the plaque design chosen, the fabrication process parameters defined, and the subsequent testing methods that parts would be assessed with, are described.

1.3 Background – Materials & Design

In this study, composite test plaques were created based on a defined standard layup design using orientation of the fiber reinforcement component similar to that found in current wind turbine blades and military ballistic applications. Fiber reinforced composite (FRC) materials are currently being used in both wind and military applications to maintain the strength comparable to metal yet a fraction of the weight. Fiber reinforced composites are composed of two components, a matrix component and

[i] This level of composite void content was defined as an upper limit benchmark by the project team. It was established by Chris Norfolk's ATI team members and ASTM 2734 as a threshold value that deemed samples suitable for further testing. Void content samples below this value had not repeatedly been demonstrated prior to this project's start (July 2010). Thus, this goal, of establishing a manufacturing process that could repeatedly yield low void samples, became one of the primary target outcomes for the project.

a reinforcement component as shown in the schematic in **Figure 1**, Here, the grey circles in the simplified diagram represent the fiber (reinforcement) component surrounded by the white matrix component as viewed in an abstract cross section view, seen from the fiber end, to aid in component identification. The matrix component is a continuous phase, which forms the binding interface between the discontinuous glass fibers/fabric reinforcement. The reinforcement component gives the strength and stiffness properties of the composite, while the matrix component provides the stress transfer through rigidity and protects the fiber component from environmental conditions.

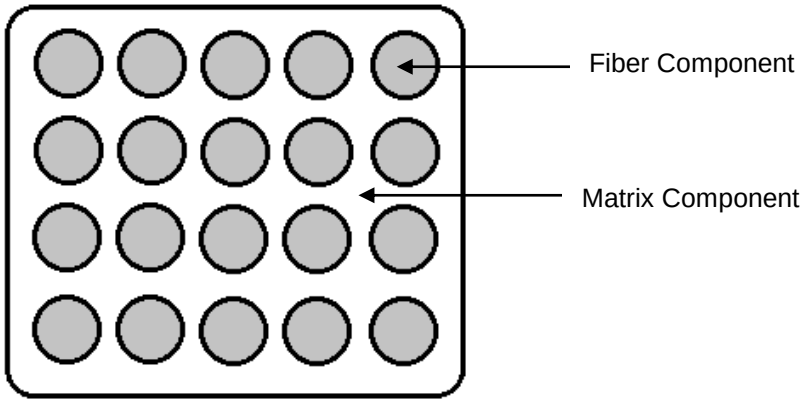

Figure 1: Diagram of Matrix and Fiber Components

The matrix component for this study is a two part epoxy resin produced by *Applied Polermeric Inc* and the reinforcement component is a woven glass fabric discussed more in section 1.3. Key properties for the resulting composite are obtained based on the individual properties of the starting components. When the combined fiber/matrix system is fabricated, the key composite characteristics of interest may

include ultimate tensile strength, elastic modulus, fiber volume fraction, density, and void content. Each of these properties was evaluated for the composite plaque materials fabricated in this study and the results are discussed in the following sections.

In both targeted applications, wind and military, the composite part's load-related performance is determined by measurement of the ultimate tensile strength and Young's modulus of the composite structure, which are important indicators of the material's ability to withstand load. This load could be related to a shear, tensile or compressive force, though for purposes of this study, tensile behavior was the only stress state evaluated. The Young's modulus, E, of the composite in relation to the stress strain curve of the components of a composite, is shown in **Figure 2.** Modulus is measured as the initial slope of these curves. As shown in **Figure 2**, the resulting modulus of the FRP (fiber-reinforced polymer) composite is midway between that of its constituents: the glass fiber is much stiffer than the matrix resin.

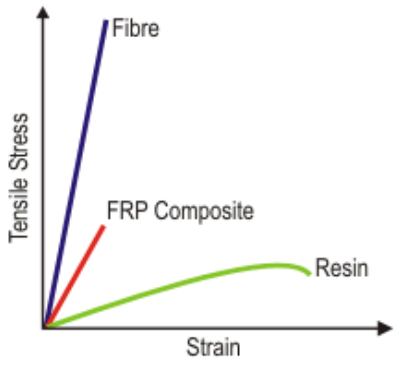

Figure 2: Demonstrating the modulus comparison between components

The composite's multi-layer design (defined by the number of ply or layers of fabrics, fabric type and weave orientation) will affect the strength and toughness behavior of the final composite. These resulting properties will dictate how the applied

stress will be distributed throughout the composite in use; thus, the sample's response will be dictated by the intermediate stress strain curve and initial modulus demonstrated as the red curve in **Figure 2**.

Composite materials have found use in wind turbines by providing large, load-bearing capabilities in a lighter weight component (as compared to a single phase material) for land-based and off- shore wind energy sources. Composite designs are used in multiple parts of the manufacturing processes including the base models of the size and shape, mold production, and the blade skin.[9] Blades are based on glass fiber-reinforced polymer matrix structures created using largely manual assembly processes.

Along with composites being fabricated on large model scales, composite panels are produced for impact resistance applications. Recently both carbon-fiber polymer matrix along with glass-fiber polymer composites have gained wider use in military impact protection applications, as well as in lower load bearing applications in naval vessels (for hatch structures, decking, and more recently, exterior hulls).[11] The push towards composite layup and design research is due to advantageous mechanical properties (per weight) as compared to the much heavier alternative metal structures, which can be both difficult to manufacture and costly to maintain. Composite materials are usually lighter in weight, and have less thermal expansion when exposed to temperature variations. They can also be molded into complex shapes without the waste and the conformity difficulties associated with metals. Multiple methods can be used in the fabrication of composite materials for wind blade applications or impact resistance plaques, though in this effort the VARTM (Vacuum Assisted Resin Transfer

Molding) method was solely used for part assembly. The details of the VARTM process will be discussed below.

The VARTM process uses a vacuum system to draw epoxy resin (matrix component) through a fabric (reinforcement component) until the fabric component is sufficiently wetted and residual air is removed. It is a relatively new composite manufacturing method that minimizes health and environmental issues traditional to wet layup process, wherein resin is applied by hand to the part, layer by layer. This requires handling and brushing of the resin onto the part, which is open to the atmosphere and to the worker, which impacts health and results in large emissions to the atmosphere.

Another composite fabrication technique is the prepreg method which relies on the process of physically impregnating the woven fabric component with a melted or solvent based polymeric precursor resin which is cured under increased temperatures and pressures. The prepreg materials are fabricated to specific fiber to resin volume ratio that is dependent on the application being used and farther layup varies. The differences between prepregs and VARTM, traditionally, are that prepregs require the application of positive pressure on the system, which serves to consolidate the part and move resin from the fiber surface to the voids. VARTM accomplishes the same using negative pressure. Positive pressure and temperature are normally applied by an autoclave, which is an expensive piece of equipment. VARTM is thought to increase the fiber/matrix interaction and decreases the percent of voids, which allows the composite matrix to efficiently transfer stress throughout the composite component. A key requirement to successfully utilizing VARTM is that the resin can thoroughly wet the glass fabric, thus displacing trapped air. As will be discussed, fibers that make up the

reinforcing glass fabrics in the composite are processed using a sizing coating, which serves to protect and strengthen the glass filaments as they pass through the yarn fabrication steps of their formation. We examined the differences of sizing chemistry on the various glass types to determine if the added sizing played affected final composite physical properties.

Different types of resins, including epoxy resins, polyester resins, vinyl ester resins, phenolic resins, and acrylics resins may be used in the VARTM process. Each resin has its own attributes (chemical and physical) and must possess good thermal and chemical stability as well as key viscosity behavior, during the VARTM process. Additionally, curing behavior varies with resin chemistry.

The material used as the reinforcement component in the VARTM process can vary, and fibers made from glass, carbon, to aramid polymers have been used. Selection of the fiber is often is based on a comparison of material properties vs. weight or density, as well as cost. As a reference point, the prices of composites are typically defined by the materials used, equipment used for fabrication, labor costs, and cost for testing of fabricated samples. Of these contributors, the highest cost driver is frequently equipment or tooling required for fabrication, followed by labor costs, and the varying cost of materials. For example of material variation costs, the price for square yard of carbon fiber plain woven fabric averages around \$35.00/yd^2, which is much higher than either plain woven glass (averaging around \$10.00/ yd^2) or the cost of the polymer epoxy resin at about \$12.00/kg.[31] The choice material involves choices between advantages and disadvantages depending on the function of the composite and thus the physical properties required to make the composite robust in the application and

environment of use. Tensile properties of composite structures based on various types of reinforcement materials (glass, aramid or carbon) and compared to other structural materials, are shown below in **Figure 3**.

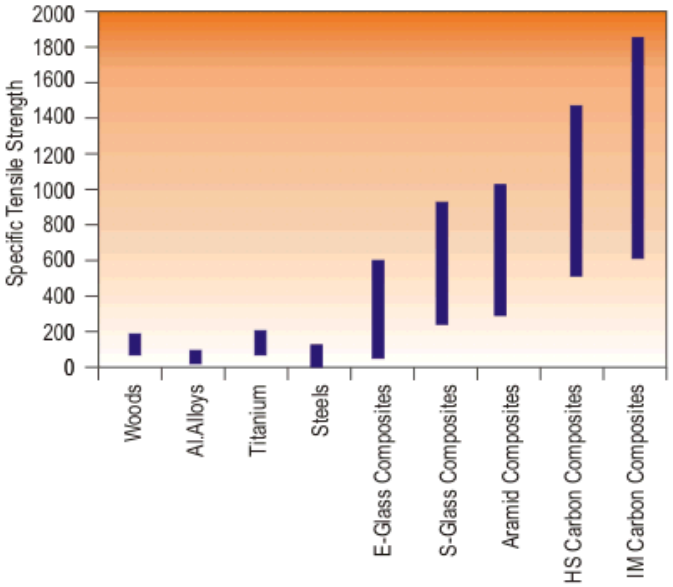

Figure 3: Comparison of tensile strength ranges between common composite materials

Glass is the most commonly used material as the reinforcement component of composites today due to its high density, low cost, good handling, and mid-range strength. There are differing grades of glass that are made into fibers that are typically differentiated by the constituents in the glass (and the amount of impurities). The mechanical properties of E-Glass, R-Glass, and S-Glass track with glass purity (low to high), thermal properties, and cost. In comparison, carbon fiber, the most expensive of the composite reinforcement materials, contains the best properties of the materials alternatives and thus, commands high prices. Carbon has some very beneficial properties to the overall structure that makes it unique, such as it's conductive nature, has high fatigue resistance, low impact resistance, a low coefficient of thermal expansion, a range of strengths, and also, low density.[12] Aramid fiber materials used

for reinforcement provide extremely high tensile strength and abrasion resistance. A comparison between reinforcing material properties are shown below in **Table 1**.

Table 1: Comparative properties of composite reinforcement materials: Grading (A-Best B-Average C-Poor)

Property	Aramid	Carbon	Glass
High Tensile Strength	B	A	B
High Tensile Modulus	B	A	C
High Compressive Strength	C	A	B
High Compression Modulus	B	A	C
High Flexural Strength	C	A	B
High Flexural Modulus	C	A	B
High Impact Strength	A	C	B
High Shear Strength	B	A	A
High In Plane Shear Strength	B	A	A
Low Density	A	B	C
High Fatigue Resistance	B	A	C
High Fire Resistance	A	C	A
High Thermal Insulation	A	C	B
High Electrical Insulation	B	C	A
Low Thermal Extension	A	A	A
Low Cost	C	C	A

This study utilized glass fabric as the reinforcing component and as stated above, there are three types of glass fiber grades, E-glass, S-glass, and R-glass. Each possesses advantages and disadvantages in regards to strength, modulus, and cost. The most widely used fiber component is woven E-Glass (electrical glass) because of the lower cost per yard of fabric, minimal moisture absorption rate, and effective mechanical properties per cost. S-glass (strength glass) has a greater tensile strength, modulus and elongation than the E-glass. The R-glass possesses an intermediate level of mechanical properties between the E and S glass.

Composites produced from the VARTM process can vary with the type of fabric used, the fabric weave, and the stacking sequences and orientations along with differing fabric weave types. The choice of composite design is dependent upon the desired application. There are multiple types of fabric layer structure and designs that can be used in composite design such as 0°/90° weave, unidirectional 0° only, braided ±30° braids, 0°/±45°/90°quasi-isotropic design.[12] Each of these fabric constructions have differing properties that have advantages and disadvantages depending on application or specific testing results being studied. For example of specific testing on individual components' properties, the unidirectional fabric designs has very high strength and stiffness in the 0° angle, yet considerably lower for the 90° angle, which is beneficial for testing individual tension data in regards to just the fiber component or just the matrix component tension strength. The quasi-isotropic design provides a more complete balance across the composite structures allowing stress to be distributed equally in all four directions.[12] The fabric orientation and stacking sequence is described in further detail in the following section.

As discussed above in section 1.2, composite materials are based on a matrix material with a reinforcing material embedded within it. In the present study, glass fiber fabric (plain weave) was incorporated into an eight-layer design, and infused with an epoxy resin. The specific attributes of the components used in the composite assembly process are discussed in this section.

1.3.1 Reinforcement (Fiber) Component- Glass Fibers & Woven Glass Fabric

The three glass types used in this study are based on fibers fabricated from bulk glass materials with differing chemical compositions. As seen in **Table 2**, the basic glass chemistry of the E, S and R glass fibers (which were woven into fabric form) used in this study, were different. These subtle compositional differences are known to affect both individual fiber properties and resulting glass fabric properties. In addition to the glass chemistry and fiber properties (assuming fiber fabrication procedure does not vary between glass types, thus yielding common formation-induced attributes regardless of glass type,) one might expect a common plain weave design from different glass types will result in woven fabric mechanical properties that are defined largely by the glass chemistry of the fiber type. For example, the higher amounts of silicon dioxide in the S-glass contributes to the increased strength and modulus displayed for the glass fibers from these glasses, as shown in **Table 3**.

As can be seen in **Tables 2** and **3** below, the variation in modifier type (alkali or alkaline earth oxides) or intermediates (aluminum oxide) will modify the extent of cross-

linking in the silicate network across these three types of glasses. Thus, while chemically similar, their compositionally-determined structure and properties, are indeed tied to resulting performance in both fiber and fabrics made from them. In discussing glass fabric properties for use as the reinforcement component in composites, it is presumed that individual glass fiber fabrication methods differ little; thus the properties of the fiber will be largely determined by the glass chemistry and structure. We can therefore assume that glass fiber chemistry type and any variation (none was used here) in fabric weave, will largely determine the overall fabric performance in the composite.

Table 2: Chemical composition of the three types of glass

Main Oxides (wt%)	E-Glass	S-Glass	R-Glass
SiO_2	52-62	64-66	55-60
Al_2O_3	12-16	24-25	23-28
B_2O_3	5-10	-	<0.2
CaO	16-25	**0-0.1**	20-24
MgO	0-5	**9.5-10**	1-4
Na_2O, K_2O	0-2	0-0.2	0-2
Fe_2O_3	0.05-0.4	0-0.1	0-0.8

Table 3: Individual Glass Filament Mechanical Properties of three glass types

Individual Glass Fiber Properties	E-Glass	S-Glass	R-Glass
Density (g/cc)	2.58	2.46	2.54

As defined by ASTM standard 1505			
Avg. Filament Diameter (µm) As measured by optical microscopy	17.14	9.76	12.26
Softening point (°C) As defined by ASTM C338	846	**1056**	952
Tensile Strength 23°C (MPa)	3445	**4890**	4135
Tensile Modulus 23°C (GPa)	72	**87**	86
Elongation (%)	4.8	**5.7**	4.8

As seen in Table 2, there are differences in the bulk composition of the glass fibers. For example magnesium oxide as opposed to the calcium oxide is used in the E-glass and R-glass formulations. Magnesium oxide is used in larger amounts in S glass instead of boron oxide (another former). In essence different formulations have been shown to affect important mechanical properties, such as elongation, tensile strength, and modulus. In terms of fiber manufacturing, higher silica content along with modifier type can increase not only the glass melting temperature but also the initial softening point of the glass, requiring fiber extrusion to be carried out at higher temperature. This then requires more energy in manufacturing, making the S-Glass more expensive to produce than the lower grades (E and R) glass types.

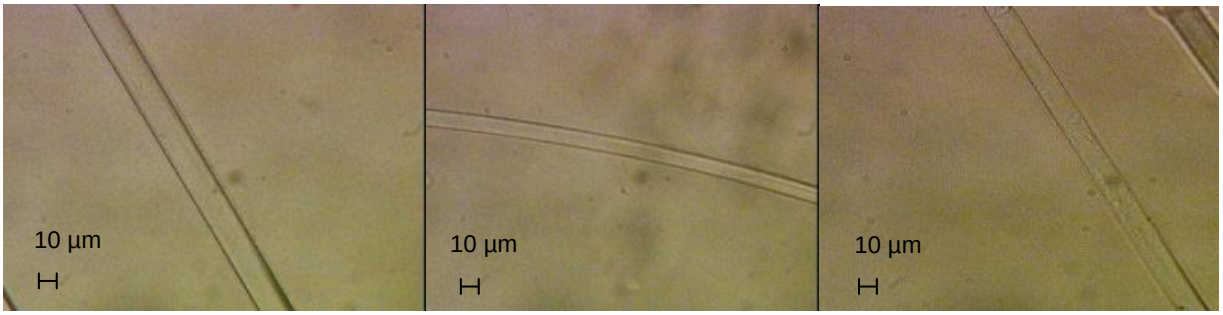

Figure 4:Optical micrographs of a) E-Glass b) S-Glass c) R-Glass to determine average fiber diameters

Figure 4 displays three optical micrographs obtained from inspection of E, S, and R-Glass fabric to determine the average fiber diameter. The fiber diameter is important when considering resin infusion and fiber wetting properties. Fiber diameter measurements were performed using optical microscopy. The results in **Table 4** show that the S-Glass has the smallest fiber diameter at approximately ten microns. This smaller fiber diameter translates into to an increased surface to volume ratio leading to increased interfacial area between the matrix and the fibers. This will presumably affect fiber substrate wetting behavior

Table 4: Fiber diameter data as determined by optical microscopy

Fiber Diameter	Average (microns)	Standard Deviation (microns)
E-Glass (um)	17	0.8
S-Glass (um)	10	0.4
R-Glass (um)	12	0.7

As stated earlier, this study investigates the resulting mechanical properties of composite plaques fabricated using SC-15 two part epoxy resin infused around a glass fiber fabric. The glass fabric consisted of one of three glass fiber types, either E, S, or R-glass, each possessing slightly differing chemical, physical and mechanical properties. A two-dimensional (0° Weft/90°Warp) plain woven glass fabric is used as

the fiber component, which is glass yarn woven in a design where the warp and weft are equal with regards to the fabric's directional strength properties, number of yarns per inch, linear density of the yarns used in warp and weft. For these reasons, it is assumed that the fabric's mechanical properties (realized from the fiber properties in **Table 3**) are primarily used in load bearing direction, and aligned accordingly during composite fabrication.

The fabric design for each of the three glass types (E, S, & R-glass) were held constant to specifically target differences between the glasses unaffected by weave design. The areal density, which is the dry weight (oz) per square yard of woven fabric, is held constant for each glass fabric type used at 24 oz/yd^2. The weave density of the fabric is defined as the number of filament bundles in the warp and weft direction per square inch of fabric, which can be used to calculate individual number of fibers in the each bundle.[8] The weave density for each of the plain woven glass fabric types were approximately constant at 5 x 5 warp/weft per square inch, though the E-glass is a slightly looser construction it is within the experimental error margins to be considered constant. All fabrics layers were layed-up using the same stacking sequence i.e, an eight-layer fabric design. The term stacking sequence refers to the order in which the orientation of eight fabric layers are constructed or stacked, with respect to their weave direction. For the stacking sequence used in this study load bearing stress can be equally distributed uniformly in all four angle directions(0°/90° & ± 45°). A schematic of a plain weave fabric is shown in **Figure 5**.

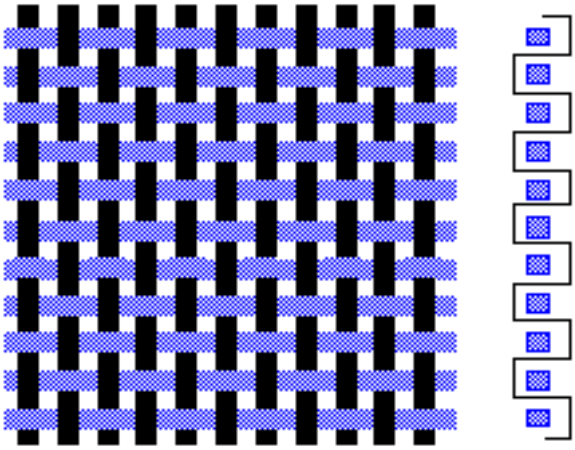

Figure 5: Diagram of Plain Weave (left) with over-under yarn pattern (right)

The stacking sequence of the eight-layer woven glass cut at the 0°/90° and ±45° angle orientations is displayed below in **Figure 6**.

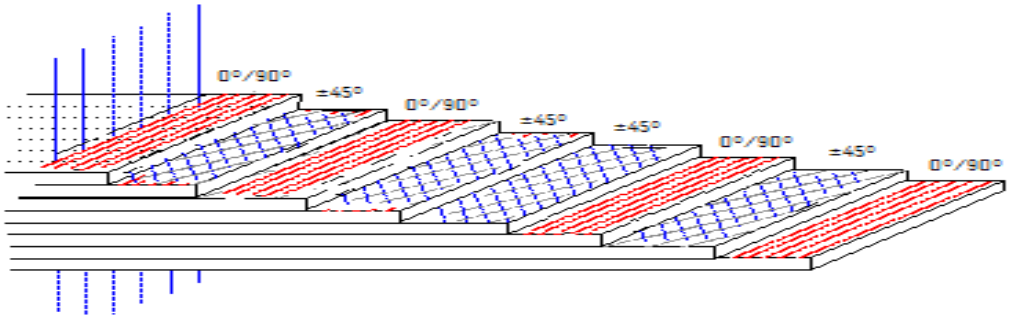

Figure 6: Woven glass stacking sequence for the eight layer layup design

The typical types of composites used in the targeted applications (wind and military) investigated in this study are composed on a multi-layered glass woven fabric with a polymer matrix component. The multi-layered glass fibers serve as impediments for crack propagation under stress in load bearing applications combined with the high impact toughness of the epoxy matrix.[34] The multi-layered fabric structure in the

applications investigated are of alternating weave angle orientation between each layer in the stacking sequence giving complete distribution of stress in all angles of fabric orientation when a load is applied (0°/90° & ± 45°). The low weight to strength properties of fiber reinforced composites serve as a more suitable material than the heavier metal alternatives.[30] The typical glass/epoxy composite designs for the applications, described in farther detail in chapter 2, will access these properties to decrease the weight to strength ratio and maintain the properties needed for the desired applications.

1.3.2 Matrix Component- SC-15 Two Part Epoxy Resin

The matrix component focused on in this study for the glass-resin composites in the VARTM process was SC-15 two part epoxy resin. SC-15 resin is composed of two parts defined as part A: diglycidylether of bisphenl A (60-70%) + aliphatic diglycidylether (10-20%) + epoxy toughener (10-20%) and part B: hardener + cycloaliphatic amine (70-90%) + polyoxylalkyamine (10-30%)[25]. The resin has a low viscosity, which is needed for practical infusion times and maximum fiber wetting to obtain a quality composite. The SC-15 resin has a relatively long pot life before curing making processing manageable and allowing complete wetting of fiber component before cure. SC-15 is specifically designed as a high impact loading resin making it ideal for both wind blade and ballistic applications.

Table 5 shows the mechanical properties of the SC-15 resin used in this fabrication process. A low viscosity is necessary for complete fiber wetting in the woven glass reinforcement during the infusion of the resin. Low resin viscosity enables residual air bubbles to rise upwards to the distribution layer and should result in a lower void content. The relatively long pot life of the SC-15 resin makes for ease of mixing, degassing, and infusion processes without gelling. The data presented in **Table 5** show some of the physical properties of the SC-15 resin precursor material.

Table 5: SC-15 two part epoxy resin properties Full set of properties found in Appendix A

Resin Type	Cured Density (g/cc)	Viscosity @ 77°F (cP)	Tg (Wet) (°F)	Tg (Cured) (°F)	Elongation (%)	Tensile Strength (ksi) / MPa	Young's Modulus (Msi)
SC-15 Two Part Epoxy Resin	1.09	300	178	220	6.0	9.0	3.8

When making a composite several properties are of interest, one of which is the matrix component that plays key roles in quality plaque fabrication. The void content is of interest as voids can negatively affect the strength, and other mechanical properties of the composite. The nature of the matrix has a large influence on the final void content of fabricated samples. Our targeted void content was 2% or less of the total sample volume. Several steps were taken to minimize void volume and these as discussed in further detail in the next section.

Chapter 2 - Literature Review

This study examines fiber reinforced composite material mechanical properties realized for plaques fabricated using a single layup design and an optimized manufacturing process using the materials described in Chapter 1. Discussed in this chapter is a short overview of the role of composites in two distinctly different applications that are of interest to this study – wind power and military applications. Also described are the testing methods used to assess composite material morphology and those attributes that are believed to impact mechanical properties of interest.

2.1 Composite Applications

The use of fiber reinforced composites materials varies in different applications. However, they serve the main function of reducing weight compared to alternative metal materials for structural applications and impact resistance while maintaining the strength needed for the application.

2.1.1 Wind Turbine Blades

Off-shore wind energy projects have become increased as a source of renewable energy that is cost effective and feasible. Wind represents an environmentally sustainable source of energy that is cost effective. Economic projections have shown that offshore wind energy has potential revenue in excess of $100 billion dollars in the materials and construction industry over the course of the next 30 years.[29] The need for stronger, lighter, cheaper materials is higher than ever with the renewable energy movement, which provides motivation for innovative research and design. Off shore

wind energy has seen significant reductions in manufacturing costs over the last decade, but advanced material research is required to lower total costs, in order to compete with nuclear and fossil fuel based energies.

Increased power requirements have driven the push for larger blade sizes, which requires lighter, stronger materials. The graph in **Figure 7** shows the growth trend of wind turbine size and power output over the last 30 years and visually displays the exponential increase in these areas, which motivates blade material research. There are several areas in which the blade structure is made up of glass/matrix composite materials, which are needed to support the structural load of the blade and environmental forces.[6] This study relates the key properties that the composite materials used in wind blade applications must maintain and the fabrication process that can produce high quality composite samples.

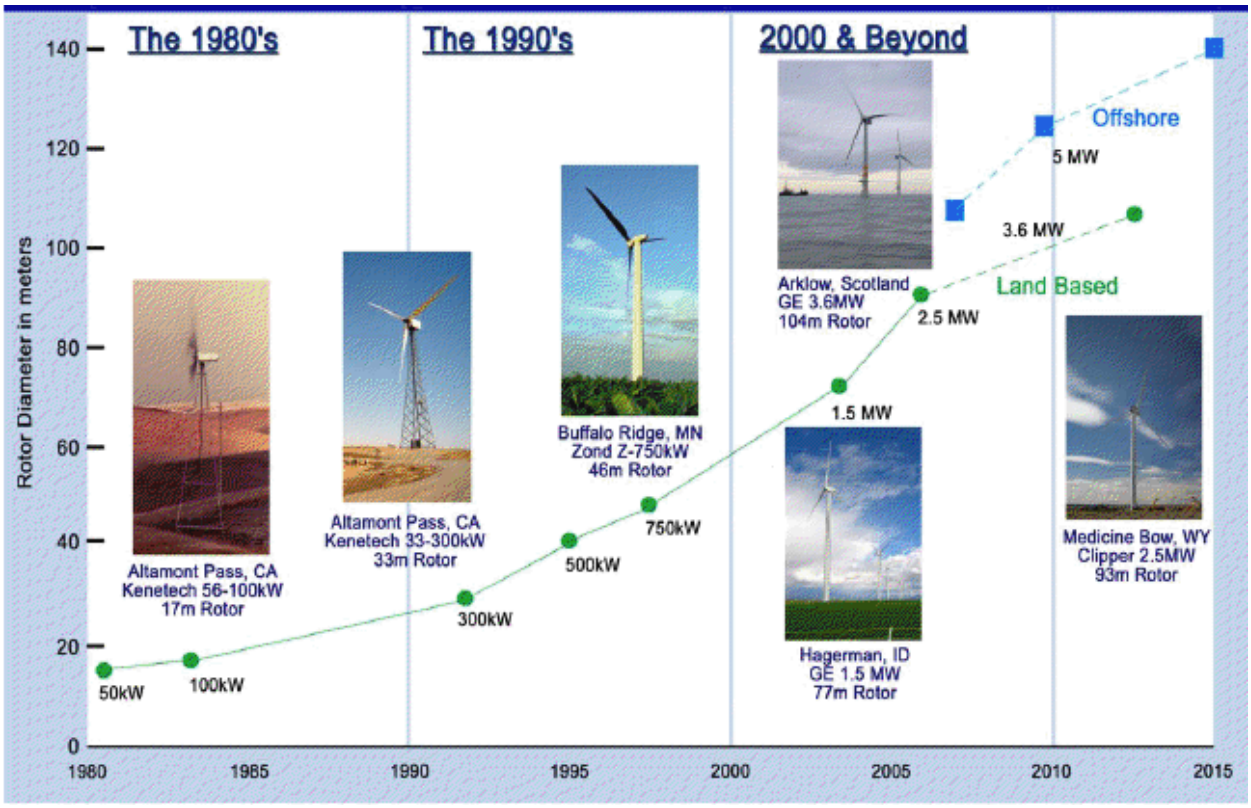

Figure 7: Current growth trend in wind turbine size and wattage

The composite system for a wind turbine blade has to maintain a structural load and have high fatigue life, survive in a high shear (stress) environment, possess good impact resistance, and maintain these mechanical properties over a broad range of temperatures and environmental conditions. Hence, key properties of composites for wind applications, which this study's findings support, include composite density, (which when considering the density of the structure's constituents includes fabric, resin and void density), void content, fiber volume fraction and Young's modulus. Each of these properties are discussed in more detail in section 2.2.1.

The main reason for increased use of composite materials in wind blade parts is to increase the system lifecycle by utilizing materials with greater fatigue properties.[5] Fatigue fracture is a result of repetitive loading at levels below the ultimate strength of the material. Even though the loading is less than the ultimate strength, damage accumulates in the part, resulting in failure at loading levels less than the ultimate strength. Multi-layered fiber reinforced composites increase fatigue resistance due the multiple layers of the fiber component preventing the crack from progressing until material failure. The matrix component, which is exposed to the environment, must survive temperature fluctuations, humidity, precipitation (rain, ice), and chemical degradation from UV (sun) exposure. A high level of interfacial bonding between the matrix and the fibers is insured by appropriate choice of sizing for the resin used matching the reinforcement component.[6]. Methods for testing large blade fatigue life include imposing a mechanical load normal to the composite blade surface layer with automated hydraulic cylinders in cycles.[10]

The current state-of-the-art blades are approaching 85-105 meters in length per blade.[10] This study employed the current blade manufacturing process, which is the VARTM (Vacuum assisted resin transfer molding) process , shown below in **Figure 8**.

Figure 8: Set up of VARTM process rotor blade for wind turbine

The photographs in **Figure 8** demonstrate the extent to which the VARTM process can be scaled up.[5] The rotor blades are infused by parallel resin channels and cured in one half sections molded to the overall blade design shape.

2.1.2 Ballistic Impact Protection

Multi-layered glass fiber reinforced composites are currently being researched and implemented for impact protection on Humvee and other armored vehicles, in parts such as door panels and hoods to reduce weight . Previous armored vehicle protection was manufactured primarily of heavy steel structures to serve as both load bearing structures and impact protection. The weight of the vehicle plays key roles in both vehicle speed and maneuverability as well as cost reduction in fuel consumed.[32] A term which the military refers to as “deploy-ability” is an important consideration where use of

composite can play an important role; it refers to the fact that the weight of a vehicle can affect the viable transportation methods for getting the vehicle to the battlefield, which can then affect the time required to deploy the asset. This latter issue, deploy-ability, is going to be a huge driver when the Army designs their next tank. When compared to the metal alternative, composites offer multiple advantages as armored parts by reducing the weight by 27%, increases the survival rate of personnel by reduction in fragmentation, reducing manufacturing costs approximately 20%, improving cabin noise resistance, and providing better thermal insulation.[33]

Composite materials play two main roles in the manufacturing of armored military vehicles, one being the load bearing structural component and the other being impact resistance against multiple types of projectiles. The maintaining of the structural load properties after a projectile has caused damage to the material is another key requirement for ballistic composite parts. The composite structure must also withstand impact from a wide range of projectiles from bullets to explosive shrapnel; therefore, the ultimate fracture stress of the composite is a key property to be investigated.[32]

2.2 Mechanical Properties in Composite Applications

2.2.1 Wind Turbine Blades Properties

Composite materials in the wind blade applications serve as a load bearing structure as the outside skin of the blade as discussed in the previous section. Key properties that were investigated in this study were those directly affecting the load bearing properties, which were ultimate tensile strength, Young's modulus, and void

content. Knowing composite load bearing data and how it relates to the materials used in fabrication, size limitations and design can be determined for wind blade applications.

The ultimate fracture strength must be able to surpass the external loads being applied such as the structural weight of the blades and parts themselves, wind and environmental forces, and maintain these properties over time and ranging temperatures.[21] The modulus plays important roles for the impact resistance of the blades, by withstanding impacts of hail, sleet, and birds without causing mechanical failure. Ultimate fracture stress and modulus are key properties to be investigated in composite research in order to continue the growth trend in wind blade size in **Figure 7**. Voids cause points of weakness during delamination or crack propagation in the composite system, resulting in a lower fatigue life. Fatigue life is a key property in wind turbine blade testing. It was not possible to investigate fully the fatigue characteristics of the composites fabricated for this study, due to time and funding constraints. The fatigue life for composite parts are modeled to last at minimum 20 years, and average ranging from 20-30 years, but all composite parts vary in time due to differing environmental effects and external loads.[18]

2.2.2 Ballistic Impact Properties

Ballistic testing on fiber/matrix composites differs from other common material mechanical testing methods and the understanding of the impact mechanism between projectile and material does not always exist. Current research and experimental models have approximated material mechanical properties based on high strain rate testing.

Modeling ballistic impacts on composite structures varies with the application involved and the specific researcher. A first level screening tool developed by Cunniff, defines a dimensionless fiber property (U*), which is the product of specific fiber toughness multiplied by strain wave velocity shown in **Equation 1** below. This equation provides researchers with a first approximation of how material properties contribute to U*.

$$U^* = \frac{\sigma\varepsilon}{2\rho}\sqrt{\frac{E}{\rho}} \tag{1}$$

In equation 1, the variable E is the composite Young's modulus, σ is the ultimate fracture stress, ε is the ultimate fracture strain, and ρ is the composite density, all of which were investigated in this study. These experimentally determined values can be found from the testing results for materials prepared in this study, in **Table 14**.

The ASTM standards that are commonly used to investigate these two distinct applications are similar. Those ASTM standards used to assess material part mechanical properties in fiber reinforced composites are listed below. Although not all of these tests were performed in the present study due to cost and sample quantity restrictions, the first five tests in **Table 8** were performed and are discussed in more detail in Chapter 3.

Table 6: ASTM Standards for testing physical properties of glass/resin composites. Test shown in BOLD were used in this study.

1 ASTM 2734 Void content	**2 ASTM 2584 Fiber volume fraction**	**3 ASTM 792- Density**	**4 ASTM 3039 Tensile testing for polymer matrix composites**
5 ASTM 6484 Compression testing for polymer matrix composites	6 ASTM D 696- Dimensional Stability	7 ASTM 1269- Specific Heat	8 ASTM 1225- Thermal Conductivity
9 ASTM E 84- Flammability and Smoke Generation	10 ASTM D 149- Electrical Properties	11 ASTM D 3518- In-Plane Shear Strength and Modulus	12 ASTM D 5379- Out of Plane Shear Strength and Modulus
13 ASTM D 2344- Short Beam Shear Strength	14 ASTM D 790- Flexural Strength	15 ASTM D 5528- Fracture Toughness	16 ASTM D 3479- Fatigue

In the next chapter the first five of these properties' experimental procedures will be explained in more detail and the results are presented in Chapter 4.

Chapter 3 - Experimental Procedure

In order to assess the physical property uniformity this study aims to quantify a set of tests were conducted on specimens meeting the void content acceptance criteria. The statistical analysis of the tests, obtained by comparison of the standard deviation within each test across fabric types, was chosen to evaluate if the optimized VARTM assembly process developed in this study yielded high quality specimens. These values then could be compared to the typical property values for the glass type shown in the previous section (**Table 3**), The key questions the study aimed to answer are repeated here:

1. How does the type of glass (E-Glass, S-Glass, and R-Glass) in the composite matrix influence the (mechanical) property values of individually tested composite samples?

2. How does the composite property uniformity vary with glass/fabric type, within and across different sets of test samples?
3. Does the defined composite plaque design and resulting mechanical property performance meet criteria and specifications (as defined by ASTM Standards and/or industry accepted metrics) for use in the defined application (military system or wind application)

An optimized manufacturing process was developed and post-fabrication testing was employed to assess physical property variations that would provide an assessment for sample set uniformity. This chapter discusses the specific attributes of the materials used, the testing methods employed to assess material and property uniformity within

and across sample types and lastly, the specifics of military and wind test standards are described and the key property attributes required to assess whether resulting plaques would meet such standards are discussed.

As discussed in Chapter 2, the following key mechanical properties are important in both wind turbine and military protection applications. To assess these properties from a statistical significant number of samples, prepared using an optimized plaque fabrication protocol, this study employs the following materials and design approach. I have defined an experimental matrix that yields a statistical significant sample set to from which to assess variation, if present, among the composite plaque samples.

3.1 Materials & Process

Composite plaques fabricated for this thesis were based on the use of a VARTM process for a single layup design. The goal of the effort aimed to evaluate the quality of the fabrication process, for a consistent design to result in high quality composites. High quality composites for purposes of this study, were defined as having low void content, high ultimate tensile strength, and high modulus, A three month trial and error optimization period was conducted programmatically changing the process variables which lead to the experimental design and sample dimensions shown below in **Table 6**. Further details of the process are summarized in Appendix D.

Table 7: Experimental design for quality composite samples

Material	**Matrix**	**Fabric Design**	**Layer Construction**	**Thickness**	**Sample Dimensions**
E-Glass S-Glass R-Glass	SC-15	Plain Weave 0°/90° and ±45°	8 Layers – 0°/90°, ±45°, 0°/90°, ±45°,±45°, 0°/90°, ±45°, 0°/90°	0.25 in	14'' x 36''
Epoxy Mixture	**Tack Strips**	**Non-Stick Layer**	**Breather Layer**	**Distribution Layer**	**Infusion Temp**
Part A: 1923.0 g Part B: 576.0 g	20.5 x 45.0 in.	3 Layers-18.5 x 43.25 in.	1 Layer-18.5 x 43.25 in.	1 Layer-14.5 x 40 in.	97-107°F

A) Reference Layup Process

The study has aimed to evaluate property uniformity utilizing a single layup composite plaque design, three sources of glass fibers and a single, industry accepted resin precursor. All other process variables were held constant to adequately quantify trends within and across glass types. The fabricated composite sample dimensions and constants are listed above in **Table 6** and the stacking sequence of the materials used is displayed in the **Figure 9** to aid in defining the VARTM process explained in this chapter.

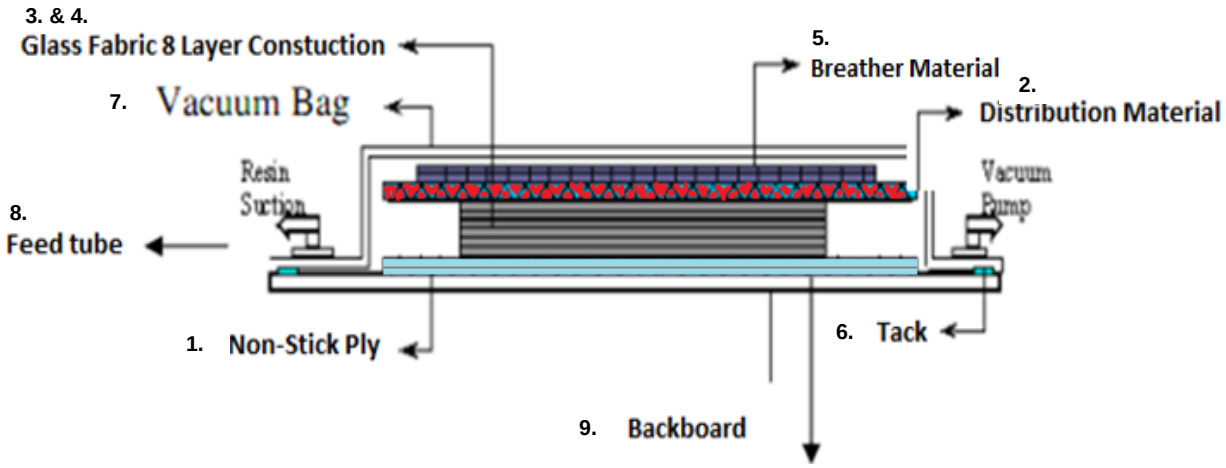

Figure 9: Schematic display of Overall Composite System

The material layers depicted in **Figure 9** are listed below. The central gray layers of the eight layer fabric stack shown in **Figure 6**, is shown within the overall composite design, in **Figure 9**. Each of the parts of the layup design that make up the resulting composite, are defined here, and discussed below. The numbers of each of the components in the list below correspond to parts shown in **Figure 9**.

1. Teflon/Nylon Non-Stick fabric
2. Distribution fabric
3. 4 Sheets Plain Weave 0° E-Glass fabric
4. 4 Sheets Plain Weave ±45° E-Glass fabric
5. Non-woven breather fabric
6. Two-Sided Adhesive tack
7. Vacuum bagging
8. Vacuum tubing
9. Aluminum Backboard
10. SC-15 Epoxy (100:30g w/w Part A to Part B)
11. Curing Oven
12. Resin Trap

*The source for the material/component definitions above are listed in **Appendix B**.

The process was completed in the fabrication order shown below in **Figure 10**. The sample sets (for each fabric type) were fabricated separately to ensure property uniformly across sample sets of each type of class to obtain a repeatable fabrication process. As shown in **Figure 10**, the final assembly sequence which changed the type of fabric used (rather than making all of one type first, and then sequentially moving onto the second and third fabric type), ensured any subtle process variation (realized through repeated use of the process) were averaged out. In total, 12 composite plaques utilizing each fabric type (E, S, R) for a total of 36 plaques were ultimately completed using the final optimized process. These 36 samples formed the test specimens for subsequent testing.

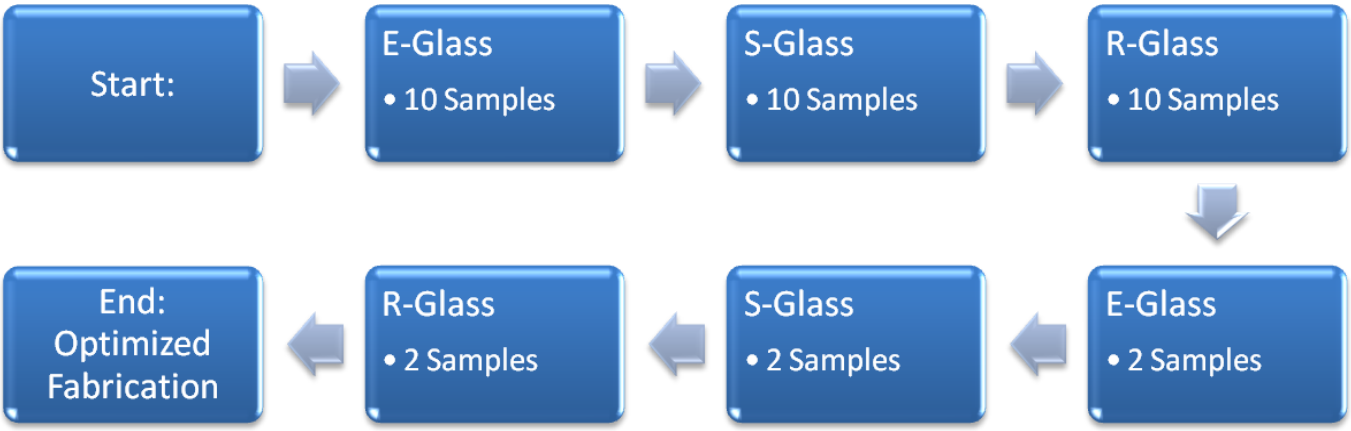

Figure 10: Optimized order of fabrication composite samples; in total, 12 plaques from each glass type were assembled.

B) Layup Process Details

To ensure assembly repeatability leading to uniformity in resulting plaque properties, each step in the composite lay-up process was refined and subsequently reproduced for the 36 plaques made. The details of each step are discussed here.

a) Cutting and preparing the fabric

The first step in the VARTM process is cutting and preparing the woven glass fabric layers in the specified orientation and dimensions. This step is important to insure accurate measuring and cutting of orientation angle with minimal skewing while handling and constructing the indicated stacking sequence.

1. Cut 4 panels of the 0°/90° woven E-Glass
2. Cut 4 panels of the ±45° woven E-Glass
3. Lay the cut fabric sheets on the backboard in the order shown in **Figure 6.**

Shown previously in **Figure 6** are the resulting eight layers of glass fabric used in the design. This structure makes up fiber component as the inner-most region of **Figure 9.**

b) Laying up the composite on the backboard

After preparation of the glass fabric was complete, the laying up of the composite with the materials indicated below was the next step in the VARTM process. This step was vital to the fabrication quality of composite samples and careful handling and assembling of each material was needed. Photographs of the process steps are show in **Figure 11** and the process of laying up the composite is displayed in the flow chart in **Figure 12.**

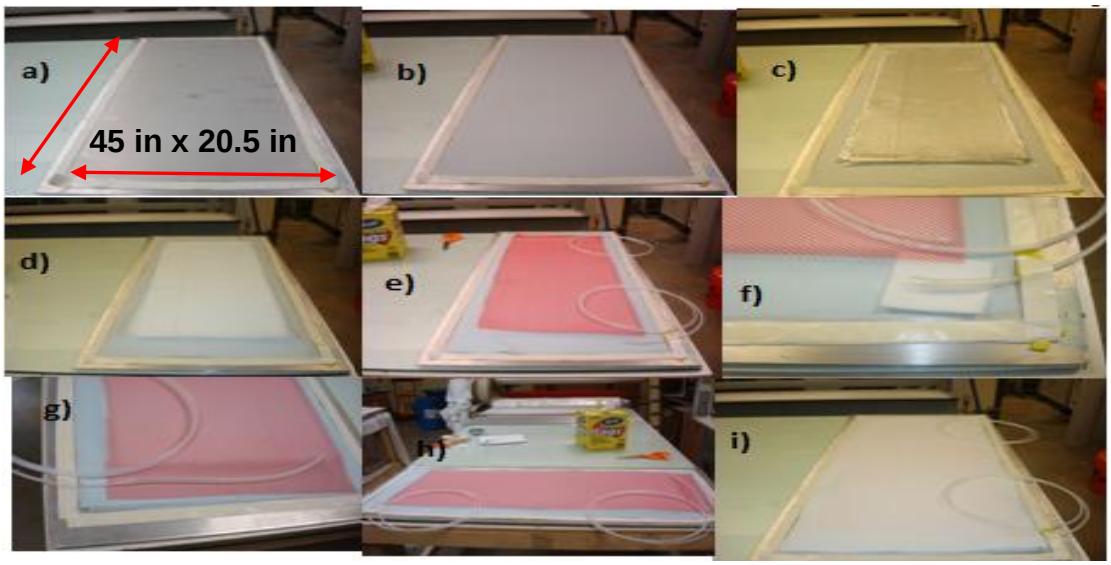

Figure 11: Photographic display of VARTM process steps. The dimensions of the completed lay up prior to infusion (dimensions of specimen 20.5 in wide x 45 in long.)

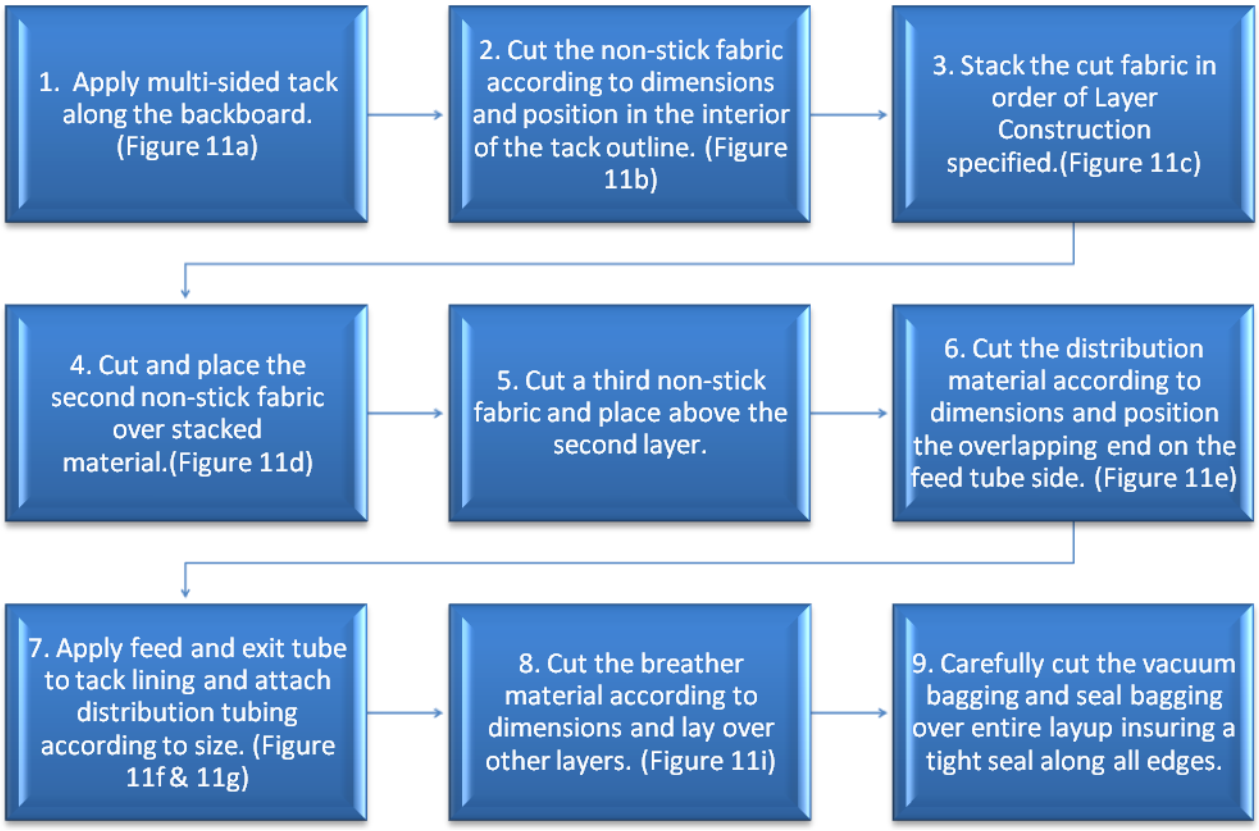

Figure 12: Optimized layup design process steps that coincide with the photos shown in Figure 11

The proper stacking and careful cutting of the materials used in preparation of the composite specimen affects the final quality by allowing proper displacement and transport of residual air bubbles through the resin. The majority of the material dimensions and material placement as described in **Figure 12** were held constant throughout the process, the exceptions being the breather layer and the non-stick layer.

The breather material serves as a porous layer to prevent the vacuum bag from sticking to the distribution material and aids in surfacing air bubbles. The initial breather layer was constructed as multi-layered narrow strips boarding the outline of the specimen. This non-uniform wetting of the layup during the infusion due to the absorption of resin in the breather material along the sides was observed. The adjustment to the stacking preparation was to apply a single layer of breather material that covered the entire area (final dimension shown in **Table 6**) of the specimen lead to a more uniform infusion and wetting of the fibers during infusion.

The addition of one additional non-stick layer was implemented to aid in the debagging step of the process. The original process of two non-stick layers, one on the bottom of the glass stacking structure and one layer on top, which made debagging troublesome. The addition of the third non-stick layer was placed on top of the previous first layer creating a two ply non-stick layer that is easily removed from the adjacent non-stick layer opposed to the initial removal of the nonstick from the surface of the composite when debagging.

c) De-bulking the specimen

This step of the process is the loading the specimen in the oven and the removing of air in the constructed composite specimen though physical compaction. It is important that the entire system is vacuum sealed and debulking is preformed to remove most of the residual air in the system so as to minimize void content. The heating of the materials in the composite specimen is also important since the viscosity of the resin could change on contact with other colder materials in the stacked lay-up. It was determined that it was best to do this step inside a curing oven. In this way a vacuum could be continuously applied to the system throughout the curing process with the curing oven doors closed. Steps in the process included:

1. Take the vacuum tube that leads from the backboard and attach to the resin trap.
2. Attach secondary vacuum tubing from resin trap to a vacuum source.
3. Clamp the end of the feed tube to allow the vacuum to build.
4. Engage the vacuum source and allow the system to de-bulk for 15 minutes before introducing the resin. This ensures that excess air leaves the system.

The end result of the de-bulking phase should be an approximately constant vacuum (25 psi) held in the vacuum bag and the formation of a tightly fit vacuum on the specimen. There should be no sounds of air leakage from any point in the system and can leaks can also be identified by any drop in pressure is due to air intake somewhere

in the vacuum sealing. Trapped air that remains in the stack following de-bulking (if any) serves as a possible void formation location during resin infusion.

d) Mixing and Degassing of SC-15 resin

The process parameter that most strongly affected the optimization of the VARTM process was the addition of the degassing phase to the epoxy resin before the infusion into the specimen. The degassing of the resin was the process of pouring the mixed resin parts into a sealed resin pot attached to a vacuum line and vacuum source. A 30 minute hold under vacuum was allowed to remove as many residual air bubbles suspended in the resin as possible. Removal of initial air bubbles before infusion ultimately lead to reduced voids entrapped in the specimen during infusion. Prior to implementing a degassing phase, the fabricated composite samples had visible surface voids indicating that internal void content was unacceptably high. The degassing phase was conducted as followed:

1. Follow safety procedures to load the Part A SC-15 resin into pouring position.
2. Place the scale and bucket container below the container and measure out 1923.0 grams of Part A then poured slowly at a tilt into the Degassing container pot.
3. Follow safety procedure (Located in lab) to load Part B SC-15 resin into pouring position.
4. Measure out 576.0 grams of Part B and pour slowly at a tilt into the Degassing pot.

5. The mixture was stirred exactly 100 times over 1-2 minutes time.
6. The lid was sealed to the pot and the vacuum line was attached.
7. Vacuum was employed for 30 minutes at a reduced pressure of approx. 25 (psi).

The amount of resin mixture was adjusted to reduce waste and aid in the minimization of the void content in the composite. The process of letting the resin flow for a further 15 minutes after complete wetting of the system required more resin than initially calculated for complete wetting. This increases the cost of resin per sample being fabricated but results in a lower void content. This step was necessary for the fabrication of high quality samples. In addition, the extra time allowed for the resin to flow through the system also gave more time for residual trapped air bubbles to be transported out of the distribution material.

e) Infusing and Curing the composite

The objective of the infusion step of the process is to infiltrate the glass fabric with the resin matrix. Wetting of the glass fiber is accomplished by the applied vacuum. The infusion process is described below:

1. Take feed tube leading from the backboard and place into resin reservoir.
2. Remove feed tube clamps to allow the vacuum access to the resin.
3. Set Oven to Infusion Cycle. Wait for the resin to completely wet the system and be drawn into the vacuum tube on the opposite side.
4. Allow to infuse until the resin has completely wetted the system and is flowing into the resin trap

5. As the backboard is already in the oven, ensure that the feed tubing has access to the reservoir. Also ensure that all openings have been sufficiently insulated to prevent heat loss.
6. Ensure that the vacuum tubing has a way to exit the oven and is connected to the vacuum source while the oven doors are closed.
7. Close the oven and start the cure cycle. The complete, stepwise cycle for the cure process is shown in **Table 7**.

*Note: Air bubbles in the vacuum tube leading to the resin trap are expected. They are the result of dissolved air in the resin coming out of solution.

Table 8: SC-15 Two Part Epoxy Curing and Post Curing Temperature Cycles

Cycle	Time 1	Time 2	Time 3	Time 4	Time 5
Infusion	Ramp to 95°F hold for 100 mins.	Set to Cure Cycle			
Cure	Initially 95°F. Hold for 10 mins.	Ramp to 140°F over 90 mins. Hold for 120 mins.	Ramp to 250°F over 28 mins. Hold for 180 mins.	Cool down to 70°F over 36 mins. Hold for 10 mins	Set to Post-Cure Cycle
Post-Cure	Ramp to 180°F over 90 mins. Hold for 14.5 hrs.	Remove from oven			

The infusion and cure cycle temperatures in **Table 7** are set specifically for the SC-15 two part epoxy resin; other resins should have different cure cycles.

f) De-bagging the composite

The de-bagging step of the process is where the cured composite plaque is separated from the other materials used during fabrication, including the vacuum bagging, spacer material, distribution material and the non stick layers. The removal steps are described below:

1. Turn off the vacuum and disconnect the vacuum tube from the resin trap.
2. Remove the backboard from oven.
3. Remove the vacuum bagging, adhesive strip, all tubing, distribution material, spacer material, and non-stick fabric from the composite. The nonstick fabric should allow this process to be relatively easy.

1.3.3 Sources of Error or Uncertainty

In the laying up step of the composite specimen preparation, there are several sources of error or uncertainty brought on by the following variables. The impact of each are briefly discussed as they could influence final part quality and physical/mechanical property uniformity, a primary target outcome of this project:

1. Difference in Fabric Orientation Angle

When preparing glass laminates, the exact angle of orientation of the glass fabric when the pattern is drawn and cut can be skewed from initial angle measurements due to handling and cutting. Delicate handling and cutting along with careful placement of the glass laminate when laying up the specimen can minimize the changing of the fabric orientation angle. The skewed angle offset from the initial pattern can disrupt overall

stress distribution when a stress load is applied or ballistic impact on the composite material. Use of a sharp cutting device is required to minimize skewing.

2. Residual air

In the degassing stage of the process, residual air not removed from the resin must be accounted for as a source of uncertainty. The small amounts of remaining air as residual small bubbles in the degassed resin are variable in two ways, visual bubbles on top of the degassed resin and dissolved air in the resin. To minimize air bubbles into the system, feed tubing is placed at the bottom of the feed bucket since bubbles naturally rise to the top of the volume. The amount of dissolved air in the resin after 30 minutes of degassing was unknown and therefore it was considered to be a source of uncertainty.

3. Mixing of the Resin

In an attempt to mitigate any thixotropic effects i.e., viscosity variation due to shear thinning, 100 manual stirs of the 100:30(g) Part A and Part B mixtures of the two part resin were made. Even though the number of stirs was held constant, there is a level of error in mixing uniformity between each sample due to human error. To minimize variable mixing, we used similar stirring rod along with an electronic constant stirring motion.

4. Temperature of Infusion/Cure

The oven used during the experiment had minor fluctuations of ±3°F from the initial set infusion temperature of 97°F. These temperature changes may affect the viscosity of the resin, which can in turn affect the overall infusion process. To minimize fluctuations in temperature, keep door opening to a minimum and keep oven damper levels constant.

1.3.4 Assembly Process Optimization – Final Result

The process described in the previous section indicates the order and key process parameters in each phase of the VARTM process used to fabricate uniform samples acceptable for mechanical testing. The process described above and shown in **Figure 13** was optimized over a three month period. To fabricate a single plaque, the total fabrication time was approximately 2.5 hours.

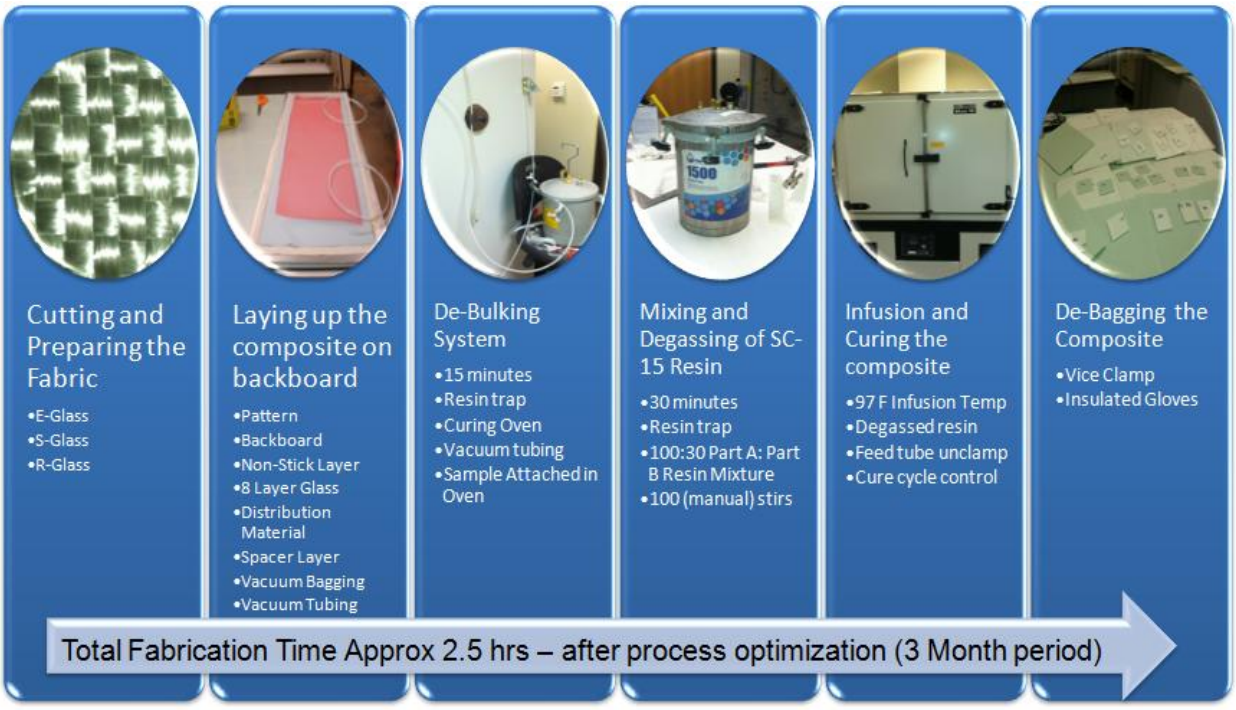

Figure 13: Flowchart of Optimized VARTM Process

Using the design discussed in section 1.3.3, each aspect of the composite fabrication process was reviewed for repeatability. While some steps (i.e., cutting the fabric and laying it up in the stated 0°/90° and ±45° orientations) is readily repeated regardless of fabric type (E, S or R glass), small variations in each step can impact resulting within-plaque properties (possible variations, for example at the edge of the plaque versus the middle region) and plaque-to-plaque property variation within glass type sets. As this study aimed to assess both within plaque property variation and plaque-to-plaque variation, special care was taken to control and repeat specifics of each step in the process. Details of the steps, and where minor variations might affect the noted variations in the resulting plaques (36) that were fabricated and tested for their physical properties, are discussed in the previous section.

The VARTM process took into account multiple parameters that were added and/or adjusted to lead to the repeatable fabrication process for quality samples for testing. All of the parameters served a different function in the process and the altering of any of the parameters above in **Figure 13** will affect the final composite sample. The detailed definitions of the materials used are described in **Appendix B**.

The key parameters that were investigated that affected the quality of the sample were the addition of the degassing phase, dimensions and placement of the spacer material, and the amount of resin needed for low void content in the resulting composite plaque. As quality control was an important aspect in fabrication, the resulting changes were necessary to meet the acceptance criteria of <2% void content.

3.2 Methods

The summary of properties evaluated in this study are shown below in **Table 9** with their corresponding ASTM test methods, instruments used to perform the testing, and the objective for conducting the tests.

Table 9: Property Evaluation and Data Analysis of ASTM Standards

Properties Examined	ASTM Standard utilized	Instrument Used / Number of Specimens Tested	Investigative Reasoning

P1- Void Content (%)	ASTM 2734	Fiber Burnout: Electric Muffle Furnace 4 Composite Specimen	Voids represent trapped air bubbles in composite matrix. Voids are believed to cause initial crack propagation and resulting in mechanical failure under increased stress.
P2- Fiber Volume Fraction (%)	ASTM 2584	Fiber Burnout: Electric Muffle Furnace 4 Composite Specimen	Achieving the optimum amount of fiber component while maintaining strength reduces overall weight of material. Defines what fraction of composite is fiber in form
P3- Density (g/cc)	ASTM 792	Archimedes Method 4 Composite Specimen	Reducing weight while maintaining strength in composites is required for larger wind blade structures and lighter ballistic paneling for vehicle parts
P4-Young'S Modulus (ksi) / (MPa)	ASTM 3039	Instron Tensile Testing DIC Camera Detection 7in Gauge Length 6 Composite Specimens	Modulus is a key property in ballistic impact performance. Increased modulus aids in high strain rate impacts from projectiles.
P5- Tensile Strength (ksi) / (MPa)	ASTM 3039	Instron Tensile Testing DIC Camera Detection 7in Gauge Length 6 Composite Specimens	Both load bearing wind application structures or ballistic protection requires high fracture stress resistance

*****Other important properties not examined in this study :

- Fatigue Life- ASTM 3479
- Compression Testing- ASTM 6484
- Fracture Toughness- ASTM 5528
- In-Plane Shear Strength- ASTM 3518
- Dimensional Stability- ASTM 696

To assess the quality of the optimized composite manufacturing process developed for the glass fiber reinforced epoxy resin materials examined in the present study, quantitative measurements on resulting parts were made. 36 plaque specimens were fabricated and qualitatively shown to have sufficiently low (<2%) void content to warrant further testing. Results of tests performed to assess (a) within- plaque, (b) plaque-to-plaque property uniformity (to assess assembly process) and to quantify

variation between plaques fabricated with (c) differing types of glass fabric are presented here. In all cases, the optimized fabrication procedures described in section 1.3 were employed on identical size plaques, of the single defined design (shown in **Figure 6**).

Table 10: Outside testing names and locations

	P1-Void Content	**P2- Fiber Volume Fraction**	**P3- Density**	**P4- Ultimate Tensile Strength**	**P5-Young's Modulus**
Test Name	Fiber Burnout	Fiber Burnout	Archimedes	Instron-DIC Camera Detection	Instron-DIC Camera Detection
Company/Location	Army Research Laboratory - Aberdeen Proving Ground, MD	Army Research Laboratory - Aberdeen Proving Ground, MD	Army Research Laboratory - Aberdeen Proving Ground, MD	Army Research Laboratory - Aberdeen Proving Ground, MD	Army Research Laboratory - Aberdeen Proving Ground, MD
Number of Samples	Four	Four	Four	Six	Six
Sample dimensions	25.0 x 5.5 mm	25.0 x 5.5 mm	25.0 x 5.5 mm	25.0 x 5.5 mm	25.0 x 5.5 mm

Tests carried out include experiments on fabric properties (evaluation of differences in fiber diameter, wetting behavior and sizing chemistry) as well as void content via SEM (performed within Clemson's MSE analytical laboratory (K. Ivey) and Electron Microscopy Center, respectively), as well as numerous analyses performed at external laboratories. The up to date summary of tests performed and locations are shown in **Table 10.**

The sizing added to the glass fibers plays a key role in the interfacial bonding between the matrix and the fiber components as well as the wetting behavior during

infusion. To investigate the sizing composition and relative quantity added to each of the glass types, a micro FT-IR experiment was conducted. Samples from each of the glass fabrics were prepared by removing a fiber bundle from the edge of the fabric that was then placed in an appropriately labeled vial. The glass sizing was stripped from the glass by adding a chloroform solvent at room temperature and allowing a hour to dissolve the sizing. Once the sizing was dissolved, a liquid film was cast on a KBr window and inserted into the FT-IR instrument. The resulting FT-IR graphs and targeted peaks were then identified by matching to reference patterns.

The first key property investigated in this study was the resulting void content and the optimized parameters to minimize voids. Void content was measured by the fiber burnout method using ASTM 2734. Four composite samples of each glass type were evaluated for void content and fiber volume fraction using the fiber burn-out test method. Samples were shipped out and measurements performed at the Army Research Laboratories (ARL) at the Aberdeen Proving Ground, Maryland. Tests were conducted by Jim Wolbert. Due to costs of testing and limited testing samples only four specimens of each glass type were tested to determine composite void content and fiber volume fraction. The sample dimensions cut for testing varied throughout each sample set of 12 fabricated The fiber burnout method involves weighing the composite sample, burning off the matrix component using a furnace that burns the matrix in an oxidizing environment. Previous known data on matrix degradation was used to determine time and temperatures needed for complete volatilization of the resin during the fiber burnout method. Remains of char (ash) of the resin can be calculated from known char values. The fiber and matrix fractions measured were used to calculate the

remaining void fraction. Voids in the composite matrix represent trapped air bubbles which remain following infusion and cure, that serve as weaknesses in the composite system. Voids are believed to be weak links where crack initiation and subsequent propagation can occur, resulting in mechanical failure under increased stress of an applied load. Along with initial crack propagation, void presence in the system decreases the interfacial bonding between the matrix component and the fiber components resulting in the partial delaminating of the composite structure. When a load is applied to a composite structure weakened by increased void content, the composite sample mechanical properties result in failure at a lower value.

ASTM 792 testing procedure was followed to measure the composite sample densities when using different glass types. The method for obtaining the composite density was measured using the Archimedes method of water displacement where the composite sample and composite sample in water masses were measured to calculated density.

The tensile data was obtained by using an Instron Tensile Tester set on a 7 inch specimen gauge length attached to a Digital Image Correlation (DIC) camera detector. The DIC camera is the post test analysis to determines the stress level at which the material fails is the ultimate strength, and is determined by monitoring the maximum stress applied. The DIC camera measures displacement during the test, which is used to calculate strain, which is then used to calculate modulus. The test measures the relationship of increasing strain on the composite material to the resulting stress until ultimate failure. The ultimate tensile strength is the breaking point of failure when a given strain is induced. The resulting stress exceeded the material limits and the

modulus is the initial slope of the stress-strain curve. The Young's modulus of the composite is determined by initial slope of stress versus strain cure, displayed in **Figure 2**. Reported below are the findings realized by each of the tests performed on composite plaques.

Chapter 4 – Results and Discussion

In this chapter, the results from the experiments described in Chapter 3 are presented and analyzed. Resulting trends from the data collected were established in order to adequately answer the three questions investigated in this study.

4.1 Glass Fiber Sizing Analysis

Composite materials strength and toughness are derived from the interfacial bonding between the matrix component and the reinforcement fiber component . Thus, the interfacial interaction between the glass fibers and the epoxy resin is important to understand when fabricating quality composite samples. The fabric wetting during VARTM processing is also related to the fabric's surface chemistry. To get insight into the wetting behavior of the glass fabric surface, an effort was made to remove the fabric's sizing to evaluate the chemical composition of the sizing used. Eight-layer structures were assembled to test wetting by exposure to SC-15 resin, but these samples did not absorb resin in a measurable manner, and thus these results are not presented here. Analysis of the sizing provided composition information. Micro FT-IR comparison of sizing composition for the three glass types is displayed in **Figure 14.**

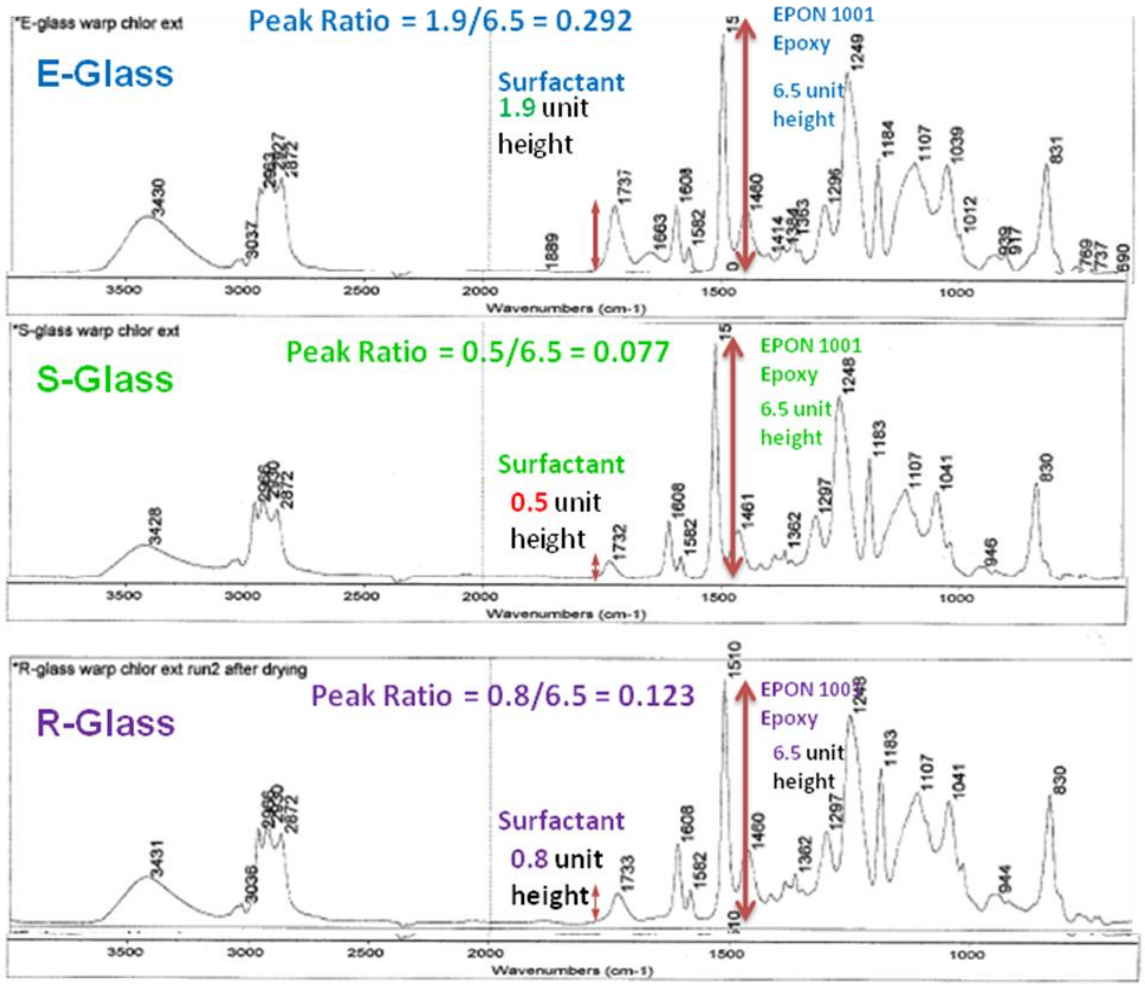

Figure 14: Micro FT-IR comparison of the three glass fibers

The height of the lines represent the peak ratio, which is taken as ratio of intensity of the surfactant 1730cm^{-1} peak to the normalized 1510cm^{-1} epoxy peak intensity. Results show as highest quantity of surfactant on lowest quality (i.e. modulus, strength) E-glass. The peaks matched the FT-IR software reference library as being EPON 1001 epoxy resin and a (Poly(alkenyl:alkanyl) ester surfactant.

4.2 Void Content & Fiber Volume Fraction

The measurements from the fiber burnout testing not only quantity of glass and matrix in the resulting composite structures, but can be used to calculate the fraction of voids. The fiber burnout results less than 1-2% total void fraction as defined in ASTM 2734 in each of the samples tested meets the industrial and military standards for a quality composite structure as seen below in **Table 11**.

Table 11: Volume Fraction and Void Percentage in different grades of glass. Each coupon is 1 in x .20 in and has an average mass as shown.

Number of specimens, glass type and nominal coupon size and average mass	**Fiber Mass Per sample (g)**		**Resin Mass (g)**		**Fiber Volume Fraction (%)**		**Resin Volume Fraction (%)**		**Void Volume Fraction (%)**	
	Avg.	**Std. Dev.**	**Avg.**	**Std. Dev.**	**Avg.**	**Std. Dev.**	**Avg.**	**Std. Dev.**	**Avg.**	**Std. Dev.**
E-Glass (4 Specimens)	2.50	0.04	0.92	0.03	55.3	0.9	45.5	0.7	<0.01	0.0
S-Glass (4 Specimens)	2.37	0.01	1.07	0.02	49.6	0.3	48.9	0.5	1.4	0.2
R-Glass (4 Specimens)	2.44	0.02	1.07	0.03	50.0	0.4	48.8	0.9	1.2	0.7

The S-Glass had the highest resulting void content. This was believed to be due to the small fiber diameter, resulting in the highest surface area for the fabric(more fibers per bundle), and increasing the tightness of the weave. The increased weave tightness increased resistance of flow through the fabric, resulting in increased trapped voids. This is consistent with the results of the E-glass, which has the loosest construction of woven fabrics used, which resulted in the lowest void content of all samples, reported as zero percent void content. The low standard deviation from the

average void content demonstrates the uniformity between glass fiber type sample sets (E, S, or R) and within each set of fabricated samples(12 samples/set). The results from the fiber burnout testing answer all of the initial three questions whether the type of glass affects the void content, whether the void content was uniform throughout sets of composite samples, and achieving a void content lower than 2% meets the standards specified for composite use in the specified applications.

Sources of increased voids that can be found in the void content results may be due to the residual air left in the resin after the degassing stage, remaining air in the system after degassing, or the introduction of air through unsealed vacuum bagging. Any extra residual air added to the system has the chance of being trapped in the fibers creating a void so minimizing all residual is key to resulting in low void content.

4.3 Composite Density Results

Before each of the same four composite samples of each glass type that were evaluated using the fiber burn-out test method, the sample's density was measured. The density results shown in **Table 12** confirm the trends seen in the average fiber diameters of the three glass types and that the type of glass affect the overall composite density. The smaller the average fiber diameter in the glass types, results in a hindered packing of the fibers when compacted upon vacuum and resin wetting. The results show that the S-glass has less weight per volume of the sample compared to the E and R-glass. The small values obtained for the standard deviations from the average demonstrate the sample uniformity between sets answering the second initial question of this study.

Table 12: Composite sample density comparison between glass types (+/-.1%)

Composite	Avg. Composite Density (g/cc)	Std. Dev Composite Density (g/cc)
E-Glass/SC-15 (4 Specimens)	1.922	0.022
S-Glass/SC-15 (4 Specimens)	1.794	0.004
R-Glass/SC-15 (4 Specimens)	1.826	0.006

Sources of variety in the density measurements are due to the skewing of the fabric orientation angle from the initial degrees outlined can prevent layer packing compared to perfectly in lined fiber bundle angles of 0°/90° and ±45° orientations. Also the variable tightness of the weave affects density measurements due to the increased ability to pack better.

4.4 Ultimate Fracture Strength & Young's Modulus

The results shown below in **Table 13** again answer the initial questions asked in this study whether the glass type affect the composite properties and uniformity between sample sets. The quality of the surface finish plays an important role with composite testing measurements. The visible quality of surface finish on the composites with no apparent surface voids or roughness was important when screening for quality fabricated samples before being shipped and costs of further testing. The surface finish quality is important to maintain the mechanical properties needed for the given applications, due to imperfections in the surface finish will be points of stress where surface cracks form when a load is applied.

Table 13: Fracture stress and Young's Modulus results from fabricated samples

Composite	Avg. Ultimate Fracture Strength (ksl) / (MPa)	Std. Dev Ultimate Facture Strength (ksi) / (MPa)	Avg. Young's Modulus (Msi) / (GPa)	Std. Dev Young's Modulus (Msi) / (GPa)
E-Glass/SC-15 (6 specimens)	32.68 / 225.30	0.757 / 5.22	1.59 / 10.96	0.05 / 0.36
S-Glass/SC-15 (6 specimens)	53.42 / 368.34	1.796 / 12.38	2.05 / 14.15	0.05 / 0.36
R-Glass/SC-15 (6 specimens)	45.81 / 315.85	0.964 / 6.65	1.99 / 13.73	0.04 / 0.31

As expected from the trends of the increased S-glass individual fiber properties in **Table 3**, the S-glass composite samples had the highest fractures strength and modulus when compared to the E and R-glass. The results from the fracture strength and modulus data answers the first initial question of whether the type of glass used affect the mechanical properties of the composite sample.

4.4 Summary of Glass Properties

In order to significantly analyze the results of the repeatable composite fabrication process, it is demonstrated to answer the three initial questions restated below by observing the comparative trends that can be concluded from the summary data in **Table 14**:

1. Whether the type of glass (E-Glass, S-Glass, and R-Glass) influences the property values of individually tested samples.
2. Whether the type of glass influences the property uniformity throughout the set of tested samples.

3. Whether the defined composite plaque design and resulting performance, as defined by ASTM Standards or industry accepted parameters, is adequate for use in the defined military application or wind specific application.

The fracture strength and Young's modulus testing results in **Table 13** answers the question to whether the type of glass influences the property values of the composite samples. The results conclude that S-glass, as the fiber component, significantly increases overall composite fracture strength and modulus as compared to E-glass and R-glass. These results were expected to due to the specific function of the S-glass fabrication being high strength glass and the individual fiber properties in **Table 3** being greater than the other types of glass(E & R). The other two glass types followed the same trend in individual fiber properties affecting the composite properties. Although only a limited number of samples were tested for void content, the standard deviations for each population was less than 3% of the mean and all data points passed the Students' T test, indicating a lack of outliers in the data.. These low standard deviations values obtained answer the second question in the motivation that the uniformity across sample sets is true and the optimized process is deemed repeatable.

Table 14: Summary of Glass Properties Across Glass Types

Composite	**Avg. Composite Density (g/cc)**	**Std. Dev Composite Density (g/cc)**	**Avg. Void Content (%)**	**Std. Dev Void Content (%)**
E-Glass/SC-15 (4 Specimens)	1.922	.022	<0.01	0.0

S-Glass/SC-15 (4 Specimens)	1.794	.004	1.4	0.2
R-Glass/SC-15 (4 Specimens)	1.826	.006	1.2	0.7
Composite	**Avg. Ultimate Tensile Strength (ksl) / (MPa)**	**Std. Dev Ultimate Tensile Strength (ksi) / (MPa)**	**Avg. Young's Modulus (Msi) / (GPa)**	**Std. Dev Young's Modulus (Msi) / (GPa)**
E-Glass/SC-15 (6 specimens)	32.68 / 225.30	0.757 / 5.22	1.59 / 10.96	0.05 / .36
S-Glass/SC-15 (6 specimens)	53.42 / 368.34	1.796 / 12.38	2.05 / 14.15	0.05 / .36
R-Glass/SC-15 (6 specimens)	45.81 / 315.85	0.964 / 6.65	1.99 / 13.73	0.04 / .31

The last question was whether the composite plaque design and resulting performance met standards or industry accepted parameters for use in the defined military application or wind specific application. Shown below in **Table 15** are the common mechanical property value ranges for the materials used as the fiber reinforcement component. This data contains values that are obtained from a variety of composites designs and processing techniques, hence serve only as a reference point. The composites fabricated from the optimized process were within the mechanical properties ranges shown in **Table 15**, therefore meeting the standards given for the targeted application.

Table 15: Industry defined mechanical properties ranges for common materials used in targeted applications. Note that these ranges have been measured for a diverse set of composite designs.

Industry Used Fiber Reinforcement Material	Composite Ultimate Fracture Strength Range (MPa)	Composite Young's Modulus Range (GPa)	Composite Void Content Range (%)
E-Glass	100-600	10-50	1-2
S-Glass	300-1100	15-55	1-2
Carbon	600-1500	75-150	1-2

A key selection criteria for use in these two (military and wind) applications is the material performance to cost ratio.

Table 16: Composite cost analysis comparison for various glass types. Property ranking is based on material property values of ultimate tensile strength and Young's modulus per cost of plaque fabrication;

Composite	Cost of Fabric per Plaque (size assumed = 14 x 36 in times 8 layers	Cost Resin per Plaque (mass assumed 2500g for each plaque)	Total Cost per Plaque (calculated based on measured vol fraction of each material)	Ultimate Tensile Strength / Plaque Cost Ranking (MPa / $)	Modulus / Plaque Cost Ranking (GPa / $)
E-Glass	$ 7.46	$ 30.53	$ 37.99	5.93	0.29
S-Glass	$ 46.65	$ 30.53	$ 77.18	4.77	0.18
R-Glass	$ 27.65	$ 30.53	$ 58.47	5.40	0.23

The properties per cost displayed in **Table 16** show the ranking value of the resulting composite ultimate fracture strength and modulus calculated to the most expensive glass type per unit of property, S-glass. These results show that the E-glass has the highest ranking of both ultimate fracture strength and modulus demonstrating the best properties to cost ratio. These results follow the trend that for most structural applications, E-glass meets the load bearing standards for the cheapest amount in material cost. The S-glass having the worst property to cost ratio meaning to achieve greater strength requirements for high impact resistance it cost 25% more per plaque for the E-glass strength properties and 58% more than the modulus. The R-glass has an intermediate property per cost ranking between the S and E-glass.

Chapter 5 - Conclusions and Future Work

The key questions that were investigated in this study were to determine if the mechanical properties of the composite samples were affected by the glass type used as the fiber component, whether the uniformity within the sets of glass types on a optimized VARTM process, and if the values obtained from the specified testing methods meet those of industry or military standards.

The mechanical and physical property test data showed that the optimized VARTM process described in the experimental chapter of this thesis produced uniform, low void, quality composite sample plaques.

Small standard deviations observed in the void fraction data and the corresponding physical property data, also confirm that the optimized process developed yielded excellent material repeatability. Key process parameter that impacted most on the final quality samples was the degassing phase as demonstrated by the very low void contact and low standard deviations of mechanical measurements

In conclusion, it has been shown that:

1) A highly repeatable, optimized composite plaque assembly process based on glass-fabric reinforced epoxy resin has been developed and used to produce high quality composite materials with repeatable properties, within fabric type.

2) Resulting composite material produced exhibited low void content material (< 2%) with mechanical properties comparable to other glass FRC materials. While absolute values cannot be compared directly due to the difference in composite

design used here as compared to others in literature, the relative ultimate fracture strength and modulus values were 225.30 MPa / 10.96 GPa for E-glass, 368.34 MPa / 14.15 GPa for S-glass and 315.85 MPa / 13.73 for R-glass.

3) Assessment of composite physical properties (density, void content, fiber and resin volume fraction) showed excellent uniformity within glass type.

4) Mechanical properties as determined by six Instron tensile tests showed ultimate fracture strengths and modulus values of 225.30 MPa / 10.96 GPa for E-glass, 368.34 MPa / 14.15 GPa for S-glass and 315.85 MPa / 13.73 for R-glass. These values while approximately equal to would be comparable to those material values required in wind and military applications described here.

Future Work

i) Future Composite Designs

Future work in this research will include the hybrid mixture of Glass/Carbon-Polymer and Glass/Aramid-Polymer composite matrix fabrication and testing. The increased stiffness of the carbon reinforcement versus weight between carbon and glass are used in aerospace composite applications. The original layup and design would serve as a basis or comparative fiber material.

Table 17: Future Composite Layup and Design

Sample	Material	Matrix	Fabric Type	Layer Construction	Target Thickness
1-Ref	E-Glass	SC-15	Plain Weave 0°/90° and ±45°	8 Layers – 0°/90°, ±45°, 0°/90°, ±45°,±45°, 0°/90°, ±45°, 0°/90°	0.25 in

2	S-Glass	SC-15	Plain Weave 0°/90° and ±45°	8 Layers – 0°/90°, ±45°, 0°/90°, ±45°,±45°, 0°/90°, ±45°, 0°/90°	0.25-0.30 in
3	S-Glass/Carbon (50:50)	SC-15	Plain Weave 0°/90° and ±45°	8 Layers – 0°/90°(C), ±45°(G), 0°/90°(C), ±45°(G),±45°(C), 0°/90°(G), ±45°(C), 0°/90°(G)	0.25-0.30 in
4	S-Glass/Aramid (50:50)	SC-15	Plain Weave 0°/90° and ±45°	8 Layers – 0°/90°(I), ±45°(G), 0°/90°(I), ±45°(G),±45°(I), 0°/90°(G), ±45°(I), 0°/90°(G)	0.25-0.30 in
5	S-Glass	SC-15 w/ Nano-Clay	Plain Weave 0°/90° and ±45°	8 Layers – 0°/90°, ±45°, 0°/90°, ±45°,±45°, 0°/90°, ±45°, 0°/90°	0.25-0.30 in
6	S-Glass/Aramid (50:50)	SC-15 w/ Nano-Clay	Plain Weave 0°/90° and ±45°	8 Layers – 0°/90°(I), ±45°(G), 0°/90°(I), ±45°(G),±45°(I), 0°/90°(G), ±45°(I), 0°/90°(G)	0.25-0.30 in
7	Carbon/Aramid (50:50)	SC-15	Plain Weave 0°/90° and ±45°	8 Layers – 0°/90°(I), ±45°(C), 0°/90°(I), ±45°(C),±45°(I), 0°/90°(C), ±45°(I), 0°/90°(C)	0.25-0.30 in
8	S-Glass	SC-15	Plain Weave 0°/90° and ±45°	10 Layers – 0°/90°, ±45°, 0°/90°, ±45°, 0°/90°, ±45°, 0°/90°, ±45°, 0°/90°, ±45°	0.30-0.35 in

Appendices

Appendix A

Matrix SC-15: Toughened Epoxy Resin System

Product Description

SC-15 is a very low viscosity two-phase toughened epoxy resin system. SC-15 was specifically developed for Vacuum Assisted Resin Transfer Molding (VARTM) processes. The pot-life and viscosity have been tailored to allow infusion at 77ºF. This resin system works very well in structural and ballistic applications that require good damage resistance.

Application

Infuse preform at 75-80ºF. Allow resin to vitrify at 77ºF overnight or 140ºF for two hours. Post-cure four hours at 200ºF (ramp temperature to 200ºF with a rate 2-4 ºF/min). If composite part is removed from mold and post-cured freestanding, use a 25ºF/hr ramp or step from 140ºF.

PHYSICAL PROPERTIES

Viscosity @ 77ºF		
	Mixed	300 cP
	Resin	590 cP
	Hardener	65 cP
Cured Density:		1.09 g/cm3
Wt. Gal:	Resin	9.42 lbs/gal
	Hardener	8.02 lbs/gal
Mix Ratio:	By Weight	100R : 30H
	By Volume	100R : 35H

CURED RESIN MECHANICALS

Tg (dry)	220°F
Tg (wet)	178°F
Modulus E' at ambient	390 ksi
$G_{ic,}$ in-lb/in^2	5.65 in-lb/in^2
Elongation	6.0%
Tensile Strength	9.0 ksi
Tensile Mod	3.8 msi
Kic	1400 psi-in$^{.5}$
% water pickup (10 days @ 180°F)	1.7

NEAT RESIN

Temp, °F	Storage Modulus (Dry), MPa
85	1970
180	1180

ADHESIVE PROPERTIES

T Peel, lbs/in^2	
RT	18

Aluminum Lap Shear	
RT	3900 psi
160°F	2050 psi

S-2 Woven Roving

$G_{ic,}$ J/M^2 (ASTM D 5528-94a) Initiation – 688 Propagation - 1104

** Need to add glass beads or equivalent for bond line control.

Tg Dry, °F	212
Tg Wet, °F (after 400 hrs @ 160°F)	183
Toughness	High
Tensile Str, psi	8,100
Tensile Mod, msi	3.8
% elongation	6.0
Viscosity, cps (77°F)	300

Appendix B
Laboratory Definitions

Woven fiberglass fabric and prepolymer matrix- These are base materials for the composite itself and are therefore subject to change depending on the desired material properties. Generally, the fiber has much more impact on the material properties than does the matrix.

Backboard- This is a flat rigid plate that allows the entirety of the specimen to rest on it with enough room around the edge to seal in all the excess materials used. (e.g. vacuum tubing, feed tubing, spacer material, etc.) It is also coated in some form of nonstick material such as Teflon to prevent the cured epoxy from sticking to the board.

Pattern- This is a square or rectangular sheet of rigid material of the size you desire the final composite to be. It is used to cut out sections of the woven fiber to the desired size.

Multi-sided adhesive- This is some form of tack or strong tape that allows the vacuum bagging to be adhered airtight to the backboard. Having tack rather than tape allows kneading out of any accidental air leaks that occur as the vacuum bagging is laid.

Non-stick fabric- This is a material that will prevent the impregnated specimen and any excess resin from adhering to any of the other components once cured.

Vacuum tubing- This is a tube that will go through the tack strip and allow the vacuum access to the specimen. This tubing will be placed inside a short length of distribution tubing to prevent the vacuum bagging from sealing off the vacuum tubing.

Feed tubing- This is a tube that will allow the resin access to the specimen. This will be placed very close to a long length of distribution tubing to allow the resin access to the entire length of the specimen.

Distribution tubing- This is some form of tubing the will allow the resin to travel the length of the specimen without the vacuum bagging restricting resin flow. It also should have slots or holes to allow the resin to escape along the length of the specimen rather than just at the ends.

Distribution material- This is some form of material that will allow the resin to flow over the top of the specimen.

Spacer material- This is some form of porous material that will prevent the vacuum bagging from sealing with the backboard and prevent the resin from flowing to the vacuum tube.

Resin trap- This is some form of can that can withhold resin from reaching and ruining the vacuum source. It allows the resin to be drawn up the feed tube without directly

accessing the vacuum source. It also must be made of a material that can withstand the heat of polymerization caused by the epoxy resin curing

Curing oven- This is some form of oven that can be programmed to follow a particular firing cycle. It also must large enough to contain the entire backboard system, and allow the vacuum tubing access to the exterior of the oven during firing. This ensures that the vacuum source is able to keep the system under pressure throughout the curing

Appendix C

ASTM Testing Standards

ASTM 792- Density

Determine the mass of a specimen of the solid plastic in air. It is then immersed in a liquid, its

apparent mass upon immersion is determined, and its specific gravity (relative density)

calculated.

Condition the test specimens at

23 ± 2°C and 50 ± 5 % relative humidity for not less than 40 h prior to test

Conduct tests in the standard laboratory atmosphere of 23 ± 2°C and 50 ± 5 % relative humidity,

Analytical Balance—A balance with a precision of 0.1

mg or better is required for materials having densities less than

1.00 g/cm3 and sample weights less than 10 grams. For all

other materials and sample weights, a balance with precision of

1 mg or better is acceptable. The balance shall be

equipped with a stationary support for the immersion vessel

above the balance pan.

Thermometer—A thermometer readable to 0.1°C or

better.

ASTM 2584 Fiber volume fraction

This test method covers the determination of the ignition loss of cured reinforced resins. This ignition loss can be considered to be the resin content if only glass fabric or filament is used as the reinforcement of an organic resin that is completely decomposed to volatile materials under the conditions of this test and the small amount of volatiles

(water, residual solvent) that may be present is ignored, the ignition loss can be considered to be the resin content of the sample

Needs the following furnace

Electric Muffle Furnace, capable of maintaining a temperature

of 565 ± 28°C (1050± 50°F).

Condition the test specimens at 23± 2°C

(73.4 ± 3.6°F) and 50 ± 5 % relative humidity for not less

than 40 h prior to test in accordance with Procedure A of

Practice D618 for those tests where conditioning is required.

ASTM 4065 Glass transition temperature determination and degree of cure

A specimen of known geometry is placed in mechanical oscillation either at fixed or natural resonant frequencies. Elastic or loss moduli, or both of the specimen are measured while varying time, temperature of the specimen or frequency of the oscillation, or both the latter. Plots of the elastic or loss moduli, or both, are indicative of viscoelastic characteristics of the specimen. Rapid changes in viscoelastic properties at particular temperatures, times, or frequencies are normally referred to as transition regions.

Unless otherwise specifie in the appropriate material specification, condition the specimen at a set temperature of 23°C [73°F] that is maintained ±2°C [±4°F] and at a set relative humidity of 50 % that is maintained ±5 % for not less than 40 h prior to test in accordance to Procedure A of Practice D618, for those tests where conditioning is required.

The function of the apparatus is to hold a plastic specimen of uniform cross section, so that the specimen acts as the elastic and dissipative element in a mechanically oscillated system. Instruments of this type are commonly called dynamic mechanical or dynamic thermomechanical analyzers. They typically operate in one of seven oscillatory modes: (*1*) freely decaying torsional oscillation, (*2*) forced constant amplitude, resonant, flexural oscillation, (*3*) forced constant amplitude, fixed frequency, compressive oscillation, (*4*) forced constant amplitude, fixed frequency, flexural oscillation, (*5*) forced, constant amplitude, fixed frequency, tensile oscillation, (*6*) forced constant amplitude, fixed frequency, torsional oscilla-tion and (*7*) forced constant amplitude, fixed frequency, or variable frequency dual cantilever.

The apparatus shall consist of the following:

Clamps—A clamping arrangement that permits grip- ping of the sample.

Oscillatory Deformation (Strain)—A device for applying an oscillatory deformation (strain) to the specimen. The deformation (strain) shall be applied and then released, as in free-vibration devices, or continuously applied, as in forced- vibration devices

Detectors—A device or devices for determining dependent and independent experimental parameters, such as force (stress or strain), frequency, and temperature. Temperature shall be measurable with an accuracy of ±1°C, frequency to ±1%, and force to ±1%.

Temperature Controller and Oven—A device for controlling the specimen temperature, either by heating (in steps or ramps), cooling (in steps or ramps), or maintaining a constant specimen environment. Any temperature programmer should be sufficiently stable to permit measurement of sample temperature to 60.5°C.

Nitrogen or other gas supply for purging purposes.

Calipers or other length-measuring device capable of measuring to an accuracy of ±0.01 mm.

ASTM 2734 Void content

The densities of the resin, the reinforcement, and the composites are measured separately. Then the resin content is measured and a theoretical composite density calculated. This is compared to the measured composite density. The difference in densities indicates the void content. A good composite may have 1% voids or less, while a poorly made composite can have a much higher void content. Finite values under 1 % should be recognized as representing a laminate density quality, but true void content level must be established by complementary tests or background experience, or both.

Condition the test specimens at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 10 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D618,

The volume of each specimen shall not be less than 2 cm3 (0.125 in.3).

The tolerance on the accuracy of the micrometer measurements shall be 60.0013 cm (60.0005 in.).

ASTM 3039 Tensile testing for polymer matrix composites

This test method is designed to produce tensile property data for material specifications,

research and development, quality assurance, and structural design and analysis. Factors that influence the tensile response and should therefore be reported include the following: material, methods of material preparation and lay-up, specimen stacking sequence, specimen preparation, specimen conditioning, environment of testing, specimen alignment and gripping, speed of testing, time at temperature, void content, and volume percent reinforcement. Properties, in the test direction, which may be obtained from this test method include the following:
Ultimate tensile strength, Ultimate tensile strain, Tensile chord modulus of elasticity, Poisson's ratio, and Transition strain.

For typical specimen geometries, an instrument with an accuracy of ±2.5 μm [±0.0001 in.] is adequate for thickness measurement, while an instrument with an accuracy of ±25 μm [±0.001 in.] is adequate for width measurement.

The testing machine shall be in conformance with Practices E 4 and shall satisfy the following requirements:

1. The testing machine shall have both an essentially stationary head and a movable head.

2. The testing machine drive mecha- nism shall be capable of imparting to the movable head a controlled velocity with respect to the stationary head.

3. The testing machine force-sensing device shall be capable of indicating the total force being carried by the test specimen. This device shall be essentially free from inertia lag at the specified rate of testing and shall indicate the force with an accuracy over the force range(s) of interest of within 61 % of the indicated value. The force range(s) of interest may be fairly low for modulus evaluation, much higher for strength evaluation, or both, as required.

4. Each head of the testing machine shall carry one grip for holding the test specimen so that the direction of force applied to the specimen is coincident with the longitudi- nal axis of the specimen. The grips shall apply sufficient lateral pressure to prevent slippage between the grip face and the coupon. If tabs are used the grips should be long enough that they overhang the beveled portion of the tab by approximately 10 to 15 mm [0.5 in.]. It is highly desirable to use grips that are rotationally self-aligning to minimize bending stresses in the coupon.

5. Poor system alignment can be a major contributor to premature failure, to elastic property data scatter, or both. Practice E 1012 describes bending evaluation guidelines and describes potential sources of misalignment during tensile testing.

Force-strain data, if required, shall be determined by means of either a strain transducer or an extensometer.

When conditioning materials at nonlaboratory environments, a temperature/vaporlevel-controlled environmental conditioning chamber is required that shall be capable of maintaining the required temperature to within ±3°C [±5°F] and the required relative vapor level to within ±3 %. Chamber conditions shall be monitored either on an automated continuous basis or on a manual basis at regular intervals.

An environmental test chamber is required for test environments other than ambient testing laboratory conditions. This chamber shall be capable of maintaining the gage section of the test specimen at the required test environment during the mechanical test.

ASTM 6484 Compression testing for polymer matrix composites

ASTM D 696- Dimensional Stability

ASTM 1269- Specific Heat

ASTM 1225- Thermal Conductivity

ASTM E 84- Flammability and Smoke Generation

ASTM D 149- Electrical Properties

ASTM D 3518- In-Plane Shear Strength and Modulus

ASTM D 5379- Out of Plane Shear Strength and Modulus

ASTM D 2344- Short Beam Shear Strength

ASTM D 790- Flexural Strength

ASTM D 5528- Fracture Toughness

ASTM D 3479- Fatigue

*Sources- Mil Handbook 17,

Appendix D

Detailed Layup Process

3.2 Cutting and preparing the fabric

Materials needed:

Pattern

Backboard

Plain Woven E-Glass

Lay the pattern over the fiber weave and orient so that the edges of the pattern align with the 0° and 90° fabric directions. Use a marker to draw around the pattern then cut four panels at this alignment. Realign the pattern over the fabric oriented so that the edges of the pattern align with the ±45° fabric directions. Again marker out and cut 4 panels at this alignment. Lay the cut fabric sheets on the backboard in the order 0°/90° sheet, then ±45° sheet, followed by another 0°/90° sheet, then ±45° sheet, followed by another ±45° sheet, then a 0°/90° sheet, then ±45° then a final 0°/90° sheet. The final arrangement should be made up of 8 layers and should look like the following layup design in **Figure 4**. This arrangement of layers will give the final composite a balanced, quasi-isotropic, and good mechanical properties vs weight.

3.3 Laying up the composite on the backboard

Outline the backboard with the multi-sided adhesive tack, leaving two inches between the specimen in the center and the adhesive on the outside perimeter of the

backboard. Remove the ordered glass layers from the backboard and place a layer of non-stick fabric cut to 18.5 x 43.25 in. down on the backboard. Then place the ordered glass layers on top of the non stick fabric. Place a second layer of nonstick fabric cut to 18.5 x 43.25 in. on top of the ordered glass layers. Ensure that this layer covers the top of the entire section and comes to within a .5 inches of the outlining tack strip. Cut the distribution material to 14.5 x 40 in. and position so that it covers the glass layers only and extends past the bottom edge of the glass layer to the edge of the adhesive tack. Cut needed length and place the feed tube on one corner of the backboard so the feed tube lies between the backboard and the adhesive strip. Lay a short second layer of adhesive tack on top of the tubing to allow the vacuum bagging to properly seal. Also, ensure the feed tube is long enough to access the resin reservoir. Run distribution tubing along the width of the glass layers on the same end as the feed tubing. Ensure the end of the distribution tubing is overlapping the end of the feed tubing to allow the resin to flow easily into the distribution tubing. Place the vacuum tubing in between layers of the spacer material at one corner of the specimen. Dress it so that it leaves the backboard over the adhesive strip. Lay a short second layer of adhesive on top of the tubing to allow the bagging to properly seal. Overlap a 4.5 inch amount of distribution tubing on the end of the vacuum tubing to prevent the bagging from sealing around the end of the vacuum tubing. This will allow the vacuum full access to the interior of the system Also ensure the vacuum tube is long enough to access the resin trap. Cover the entire system with vacuum bagging and push the vacuum bagging onto the adhesive tack. Ensure that there are no leaks or pleats in the bag along the bagging/adhesive

tack interface. This will prevent leaks from occurring in the vacuum and unwanted air intake.

3.4 De-bulking the specimen

Materials needed:

Backboard with specimen and vacuum bagging laid as directed in Step B

Vacuum source

Resin trap

Secondary vacuum tubing

Curing oven

It is best to perform this step inside the curing oven so that the vacuum can be continuously applied to the system throughout the curing process with the oven doors closed. First, take the vacuum tube that leads from the backboard and attach to the resin trap. Then attach secondary vacuum tubing from resin trap to vacuum source. Clamp end of feed tube to allow the vacuum to be sealed. Turn on the vacuum source and allow the system to de-bulk for 30 minutes before introducing the resin. This allows excess air to leave the system.

3.5 Mixing and Degassing of SC-15 resin

To adequately infused an entire panel the size of the reference sample the resin is mixed with the following amounts, other size samples need to be 3:1 Part A:Part B.

Follow safety procedures to load the Part A SC-15 barrel of resin into pouring position. Place the scale and bucket container below the container and measure out 1923.0 grams of Part A then poured slowly at a tilt into the Degassing container pot. Follow safety procedure to load Part B SC-15 resin container into pouring position.Measure out 576.0 grams of Part B and pour slowly at a tilt into the Degassing pot. It is important that the mixture should be stirred exactly 100 times to obtain uniform mixing and optimum viscosity. Seal the lid to the Degassing pot and attached the vacuum line. Turn on the vacuum and begin degassing for 30 minutes.

Note: Chem goggles, gloves and lab coat should be worn during this entire process. Epoxy resin is toxic to eyes.

3.6 Infusing and Curing the composite

Materials needed:

Resin-infused backboard system

Curing oven

To initiate the infusion step of the process, take feed tube leading from the backboard and place into resin reservoir. Remove feed tube clamps to allow the vacuum access to the resin. Wait for the resin to completely wet the system and be drawn into the vacuum tube on the opposite side. The infusion time varies from 70-100 minutes depending on tube size and vacuum pressure. Keep the vacuum source on to keep the sample under pressure while the resin is cured and ensure that there is always resin in the reservoir to prevent air from being introduced into the system. Once the resin has completely wetted the system and is flowing into the resin trap start the cure

cycle. Air bubbles in the vacuum tube leading to the resin trap are expected. They are the result of dissolved air in the resin coming out of solution.

As the backboard is already in the oven, ensure that the feed tubing has access to the reservoir. Also ensure that all opening have been sufficiently insulated to prevent heat escape. Ensure that the vacuum tubing has a way to exit the oven and is connected to the vacuum source while the oven doors are closed. Close the oven and start the cure cycle.The infusion, cure, and post cure cycle should be programmed to the following pattern in **Table 3**:

To begin the debagging step, turn off the vacuum and disconnect the vacuum tube from the resin trap. Remove the backboard from oven. Carefully remove the vacuum bagging, adhesive strip, all tubing, distribution material, spacer material, and non-stick fabric from the composite. The nonstick fabric should allow this process to be relatively easy.

Appendix E

Fiber Burnout Testing Results

Densities:

e-glass	2.54	g/cc	SC-15	1.139	g/cc
s2-glass	2.49	g/cc			
R-glass	2.54	g/cc			

S2/SC-15 composites

SPECIMEN I.D.	CRUCIBLE	CRUCIBLE + GLASS	COMPOSITE WEIGHT	COMPOSITE WGT IN H2O	COMPOSITE DENSITY	FIBER WEIGHT	RESIN WEIGHT	v_f	v_r	v_v
	(g)	(g)	(g)	(g)	(g/cc)	(g)	(g)	(%)	(%)	(%)
a	68.3163	70.6982	3.4589	1.5371	1.7958	2.3819	1.0770	49.7	49.1	1.2
b	67.4371	69.8088	3.4162	1.5203	1.7979	2.3717	1.0445	50.1	48.3	1.6
c	64.4382	66.7981	3.4385	1.5224	1.7905	2.3599	1.0786	49.4	49.3	1.3
d	73.3486	75.7130	3.4382	1.5215	1.7898	2.3644	1.0738	49.4	49.1	1.5
				MEAN	1.7935		MEAN	49.6	48.9	1.4
				STD. DEV.	0.0040		STD. DEV.	0.3	0.5	0.2

E/SC-15

SPECIMEN I.D.	CRUCIBLE	CRUCIBLE + GLASS	COMPOSITE WEIGHT	COMPOSITE WGT IN H2O	COMPOSITE DENSITY	FIBER WEIGHT	RESIN WEIGHT	v_f	v_r	v_v
	(g)	(g)	(g)	(g)	(g/cc)	(g)	(g)	(%)	(%)	(%)
2	107.5436	110.0077	3.3500	1.6268	1.9397	2.4641	0.8859	56.2	45.0	-1.2
3	105.7601	108.2340	3.4152	1.6414	1.9211	2.4739	0.9413	54.8	46.5	-1.3
4	109.1970	111.7424	3.4917	1.6499	1.8916	2.5454	0.9463	54.3	45.0	0.7
5	113.7219	116.2191	3.4047	1.6499	1.9359	2.4972	0.9075	55.9	45.3	-1.2
				MEAN	1.9221		MEAN	55.3	45.5	-0.7
				STD. DEV.	0.0219		STD. DEV.	0.9	0.7	1.0

R/SC-15

SPECIMEN I.D.	CRUCIBLE	CRUCIBLE + GLASS	COMPOSITE WEIGHT	COMPOSITE WGT IN H2O	COMPOSITE DENSITY	FIBER WEIGHT	RESIN WEIGHT	v_f	v_r	v_v
	(g)	(g)	(g)	(g)	(g/cc)	(g)	(g)	(%)	(%)	(%)
D	108.7819	111.2472	3.5655	1.6266	1.8348	2.4653	1.1002	49.9	49.7	0.3
K	106.3608	108.7896	3.4528	1.5652	1.8251	2.4288	1.0240	50.5	47.5	1.9
W	117.8736	120.3115	3.5037	1.5822	1.8194	2.4379	1.0658	49.8	48.6	1.6
Z	115.0844	117.4990	3.4863	1.5794	1.8242	2.4146	1.0717	49.7	49.2	1.0
				MEAN	1.8259		MEAN	50.0	48.8	1.2
				STD. DEV.	0.0065		STD. DEV.	0.4	0.9	0.7

Appendix F

Fiber Micro-graph diameter results

	E Glass (microns)	S Glass (microns)	R Glass (microns)
Sample 1	17.945	9.569	11.011
Sample 2	15.369	9.732	12.015
Sample 3	17.974	10.049	12.15
Sample 4	17.015	10.35	13.605
Sample 5	16.749	9.624	11.21
Sample 6	16.184	9.487	12.526
Sample 7	18.072	10.338	13.057
Sample 8	17.469	8.978	12.036
Sample 9	17.458	9.832	12.526
Sample 10	17.145	9.644	12.437
Average	**17.138**	**9.7603**	**12.2573**
Std Dev	**0.858968244**	**0.411792572**	**0.774726052**

References

1. Advanced Composite Group.*Prepregs from advanced composites group- what is prepreg?*http://www.advanced-composites.co.uk/intro%20to%20advanced%20composites/prepregs%20and%20introduction%20to%20advanced%20composites.html
2. Al Chan, Grey Chapman, David Hartman.Lightweight composite integrated structural armor. *Owens Corning Science & Technology Center,*
3. AZO Composites.Composites: A basic introduction.
4. Brouwer, W. D., van Herpt, E. C. F. C., & Labordus, M. (2003). Vacuum injection moulding for large structural applications. *Composites Part A: Applied Science and Manufacturing, 34*(6), 551-558. doi:DOI: 10.1016/S1359-835X(03)00060-5
5. Bulent Eker, Aysegul Akdogan and Ali Vardar.Using composite material in wind turbines blades.
6. Calvert, S. (June 15, 2009). Materials science in modern wind turbines. *Wind Technology Application Team Lead,*
7. Carlson, T., Ordéus, D., Wysocki, M., & Asp, L. E. (2010). Structural capacitor materials made from carbon fibre epoxy composites. *Composites Science and Technology, 70*(7), 1135-1140. doi:DOI: 10.1016/j.compscitech.2010.02.028
8. Cheeseman, B. A., & Bogetti, T. A. (2003). Ballistic impact into fabric and compliant composite laminates. *Composite Structures, 61*(1-2), 161-173. doi:DOI: 10.1016/S0263-8223(03)00029-1
9. Christou, P. (2007). Advanced materials for turbine blade manufacture. *Reinforced Plastics, 51*(4), 22-24. doi:DOI: 10.1016/S0034-3617(07)70148-0
10. Clean Energy Sector Development. (April 2010). Upsizing blade test regimes. *Composites Technology,*
11. Eamon, C. D., & Rais-Rohani, M. (2009). Integrated reliability and sizing optimization of a large composite structure. *Marine Structures, 22*(2), 315-334. doi:DOI: 10.1016/j.marstruc.2008.03.001
12. F. C. Campbell. (2004). *Manufacturing processes for advanced composites* Elsevier Ltd.
13. Ghiasi, H., Fayazbakhsh, K., Pasini, D., & Lessard, L.Optimum stacking sequence design of composite materials part II: Variable stiffness design. *Composite Structures, In Press, Corrected Proof* doi:DOI: 10.1016/j.compstruct.2010.06.001
14. Ghiasi, H., Pasini, D., & Lessard, L. (2009). Optimum stacking sequence design of composite materials part I: Constant stiffness design. *Composite Structures, 90*(1), 1-11. doi:DOI: 10.1016/j.compstruct.2009.01.006
15. Gurit AG.0/90 degree fabrics.
16. Gurit AG.Multiaxial fabrics.
17. Gurit AG.Unidirectional fabrics.

18. Jureczko, M., Pawlak, M., & Mężyk, A. (2005). Optimisation of wind turbine blades. *Journal of Materials Processing Technology, 167*(2-3), 463-471. doi:DOI: 10.1016/j.jmatprotec.2005.06.055
19. Luo, X., & Chung, D. D. L. (2001). Carbon-fiber/polymer-matrix composites as capacitors. *Composites Science and Technology, 61*(6), 885-888. doi:DOI: 10.1016/S0266-3538(00)00166-4
20. Marsh, G. (2006). Wind energy – the offshore conundrum. *Reinforced Plastics, 50*(4), 20-24. doi:DOI: 10.1016/S0034-3617(06)70972-9
21. Marsh, G. (2007). Tooling up for large wind turbine blades. *Reinforced Plastics, 51*(9), 38-40, 42-43. doi:DOI: 10.1016/S0034-3617(07)70281-3
22. Naik, N. K., Shrirao, P., & Reddy, B. C. K. (2006). Ballistic impact behaviour of woven fabric composites: Formulation. *International Journal of Impact Engineering, 32*(9), 1521-1552. doi:DOI: 10.1016/j.ijimpeng.2005.01.004
23. Pihtili, H. (2009). An experimental investigation of wear of glass fibre–epoxy resin and glass fibre–polyester resin composite materials. *European Polymer Journal, 45*(1), 149-154. doi:DOI: 10.1016/j.eurpolymj.2008.10.006
24. Reichl, M. (2007). Composites turn the blades. *Reinforced Plastics, 51*(4), 18. doi:DOI: 10.1016/S0034-3617(07)70147-9"
25. Rosse, D. (2007). *Focus on materials science research* Nova Science Publishers. Retrieved from http://books.google.com.proxy.lib.clemson.edu/books?id=hfKFHCis4wEC
26. Savage, G. (2010). Formula 1 composites engineering. *Engineering Failure Analysis, 17*(1), 92-115. doi:DOI: 10.1016/j.engfailanal.2009.04.014
27. Thiruppukuzhi, S. V., & Sun, C. T. (2001). Models for the strain-rate-dependent behavior of polymer composites. *Composites Science and Technology, 61*(1), 1. doi:DOI: 10.1016/S0266-3538(00)00133-0"
28. Veers, P.Research directions in wind turbine blades: Materials and fatigue. *Sandia National Laboratories, Wind Energy Department,*
29. W. Musial and S. Butterfield. (2006). Energy from offshore wind.
30. Fecko, D. (01 April 2006). High strength glass reinforcements still being discovered. *Reinforcedplastics.Com,*
31. Gammon L.M. and Hayes B.S. (2010). *Optical microscopy of fiber-reinforced composites* ASM International.
32. Hogg, P. J.Composites for ballistic applications. *Department of Materials,*
33. Schwartz, M. (1992). *Composite materials handbook* (Second Edition ed.). New York: McGraw-Hill Inc.
34. Kedward, K. T.ENGINEERING PROPERTIES OF COMPOSITES. *Chapter 35,*