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Creep Rupture and Life Prediction of Polymer Composites

Saurabh Batra

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

**Master of Science
in
Chemical Engineering**

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Morgantown, West Virginia
2009

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tension, SEM

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Abstract

Lack of long term performance data inhibits the widespread use of composites. The long-term response/performance of composite materials and structures exposed under service conditions (load, time, temperature, water, and others) must be established. Creep response under sustained load results in rupture but the time to rupture depends on environmental conditions. Creep rupture tests at higher fractions of ultimate failure load are often used to determine the time necessary to produce failure. Creep data are generated first in terms of deformation/strain versus time up to rupture for each test under a fraction of rupture load at a given temperature. Then the creep data from a large number of tests at various stresses and temperatures are used to develop log-log scale plots of applied stress versus time to failure and the data trend can then be used to extrapolate time to failure (stress rupture, life) under lower fractions of rupture loads for general design purposes. Such an approach is referred to as development of Master curves.

This work, at the Constructed Facilities Center of West Virginia University, focuses on characterization of creep rupture behavior of composite materials using both experimental and theoretical approaches. Composite materials with varying fiber orientations, thickness and manufacturing process were first evaluated under static (tension, compression and bending) loads for their mechanical properties under various environmental conditions. Their flexural creep behavior was also investigated under varying temperatures, moisture contents and loads. Static property data were then used to predict creep curves based on Time Temperature Superposition (TTS) Principle and Miyano's Accelerated Testing Methodology (ATM) approach (Miyano and Nakada, 2008). The predicted creep curves were finally validated through experimental creep data. In addition, Findley Power Law was employed to describe creep strain –time dependence. Our studies demonstrated that the predicted master curves are in good agreement with the experimental data and show great potential to estimate service life of the given composite, thus avoiding cumbersome and time consuming creep tests; and the Findley Power Law is capable of well representing creep strain responses at secondary creep phase for one of the composite materials studied.

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CHAPTER 1

INTRODUCTION

1.1 Background

Fiber Reinforced Composites (FRP) consists of fibers with high strength and modulus, which are bonded to a polymer matrix. Hence both fibers and matrix retain their physical and chemical properties, producing a combination of properties which are superior to its constituents and cannot be achieved by either fiber or matrix alone (Mallick, 2008).

The most common forms in which fiber-reinforced composites are used in structural applications are laminates, manufactured by stacking a number of thin layers of fibers and matrix and consolidating them into a sheet of desired thickness. Fiber orientation of each layer as well as stacking sequence of different layers in a composite laminate can be controlled and designed to generate varied physical and mechanical properties for the composite laminate.

Most important characteristic of FRP materials is its adaptability for any particular design situation. The mechanical properties of FRP materials can be easily altered by changing the type of fiber, the type of resin, the fiber volume fraction, and fiber orientation. One of the more popular and cost effective method of producing FRP composites is through pultrusion. The pultrusion process has the ability to construct shapes similar to what is found in construction materials as I-shapes, channels, bars, angles, tees, and tubular sections.

FRP composite plates and sheets have been used for years to strengthen existing structures but the lack of reliable design criteria for FRP structural sections has reduced the acceptance of these materials by structural engineers (Smith, 2005). The major hurdle to the development of a reliable design criterion is due to lack of understanding of the behavior of FRP materials under sustained loading. Besides, types of FRP materials proposed for use in civil applications have shown a much greater sensitivity to not only temperature variations but also for change in moisture conditions when compared to the traditional construction materials (Liao et. al., 1998). At elevated temperatures and moisture penetration, the resin matrix tends to soften, hence, decreasing the ultimate strength and the modulus of elasticity. A reliable methodology, to assess how these

mechanical properties are diminished due to elevated temperatures, is needed in order to develop accurate predictive model of structural behavior under sustained load over a service life.

1.2 Scope and Objectives

- To evaluate mechanical properties at varying environmental conditions i.e. temperature, moisture, time and load of FRP composites.
- To investigate flexural creep and its dependence on time, temperature, moisture and fraction of failure load.
- To model experimental creep strain responses using Findley Power Law.
- To model the creep rupture through Miyano's Accelerated Testing Methodology using Time-Temperature Superposition Principle.

1.3 Organization of Thesis

Chapter 2 provides an overview of different composite materials and manufacturing processes available. This chapter also introduces various creep theories developed and used with varying conditions of temperature and moisture.

Chapter 3 discusses the constituent materials and manufacturing processes used in this study for fabrication of composites. Constituent materials include fiber and resin matrix with their part construction.

Chapter 4 explains all the laboratory tests, test set ups, test procedures according to ASTM and their significance in mechanical characterization of the composite system.

Chapter 5 discusses the results of experiments conducted on the specimens. This chapter includes the test results and discussions on the mechanical properties (static and creep) of the pultruded and compressed samples.

Chapter 6 evaluates the results to predict and verify creep responses and creep rupture properties of various composites tested using different models.

Chapter 7 concludes the results and discusses recommendations for future work.

Figure 1.1 Structure of thesis and scope of work

CHAPTER 2

LITERATURE REVIEW

2.1 Composites

Composites are combinations of two materials in which one of the materials, called the reinforcing phase, is in the form of fibers, sheets, or particles, and are embedded in the other materials called the matrix phase. The reinforcing material and the matrix material can be metal, ceramic, or polymer. Typically, reinforcing materials are stronger with low densities while the matrix is usually a ductile, or tough, material. If the composite is designed and fabricated correctly, it combines the strength of the reinforcement with the toughness of the matrix to achieve a combination of desirable properties not available in any single conventional material. However, such composites are often more expensive than the conventional materials.

It has properties and characteristics that are enhanced from its constituent materials, though constituent materials maintain separate identities in the composite (Mallick, 1997). The reinforcements are the major load resisting components of a composite. The polymer matrix holds the reinforcements in place, protects the reinforcements from an adverse environmental condition and also acts as a path for stress transfer between fibers. In this research work, GFRP-Glass Fiber Reinforced Polymer Composites are used for developing structural products. As the name implies, glass fibers are used as reinforcements and vinyl ester is used as polymer matrix. The mechanical properties of a composite not only depend on the reinforcement and polymer matrix, but also on the interface between reinforcement and the matrix, which plays a critical role in the performance of the composite material. Without a strong interfacial bond between the reinforcement and matrix, the reinforcements can not effectively transfer shear forces and hence, composite material can not effectively resist the external load. A schematic representation of reinforcement, matrix and interface in a composite material is shown in Figure 2.1. The interfacial bond strength can be improved by fiber surface treatment (Mallick, 1997).

Figure 2.1 Schematic representations of reinforcement, matrix and interface
(Mallick, 1997)

Some of the advantages of composite materials are:

Light Weight: Composite parts offer more strength per unit of weight than any unreinforced plastic or most metals. For some parts, the reduction can be significant. This can reduce the shipping costs; the labor needed to install parts, and also reduces injuries from installing large, heavy components.

Design Flexibility: Parts molded with polyester or vinyl ester composite materials can reproduce almost any shape as desired. The molding process allows for a more aesthetically pleasing part to be designed than is often possible when the part was fabricated in metal, thermoformed, or made from another material or process.

Part Consolidation: A well designed composite part can easily eliminate the assembly of many parts by molding as one complete piece. The result is a finished unit with fewer components that would reduce the time and money spent for assembly.

Dimensional Stability: Products made from composite materials offer a greater degree of dimensional stability when compared to thermoplastics, wood, and metals. Composites can maintain their shapes even under intense mechanical and environmental stresses.

High Strength: Composite parts can be designed to provide a wide range of impact, tensile and flexural strength properties, depending upon specific requirements of the application. Each compound is custom made to meet user requirements for strength as well as other properties.

Corrosion Resistance: Composites do not rust or corrode. In addition, they offer various levels of chemical and environmental resistance, depending upon the resin system chosen for the application.

Low Electrical and Thermal Conductivity: Composites can offer a wide range of insulating properties to meet specific requirements for electrical and thermal resistance.

2.1.1 Design of Fiber Reinforced Polymer Composites

Mechanical properties of a composite are constituted by the individual properties of its constituents such as fibers, resin etc. Effective reinforcement requires good bonding between the fibers and the matrix. For a unidirectional composite, the longitudinal tensile strength is quite independent of the fiber-matrix bonding, but the transverse tensile strength and the flexural strength (for bending in longitudinal or transverse directions) increases with increasing fiber-matrix bonding. On the other hand, excessive fiber-matrix bonding can cause a composite with a brittle matrix (e.g., carbon and ceramics) to become more brittle, as the strong fiber-matrix bonding causes cracks to propagate in the direction perpendicular to the fiber-matrix interface without being deflected to propagate along this interface. In the case of a composite with a ductile matrix (e.g., metals and polymers), a crack initiating in the brittle fiber tends to be blunted when it reaches the ductile matrix, even when the fiber-matrix bonding is strong. Therefore, an optimum degree of fiber-matrix bonding is needed for brittle-matrix composites, whereas a high degree of fiber-matrix bonding is preferred for ductile-matrix composites. In conclusion, it is utmost important that in a given composite fiber and matrix shows a good compatibility.

The mechanisms of fiber-matrix interaction include chemical bonding, van der Waals bonding, and mechanical interlocking. Chemical bonding gives the largest bonding force, provided that the density of chemical bonds across the fiber-matrix interface is sufficiently high. This density can be increased by chemical treatments of the fibers or by

sizing on the fibers. Mechanical interlocking between the fibers and the matrix is an important contribution to the bonding if the fibers form a three-dimensional network. Otherwise, the fibers should have a rough surface in order to form a small degree of mechanical interlocking. Both chemical bonding and van der Waals bonding require the fibers to be in intimate contact with the matrix. For intimate contact to take place, the matrix or matrix precursor must be able to wet the surfaces of the carbon fibers, during infiltration of the matrix or matrix precursor into the carbon fiber preform. Chemical treatments and coatings can be applied to the fibers to enhance wetting. The choice of treatment or coating depends on the matrix. Another way to enhance wetting is the use of a high pressure during infiltration (Bambal, 2007). A third method is to add a wetting agent to the matrix or matrix precursor before infiltration. As the wettability may vary with temperature, the infiltration temperature can be chosen to enhance wetting. The occurrence of a reaction between the fibers and the matrix helps the wetting as also bonding between the fibers and the matrix. However, an excessive reaction degrades the fibers, and the reaction products may be undesirable for the mechanical, thermal, or moisture resistance properties of the composite. Therefore, an optimum amount of reaction is preferred.

2.1.2 Fibers

2.1.2.1 Introduction

Fibers are the principal constituents in a fiber reinforced composite. They generally have the highest volume fraction in the composite material. Fibers share a major portion of the load acting on the composite. Orientation of the fibers can influence the following properties of a composite:

1. Specific gravity
2. Tensile strength and modulus
3. Compressive strength and modulus
4. Fatigue strength
5. Thermal and Electrical Conductivity

2.1.2.2 Glass Fibers

Glass fibers are the most commonly used fibers for polymer composite manufacturing because of its various advantages like low cost, high chemical resistance, good insulating properties and high tensile strength. It also has some disadvantages namely low fatigue resistance, high specific gravity, low tensile modulus and high hardness those can cause abrasion to molding and cutting tools.

Three different types of glass fibers are generally available, namely, E-glass, S- glass and C-glass. Out of these three glass fibers, E-glass is the cheapest, more commercially available and widely used in Industrial applications. S-glass is the improved version of E-glass, developed with higher tensile strength, making it more costly than E-glass and it finds its use in aircraft components and missile casings. C-glass was developed for chemical applications. Hence, it has greater resistance to corrosion and acids. Glass fibers have a three dimensional structure having long network of silicon, oxygen, calcium etc., arranged in a random fashion which makes the glass fibers amorphous and isotropic. Typical composition of E-glass and S-glass can be seen in Table 2.1

Table 2.1 Composition of glass fibers (in weight percent), (Mallick, 1997)

Type	SiO ₂	Al ₂ O ₃	CaO	MgO	B ₂ O ₃	Na ₂ O
E-glass	54.5	14.5	17	4.5	8.5	0.5
S-glass	64	26	--	10	--	--

2.1.3 Resin System

Large number of commercial resins is available for manufacturing composites. Selection of the suitable resins depend upon their compatibility with fibers, manufacturing process, and property requirements. Resins are broadly classified in the following categories:

1. **Thermosetting** resins can operate in the temperature range of 125-350⁰C. Phenolics, polyesters, polyamide etc. are few examples of thermosetting resins.
2. **Thermoplastic** resins operate in limited temperature range (near ambient). Acrylonitrile-butadiene-styrene (ABS), PVC, polycarbonates, Acrylics, Acetal Copolymers, Acetal Homopolymers etc. are few examples of thermoplastic resins.

2.1.3.1 Selection of Resin

Selection of resin is very important as it affects interlaminar shear as well as in plane shear properties (azom.com, article ID=997). Some of the important properties on the basis of which resin classification for a particular composite can be done are:-

1) Adhesive Properties

Adhesion of the matrix to the fiber is very important to enhance the properties of a composite. Polyesters generally have low adhesive properties. Vinyl ester is an improvement over polyesters but epoxy systems offer even better performance. The increased performance in epoxies is because of the presence of polar hydroxyl and ether groups. The adhesive properties are not solely responsible for strength of the bond but surface coating of fiber is also an important parameter.

2) Mechanical Properties

The most important mechanical properties are tensile strength and tensile modulus for any resin. A comparative study of tensile strength and stiffness for polyester, vinyl ester and epoxy is shown in Figure 2.2, with post curing for 7 days at 20°C and for 5 days at 80°C.

Figure 2.2 A comparative study of Tensile Strength and stiffness of different resins
(<http://www.azom.com/details.asp?ArticleID=997>)

The amount of shrinkage due to rearrangement and reorientation in the liquid phase, during the curing period in a resin, deteriorates the mechanical properties of the resin. Epoxy undergoes a small amount of rearrangement without releasing any volatile by products and is responsible for better mechanical properties of epoxy.

3) Micro Cracking

Before reaching the ultimate strength, the laminate reaches a stress level where the resin begins to crack from the fibers which are not aligned with the load. The spreading of these cracks throughout the matrix is known as micro cracking. Hence, for long durability of the structures, the composite the load should not increase to the point where micro cracking commences. Figure 2.3 is a stress strain curve which shows the micro cracking and ultimate tensile strength. Hence, for brittle systems, this point occurs much before the ultimate failure, thus, limiting the resin ability to reach high strain values. Also, if a structure is under varying environmental conditions like water, it can lead to absorption of water, and hence, considerably reducing the properties of the composite. Therefore, a resin with good wet out properties is required.

Figure 2.3 Stress-Strain curve of a typical fiber Reinforced polymer composite
(<http://www.azom.com/details.asp?ArticleID=997>)

4) Fatigue Resistance

Fatigue failure results from accumulation of damages such as resin toughness, amount of voids, resistance to micro cracking and various other defects during manufacturing.

5) Degradation From water Ingress

Resins generally absorb water which affects the resin-fiber bond in a composite. This might lead to a gradual long term loss in mechanical properties. Resins such as polyester and vinyl ester are prone to degradation in water due to a hydrolysable ester group. Hydrolysable components in a resin can also include dirt and debris which might get trapped during manufacturing.

2.1.3.2 Vinyl Ester

Vinyl ester resins are generally cheaper as compared to epoxy and are more durable than the polyester resins. Vinyl ester resins offer better resistance to moisture absorption when compared to polyester resin since vinyl ester is a hybrid form of polyester resin toughened with epoxy. Vinyl ester is extremely sensitive to temperature and atmospheric moisture while curing. The continuous curing of vinyl ester makes the bond weaker and in some cases, the resin can be easily peeled off. Vinyl ester generally bonds very well with glass fibers as compared to Kevlar or carbon fibers.

Epoxy Vinyl ester resin is brominated bisphenol-A to offer maximum fire retardation combined with enhanced chemical resistance and toughness. Typically, MEKP (Methyl Ethyl Ketone Peroxide) is used as an initiator or “catalyst” for the room temperature free radical curing of unsaturated polyester and vinyl ester resin. Vinyl ester resin used to make FRP composite sheets was Derekane.

Since Vinyl ester resin is epoxy based, it is generally preferred over other thermoset resins because of its excellent thermal and mechanical properties and easy handling at room temperature similar to epoxy resin. It also offers a good chemical resistance and greater control over cure rate and reaction conditions (Manab, 2008). Vinyl ester can be cured both at room temperature and high temperature. Elevated temperature curing is generally preferred by large scale manufacturers to increase production rate. High

temperature also decreases the viscosity of vinyl ester resin making it easy to handle and process.

Figure 2.4 shows the chemical structures of Vinyl ester oligomers, Cobalt Naphthenate and MEKP. In Vinyl Ester Resin, unsaturated oligomers are kept dissolved in styrene to decrease the viscosity for easy handling and process ability.

(a)

(b)

(c)

Figure 2.4 Chemical structures of (a) Vinyl Ester (b) Cobalt Naphthenate (c) MEKP (Ziaee et. al., 1998)

Epoxy based vinyl ester resins are methacrylate epoxies as can be seen in Figure 2.4. Due to the epoxy backbone there is no weak chemical link susceptible to chemical attack making vinyl ester resistant against chemical attacks. For the curing of the vinyl ester resin first Cobalt Naphthenate is added to the resin as a promoter because they decompose into free radicals. Cobalt Naphthenate acts like a catalyst and is not consumed during the reaction. MEKP is used to provide free radicals after the peroxides linkage breaks and helps to initiate the reaction. MEKP can be referred to as a true catalyst because it is being consumed in the reaction. Since MEKP decomposes very slowly, it is difficult to cure the vinyl ester resin without a promoter. Hence Cobalt Naphthenate is generally used as a promoter to decompose MEKP into free radicals. Thus, Cobalt Naphthenate is the true catalyst for vinyl ester curing process as it is not consumed during the reaction.

Commercially available Vinyl ester resins, Derakane Momentum with Permek N: 0.8 and cobalt naphthenate: 0.06 as additives were used to make FRP composite sheets through Pultrusion. Derakane Momentum 411-350 Epoxy Vinyl ester resin offers higher reactivity, easier workability, reduced stress cracking, shorter curing time, light color and longer shelf life. It is based on bisphenol-an epoxy resin, hence, it provides resistance to a

wide range of acids, alkalis, bleaches and solvents. Table 2.2 lists the typical properties of the resin.

Table 2.2 Vinyl Ester data sheet for Derakane 411-350 Resin (Ashland Co.)

Typical Properties for VE Resin	
Dynamic Viscosity @ 25°C (77°F), <i>mPa.s</i>	370
Styrene Content, %	45
Density @ 25°C (77°F), <i>g/ml</i>	1.046
Commercial Warranty, dark, @ 25°C (77°F), <i>months</i>	12
Typical Room-Temperature Properties of Clear Castings Made with DERA KANE 411-350 Resin	
Tensile Strength, <i>MPa/psi</i>	86/12,000
Tensile Modulus, <i>GPa/unit 10⁵ psi</i>	3.2/4.6
Tensile Elongation, %	5.0-6.0
Flexural Strength, <i>MPa/psi</i>	150/22000
Flexural Modulus, <i>GPa/unit 10⁵ psi</i>	3.4/4.9
Specific Gravity	1.14
Heat Distortion Temperature, °C (F°) <i>at 1.82 Mpa (264 psi) applied stress</i>	105/220
Barcol Hardness	35

2.1.4 Glass Fiber Reinforced Polymer Composites

Fibers are the major load carrying component of a fiber reinforced composite. Fibers are used in composite because of the following advantages: (a) fibers are light weight, (b) fibers are stiff and strong, (c) fibers are stronger than the bulk material because of the preferential orientation of the molecules along the fiber direction and also that (d) a fiber contains less number of defects as opposed to the bulk material (Barbero, 1998). Fibers may be continuous or discontinuous like chopped or short fibers. Continuous fibers are preferred in structural applications because the composite with discontinuous fibers can undergo a large creep deformation. Different types of fibers are available such as glass, carbon, Kevlar (organic), boron, alumina etc. Choosing the type of fiber depends mainly on the following factors: (a) mechanical properties, (b) environmental conditions and (c) cost of fiber. In our application of FRP composite, we have chosen glass fibers as the reinforcing component because of their: (a) good tensile strength, (b) high strength to weight ratio, (c) little moisture absorption, (d) high Electrical resistance and (e) economical feasibility for mass production.

Fibers are available in various forms such as roving, strands, tow and yarns. Depending upon construction and orientations, fiber/fabrics are classified into unidirectional (1D), bi-directional (2D) and even advanced fabrics (3D) including mats. Unidirectional fibers run in one direction (0^0 directions) only, and can be used to mainly resist tensile loads in that (fiber) direction. Mats are of two types: chopped strand mat and continuous strand mat. In case of multidirectional fabrics, multiple layers of continuous roving are held together by stitching in the plane direction of the fabrics. 2D fabrics are classified into bi-directional (e.g., 0/90, +45/-45 etc.), tri-directional (0/45/-45; 90/+45/-45; etc) and quadric-directional fabrics (0/90/+45/-45). For our laboratory research, we have used unidirectional fabric and tri-axial fabric system with vinyl ester resin manufactured by pultrusion.

2.1.5 Manufacturing Method

Pultrusion was used for manufacturing and production FRP composites. The composites used in this study were unidirectional E-glass/Vinyl ester composites and Poly-Urethane based tri-axial glass fiber composites and these were prepared using pultrusion. Pultrusion method of manufacturing is discussed below:

2.1.5.1 Pultrusion Process

Pultrusion is a continuous manufacturing process used to produce lengths of fiber reinforced polymer structural shapes with constant cross-sections. Due to the continuous nature of the process, composites of any desired length can be produced. Raw materials are a liquid resin mixture (resin, fillers and some specialized additives) and flexible reinforcing fibers. The process involves pulling these raw materials rather than pushing as in the case of extrusion, through a heated steel forming die, using as a continuous pulling device. The basic process consists of drawing the reinforcement through a resin bath followed by a die of required shape to remove any excess resin or entrapped air. The material is then pulled through the oven for resin curing. Process speed varies depending upon the nature of the resin and the geometry required; faster line speeds are desirable. These lead to increase in process efficiency and economics. Pultrusion can be used to produce composites with higher fiber volume fraction, thus, providing higher mechanical properties (about.com). Commercial applications for pultruded composites are mostly

standard structural shapes like I-beams, channel sections, nonconductive ladder rails, tool handles, grating and window framing.

Pultrusion process can be divided into four basic steps described below and the schematic of pultrusion process is shown in Figure 2.5.

1. Reinforcements

The first step in the process is to set up the reinforcements for pulling. Roving can be used for unidirectional orientation of the structures or fabrics can be used to add off-axis fibers. Since the entire cross-section is pulled at once, instead of building it up as a traditional lay-up, a large number of spools are used. A large number of strands could end up in tangles, hence, after leaving the creels; the fibers are passed through an alignment card which also prevents twisting of the roving.

2. Resin Impregnation

After the fibers are aligned, they pass through a resin bath, to achieve a complete wet-out. The strands are passed through a series of rollers which flatten and spread out individual roving. A long resin bath is also used to achieve a good wet-out. Different resins can be used depending upon the property requirements. Polyesters, vinyl esters and epoxies are most commonly used. The resins used in pultrusion should have a fairly low viscosity for good and rapid impregnation, a long pot life for continuous processing and a short cure time to achieve full cure during the time the pultrusion is in the die.

3. Pultrusion Die

Die is the most important part of the process; the impregnated fibers enter the die and exit as the required cured part. The die helps in maintaining fiber alignment, compresses the fibers to the desired volume fraction and cures the composites in a relatively short period of time without damaging the fibers.

4. Pulling and Cutoff

The pulling station is located at the end of the pultrusion line. A set of padded clamps are used to grip the cross section. The grips are translated horizontally to pull the section. Two sets of grips are used to make the process continuous, with one pulling the cross section till the other one travels back to its initial position. Pulling force used is of the order of six to eight inches or of higher dimensions.

A radial cutoff saw is located beyond the pulling station. The saw is mounted on the table that moves with the pultrusion to achieve clean cuts even as the pultrusion continues to travel.

Figure 2.5 Manufacturing line for typical pultrusion process (Pultrusion Industry Council), (<http://www.olemiss.edu/depts/compmat/>)

2.2 Long Term Durability of FRP and Life Predicting Models

The most significant technical obstacle preventing the extensive use of FRP composites is lack of long term durability and performance data. Most of the data for glass reinforced composites involve short fiber materials and have not been collected at the high loading levels associated with many structural applications. Hence, there is much concern about the long term behavior of composites, such as creep rupture and environmental aging due to various service environments (Liao et. al., 1998). Hence, research studies are needed, especially for polymer composites manufactured using pultrusion due its low cost for large volume process ability.

A fiber reinforced composite in an infrastructure application may or may not sustain constant or repeated loading for prolonged period of time under different environmental exposure. The service conditions such as environmental effects, creep, stress rupture, stress corrosion, fatigue and environmental fatigue are very important while designing infrastructure systems.

2.2.1 Introduction to Creep

Creep is defined as increase in strain with time at a constant stress level. In polymers, creep occurs due to a combination of elastic deformation and viscous flow of polymer molecules, commonly known as viscoelastic deformation. Due to viscoelasticity, FRP's are more susceptible to creep than the conventional materials such as metals. Creep strains lead to nonlinear response with time, as is seen in Figure 2.6. When stress is released after a period of time, it is expected that the elastic deformation is immediately recovered whereas creep strain is recovered partially.

Creep strains in polymers and polymer matrix are largely dependent upon the percent of rupture stress induced in a member and temperature. Most polymers exhibit large creep strains at room temperature and at low stress levels. At high temperatures or high stress levels, creep phenomenon becomes more critical. Generally, highly cross-linked thermoset polymers exhibit lower creep strains as compared to thermoplastic polymers (Mallick, 2008). However, load in FRP composites is carried primarily by the fibers, which behave elastically; hence creep is not that significant in composites as compared to

bulk polymer matrix, particularly along fiber direction, which is designed typically to be the primary load path.

Reinforcement content, direction of reinforcement and structure of material, influence the creep behavior of FRP, but polymer matrix is the major cause of creep in FRP composite; Creep due to glass fiber is considered insignificant. Deformation of molecules, with molecular segments changing conformations and sliding past one other, is the cause of creep in polymeric materials. Large deformations can cause chain ruptures predominant in thermosets since chains are cross linked to a network. Hence, mechanical properties tend to change as function of time for which load is applied, and as function of temperature, moisture and other environmental factors. These properties tend to alter the creep behavior due to their effect on molecular activity in polymers. High loading levels damage the fiber-matrix interfacial regime, leading to slip between the matrix and the fiber. Fiber rupture causes high fiber stresses in the surroundings intact fiber leading to elongation and hence, increases the rate of creep.

Figure 2.6 Representation of creep strain and recovery strain in a polymer
(Mallick, 2008)

2.2.1.1 Creep compliance

Creep behavior of polymer matrix or composites under uni-axial stress is represented by creep compliance, given by Equation 2.1.

$$\text{Creep compliance} = D(t) = \frac{\epsilon(t)}{\sigma} \quad (2.1)$$

Where, σ is the constant stress applied and $\epsilon(t)$ is the strain as a function of time

Figure 2.7 Tensile creep curves for SMC-R25, polyester laminates (Mallick. 2008)

Figure 2.7 shows a typical creep curve for an SMC R-25 laminate at various stress levels; its creep compliances can be determined by slopes of these curves. Generally, creep compliance increases with stress levels, temperature and time. It is also a function of fiber orientation, angle θ . For $\theta = 0^\circ$ creep compliance remains constant indicating negligible creep in unidirectional laminate in fiber direction. Creep in other fiber orientation can be significant.

Temperature dependence of creep compliance is also influenced by fiber orientation angle. Creep in composites is governed by creep in fibers if the fibers are in loading direction. But for other orientations, creep due to matrix is dominant. Hence, for off axis, laminates creep compliance is proportional to temperature. Table 2.3 gives the creep compliance of unidirectional laminates with different fiber orientation and temperature.

**Table 2.3 Creep compliance of unidirectional E-Glass-Epoxy laminates
(Mallick, 2008)**

Temperature	Property	Fiber orientation angle			
		30 ⁰	45 ⁰	60 ⁰	90 ⁰
28 ⁰ C	Tensile Strength(MPa)	278	186.4	88.8	55.2
	1-h compliance(10 ⁻⁶ MPa)	0.0883	0.2065	0.3346	0.5123
	10-h compliance(10 ⁻⁶ MPa)	0.1300	0.3356	0.6295	1.4390
75 ⁰ C	Tensile Strength(MPa)	230	162.8	74.8	43.2
	1-h compliance(10 ⁻⁶ MPa)	0.1217	0.2511	0.4342	0.6689
	10-h compliance(10 ⁻⁶ MPa)	0.1461	0.3841	0.6539	1.5377
100 ⁰ C	Tensile Strength(MPa)	206	134.4	67.7	40.2
	1-h compliance(10 ⁻⁶ MPa)	0.1460	0.3586	0.6931	0.7728
	10-h compliance(10 ⁻⁶ MPa)	0.1751	0.5739	1.2084	1.9031

2.2.2 Creep Models based on Stress variations (Liao et. al., 1998)

2.2.2.1 Findley's Power Law Model

In Findley's power law model the creep strain is represented in power law form, given by Equation 2.2.

$$\epsilon = \epsilon_0 + m (t / t_0)^n \quad (2.2)$$

Where, ϵ is the total strain, ϵ_0 is a stress dependent, time independent elastic strain, t is the time, and m and n are creep parameters which are determined experimentally, t_0 is the scaling factor generally taken as unity.

Yen and Williamson (Yen et. al., 1990) have shown for glass-polyester sheet molding compound, the creep parameter m is temperature and stress dependent. Whereas Augl (Augl et. al., 1987) has shown that n is slightly stress and temperature dependent for dry epoxy resin and its stress-dependency is less pronounced with longer aging time. Augl also found that n is linearly dependent on moisture content of epoxy. Hence to account

for stress dependency of both elastic strain ϵ_0 and time dependent strain ϵ_t , the following Equation 2.3 was used (Findley, 1960):

$$\epsilon = \epsilon_0 \sinh (\sigma / \sigma_0) + m (t / t_0)^n \sinh (\sigma / \sigma_t) \quad (2.3)$$

Where σ_0 , t_0 and σ_t are experimentally determined, and σ is applied stress. For very low stresses ($\sigma \ll \sigma_0$ and $\sigma \ll \sigma_t$), Equation 2.3 can be written as:

$$\epsilon = \epsilon_0 (\sigma / \sigma_0) + m (t / t_0)^n (\sigma / \sigma_t) \quad (2.4)$$

Rearranging Equation 2.4 we get

$$\frac{\sigma}{\epsilon} = E_v = \frac{E_0 E_t}{E_t + E_0 (t / t_0)^n} \quad (2.5)$$

Where $E_0 = \sigma_0 / \epsilon_0$, $E_t = \sigma_t / m$, and E_v is known as viscoelastic modulus. Analogy for Equation 2.2 for shear deformation is:

$$\gamma = \gamma_0 + m_g (t / t_0)^{n_g} \quad (2.6)$$

Where γ is the total shear strain, γ_0 is the initial shear strain, m_g and n_g are parameters related to shear deformation under creep. Hence, the time dependent shear modulus can be written similar to Equation 2.4 as:-

$$\frac{\tau}{\gamma} = G_v = \frac{G_0 G_t}{G_t + G_0 (t / t_0)^{n_g}} \quad (2.7)$$

Where τ is the shear stress, $G_0 = \tau_0 / \gamma_0$, $G_t = \tau_t / m_g$, τ_0 and τ_t are constants.

Findley's Model has been verified by a 26 year long (Findley et. al., 1987) experiment on polyvinyl chloride and polyethylene in which predictions were based on a short term 1900 h creep data and it proved to be in a good agreement with the long term 26 year data.

Compression creep of pultruded FRP composites was investigated by Scott and Zureick (Scott et. al., 1998) at three different stress levels and time durations of up to 10,000 hrs. The material system they used was vinyl ester matrix with unidirectional E-glass roving and continuous filament mat (CFM) with a fiber volume fraction of 30% and filler content of 5% by volume. According to their study, it was observed that stress level of 60% is when the material begins to show non linear behavior. They modeled the data using Findley power law, but since Findley's model was based on unreinforced thermoplastic material and while they used a low fiber volume fraction of 30% the authors asserted that the creep behavior would primarily be matrix driven. Hence, Findley's model would provide an accurate approximation.

2.2.2.2 Boltzmann-Volterra Model

According to Boltzmann superposition principle, which is only valid only for viscoelastic regime, states the effect of change in applied stress as the algebraic sum of the time-dependent strain resulting from the new stress and the time dependent strain in the system had the change not been imposed.

Maksimov, et. al. (1975) have used the Boltzmann-Volterra theory to describe the viscoelastic behavior of polymers and polymer composites, in which the stress-strain relationship can be described in the form of Volterra integral equation.

$$\varepsilon = J_0 \sigma(t) + \int_0^t J_t(t-\tau) \frac{d\sigma(\tau)}{d\tau} d\tau \quad (2.8)$$

Where J_0 is time independent creep compliance and $J_t(t)$ is time dependent creep compliance. This method can be applied to polymeric material under shear or under a combined tensile and shear behavior.

Its successful prediction for viscoelastic behavior of polyester, from short term results, was with experimental data for 6 years with varying temperature and humidity conditions, was done by Maskinmov, et. al. (1975)

2.2.2.3 Schapery Model

Schapery's nonlinear viscoelastic theory is derived from the fundamentals of irreversible thermodynamic principles. Hence, Schapery model gives an accurate description of creep behavior for polymers and their composites. It is considered to be an analytical form of a time-stress superposition principle (Schapery et. al., 1969).

Hence for Uni-axial loading under isothermal conditions, creep strain is expressed as:

$$\varepsilon = g_0 D_0 \sigma + \int_0^t D_t (\psi - \psi') \frac{dg_2 \sigma}{d\tau} d\tau \quad (2.9)$$

Where D_0 is the initial creep compliance and D_t is the transient creep compliance. ψ and ψ' are reduced time parameters and g_0 , g_1 and g_2 are stress dependent material parameters. In case of linearly viscoelastic materials or under low stresses, the Schapery model reduces to Boltzmann superposition model (Equation 2.8).

2.2.3 Effect of Moisture and Temperature on Creep

Moisture uptake and temperature can significantly affect the physical and mechanical properties of polymer or polymer composite. Therefore, it can be assumed that a laminate strength is a function of temperature, moisture and strain rate.

Similar to temperature and time, moisture and time also have an interchangeable effect on creep behavior of laminates and polymers (Liao et. al., 1998). Creep compliance usually increases with increase in temperature and moisture content with time (Figure 2.8). Moisture absorption in a poly matrix plasticizes the resin and leads to the development of residual stresses. Generally, moisture absorption level is history dependent; hence, the moisture absorption will be different for varying temperatures.

**Figure 2.8 Effect of time temperature and moisture on creep compliance
(Liao et. al. 1998)**

The effect of water immersion on compressive creep was investigated by Panasyuk, et al. (1987) for unidirectional glass reinforced composite. It was found that there was a limiting creep deformation ($d\epsilon/dt = 0$) for low stress levels once the limit was reached and for stress levels of 85% or above of the ultimate static strength, a steady creep rate was observed ($d\epsilon/dt = \text{constant}$). But in a liquid medium, both limiting and steady creep were observed at significantly low stress levels and further that the rate of creep also showed considerable increase. This behavior was attributed to redistribution of stresses between glass fibers and polymer matrix. The damaged and undamaged reinforcing parts and plasticizing also played a part in the change of behavior observed in the moisture. However, at elevated temperatures, the response pattern of composite was retained but range of limited creep became constricted.

Effect of moisture, temperature and stress on creep behavior glass epoxy composite was studied by Wang et. al. (1980). During this study unidirectional laminates were used and were loaded at different angles (of 0° , 45° and 90°) with respect to fiber direction. It was observed that 0° laminates show minimum creep deformation when compared to 45° and 90° laminates but a significant creep deformation was observed at elevated temperature of 102°C . Creep of 45° and 90° even at low stresses was strongly influenced by moisture and temperature. Table 2.4 shows a list of creep studies on FRP composites with varying temperature and environmental conditions.

Table 2.4 Selected Creep studies on different FRP composites

Creep Studies on FRP						
Material	Structure	Loading Mode	Temperature range ($^{\circ}\text{C}$)	max creep time (h)	Environment	Reference
E-glass/ polypropylene	chopped-fiber bar	tension	23-80	300	50% R.H.	Cessna, et al. (1971)
glass/polyester	random fiber mat	tension	25	719	air	Howard, et al. (1987)
E-glass/epoxy	laminates	tension	20	556	60% R.H.	Weidmann, et al. (1974)
glass/epoxy	woven fabric	flexural	50	1	air	Zhang, et al. (1989)
E-glass/epoxy	laminates	tension	RT-150	50	air	Bhatnagar, et al. (1981)
glass/epoxy	laminates	tension	24-102	17	0-1% moisture	Wang, et al. (1980)
glass/polyester	SMC	tension	23-149	3	ambient	Yen, et al. (1990)
E-glass/vinyl ester E-glass/polyester	pultruded laminates	flexural	ambient	100	ambient	Levy, et al. (1993)
glass/epoxy- phenolic	rod	compression	20-120	9735	air, water	Panasyuk, et al. (1987)
glass/epoxy	laminates	compression	20	168	ambient	Irion, et al. (1981)
glass/epoxy	pultruded rod	compression	21	840	ambient	Spence, et al. (1990)

2.2.3.1 Time-Temperature Stress Superposition Principle (TTSSP)

For an infrastructure, designing a creep deformation is a tough task requiring collection of data for many years, time consuming, costly and impractical and hence, with predictive model based on superposition theory of viscoelasticity long term data can be obtained by using many short term accelerated tests. Like temperature, increased stress also shows acceleration in deformation.

Time-Temperature Stress Superposition is based on:

- 1) Increase in temperature and stress accelerates viscoelastic deformation process.
- 2) Creep deformation curves are similar in shape for different conditions.
- 3) A shift is observed on log time scale due to change in stress or temperature.
- 4) All these deformation curves at different temperature and time can be combined into a smooth curve, known as master curves which represent long term viscoelastic behavior at a given condition.

From the free volume theory, there is Doolittle equation which gives a relationship between the coefficient of viscosity η and the volume fraction f and is given by:

$$\ln \eta = \ln A + B \left(\frac{1}{f} - 1 \right) \quad (2.10)$$

where A and B represent material constants.

The stress acts on the free volume of material like temperature according to superposition principle. Assuming change (LI Zhi-da et. al., 2007) in free volume fraction is linearly dependent on change in stress and temperature, free volume fraction can be written in a linear equation as:-

$$f = f_0 + \alpha_T (T - T_0) + \alpha_\sigma (\sigma - \sigma_0) \quad (2.11)$$

Where α_T is coefficient of thermal expansion, α_σ is coefficient of expansion of stress, σ_0 and T_0 are reference stress and temperature and f_0 is the free volume fraction at reference temperature and stress.

Assuming a temperature and stress shift factor $\phi_{T\sigma}$ that satisfies the following equation:

$$\eta(T, \sigma) = \eta(T_0, \sigma_0) \phi_{T\sigma} \quad (2.12)$$

Combining Equations 2.10, 2.11 and 2.12 we obtain:

$$\log(\phi_{T\sigma}) = -C_1 \left[\frac{C_3(T - T_0) + C_2(\sigma - \sigma_0)}{C_2 C_3 + C_3(T - T_0) + C_2(\sigma - \sigma_0)} \right] \quad (2.13)$$

Where $C_1 = B/(2.303f_0)$; $C_2 = f_0/\alpha_T$; $C_3 = f_0/\alpha_\sigma$.

When Stress is equal to reference stress Equation 2.13 becomes WLF equation which was first proposed by Williams, Landel and Ferry for time temperature superposition. Similarly, when Temperature is kept at reference temperature, the Equation 2.13 becomes time stress superposition.

Now the stress shift factor at constant temperature ϕ_σ^T and temperature shift factor at constant stress ϕ_T^σ are defined such that:-

$$\eta(T, \sigma) = \eta(T, \sigma_0) \phi_\sigma^T = \eta(T_0, \sigma_0) \phi_T^{\sigma_0} \phi_\sigma^T = \eta(T_0, \sigma) \phi_T^\sigma = \eta(T_0, \sigma_0) \phi_\sigma^{T_0} \phi_T^\sigma \quad (2.14)$$

Therefore,

$$\log(\phi_\sigma^T) = -C_1 \frac{C_2}{C_2 + (T - T_0)} \left[\frac{C_2(\sigma - \sigma_0)}{C_2 C_3 + C_3(T - T_0) + C_2(\sigma - \sigma_0)} \right] \quad (2.15)$$

$$\log(\phi_T^\sigma) = -C_1 \frac{C_3}{C_3 + (\sigma - \sigma_0)} \left[\frac{C_3(T - T_0)}{C_2 C_3 + C_3(T - T_0) + C_2(\sigma - \sigma_0)} \right] \quad (2.16)$$

Now from Equations 2.12 and 2.14 we can obtain:-

$$\phi_{T\sigma} = \phi_T^{\sigma_0} \phi_\sigma^T = \phi_\sigma^{T_0} \phi_T^\sigma \quad (2.17)$$

Hence, through Equation 2.17, we can obtain behavior under any Temperature T and stress σ by shifting the time-dependent properties of viscoelastic materials at varying temperature and stress levels by shifting along the time scale to construct a master curve with a larger time scale at a given temperature, T_0 , and stress level σ_0 .

TTSSP was found to work for long term creep analysis by generating master curves, Ma et al (1997) examined the stress and temperature effects on accelerated creep responses of composite laminates. Distribution of relaxation times in HDPE was examined using TTSSP by Lai et. al.(1995). Also that PMMA's crazing damages under creep were studied by Li Zhi-da et. al.(2006) using TTSSP.

CHAPTER 3

MATERIAL AND MANUFACTURING PROCESS

3.1 Introduction

This chapter deals with a brief overview of constituent materials such as type of fiber, polymer matrix, fiber configuration and manufacturing methods. Also, the coupon preparations for tension, compression, bending and creep test etc. are also mentioned.

3.2 Unidirectional Pultruded Samples

These samples were manufactured through pultrusion in the form of thin sheets (0.134" thick) by Kazak composites Co. For this study, E-glass/vinyl ester fabric-resin system was chosen due to its effectiveness as a material of construction and because of its high mechanical performance in terms of strength, durability and low cost. The fiber orientation in all sheets was unidirectional. Hence, only one direction had fibers. The properties of the composite in fiber direction (0^0) was due to both matrix and fiber contribution whereas in the transverse direction (90^0) the properties of the composite were matrix driven. The fiber volume fraction and weight fraction were experimentally measured to be 61% and 79.42%. These unidirectional pultruded samples were tested for the static properties and creep properties with varying environmental conditions of temperature and moisture.

3.3 Quasi Isotropic Compressed samples

These samples were also manufactured by Kazak Composites Co. in the form of thin laminates (0.09" thick) using compression molding technique. These samples were prepared using E-glass/vinyl ester fabric resin system. The fiber orientation in the sheets was $[90/+45/0/-45]$ and equal amount of fiber was used in each direction. The strength of these samples was the same in both directions due to its isotropic nature because of the same amount of fabric in each direction. These samples were used to perform static tension tests and tensile creep tests. A quarter inch hole was drilled in the center of the

sample to study the failure induced due to hole. The measured fiber volume and weight fraction for these samples were 59.74% and 79%.

3.4 Tri-axial Pultruded Samples

These samples were manufactured by Creative pultrusion Inc. in the form of sheets with two different thicknesses (1/2" and 1/4") using pultrusion. A brief description of its part construction can be seen in Figure 3.1 and Figure 3.2. The resin fiber system used for these samples was Poly-Urethane/E-glass. As seen from the figures, the fiber orientation was tri-axial with almost equal amount of fiber in each direction. Half inch thick samples had a fiber volume fraction of 46.6% and fiber weight fraction of 71.05%. Quarter inch thick samples had a fiber volume fraction of 46.49% and fiber weight fraction of 66.1%. This system of composite was used to study the three point bending and flexural creep behavior.

Figure 3.1 Part construction of the tri-axial composite for half inch thick samples as provided by Creative Pultrusion

PART CONSTRUCTION
 12" x 1/4" FLAT STRIP
 PART # FT191.794 DIE # 287
 DATE: 10-16-08

1) CN 1501 IS A 1.5 OZ CFM SEWN TO A POLYESTER VEIL
 2) E-TTXM 4008 IS A TRIAXIAL 24 OZ/YD² IN THE +/-45° DIR. AND
 16 OZ/YD² IN THE 90° DIR. WITH 6.75 OZ/YD² CHOP ON ONE SIDE.

Figure 3.2 Part construction of the tri-axial composite for quarter inch thick samples as provided by Creative Pultrusion

3.5 Sample Preparation

All the samples were cut in required dimensions using diamond saw. Tabs were for the tension and compression tests and were cut in required dimensions and these were glued using Plexus MA 310 on each end of the sample. The sample dimensions and tab dimensions are mentioned in more detail in chapter 4.

CHAPTER 4

EXPERIMENTATION

This chapter gives an introduction to coupon level testing used for the mechanical characterization and to determine the physical properties of the composite samples. The mechanical testing mainly includes tension, bending, compression and creep on coupon level samples. Experimental setup, testing equipment and procedure for data analysis are briefly discussed. Also, physical characterizations such as fiber volume fraction through burn out test, void content through SEM are also narrated in this chapter.

4.1 Testing Equipments

This section deals with different apparatus used for mechanical testing of composites. Important parts of these equipments and their working principles are discussed. The major equipments used for testing include,

1. Instron 8501 Machine for conducting bending, tension and compression tests
2. Data Acquisition System for recording strain, and load data
3. Environmental Chamber and moisture variation
4. Creep test frame for tensile and bending creep

4.1.1 Instron Model 8501 Servo-Hydraulic Material Testing System

Instron-8501 with the maximum load capacity of 1000-kN or 22 kip can be used to conduct the broad range of static tests such as compression, tension, shear, fatigue and bending. A control panel was used to maintain the uniformity of the applied load rate. Important components of Instron-8501 are listed below.

- Strength-Testing Machine
- Mechanical Clamps
- Hydraulic Power Supply
- Control Tower
- Control Panel
- Computer and Dedicated Data Recording Unit

This equipment setup was used to carry out bending, compression and tension. During a test, following parameters can be controlled:

- Position (by controlling the travel of the hydraulic piston)
- Load (force applied to the tested sample)
- Deformation (distortion of the tested sample)

The Instron unit consists of a two-column load frame with movable crosshead. Auxiliary hydraulic lift cylinders help to regulate the position of the crosshead. Hydraulic column clamps fix the crosshead in the desired position. A load cell, mounted on the crosshead, could measure load measurement in the range of ± 100 kN. The force applied to the specimen is sensed by in-built strain gage type load cell placed on the stationary top cross-head, where the analog signals from the load cell are amplified and converted into a digital signal. The machine is operated by the control panel of the computer, where the readings are displayed and are transmitted to the data acquisition computer for data collection using WaveMaker[®] software. The displacement is determined from grip position. The data were processed for evaluating mechanical properties. Figure 4.1 shows a typical equipment setup for a bending test.

Figure 4.1 Instron model 8501 servo-hydraulic material testing system

4.1.2 Data Acquisition Unit

It is an independent unit and is not a part of the Instron-8501; it consists of a computer system and Model 5000 scanner (Vishay) as seen in Figure 4.2. It is generally used to record data by externally connecting to any testing equipment. It can collect instantaneous data such as load, deflection, strain etc. For static compression and tension tests data acquisition were used to determine the strain. Strain gage lead wires and external output of the Instron were connected to the Model 500 scanner, which was further connected to the computer. The loading rate was maintained using Instron through a programmable controller and the corresponding load versus strain data was recorded using data acquisition unit. The recorded data was then converted to an Excel sheet for data analysis.

Figure 4.2 Data acquisition unit

4.1.3 Environmental Chamber

Environmental chamber was used to provide varied environmental conditions of temperature and moisture. Figure 4.3 shows the environmental chamber mounted on Instron-8501. The chamber is heated via a mineral insulated element located next to a three speed fan at the rear chamber. The convected air is channeled using a baffle plate to the top and the bottom of the chamber and returned through the center of the baffle plate. A safety Interlock switch operated by the door hinge cuts power to the heating elements and the fan, if the door is opened during operation. The inner chamber is constructed from stainless steel with high efficiency, low thermal mass ceramic fiber insulation packed around it. The temperature range of the chamber varies from -70°C to 250°C .

Figure 4.3 Instron environmental chamber

4.1.4 Creep Frame

Two types of creep frame were made to perform tension and three point bending creep tests.

4.1.4.1 Three point bending creep

A set up for three point bending creep tests was prepared as shown in Figure 4.4. The setup consisted of two small rollers one small piece of hollow steel tube, a thin steel rod and weights. Two steel rollers were kept at the required span length of the bending creep sample to provide point contact. The small hollow piece of tube with a hole to hang the rod vertically was used to provide a point load. Then the required load was hung on the steel rod. Deflections were measured as a function of time through a dial gauge.

Figure 4.4 Three point bending creep frame

4.1.4.2 Tension Creep

The setup consisted of two plates that were welded together with a nut for dywidag bars leaving enough space for the specimen between them. Small holes were made between the plates and the sample and two screws were passed through the holes in the plate and holes in the sample to hold the sample together, this was done on both ends of the sample. Then the whole frame was kept vertically with a load cell between the frame and a strain gauge in the center of the sample to measure load and strain through data acquisition. The nut on dywidag bar was then tightened with a wrench till the required load was achieved. Load and strain were measured using data acquisition.

Figure 4.5 Tension creep frame

4.2 Testing Methodologies

Unidirectional vinyl ester/glass fiber coupon specimens were subjected to tension compression bending and creep test; also tri-axial poly-urethane based composites were tested for bending and creep test and quasi isotropic vinyl ester composites were tested for tension and creep test to determine the mechanical properties. Comprehensive summary of standard testing methodologies, results and data analysis for each of the above is listed below.

4.2.1 Tension Test

This test was used to determine elasticity and tensile strength. All the tests were carried out as per ASTM D3039 standards. The specimens subjected to tension test were of the size 10"x1". Thicknesses of test specimens were around 0.134" and 0.09". Schematic diagram of tension test specimen is shown in Figure 4.6. Test specimens were bonded with composite tabs of 3" on both the ends.

Cross Sectional View A-A

Figure 4.6 Schematic of tension test sample along with the cross sectional view A-A

The tabs distribute gripping stresses and prevent specimen failure caused by grip jaws. Commercially available bonding agent like Plexus MA310 TM, Pliogrip TM and epoxy were used for this purpose. A minimum of 24 hours curing time was allowed for the bonding agent before testing the samples. Strain gages were mounted axially along the length of the specimen. All the tension test specimens were tested with Instron 8501 with environmental chamber to maintain temperature and moisture conditions as also maintaining actuator rate nearly constant. Data of load versus strain were recorded from tension test through data acquisition unit. The data was then reduced for stress versus strain curve. The linear portion of stress versus strain plot is referred to as modulus of elasticity, a measure of stiffness of material. Results and comparison for different sets of sample are summarized in chapter 5. Typical Tension samples can be seen in Figure 4.7.

Figure 4.7 Tension test specimens

4.2.1.1 Step-wise Test Procedure for Tension Test

- I. Width and thickness of sample were measured at three different locations. Cross sectional area was calculated from average width and thickness.
- II. The end surfaces of the sample were grinded to facilitate a good bond between tabs and sample surface. Tabs of 3” length were bonded on the end surfaces. End surface of the tab was made flat after grinding the unnecessary glue on it. It also helps in holding samples tight inside the jaws.
- III. Next step of sample preparation includes mounting strain gages. For the specimen tested here, strain gages were axially mounted at mid-span along the length.
- IV. In tension test, specimen was centrally aligned between the jaws ensuring uniform force distribution. Strain gages were connected to data acquisition unit with connecting wires. Load cell of Instron machine was externally connected to the input port of data acquisition unit. During the test, loading rate was maintained constant at 0.05 in/min. All the samples were tested till failure.
- V. Data was recorded for load versus strain variations during the test. Experimental values of tensile strength and stiffness were calculated. This procedure was repeated for all the samples.

Typical plot of stress vs. strain for unidirectional pultruded sample in transverse direction is shown in Figure 4.8.

4.2.1.2 Tension Test Calculations

Tensile strength $\sigma_{ult} = \frac{P_{ult}}{wt}$ psi

Tensile modulus $E_{ss} = \frac{\sigma}{\epsilon}$ msi

Where,

P - Applied load on the specimen (lbs)

w - Average width of specimen (in)

t - Thickness of specimen (in)

$A = W \times T$ = Cross-sectional area of the specimen (in²)

E_{ss} – Calculated from slope of elastic zone of Stress Vs. Strain curve (psi Vs micro-strain)

Figure 4.8 Typical plot of stress vs. strain for E-glass/vinyl ester pultruded samples under tension test

4.2.2 Bending Test

This type of testing method is used to determine flexural properties of composite material under three point or four point bending tests. In this study, three point bending test was chosen for coupon level testing for both pultruded specimens.

4.2.2.1 Three Point Bending Tests

In case of three point bending, concentrated load P is applied at mid-span. A schematic representation of test set up is shown in Figure 4.9. This test method was used for coupon level specimen testing. Tests were carried on Instron-8501.

Figure 4.9 Schematic of 3pt. bending (mid-span type) test set up with stress and moment distribution in the component

The entire set of coupons was tested for bending in longitudinal directions to determine the flexural properties of the material. Tests were carried out as per test methods of the ASTM 790 (2005) and ASTM 6272 (2005) specifications. An over hang of 0.5" on either side was provided to prevent the specimen from slipping through the supports. All tests were displacement controlled at actuator rate of 0.05 in/min. The data from the tests were used to obtain the flexural modulus and flexural strength.

4.2.2.2 Step-wise Procedure for Bending Tests

- I. Width and thickness of the samples were measured at three different locations. Cross sectional area was calculated from average width and thickness.
- II. The sample was placed on the fixture support and was centrally aligned with 4” or 8” span.
- III. The Instron machine was calibrated with position. The loading rate of 0.05 in/min was used for all the tests. Specimens were tested till failure.
- IV. This procedure was repeated for each specimen. Experimental values of stiffness, bending strength, and bending strain for each specimen were calculated and tabulated. Specimen deflection was measured by actuator displacement. A typical load-deflection curve for composite coupon specimens is shown in Figs 4.10.

The formula for computing flexural modulus as per ASTM D790 and D6272 is based on the equation, which implicitly assumes that all the deformation is contributed by bending and any shear deflection is ignored. Figure 4.11 shows typical test specimens used for the three point bending test.

Bending modulus (from load-deflection curve)

$$E_x^B = \left(\frac{P}{\delta} \right) \frac{L^3}{4 \times w \times t^3} \quad \text{msi}$$

The flexural strength

$$\sigma_{ult} = \frac{3PL}{2wt^2} \quad \text{psi}$$

Where,

P - maximum load (lbs)

w - width of specimen (in)

t - thickness of the specimen (in)

Figure 4.10 Typical plot of load vs. deflection for test specimen

Figure 4.11 Typical bending test specimens

4.2.3 Compression Test

This test was used to determine elasticity and compression strength. All the tests were carried out as per ASTM D695-02a standards. The specimens subjected to compression test were of the size 3.3"x1". Thickness of test specimen was around 0.134". Schematic diagram of tension test specimen is shown in Figure 4.12. Test specimens were bonded with composite tabs of 1.1" on both the ends.

Figure 4.12 Schematic representation of compression test sample

The tabs were used to prevent gripping stresses and specimen failure due to crushing. Commercially available bonding agents like Plexus MA310TM, PliogripTM and epoxy were used for this purpose. A minimum of 24 hours curing time was allowed for the bonding agent before testing the samples. A compression jig was applied as per ASTM to test thin samples for compression test. Figure 4.13 shows a typical compression jig mounted on Instron for testing.

**Figure 4.13 Compression test jig employed for very thin specimen for
Compression Testing (ASTM D695)**

Strain gages were mounted axially along the length of the specimen. All the compression test specimens were tested with Instron 8501 with environmental chamber to maintain temperature and moisture conditions while also maintaining loading rate nearly constant. Data of load versus strain were recorded from compression test through data acquisition unit. This data was then reduced to stress versus strain curve. The linear portion of stress versus strain plot is referred to as compression modulus, a measure of stiffness of the material. All compression coupon specimens were tested in longitudinal and transverse directions by standardized procedure using ASTM D695-02a. Typical compression samples can be seen in Figure 4.14.

Figure 4.14 Compression test specimens

4.2.3.1 Step wise Test procedure for Compression test

- I. Width and thickness of the samples were measured at three different locations. Cross sectional area was calculated from average width and thickness.
- II. The end surfaces of the sample were grinded to facilitate a good bond between tabs and sample surface. Tabs of 1.1” length were bonded on end surfaces. End surface of the tab was made flat after grinding the unnecessary glue on it. Next step of the sample preparation includes mounting strain gages. For the specimen tested here, strain gages were axially mounted at mid-span along the length.
- III. In compression test, the specimen was placed inside the compression test jig. Strain gages were connected to data acquisition unit with connecting wires. Load cell of Instron machine was externally connected to the input port of data acquisition unit. During the test, loading rate was maintained constant at 0.05 in/min. All the samples were tested for failure.
- IV. Data was recorded for load versus strain variations during the test. Experimental values of compressive strength and modulus were calculated. This procedure was repeated for all the samples.

Typical plot of stress vs. strain for unidirectional pultruded sample in longitudinal direction is shown in Figure 4.15.

4.2.3.2 Compression Test Calculations

Compressive strength $\sigma_{ult} = \frac{P_{ult}}{wt}$ psi

Compressive modulus $E = \frac{\sigma}{\epsilon}$ msi

Where,

P - Applied load on the specimen (lbs)

w - Average width of the specimen (in)

t - Thickness of the specimen (in)

A = W x T = Cross-sectional area of the specimen (in²)

E – Calculated from slope of linear portion of Stress vs. Strain curve (psi Vs micro-strain)

Figure 4.15 Typical plot of stress vs. strain for E-glass/vinyl ester pultruded sample under compression test

4.2.4 Creep Test

Creep tests were conducted on, unidirectional E-glass/vinyl ester, quasi isotropic E-glass/vinyl ester and poly-urethane based tri-axial pultruded composites. These tests were used to predict the life of the composite and its durability. Creep tests were carried out at different temperatures and moisture conditions. Two types of creep tests were performed, three point bending creep tests and tension creep tests obtain change in deflection with time.

4.2.4.1 Three Point Bending creep Test

In case of 'three point bending creep', a constant load P is applied at mid-span. A schematic representation of the test set up is shown in Figure 4.16. The specimens used were the same as three point bending test specimens as shown in Figure 4.11. This test method was used for coupon level specimen. Tests were carried on manufactured frame.

Figure 4.16 Schematic of 3pt. bending creep test set up

The entire set of coupons was tested for three point bending creep in longitudinal directions. An over hang of 0.5" on either side was provided to prevent the specimen from slipping through the supports. Different % of maximum load to failure was used for the tests; the maximum load was calculated from the static three point bending tests.

4.2.4.1.1 Step-wise Procedure for Three Point Bending Creep Tests

- I. Width and thickness of the samples were measured at three different locations. Cross sectional area was calculated from average width and thickness.
- II. The sample was placed on the fixture support and was centrally aligned with 4" or 8" span.
- III. The sample was placed on the rollers. A dial gage was kept in the center of the sample and an instantaneous constant load was applied. Specimens were tested to failure.
- IV. The specimen deflection was measured using dial gauge after particular intervals of time. Measurements were made frequently during the start of the test and once a day thereafter.
- V. This procedure was repeated for each specimen. Experimental data of deflection vs. time was obtained and creep compliance vs. time was calculated and tabulated. Specimen deflection was measured by dial gauge. A typical deflection-time curve for composite coupon specimens under creep is shown in Figs 4.17.

Figure 4.17 Typical plot of deflection vs. time for E-glass/poly-urethane pultruded sample for three point bending creep

4.2.4.2 Tension Creep Test

This test was similarly used to determine deflection with time. The specimens subjected to tension creep test were the same as the specimens of tension tests as shown in Figure 4.7. Schematic diagram of tension creep test is shown in Figure 4.18. Test specimens were bonded with composite tabs of 4" on both the ends using Plexus MA 310.

Figure 4.18 Schematic of tension creep test set up

Data of strain versus time was recorded from tension creep test through data acquisition unit. All tension creep tests were tested in longitudinal direction. Different % of maximum load to failure was used for the tests; the maximum load to failure was calculated from static tension tests.

4.2.4.2.1 Step wise Test procedure for Tension Creep test

- I. Width and thickness of the samples were measured at three different locations. Cross sectional area was calculated from average width and thickness.
- II. The end surfaces of the samples were grinded to facilitate a good bond between tabs and sample surface. Tabs of 4" length were bonded on end surfaces. End surface of the tab was made flat after grinding the unnecessary glue on it.
- III. Two holes were drilled on both sides of the sample, and a strain gages was mounted. Strain gages were axially mounted at mid-span along the length.
- IV. In tension creep test, specimen was centrally aligned between the two metal grips and screws were inserted through the metal plates passing the sample holes and the coming out from the other plate in both ends of the sample. A load cell was placed between the sample and the supporting horizontal rod as can be seen in Figure 4.18.
- V. Strain gages and load cell were connected to data acquisition unit with connecting wires. Required amount of load was calculated and the nut on the dywidag bar was tightened till the required load was achieved.
- VI. Data was recorded for strain versus time during the test. This procedure was repeated for all the samples.

4.3 Scanning Electron Microscopy

E-glass/vinyl ester pultruded composites were observed under Electron Microscope. The behavior and performance of polymer composite have to be understood in conjunction with the specific properties of its constituents (the fibers and the matrix). The interface that exists between fibers and matrix is an essential element of the composite. Adequate adhesion between the fibers and matrix (a complete wet out) is a precondition for an optimized stress transfer in fiber reinforced polymer. Some of the observations as cited in literature show,

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

- SEM helps in identifying interfacial problems, wet-out problems, crack formulations and growth, and voids in a composite that directly affect short and long-term mechanical properties.

Electron microscopy, in a high resolution mode, is necessary to obtain useful information about the fine structure of the fiber-matrix interface. The interference fringes coming from the interference between the un-deviated beam and the beam diffracted by the glass layers of the fiber have been the basis for the study of fiber-matrix interfaces. For the microstructure study by SEM, small sample dimension of cubical shape of size 0.25" was selected.

Figure 4.19 Hitachi S-4700 field emission scanning electron microscope (FE-SEM)

A Cold Field Emission Gun Scanning Electron Microscope (FEGSEM) of "below-the-lens" design capable of (manufacturer's claims) 1.5 nm resolution at 15 kV, 12 mm W.D.; and 2.5 nm resolution at 1 k, 2.5 mm W.D. Magnification ranges from 30X to 500,000X. Specimen tilt is possible at 12 mm W.D up to 45 degrees. Electron source is a cold FE gun producing high brightness ($\sim 2 \times 10^9$ A / cm²/sr) with little energy spread (0.2 - 0.3 eV). The "below-the-lens" design and large sample chamber port permit samples as large as 100 mm diameter X 17 mm thick H. Oil-free vacuum systems pump column and sample exchange. Available image modes include secondary and backscattered electron images. There are two secondary electron detectors; one above the objective lens, the other below. Digital images may be acquired in BMP, TIFF, or

JPEG file formats at 640 X 480, 1280 X 960, or 1560 X 1920 pixels. Figure 4.19 shows the Electron microscope used for the study.

4.3.1 Sample preparation for SEM Images

- I. The dimensions of the sample for SEM study as decided by the Microscopy Unit were cubical shaped of 0.25", the size was limited by the instrument.
- II. The sample was molded in cylindrical rubber mold with epoxy. Ratio of epoxy to converter was 5:1. Approximately 12-15gms of epoxy was used for each sample. Composite sample was placed in the cylindrical mold and epoxy was poured on it. Care should be taken to remove the air bubble below the sample surface, in order to avoid any sample damage during grinding step. Mold was allowed to cure for 24hrs.
- III. The cylinders with the specimens were fixed on rotating circular disc for grinding purpose. These discs are connected to rotating shaft with motor. Out of the two rotating discs, the top one has arrangement to hold cylindrical epoxy sample. The bottom disc has surface which allows mounting of different sand paper on it. Different types of sand paper were used in steps in order to achieve fine surface on sample. Sand papers with grid number of 180, 240, 320, 400 and 600 were used. Higher the grid number, smoother is the surface of sand paper. Each step of grinding was carried out for about 30 minutes.
- IV. Final surface finishing includes grinding the sample surface on white cloth by replacing sand papers. Silica solution was continuously sprayed on the white cloth mounted on bottom disc. Silica solution with different particle size 1 μ m, 0.3 μ m and 0.05 μ m, were used for this purpose.
- V. Sample was then cleaned with water and epoxy cover was removed by crushing in jaws. It must be ensured that during crushing step, sample is kept away from any damage.
- VI. The specimen was then kept in a vacuum chamber and its top surface was sputtered with gold particles. Then, the sample was taken out of the vacuum chamber and a conducting copper tape was mounted on it to avoid the formation of electron cloud while examining it under electron microscope.
- VII. This specimen was examined under SEM machine for viewing.

4.4 Physical and Morphological Properties

4.4.1 Fiber Content using burn out test

Strength of composite typically is in direct proportion with fiber content. Hence, quantitative measure of the fiber fraction in a given composite is essential. Fiber Volume Fraction (FVF) test was performed to determine the fiber volume (or weight) content of composite materials. The tests were performed as per Procedure G of ASTM D 3171. Specimens were cut to a dimension of 1" x 1" and measured to the nearest of 0.001 inches.

4.4.1.1 Test procedure and set up

Each specimen was placed in a pre-weighed crucible. The crucible (along with specimen) was kept in a preheated furnace (Isothermal Muffle Furnace, Model 550-58) at 600° C (Figure 4.20) for 3 hours. The specimens were taken out from the furnace and the crucible along with the fibers was further weighed.

Figure 4.20 Isothermal muffle furnace

4.4.1.2 Fiber volume fraction calculation

Step 1: Measure dimension of the composite sample – Length (L in cm), Width (W in cm) and Thickness (T in cm).

Step 2: Measure the weight of empty crucible (W_C in gms)

Step 3: Measure the weight of crucible + sample (W_{c+s} in gms) before burn-out

Step 4: Measure weight of fabric (left after resin burn) + crucible (W_{f+c} in gms) after burn-out

Step 5: Evaluate FVF = $\frac{W_{f+c} - W_c}{\rho LWT} \times 100$

Where, ρ is density of fiber (E-glass) = 2.522gm/cc.

4.4.2 Void Content

In order to achieve superior properties of FRP composites, it is required to have good physical bond between fibers and matrix. Also, during resin curing air entrapment should be minimum resulting into small voids. Smaller void size helps in better composite properties. Microstructure of composite such as fiber-matrix bond and void content were studied by Scanning Electron Microscope (SEM) images. A small piece of sample (0.25") was cut from composite plate in order to process SEM images. This sample after going through few sample preparation stages was tested for SEM images. Fig 4.21 shows some of the SEM images of E-glass/vinyl ester sample.

Figure 4.21 SEM images of pultruded composites

CHAPTER 5

RESULTS AND DISCUSSIONS

5.1 Introduction

In this chapter, results and analysis of experimental data are presented in the form of tables and graphs. As described earlier, specimens were tested for tension, compression, bending and creep properties as per ASTM D3039, D695, D790 and ASTM D2990 standards. Static test results and creep results of the laminates are shown in the subsequent sections. Moreover, their morphological properties such as voids, fiber-matrix adhesion viewed by scanning electron microscopy are also described in the later sections of this chapter.

The data collected through static testing as shown in this chapter were then used for life prediction of these composites; and also change in modulus with time was determined using the creep data which is very useful for design purposes. The models used to carry out these studies are described in chapter 6.

5.2 Unidirectional E-glass/VE Composites

5.2.1 Static Tests

The laminates were tested for their static mechanical properties. Coupon specimens with desired dimensions were prepared for testing under bending, compression and tension. Load-deflection and stress-strain behavior were plotted as the material's response. General trends for stress-strain and load-deflection behavior are presented in Chapter 4, in addition to providing deterministic relations through mathematical equations.

Compression test was performed both in longitudinal and transverse directions of the laminates. Tension samples were cut only in transverse direction while bending samples were cut in longitudinal direction. Longitudinal direction here refers to the direction of 0^0 fibers and transverse direction refers to the direction of 90^0 fibers (there are no 90^0 fibers). Mechanical responses under tension, bending and compression are shown in the later section. All the above mentioned tests were carried out at five temperatures, under three varying relative humidity conditions. Three replications were done for each test.

The conditioning for all samples was done for 24 hours and all samples were tested to failure. For dry conditioning and varying temperature, the samples were kept in the environmental chamber and the required temperature was maintained before and during testing. For 50% R.H. and varying temperature, a humidifier was attached to the environmental chamber and the temperature was maintained using environmental chamber before and during the test. For wet conditions, the samples were kept in a tank of water attached with a coil heater, which was connected to a thermocouple to maintain the required temperature; a stirrer was also used to prevent any localized heating.

5.2.1.1 Bending Tests

Three point bending test was performed with Unidirectional Pultruded samples using ASTM D790. All bending tests were performed in the longitudinal (0° fiber) direction with samples of the dimensions of 1/2 inch wide, 5 inch long at a span of 4 inches. These tests were done at five temperatures (20°C , 50°C , 80°C , 110°C and 140°C) and three humidity conditions (Dry, 50% R.H. and Wet), leading to a total of 15 combinations. Data of these tests were also used to determine the loading levels for creep tests as described in the later sections of this chapter.

5.2.1.1.1 Dry Conditions

Samples were tested under dry environmental conditions with varying temperature from 20°C to 140°C with an interval of 30°C . Tables 5.1 to 5.5 show the test result for each batch of samples and flexural strength and flexural modulus.

Figure 5.1 shows variation of flexural stress with temperature. There is a considerable decrease in strength with increase in temperature. As seen from the graph with an increase in temperature from 20°C to 140°C , bending stress at failure decreased from 140 ksi to 9.65 ksi, which is 93% reduction in strength. Figure 5.1 also reveals that the decrease in strength from 140 ksi at 20°C to 63.71 ksi at 80°C shows 55% reduction, but with further increase in temperature i.e. 80°C to 140°C , the reduction in strength increases rapidly, as above 100°C the polymer matrix approaches its T_g , hence further reducing the strength from 63.71 ksi to 9.65 ksi which is a 84% reduction.

Table 5.1 Bending test result for unidirectional samples @ 20⁰C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Max. Load (lbs)	Bending Stress (ksi)
B1.1	0.500	0.134	209.4	140.00
B1.2	0.500	0.134	210.3	141.00
B1.3	0.500	0.134	207.9	139.00
AVG				140.00
% STD				0.70

Table 5.2 Bending test result for unidirectional samples @ 50⁰C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B2.1	0.507	0.131	404.8	122.6	84.97	5.72
B2.2	0.509	0.131	483.8	137.9	94.77	6.76
B2.3	0.510	0.134	463.3	127.1	83.27	6.04
AVG					87.67	6.17
% STD					6.20	0.53

Table 5.3 Bending test result for unidirectional samples @ 80⁰C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B3.1	0.510	0.133	483.3	89.2	59.29	6.89
B3.2	0.511	0.133	489.3	90.9	60.41	6.50
B3.3	0.510	0.135	503.0	107.4	71.43	6.38
AVG					63.71	6.59
% STD					6.70	0.27

Table 5.4 Bending test result for unidirectional samples @ 110⁰C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B4.1	0.486	0.139	401.26	68.9	44.04	5.53
B4.2	0.487	0.139	394.73	53.1	33.89	4.82
B4.3	0.484	0.139	376.56	56.1	36.01	4.63
AVG					37.98	4.99
% STD					5.35	0.47

Table 5.5 Bending test result for unidirectional samples @ 140⁰C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B5.1	0.484	0.135	186.53	14.3	9.77	2.50
B5.2	0.483	0.136	174.79	12.5	8.26	2.24
B5.3	0.486	0.131	203.1	15.2	10.93	2.97
AVG					9.65	2.57
% STD					1.33	0.37

Figure 5.1 Bending stress vs. temperature for unidirectional samples at dry conditions

5.2.1.1.2 50% Relative Humidity

In this set of experiments, the samples were tested at 50% Relative Humidity with varying temperatures.

Tables 5.6 to 5.10 show the test results for each batch of sample and their flexural strength and flexural modulus. Figure 5.2 shows variations of bending stress with temperature and conditioned at 50% R.H. The curve in Figure 5.2 shows a similar trend as seen in Figure 5.1 for dry conditions tests. A reduction of 92.9% in strength was observed when temperature was varied from 20⁰C to 140⁰C, i.e. the stress to failure reduced from 118.01 ksi to 8.35 ksi. As was observed in the earlier section, the decrease in strength from 20⁰C to 80⁰C is 46.34% which is much less when compared to the reduction in strength from 80⁰C to 140⁰C which was found to be 86.8% reduction.

Table 5.6 Bending test result for unidirectional samples @ 20°C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B6.1	0.508	0.133	459.5	181.45	121.08	6.14
B6.2	0.501	0.135	495.16	174.33	113.37	6.21
B6.3	0.507	0.137	479.04	189.77	119.58	5.87
AVG					118.01	6.07
% STD					4.08	0.18

Table 5.7 Bending test result for unidirectional samples @ 50°C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B7.1	0.485	0.131	430.9	110.5	79.51	6.31
B7.2	0.482	0.137	474.6	138.6	90.40	5.98
B7.3	0.484	0.137	476.21	131.6	86.97	6.12
AVG					85.62	6.13
% STD					5.56	0.17

Table 5.8 Bending test result for unidirectional samples @ 80°C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B8.1	0.466	0.136	436.78	104.2	72.53	5.96
B8.2	0.485	0.137	495.71	96.1	62.33	6.44
B8.3	0.485	0.134	451.16	80.1	55.10	6.41
AVG					63.32	6.27
% STD					8.75	0.27

Table 5.9 Bending test result for unidirectional samples @ 110⁰C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B9.1	0.510	0.133	393.63	38.9	25.85	5.39
B9.2	0.510	0.133	381.09	44.0	29.30	4.66
B9.3	0.509	0.133	417.13	50.7	33.73	5.43
AVG					29.62	5.16
% STD					3.95	0.43

Table 5.10 Bending test result for unidirectional samples @ 140⁰C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B10.1	0.510	0.133	170.5	14.3	9.56	2.16
B10.2	0.510	0.133	148.79	10.7	7.14	2.06
B10.3	0.510	0.133	158.72	12.3	8.23	2.20
AVG					8.31	2.14
% STD					1.21	0.072

Figure 5.2 Bending stress vs. temperature for unidirectional samples at 50% relative humidity.

5.2.1.1.3 Wet Conditions

This set of samples was tested under wet conditions with varying temperatures from 20⁰C to 140⁰C. Tables 5.11 to 5.15 show the test results and their strength to failure along with the bending modulus. Figure 5.3 shows variations of bending stress with temperature under wet conditions.

A reduction of 95.36% in strength was observed when temperature was varied from 20⁰C to 140⁰C, i.e. the stress to failure reduced from 110.68 ksi to 5.14 ksi. Also, decrease in strength from 20⁰C to 80⁰C is 61.23% which is much less when compared to the reduction in strength from 80⁰C to 140⁰C which was found to be 91.6%.

Table 5.11 Bending test result for unidirectional samples @ 20⁰C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B11.1	0.512	0.143	477.01	193.61	110.95	5.72
B11.2	0.476	0.130	505.82	127.36	94.99	6.17
B11.3	0.482	0.131	414.59	173.87	126.12	6.12
AVG					110.68	6.0
% STD					15.5	0.24

Table 5.12 Bending test result for unidirectional samples @ 50⁰C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B12.1	0.483	0.131	441.38	86.69	62.75	6.50
B12.2	0.486	0.137	469.5	105.80	69.59	6.01
B12.3	0.484	0.136	429.71	112.64	75.34	5.63
AVG					69.22	6.04
% STD					6.30	0.43

Table 5.13 Bending test result for unidirectional samples @ 80⁰C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B13.1	0.485	0.138	410.12	81.52	52.95	5.14
B13.2	0.487	0.137	434.58	54.33	35.15	5.43
B13.3	0.488	0.139	442.13	63.80	40.60	5.39
AVG					42.9	5.32
% STD					9.12	0.15

Table 5.14 Bending test result for unidirectional samples @110⁰C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B14.1	0.486	0.128	172.12	18.57	14.00	2.70
B14.2	0.487	0.134	167.7	14.97	10.27	2.28
B14.3	0.470	0.138	153.25	14.03	9.40	1.98
AVG					11.22	2.32
% STD					2.44	0.36

Table 5.15 Bending test result for unidirectional samples @140⁰C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Slope P vs. defl. (lb/in.)	Max. Load (lbs)	Bending Stress (ksi)	E_b (msi)
B15.1	0.509	0.138	132.16	7.82	4.84	1.58
B15.2	0.475	0.134	108.13	8.41	5.91	1.51
B15.3	0.485	0.136	100.75	7.01	4.69	1.32
AVG					5.14	1.47
% STD					0.66	0.13

Figure 5.3 Bending stress vs. temperature for unidirectional samples at wet conditions.

5.2.1.1.4 Conclusions

Figure 5.4 shows consolidated test results for bending stress versus temperature at all three humidity variations. The trend of the curve is as expected i.e. decreasing in strength with increasing temperature and moisture. As seen in the above sections, there was about 90%-95% reduction in strength as temperature increased from 20⁰C to 140⁰C for all humidity variations. Also, as R.H. changed from dry to 50% R.H. conditions, 2%-22% reduction in strength was observed with temperature varying from 20⁰C to 140⁰C. Similarly from 50% R.H. conditions to wet conditions, we see a reduction in strength from 19%-62% on the same temperature range. From dry conditions to wet conditions, the strength reduction of about 20%-70% in the temperature ranging from 20⁰C to 140⁰C was observed. The large change in the strength due to moisture variation is because of large number of voids present, as shown in the later section on morphological properties. The maximum average strength of the bending data was used to determine the loading levels for the three point bending creep tests as discussed in the sections later. Also, the

bending strength and time to failure data were used to predict a creep rupture master curve using Miyano's Accelerated Testing Method (ATM) approach which was validated with bending creep data.

Figure 5.4 Bending Stress-temperature-humidity behavior of Unidirectional Pultruded Composites

5.2.1.2 Tension Test (Transverse)

Tension tests were done in the transverse direction for the unidirectional pultruded samples using ASTM D3039. Since these samples were cut in the direction perpendicular to the direction of the fibers, fibers did not contribute to the strength of the samples. The strength of the samples was primarily due to the polymer matrix. The samples for transverse tension were cut 11 inch long, $\frac{3}{4}$ inch wide with 2 inch tabs on both ends of the samples.

The conditions were kept similar to those for three point bending tests i.e. five variations in temperature (20⁰C, 50⁰C, 80⁰C, 110⁰C and 140⁰C) and three variations in humidity (Dry, 50% R.H. and Wet). Tensile data with varying environmental condition, (i.e. temperature and humidity) were generated.

5.2.1.2.1 Dry Conditions

The effect on tensile strength under dry conditions at different temperatures was measured by testing the samples to failure.

Tables 5.16 to 5.20 show the tensile test results for each batch of samples and their tensile strength. The considerable decrease in strength as compared to bending strength is due to the fact that there were no fibers to bear the load in transverse direction. Figure 5.5 shows decreasing tensile strength with increasing temperature. It is observed from the tables and graphs that as the temperature increases from 20⁰C to 140⁰C, the tensile stress to failure decreases from 1.35 ksi to 0.413 which is 69% reduction.

The reduction is more pronounced at above 100⁰C, the reduction in strength from 20⁰C to 80⁰C is 1.35 ksi to 1.11 ksi which is a reduction of 18%. This is relatively smaller when compared to reduction from 80⁰C to 140⁰C which is 1.11 ksi to 0.413 ksi, a reduction of 63%. Hence as the temperature approaches T_g of the polymer matrix, the strength is reduced considerably.

Table 5.16 Transverse tension test result for unidirectional samples @ 20⁰C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TT1.1	1.000	0.135	0.135	176.80	1.31
TT1.2	0.960	0.137	0.132	184.98	1.41
TT1.3	1.020	0.134	0.137	185.79	1.35
AVG					1.35
% STD					0.05

Table 5.17 Transverse tension test result for unidirectional samples @ 50⁰C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TT2.1	0.750	0.138	0.103	130.44	1.26
TT2.2	0.750	0.136	0.102	151.63	1.48
TT2.3	0.750	0.137	0.103	113.20	1.10
AVG					1.28
% STD					0.19

Table 5.18 Transverse tension test result for unidirectional samples @ 80⁰C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TT3.1	1.002	0.136	0.136	154.37	1.13
TT3.2	1.003	0.135	0.135	163.29	1.20
TT3.3	1.006	0.134	0.135	137.22	1.01
AVG					1.11
% STD					0.09

Table 5.19 Transverse tension test result for unidirectional samples @ 110⁰C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TT4.1	1.007	0.136	0.137	107.72	0.784
TT4.2	1.003	0.136	0.136	117.32	0.858
TT4.3	1.006	0.135	0.136	89.19	0.655
AVG					0.765
% STD					0.10

Table 5.20 Transverse tension test result for unidirectional samples @ 140⁰C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TT5.1	1.003	0.137	0.137	58.32	0.424
TT5.2	1.006	0.134	0.134	63.12	0.468
TT5.3	1.001	0.137	0.137	48.02	0.349
AVG					0.413
% STD					0.06

Figure 5.5 Transverse tension stress vs. temperature for unidirectional samples at dry conditions

5.2.1.2.2 50% Relative Humidity

In this set of experiments, the effect of 50% R.H. on tensile strength as function of temperature was examined.

Tables 5.21 to 5.25 show the test results for each batch of samples and their tensile strength. Figure 5.6 shows variation of tensile stress with temperature, conditioned at 50% R.H. The curve in Figure 5.6 shows a similar trend as was seen in above figures. A reduction of 72.9% in tensile strength was observed as the temperature varied from 20⁰C to 140⁰C, the tensile stress to failure reduced from 1.22 ksi to 0.33 ksi. As was observed in earlier sections, similar decrease in tensile strength from 20⁰C to 80⁰C was observed at 19% which is relatively smaller when compared to reduction in strength from 80⁰C to 140⁰C which was 67%.

Table 5.21 Transverse tension test result for unidirectional samples @ 20⁰C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TT6.1	1.002	0.136	0.136	196.23	1.43
TT6.2	1.004	0.137	0.137	172.21	1.25
TT6.3	1.003	0.136	0.136	135.16	0.99
AVG					1.22
% STD					0.22

Table 5.22 Transverse tension test result for unidirectional samples @ 50⁰C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TT7.1	0.746	0.137	0.102	115.19	1.12
TT7.2	0.753	0.138	0.103	150.12	1.44
TT7.3	0.749	0.136	0.102	99.48	0.97
AVG					1.17
% STD					0.24

Table 5.23 Transverse tension test result for unidirectional samples @ 80⁰C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TT8.1	0.749	0.139	0.104	100.28	0.965
TT8.2	0.741	0.138	0.102	109.55	1.073
TT8.3	0.749	0.134	0.101	93.31	0.926
AVG					0.988
% STD					0.07

Table 5.24 Transverse tension test result for unidirectional samples @ 110⁰C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TT9.1	0.749	0.136	0.102	61.06	0.600
TT9.2	0.745	0.136	0.102	76.15	0.749
TT5.3	0.749	0.137	0.102	68.61	0.670
AVG					0.673
% STD					0.07

Table 5.25 Transverse tension test result for unidirectional samples @ 140⁰C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TT10.1	0.746	0.135	0.101	43.91	0.434
TT10.2	0.749	0.135	0.101	33.61	0.332
TT10.3	0.751	0.137	0.103	24.01	0.232
AVG					0.33
% STD					0.1

Figure 5.6 Transverse tension stress vs. temperature for unidirectional samples at 50% relative humidity.

5.2.1.2.3 Wet Conditions

This set of experiments was used to study the effects of wet conditioning on tensile strength at different temperatures.

Tables 5.26 to 5.30 show the test results for each batch of samples and their tensile strength. Figure 5.7 shows variation of tensile stress to failure with temperature under wet conditions. A reduction from 1.10 ksi to 0.22 ksi in tensile stress to failure was observed, which is 80% reduction, as the temperature varied from 20⁰C to 140⁰C. Also, decrease in tensile strength from 20⁰C to 80⁰C was observed to be 29% which is relatively smaller when compared to reduction in strength from 80⁰C to 140⁰C which was found to be 71.8%.

Table 5.26 Transverse tension test result for unidirectional samples @ 20⁰C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in ²)	Max. Load (lbs)	Max. Stress (ksi)
TT11.1	0.748	0.135	0.101	111.15	1.103
TT11.2	0.753	0.135	0.102	102.23	1.005
TT11.3	0.753	0.136	0.102	122.12	1.195
AVG					1.10
% STD					0.09

Table 5.27 Transverse tension test result for unidirectional samples @ 50⁰C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in ²)	Max. Load (lbs)	Max. Stress (ksi)
TT12.1	0.743	0.135	0.097	87.82	0.902
TT12.2	0.746	0.133	0.099	102.05	1.028
TT12.3	0.745	0.134	0.100	102.91	1.030
AVG					0.987
% STD					0.07

Table 5.28 Transverse tension test result for unidirectional samples @ 80⁰C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in ²)	Max. Load (lbs)	Max. Stress (ksi)
TT13.1	0.759	0.135	0.102	74.10	0.725
TT13.2	0.755	0.136	0.103	69.29	0.675
TT13.3	0.747	0.136	0.101	96.36	0.946
AVG					0.782
% STD					0.14

Table 5.29 Transverse tension test result for unidirectional samples @ 110⁰C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TT14.1	0.756	0.137	0.103	52.62	0.509
TT14.2	0.759	0.137	0.104	61.75	0.592
TT14.3	0.760	0.138	0.104	56.94	0.544
AVG					0.548
% STD					0.04

Table 5.30 Transverse tension test result for unidirectional samples @ 140⁰C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TT15.1	0.757	0.135	0.102	19.21	0.187
TT15.2	0.759	0.136	0.103	21.26	0.205
TT15.3	0.756	0.135	0.102	27.44	0.269
AVG					0.22
% STD					0.04

Figure 5.7 Transverse tension stress vs. temperature for unidirectional samples at wet conditions.

5.2.1.2.4 Conclusions

Figure 5.8 shows combined results for tensile failure stress versus temperature at three humidity conditions. The trend of the curves is similar to that of Figure 5.4 for the bending stress to failure, i.e. decreasing strength with increasing temperature and moisture. The substantial difference between the stress to failure for bending and tension is due to the fact that tension tests were done in the direction perpendicular to the direction of fiber.

As seen in the sections above, there was 69%-80% reduction in strength as temperature increased from 20⁰C to 140⁰C for all humidity variations. Also, as R.H. changed from dry to 50% R.H., 10%-20% reduction in tensile strength was noticed for temperature increase from 20⁰C to 140⁰C. Similarly, for 50% R.H. to wet conditions, a reduction in tensile strength from 10%-35% was observed for the same temperature increase. From dry to wet conditions, strength reduction of about 20%-45% was seen for temperature increase from 20⁰C to 140⁰C.

The average maximum strength and time to failure from the tension test was used to predict tension creep curve using Miyano’s ATM approach.

Figure 5.8 Transverse tension stress-temperature-humidity behavior of unidirectional composites

5.2.1.3 Compression Test (Longitudinal)

Compression tests were done in both transverse and longitudinal directions of the unidirectional composite laminates. In this section, longitudinal compression test data is discussed while transverse compression data is discussed in section 5.2.1.4. All compression tests were done in a similar manner to three point bending tests and transverse tension tests i.e. at five different temperatures (20⁰C, 50⁰C, 80⁰C, 110⁰C and 140⁰C) and varying humidity conditions (Dry, 50% R.H. and Wet).

The compression tests were used to determine the maximum compressive stress to failure at different environmental conditions mentioned above and were tested using ASTM D695. Since the samples were thin laminates (0.133”) hence a support jig was required to test those samples. Therefore, a compression jig as per ASTM specifications was made to perform both longitudinal and transverse compression tests. The details of the jig and the test procedure are explained in chapter 4. The dimensions of the samples in the longitudinal direction were 3.13” long, ½” wide and end tabs of 1.1” long were used to prevent failure due to crushing.

5.2.1.3.1 Dry Conditions

Samples were tested under dry conditions at temperatures varying from 20⁰C to 140⁰C. Tables 5.31 to 5.35 show the test results for each batch of samples and their compressive strength. Figure 5.9 shows variation of compressive stress with temperature. As seen from the Figure 5.9, decreasing strength is observed with increasing temperature. As the temperature increases from 20⁰C to 140⁰C, there is a decrease in stress to failure from 55.23 ksi to 6.72 ksi which is a reduction of 89%. The decrease in strength from 80⁰C to 140⁰C is 75% which is much more pronounced when compared to a reduction from 20⁰C to 80⁰C which is 51%.

Table 5.31 Longitudinal compression test result for unidirectional samples @ 20°C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC1.1	0.435	0.132	0.057	3169.90	55.21
LC1.2	0.438	0.133	0.058	3454.31	59.30
LC1.3	0.438	0.134	0.059	3003.85	51.18
AVG					55.23
% STD					4.06

Table 5.32 Longitudinal compression test result for unidirectional samples @ 50°C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC2.1	0.508	0.137	0.069	2561.97	36.83
LC2.2	0.497	0.137	0.068	2414.45	35.48
LC2.3	0.506	0.132	0.066	2429.54	36.28
AVG					36.19
% STD					0.67

Table 5.33 Longitudinal compression test result for unidirectional samples @ 80°C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC3.1	0.508	0.138	0.070	1831.25	26.12
LC3.2	0.497	0.136	0.067	1954.06	28.85
LC3.3	0.507	0.137	0.069	1816.84	26.15
AVG					27.04
% STD					1.56

Table 5.34 Longitudinal compression test result for unidirectional samples @ 110⁰C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC4.1	0.500	0.134	0.067	1134.15	16.87
LC4.2	0.511	0.134	0.068	1167.09	17.05
LC4.3	0.497	0.138	0.069	1149.25	16.71
AVG					16.87
% STD					0.17

Table 5.35 Longitudinal compression test result for unidirectional samples @ 140⁰C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC5.1	0.507	0.136	0.069	531.05	7.70
LC5.2	0.503	0.135	0.068	472.73	6.96
LC5.3	0.503	0.132	0.066	367.07	5.52
AVG					6.72
% STD					1.10

Figure 5.9 Longitudinal compression stress vs. temperature for unidirectional samples at dry conditions

5.2.1.3.2 50% Relative Humidity

These set of experiments were done at 50% R.H. with the variation of temperature. Tables 5.36 to 5.40 show the test results for the each batch of samples and their compressive strength. Figure 5.10 shows variation of longitudinal compressive stress with temperature, conditioned at 50% R.H. A reduction of 92% in longitudinal compressive strength was observed with increase in temperature from 20⁰C to 140⁰C at 50% R.H., i.e. the compressive stress reduced from 38.94 ksi to 2.75 ksi. As was observed in earlier sections also, the decrease in strength from 20⁰C to 80⁰C was 37.26% which is relatively smaller when compared to reduction in strength from 80⁰C to 140⁰C which was 88.6%.

Table 5.36 Longitudinal compression test result for unidirectional samples @ 20°C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC6.1	0.509	0.143	0.072	2985.30	41.11
LC6.2	0.503	0.138	0.069	2845.33	41.11
LC6.3	0.505	0.138	0.069	2404.84	34.61
AVG					38.94
% STD					3.75

Table 5.37 Longitudinal compression test result for unidirectional samples @ 50°C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC7.1	0.508	0.137	0.069	2585.98	37.06
LC7.2	0.504	0.134	0.068	2336.92	34.53
LC7.3	0.508	0.138	0.070	2062.47	29.51
AVG					33.7
% STD					3.84

Table 5.38 Longitudinal compression test result for unidirectional samples @ 80°C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC8.1	0.498	0.138	0.069	1878.59	27.28
LC8.2	0.508	0.137	0.069	1524.55	21.92
LC8.3	0.495	0.138	0.068	1641.88	24.11
AVG					24.43
% STD					2.69

Table 5.39 Longitudinal compression test result for unidirectional samples @ 110⁰C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC9.1	0.500	0.137	0.068	1095.04	16.01
LC9.2	0.502	0.135	0.067	1115.62	16.47
LC9.3	0.501	0.135	0.068	1088.87	16.04
AVG					16.17
% STD					0.25

Table 5.40 Longitudinal compression test result for unidirectional samples @ 140⁰C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC10.1	0.505	0.134	0.068	197.60	2.91
LC10.2	0.496	0.133	0.066	179.76	2.72
LC10.3	0.499	0.137	0.068	186.62	2.72
AVG					2.78
% STD					0.11

Figure 5.10 Longitudinal compression stress vs. temperature for the unidirectional samples at 50% relative humidity.

5.2.1.3.3 Wet Conditions

This set of samples was tested at temperatures varying from 20⁰C to 140⁰C at wet conditions. Tables 5.41 to 5.45 show the test results for each batch of samples and their longitudinal compressive strength. Figure 5.11 shows variation of longitudinal compressive stress with temperature under wet conditions. Compressive stress to failure reduced from 28.72 ksi to 1.81 ksi when temperature changed from 20⁰C to 140⁰C which reduced the strength by 93.7%. Also a reduction of 45.3% was observed when temperature were changed from 20⁰C to 80⁰C which is relatively smaller than the reduction observed with temperature varying from 80⁰C to 140⁰C which was found to be 88%.

Table 5.41 Longitudinal compression test result for unidirectional samples @ 20°C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC11.1	0.506	0.134	0.068	2096.09	30.97
LC11.2	0.499	0.133	0.066	1994.54	30.07
LC11.3	0.496	0.136	0.067	1689.91	25.13
AVG					28.72
% STD					3.14

Table 5.42 Longitudinal compression test result for unidirectional samples @ 50°C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC12.1	0.502	0.131	0.066	1532.79	23.23
LC12.2	0.495	0.138	0.068	1589.73	23.31
LC12.3	0.504	0.133	0.067	1578.04	23.61
AVG					23.38
% STD					0.2

Table 5.43 Longitudinal compression test result for unidirectional samples @ 80°C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC13.1	0.497	0.138	0.069	1149.25	16.71
LC13.2	0.485	0.131	0.063	1018.00	16.03
LC13.3	0.500	0.132	0.066	951.64	14.37
AVG					15.70
% STD					1.23

Table 5.44 Longitudinal compression test result for unidirectional samples @ 110°C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC14.1	0.501	0.134	0.067	270.33	4.03
LC14.2	0.502	0.137	0.069	277.19	4.02
LC14.3	0.506	0.137	0.069	307.38	4.44
AVG					4.16
% STD					0.24

Table 5.45 Longitudinal compression test result for unidirectional samples @ 140°C and wet conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
LC15.1	0.497	0.135	0.067	139.97	2.09
LC15.2	0.501	0.135	0.067	101.54	1.51
LC15.3	0.508	0.137	0.069	126.24	1.82
AVG					1.81
% STD					0.29

Figure 5.11 Longitudinal compression stress vs. temperature for unidirectional samples at wet conditions.

5.2.1.3.4 Conclusions

Figure 5.12 shows consolidated test results for longitudinal compressive stress versus temperatures at three different humidity variations. As seen in the earlier sections, the bending and transverse tension (figures 3.4 and 3.8) also showed a similar trend. As the temperature increased from 20⁰C to 140⁰C, 89%-93% reduction in strength at all the three humidity variations was observed. As humidity changed from dry to 50% R.H. conditions, a reduction of 10%-50% in longitudinal compressive stress was seen. Similarly from 50% R.H. to wet conditions, a reduction of 20%-35% in compressive stress at all temperatures was observed. Further, from dry to wet conditions, strength reduction of 50%-75% was observed at all temperatures from 20⁰C to 140⁰C.

The maximum average strength of the longitudinal compression data along with the time to failure are used to predict a creep rupture curve using Miyano's Accelerated Testing Method (ATM) approach.

Figure 5.12 Longitudinal compression stress-temperature-humidity behavior of unidirectional composites

5.2.1.4 Compression Test (Transverse)

Transverse compression tests were performed in exactly the same way as longitudinal compression tests except that these samples were cut in the direction perpendicular to the fibers. This set of tests was also done at five different temperatures (20⁰C, 50⁰C, 80⁰C, 110⁰C and 140⁰C) and three different humidity conditions (Dry, 50% R.H. and Wet).

The tests were used to determine the maximum transverse strength to failure with varying environmental conditions. The dimensions of the samples cut in the transverse direction were 3.13” long, ½” wide and end tabs of 1.1” long were used to prevent failure due to crushing.

5.2.1.4.1 Dry Conditions

Samples were tested under dry conditions at different temperatures varying from 20⁰C to 140⁰C. Tables 5.46 to 5.50 show the test results for each batch of samples and their transverse compression strength.

Figure 5.13 shows variation of transverse compressive stress to failure with temperature. As seen from the figure, decreasing strength is observed with increasing temperature. As the temperature increases from 20⁰C to 140⁰C, there is a decrease in stress to failure from 9.83 ksi to 0.67 ksi showing a reduction of 93.2%. The decrease in strength from 80⁰C to 140⁰C was observed as 86.7% which is much more pronounced when compared to a reduction from 20⁰C to 80⁰C which was 48.6%.

Table 5.46 Transverse compression test result for unidirectional samples @ 20°C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC1.1	0.435	0.134	0.058	600.32	10.30
TC1.2	0.434	0.136	0.059	551.34	9.34
TC1.3	0.431	0.138	0.059	586.13	9.85
AVG					9.83
% STD					0.48

Table 5.47 Transverse compression test result for unidirectional samples @ 50°C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC2.1	0.518	0.139	0.072	375.99	5.23
TC2.2	0.515	0.137	0.070	472.04	6.70
TC2.3	0.517	0.139	0.072	503.61	6.98
AVG					6.30
% STD					0.94

Table 5.48 Transverse compression test result for unidirectional samples @ 80°C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC3.1	0.513	0.136	0.070	356.78	5.13
TC3.2	0.515	0.136	0.070	354.72	5.07
TC3.3	0.516	0.138	0.071	352.66	4.96
AVG					5.05
% STD					0.86

Table 5.49 Transverse compression test result for unidirectional samples @ 110°C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC4.1	0.518	0.139	0.072	170.15	2.36
TC4.2	0.517	0.140	0.072	159.17	2.20
TC4.3	0.509	0.137	0.070	179.76	2.57
AVG					2.37
% STD					0.17

Table 5.50 Transverse compression test result for unidirectional samples @ 140°C and dry conditions

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC5.1	0.519	0.138	0.072	56.26	0.78
TC5.2	0.513	0.137	0.070	41.85	0.59
TC5.3	0.521	0.141	0.073	48.02	0.65
AVG					0.67
% STD					0.09

Figure 5.13 Transverse compression stress vs. temperature for unidirectional samples at dry conditions

5.2.1.4.2 50% Relative Humidity

This set of experiments was done at 50% R.H. with the variation in temperature. Tables 5.51 to 5.55 show the test results for each batch of samples and their transverse compressive strength. Figure 5.14 shows variation of transverse compressive stress with temperature at 50% R.H. A reduction of 94.3% in strength was observed when temperature increased from 20°C to 140°C at 50% R.H., i.e. the compressive stress to failure reduced from 8.68 ksi to 0.49 ksi. As was observed in earlier sections the reduction in strength from 20°C to 80°C temperature is 48.27% which is relatively less as compared to reduction in strength from 80°C to 140°C temperature, which was 89.1%.

Table 5.51 Transverse compression test result for unidirectional samples @ 20°C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC6.1	0.512	0.142	0.073	660.73	9.08
TC6.2	0.507	0.140	0.071	579.77	8.19
TC6.3	0.509	0.141	0.072	630.54	8.78
AVG					8.68
% STD					0.45

Table 5.52 Transverse compression test result for unidirectional samples @ 50°C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC7.1	0.517	0.137	0.071	386.97	5.45
TC7.2	0.510	0.133	0.068	365.99	5.39
TC7.3	0.514	0.138	0.071	379.42	5.34
AVG					5.39
% STD					0.05

Table 5.53 Transverse compression test result for unidirectional samples @ 80°C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC8.1	0.510	0.135	0.069	288.17	4.17
TC8.2	0.509	0.136	0.069	354.72	5.13
TC8.3	0.513	0.136	0.070	290.91	4.17
AVG					4.49
% STD					0.55

Table 5.54 Transverse compression test result for unidirectional samples @ 110°C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC9.1	0.518	0.138	0.072	148.88	2.07
TC9.2	0.517	0.139	0.072	149.57	2.08
TC9.3	0.518	0.138	0.071	157.80	2.20
AVG					2.11
% STD					0.07

Table 5.55 Transverse compression test result for unidirectional samples @ 140°C and 50% R.H.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC10.1	0.515	0.134	0.069	29.50	0.427
TC10.2	0.512	0.135	0.069	41.16	0.595
TC10.3	0.512	0.136	0.070	31.56	0.452
AVG					0.49
% STD					0.09

Figure 5.14 Transverse compression stress vs. temperature for unidirectional samples at 50% relative humidity.

5.2.1.4.3 Wet Conditions

This set of samples was tested under wet conditions with temperatures varying from 20⁰C to 140⁰C. Tables 5.56 to 5.60 show the test results for each batch of samples and their strength to failure. Figure 5.15 shows variation of transverse compressive stress with varying temperature under wet conditions. Compressive stress to failure reduced from 5.99 ksi to 0.31 ksi when temperature changed from 20⁰C to 140⁰C recording a reduction of 94.8%. Also a reduction of 46.4% was observed when temperature was changed from 20⁰C to 80⁰C which is relatively lesser than the reduction observed with variation of temperature from 80⁰C to 140⁰C which was found to be 90.3%.

Table 5.56 Transverse compression test result for unidirectional samples @ 20°C and wet conditions.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC11.1	0.519	0.140	0.073	495.37	6.79
TC11.2	0.518	0.138	0.072	421.96	5.88
TC11.3	0.517	0.139	0.072	382.85	5.31
AVG					5.99
% STD					0.74

Table 5.57 Transverse compression test result for unidirectional samples @ 50°C and wet conditions.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC12.1	0.518	0.139	0.072	306.69	4.27
TC12.2	0.515	0.137	0.070	295.71	4.20
TC12.3	0.517	0.139	0.071	354.04	4.93
AVG					4.47
% STD					0.40

Table 5.58 Transverse compression test result for unidirectional samples @ 80°C and wet conditions.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC13.1	0.517	0.141	0.072	249.74	3.43
TC13.2	0.517	0.141	0.073	176.33	2.42
TC13.3	0.517	0.139	0.072	273.07	3.79
AVG					3.21
% STD					0.71

Table 5.59 Transverse compression test result for unidirectional samples @ 110°C and wet conditions.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC14.1	0.517	0.139	0.072	56.26	0.782
TC14.2	0.515	0.140	0.072	52.14	0.724
TC14.3	0.519	0.139	0.072	66.55	0.920
AVG					0.81
% STD					0.10

Table 5.60 Transverse compression test result for unidirectional samples @ 140°C and wet conditions.

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
TC15.1	0.518	0.138	0.072	25.38	0.354
TC15.2	0.515	0.141	0.072	25.38	0.350
TC15.3	0.515	0.141	0.072	18.52	0.255
AVG					0.31
% STD					0.05

Figure 5.15 Transverse compression stress vs. Temperature for unidirectional samples at wet conditions.

5.2.1.4.4 Conclusions

Figure 5.16 shows consolidated test results for transverse compressive stress versus temperatures at three humidity variations. As seen in the above sections these tests also show a similar trend. As the temperature changes from 20⁰C to 140⁰C we observe 93%-95% reduction in strength at all the three humidity variations. As humidity changes from dry to 50% R.H, conditions a reduction of 12%-25% in failure is observed. Similarly from 50% R.H. to wet conditions a reduction of 20%-60% in compressive stress is observed at all temperatures. As we move from dry to wet conditions a strength reduction of 30%-65% was observed at all temperatures.

The maximum average strength of the transverse compression data along with the time to failure was also used to predict a creep rupture master curve using Miyano's Accelerated Testing Method (ATM) approach.

Figure 5.16 Transverse compression stress-temperature-humidity behavior of unidirectional composites

5.2.2 Creep Tests

A set of short term creep tests was done at various environmental conditions. Creep tests were needed to evaluate the long term behavior of the unidirectional composites, for design purpose it is very important to have long term data in order to estimate the life of composites.

First, a couple of samples were subjected to tensile creep. The setup for tensile creep test can be seen in Chapter 4. A loading level was selected based on the maximum load to failure obtained from static tests. The required load was applied to a tensile creep sample. Since a hole had to be drilled in the sample in order to attach it to the frame, a shear failure was observed near the hole instead of a tensile failure. This was attributed to the fact that the sample was unidirectional and there was a high stress concentration near the hole. The details of those tests are given in the Appendix.

Due to the difficulty in conducting effective tensile creep tests of unidirectional samples three point bending creep tests were performed instead. These tests were similar to the three point bending static tests except for the fact that a sustained load was used. The dimensions of the samples were the same as used for static tests, which were 5 inch long and ½” wide at a span of 4”. The details of the test set up and the procedure are given in Chapter 4. The variations of environmental conditions include: 1) Dry conditions at room temperature, 2) Wet Conditions at room temperature, 3) Dry conditions at 80⁰C and 4) Wet conditions at 80⁰C.

Deflections versus time plots are used in the following sections for the discussion purposes. The loading levels were selected based on the maximum stress to failure obtained from three point bending static tests at that particular condition.

5.2.2.1 Creep at Dry Conditions and Room Temperature

In this set of experiments, the test samples were subjected to short term flexural creep loads. The loading levels were a fraction of the maximum load to failure observed in the static test for the dry conditions and room temperature.

As seen from the Table 5.1, the maximum stress to failure at the above mentioned conditions was 140 ksi. Hence 5 different loading levels were used for the short term creep tests (65%, 54%, 52%, 50%, and 40%). It turned out that 65%, 54%, 52% loadings

gave short term creep data as they reached the tertiary creep stage soon after the test was started, hence causing a creep rupture. This is attributed to the fact that the high loading levels produced microcracks leading the sample to move from primary creep region to secondary creep region and as the microcracks developed with applied constant stress, the stress concentration increased. When the stress concentration became so high that samples were not able to sustain the stresses near the crack, hence it caused the samples to fail catastrophically. Therefore, the tertiary creep is observed after a narrow secondary creep region.

40% and 50% loading levels gave large secondary creep region, which is attributed to the similar explanation of microcracking except for the fact that the stresses near the holes were not enough, leading to a failure. But with time due to the constant load and stress concentration near the crack the crack would propagate and stresses increase, which will lead to a situation, when the stresses would be high enough not to be sustained by the sample, leading to failure. Hence it can be assumed that for these particular samples under particular environmental conditions, the threshold value for the crack to propagate catastrophically is around 50%. As can be seen from Figure 5.17, the loading level at 52% is a short term test while 50% stress is not high enough to lead to a catastrophic failure.

It is also seen from the Figure 5.17 that the initial deflection decreases and the time to failure increases as the loading level decreases, hence with increase in loading level the nature of creep remains the same but the secondary creep region becomes narrower.

Figure 5.17 Deflection-time behavior of three point bending creep with constant loads at dry conditions and room temperature

5.2.2.2 Creep at Wet Conditions and Room Temperature

This set of samples was tested for their creep properties at wet conditions and room temperature. The samples were kept in a tank filled with water; the assembly of tests at these conditions is shown in chapter 4. The samples were subjected to a constant load and at the same time allowed for water diffusion. The loading levels were based on the ultimate failure loads of static tests under wet conditions and room temperature. Table 5.11 shows that the average stress to failure was 118 ksi at wet conditions and room temperature. The creep tests were conducted at 60%, 57% and 43.55% of stress to failure, where 60% and 57% loading levels were short term creep tests and 43.55% was a long term creep test.

Figure 5.18 shows deflection versus time curves for different loading levels at wet condition and room temperature.

Figure 5.18 Deflection-time behavior of three point bending creep with constant loads at wet conditions and room temperature

5.2.2.3 Creep at Dry Conditions and 80°C

These set of samples were subjected to creep test under dry conditions and 80°C. These tests were conducted inside the environmental chamber to maintain a uniform temperature of 80°C throughout the test. The assembly of this test is shown in detail in Chapter 4. In case of polymers, even a small change in temperature can have a large impact on their properties which was also observed under static tests at higher temperatures. Hence this test was done to determine the effect of temperature on the long term properties of the polymer composite.

Table 5.3 shows that the average stress to failure is 68 ksi at dry conditions and 80°C. The creep tests were conducted at 78%, 67.8% and 62.6% of stress to failure. All these tests were short term tests and the effect of temperature considerably decreased failure time.

Figure 5.19 shows deflection versus time curves for different loading levels at dry conditions and 80°C.

Figure 5.19 Deflection-time behavior of three point bending creep with constant loads at dry conditions and 80⁰C

5.2.2.4 Creep at Wet Conditions and 80⁰C

These set of samples were tested for their creep properties under wet conditions and 80⁰C. The samples were kept in a tank filled with water. The temperature was maintained using two coil heaters which were controlled by a thermocouple. The detailed assembly is shown in Chapter 4. These tests were conducted to study the effects of temperature and moisture diffusion on creep properties.

Table 5.13 shows that the average stress to failure is 42.9 ksi at wet conditions and 80⁰C for the static test. The creep tests were conducted at 74.2%, 67.9% and 63.4% of stress to failure. Figure 5.20 shows deflection versus time curves for different loading levels at wet conditions and 80⁰C.

Figure 5.20 Deflection-time behavior of three point bending creep with constant loads at wet conditions and 80°C

5.2.2.5 Conclusions

Unidirectional pultruded samples were tested for their creep properties at varying environmental conditions. Table 5.61 gives a summary of the creep tests. It also includes the details like initial deflection and time to failure, if the sample failed. Figure 5.21 shows creep rupture data for all the samples tested and failed i.e. the constant loading stress versus the time taken for the sample to fail. Figure 5.21 shows how temperature and water affect the long term properties of composite samples. It can be seen clearly that at higher stresses the failure is faster for all environmental conditions. Also the effect of temperature is much more pronounced when compared to the effect of water absorption. Major cause of creep in composites is primarily due to the matrix while the creep of glass fibers is considered negligible. The reduction in properties is attributed to the fact that moisture absorption in composite results in development of residual stresses and plasticizes the matrix. Rate of creep also considerably increases with immersion in water due to redistribution in stresses both between glass fiber and the vinyl ester matrix and

between damaged and undamaged reinforcing elements. At elevated temperatures the pattern of response for creep essentially retained but the range of limited creep becomes narrower. At higher stress levels creep damage is attributed to a slip between fiber and matrix.

The creep data was generated to verify the creep master curves generated using static data by Miyano's Accelerated Testing Methodology (ATM), which will be discussed in detail in the chapter 6.

Figure 5.21 Stress- Time to Failure of three point bending creep test at all conditions

Table 5.61 Summary of creep results for unidirectional pultruded samples (as of May 30, 2009)

Sample No.	Loading (%)	Temp (°C)	Condition	Span (in)	Initial Deflection (in)	Time Running (days)	Failed
1	65	RT	Dry	4	0.234"	0.16	✓
2	54	RT	Dry	4	0.230"	4.06	✓
3	52	RT	Dry	4	0.216"	8.02	✓
4	50	RT	Dry	4	0.203"	317.70	✗
5	40	RT	Dry	4	0.183"	317.85	✗
6	60	RT	Wet	4	0.230"	0.48	✓
7	50	RT	Wet	4	0.219"	21.83	✓
8	43.5	RT	Wet	4	0.102"	40.94	✗
9	78.1	80	Dry	4	0.207"	0.015	✓
10	67.8	80	Dry	4	0.144"	0.048	✓
11	62.6	80	Dry	4	0.123"	0.35	✓
12	74.2	80	Wet	4	0.130"	0.019	✓
13	67.9	80	Wet	4	0.113"	0.05	✓
14	63.4	80	Wet	4	0.092"	0.69	✓

5.2.3 Void Analysis using Scanning Electron Microscopy

Composite properties are generally governed by the nature of interfacial bonding between the matrix and the fiber. In order to obtain a uniform stress distribution and to prevent any stress concentration in local areas, the quality of bonding must be as high as possible so as to achieve a strong adhesion. Also to enhance performance, good adhesion between the fiber and the sizing is needed in addition to correct intrinsic physiochemical and mechanical characteristics. Therefore, microstructure properties give an insight on composite properties and manufacturing methods. Scanning Electron Microscopy (SEM) is an imaging technique which was used in this study to analyze the microstructure. Sample dimensions and testing procedure are given in Chapter 4 along with details about SEM.

The objectives of SEM analysis involve examination of fiber-matrix adhesion, analysis of voids and confirmation of the morphology of the composite system.

Using SEM the fiber orientation was confirmed within the composite specimen, all the fibers were oriented in one direction and only one plane, hence showing the fibers only in 0° as can be seen from Figure 5.22.

Figure 5.22 Fiber orientations in the unidirectional pultruded samples

Strength of fiber reinforced polymer composites is governed by the degree of compatibility between fiber and matrix. Good interface bonding results in high performance composites. SEM images in Figure 5.23 show the effective bonding

between fiber and matrix in some areas. It reveals the continuous matrix region between fibers and good interface contact.

Figure 5.23 SEM images showing good fiber-matrix adhesions for unidirectional pultruded samples

Presence of voids in composites significantly discounts the mechanical strength of the material through increasing localized stress concentration and microcrack propagation. The region with high void content is often responsible for crack propagation during loading. Voids are created due to entrapment of air inside the material during manufacturing operation or poor wet out without adequate resin. Although voids can not be completely removed from the material, void density can be effectively reduced by proper selection of curing parameters or reaction rate, applied pressure and pull rate. Figure 5.24 shows the region of voids due to poor wet out.

Figure 5.24 Unidirectional pultrusion sample showing high void surface

Figure 5.25 shows high void content due to incompatibility between the polypropylene fibers used to stitch glass fibers. This incompatibility is attributed to the fact that thermoplastics and thermosets are not compatible.

Figure 5.25 Unidirectional pultrusion samples with regions of high void density near stitch

Figure 5.26 shows the region of high and low void contents and it can be seen the void contents are higher near the polypropylene stitch and matrix interface than to glass fibers/matrix interface.

Figure 5.26 Unidirectional pultrusion samples with regions of high/low void density

It is important to understand and optimize the chemical composition and morphology of the composites because of the nature of interface i.e. fiber-matrix adhesion dictates the characteristics of the composite and its end use. Also a surface coating (gel coating) is

required to prevent any diffusion and chemical attack to accommodate the chemical attack by the matrix. The process parameters such as temperature, pressure, curing time, pull rate dictate the wet out of the fibers. Load transfer is accomplished from the generally lower-stiffness matrix to the high-modulus fibers through the interface. Fiber-matrix bonding is sufficient to accomplish this transfer for axial composite properties, but highly bonded regions are necessary to achieve high levels of off-axis and shear properties.

5.3 Quasi-Isotropic E-glass/ VE Composites with Holes

These set of samples i.e. quasi isotropic samples were used to study both static tension and tensile creep properties. A quarter inch hole was drilled in the center of all tension samples. The tension samples were 1 inch wide and 10 inch long with 2 inch tabs on both ends. These compressed tension samples were tested both for their static properties and creep properties. Since these samples were quasi-isotropic, all directions had the same amount of fiber which provided equal strength in all directions.

5.3.1 Static Tests

5.3.1.1 Tension Tests

The tension tests were conducted at four different temperatures (20⁰C, 50⁰C, 80⁰C and 110⁰C), all in dry room conditions. These test results were also used to determine the loading levels for the tensile creep tests described in later sections. The samples were preconditioned in an environmental chamber for 24 hours to attain temperature equilibrium throughout the sample. Two replications were done for each test at each temperature. Tables 5.62 to 5.64 show the test results for the each batch of samples and their strength to failure.

Figure 5.27 shows variation of tensile stress with temperature. As seen from the graph there is a considerable decrease in strength with increase in temperature. Increasing temperature from 20⁰C to 110⁰C reduced the tensile stress to failure from 32.68 ksi to 19.05 ksi which is around 42% reduction in strength. Also, as seen from Figure 5.27 the decrease in strength is 32.68 ksi at 20⁰C to 28.68 ksi at 80⁰C which is a reduction of 12% in strength, but with further increase in temperature from 80⁰C to 110⁰C, the reduction in

strength becomes significant, further reducing the strength from 28.68 ksi to 19.05 ksi which is 33% reduction in strength, as above 100⁰C the polymer matrix approaches its T_g.

Table 5.62 Quasi-Isotropic tension test samples at 20⁰C

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
QT1.1	0.989	0.089	0.088	2767.11	31.44
QT1.2	1.014	0.089	0.068	3071.75	33.92
AVG					32.68
% STD					1.75

Table 5.63 Quasi-Isotropic tension test samples at 50⁰C

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
QT2.1	1.001	0.086	0.086	2672.43	31.04
QT2.2	1.001	0.089	0.089	2727.32	30.71
AVG					30.87
% STD					0.23

Table 5.64 Quasi-Isotropic tension test samples at 80⁰C

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
QT3.1	0.977	0.089	0.087	2470.71	28.52
QT3.2	1.006	0.090	0.090	2613.42	28.85
AVG					28.68
% STD					0.23

Table 5.65 Quasi-Isotropic tension test samples at 110°C

Sample	Avg. Width (in.)	Avg. Thickness (in.)	Area (in²)	Max. Load (lbs)	Max. Stress (ksi)
QT3.1	1.011	0.089	0.090	2337.61	25.97
QT3.2	1.002	0.087	0.087	1149.93	13.14
AVG					19.55
% STD					9.07

Figure 5.27 Tensile stress to failure vs. temperature for Quasi-Isotropic samples

5.3.2 Creep Tests

A set of short term tensile creep tests was performed at room temperature under different loadings. These tensile creep tests were done using Instron 8801 with two different loading levels. The dimensions of the samples were kept the same as were used for static tension tests. Deflections versus time plots were used to reflect the results. The loading levels were selected based on the maximum stress to failure obtained from static tension tests at room temperature. As seen from Table 5.62, the maximum stress to failure at room temperature was 32.68 ksi. Two different loading levels were selected for the short term creep tests (86% and 88%). Figure 5.28 shows the deflection versus time curve for the two creep tests. The time taken for these samples to fail due to creep was 5.85 min for the 88% and 228.3 min for the 86% loading sample. It can be seen that there is significant increase in time to failure at lower loading level.

Figure 5.28 Deflection-time behavior of tensile creep with constant loads at room temperature

5.3.3 Void Analysis using Scanning Electron Microscopy

Scanning Electron Microscope was used to reveal the fiber-matrix adhesion, voids and morphology of the composite. The test procedure is similar to the tests done for unidirectional pultruded samples. The details about this test are given in Chapter 4 and Section 5.2.3.

The fiber orientation was seen using SEM. However it was difficult for the three different directions of fiber [0/+45/-45] to remain in one image. The 0° fibers are easily viewed as small full circles and +45° are smaller elliptical fibers. Figure 5.29 shows 0° as circular fibers and +45 as elliptical fibers and also that the 0° fibers look much larger than +/-45°, and that make them distinguishable as is seen from the Figure 5.29. The 90° fibers can be seen as lines of fibers running horizontally.

Figure 5.29 Fiber orientation in the QI compressed samples

SEM images in Figure 5.30 show the effective bonding between fiber and matrix. It reveals that there is a continuous matrix region between fibers and good interface contact. Figure 5.31 shows the region of voids due to poor wet out.

Figure 5.30 SEM images showing good fiber-matrix adhesions for QI compressed samples

Figure 5.31 QI compressed sample showing high void surface

It is important to understand and optimize the chemical composition and morphology because of the nature of interface i.e. fiber-matrix adhesion dictates the characteristics of the composite and its end use. The process parameters such as temperature, pressure and curing time dictate the wet out of the fibers as discussed in detail in Section 5.2.3.

5.4 Tri-axial E-glass/PU Composites

These samples were first tested to failure for their three point bending static properties. After the maximum stress was determined, they were tested for their long term responses using three point bending creep.

5.4.1 Static Tests

Samples were tested for their static mechanical properties. Coupon specimens with desired dimensions were prepared for testing under three point bending using ASTM D790. Load-deflection behavior was plotted for the bending response. These three point bending static tests were carried out to failure on Instron. All tests were done at room conditions. The purpose of the static tests was to determine the maximum stress for the samples on which creep loadings are based.

5.4.1.1 Half Inch Thick Samples

This set of half inch thick samples was tested for their bending properties. Table 5.66 shows the dimensions of the samples including the span length, bending stress to failure and bending modulus. Also, the slope of load versus deflection is shown which was used to calculate bending modulus. Figure 5.32 shows the load deflection curves for all the three samples tested. As seen from the Table 5.66, the average bending stress to failure is 84.29 ksi and average bending modulus is 2.66 msi. The failure mode observed in these samples was primarily due to delamination. This delamination was attributed to the fact that two CFM layers of fabric were used which prevented resin flow, resulting in an incomplete wet out. Considering the maximum stress to failure, four creep loadings were selected to determine the long term properties.

Table 5.66 Bending test results for tri-axial composite samples, half inch thick

Sample No.	Width (in)	Thickness (in)	Span (in)	Max Load (lb)	Max Stress (psi)	E_b (msi)
CH1	0.421	0.499	8	700.44	80.18	2.53
CH2	0.44	0.498	8	826.228	90.86	2.77
CH3	0.389	0.498	8	660.47	81.82	2.67
Avg.					84.29	2.66
%STD					5.75	0.12

Figure 5.32 Load versus deflection behavior of tri-axial composite samples, half inch thick

5.4.1.2 Quarter Inch Thick Samples

These sets of quarter inch thick samples were also tested for their bending properties. Table 5.67 shows the dimensions of the sample including the span length, bending stress to failure and bending modulus. Also, the slope of load versus deflection is shown which was used to calculate bending modulus. Figure 5.33 shows the load deflection curves for all the three test samples. As seen from Table 5.67, the average bending stress to failure, is 58.5 ksi and average bending modulus was 1.73 msi. Considering the maximum stress to failure 4 creep loadings were selected to determine the long term properties.

Table 5.67 Bending test results for tri-axial composite samples, quarter inch thick

Sample No.	Width (in)	Thickness (in)	Span (in)	Max Load (lb)	Max Stress (ksi)	E _b (msi)
CQ1	0.450	0.249	4	280.19	59.77	1.82
CQ2	0.449	0.248	4	272.82	58.80	1.63
CQ3	0.421	0.248	4	245.70	56.93	1.74
Avg.					58.5	1.73
% STD					1.443	0.09

Figure 5.33 Load versus deflection behavior of tri-axial composite samples, quarter inch thick samples

5.4.2 Creep Tests

These set of experiments were conducted to determine the long term creep responses of these samples. Since polymers are viscoelastic materials, for design purposes, it is important to determine how the material modulus changes with time. Hence a set of three point bending creep tests were done. Findley's power law was used to determine the change in strain with time as it is impossible to run creep tests for all the materials for a long interval of time used for infrastructure applications. These tests over a small interval of time can help in determining the change of modulus with time.

5.4.2.1 Half Inch Thick samples

The half inch thick samples were tested at four loading levels of 80%, 65%, 50% and 35% of failure load, where 80% and 65% were short term tests and 50% and 35% were long term tests. The creep curves are shown in Figure 5.34 in the form of deflection versus time curves. As can be seen, the 80% test lasted for a very short period of time and the sample failed in 70 min whereas the 65% test failed in 92 days. It can be seen that how a 15% decrease in loading can significantly increase the time to failure. It can also

be seen the 50% and 45% tests are still ongoing and have initial deflection much lower than 65% and 80%. The 65% sample failed due to delamination due to the similar reason of poor wet out as discussed in the Section 5.4.1.1.

Figure 5.34 Deflection-Time curves of half inch thick tri-axial samples

5.4.2.2 Quarter Inch Thick samples

Similarly, the quarter inch thick samples were tested under four different loading levels of 80%, 65%, 50% and 35%, where 80% and 65% were short term tests and 50% and 35% were long term tests. The creep curves are shown in Figure 5.35 in the form of deflection versus time curves. As can be seen the 80% test was a short term test and the sample failed in 7.5 days. It can also be seen that as the loading levels decrease, the slope of the curve and initial deflections also decrease.

Figure 5.35 Deflection-Time curves of quarter inch thick tri-axial samples

5.4.3 Conclusions

Creative Pultrusion samples were tested for their long term behavior and change in deflection with time. Table 5.68 gives a summary of the creep tests. It also includes the details like initial deflection, time to failure and whether the samples failed or not.

Major cause of creep in composites is due to the matrix and the creep of glass fibers is considered negligible. At higher stress levels of 80% loading, the creep damage is attributed to slip between fiber and matrix.

These creep data were generated to determine the change of modulus or strain with time under constant load using Findley's Power law model, hence preventing the need to conduct creep tests for long intervals of time. The curves for the Findley's power law model are shown in detail in Chapter 6 along with its verification using the experimental data.

**Table 5.68 Summary of creep results for Tri-axial pultruded samples,
(as of May 30, 2009)**

Sample No.	Dimensions (L x B x T)	Loading (%)	Span (in)	Initial Deflection (in)	Time Running (days)	Failed
1	5"x0.415"x0.25"	80	4	0.236"	7.5	✓
2	5"x0.415"x0.25"	65	4	0.200"	121	✗
3	5"x0.415"x0.25"	50	4	0.150"	138	✗
4	5"x0.410"x0.25"	35	4	0.109"	139	✗
5	10"x0.415"x0.5"	80	8	0.536"	0.05	✓
6	10"x0.420"x0.5"	65	8	0.355"	92.8	✓
7	10"x.410"x0.5"	50	8	0.315"	91	✗
8	10"x.420"x0.5"	35	8	0.153"	131	✗

CHAPTER 6

CREEP STRAIN RESPONSE AND RUPTURE PREDICTIONS

6.1 Introduction

The long term behaviors of creep and fatigue are very important for a number of applications. The need to develop creep response predictive methods to determine long term life of polymer composites is felt necessary because of various time controlled properties like strength and modulus change with time under constant load. This chapter involves prediction of long term properties of polymer composites under constant loads. Method of prediction of creep rupture and change in modulus with time under creep is suggested in this chapter; the data used for these predictions are given in chapter 5.

Various models have been developed to study creep in polymers. For example, the models used in this study are Accelerated Testing Methodology (ATM) which was developed by Miyano in 2006 (Miyano et. al., 2006) and the popular Findley's Power Law Model (Findley et. al., 1987). ATM was used to determine the creep rupture properties and Findley's model has been used to determine the change in modulus with time.

6.2 Miyano's Accelerated Testing Methodology

The most important basis of Miyano's ATM in prediction of creep rupture is time temperature superposition. It was developed mainly for polymer composites as these materials show similar failure modes under constant strain rate tests (static tests), creep tests and fatigue tests. Hence a relationship can be developed to derive one from the other. Also, the results from the elevated temperature are used to accelerate the mechanisms of mechanical degradation which occurs under loads over a long period of time. The degree of acceleration per increment of elevated temperature is found through the use of time-temperature superposition principle. This correlation was developed by Miyano in 2006 and the sections below show how to convert a Constant Strain Rate (CSR) or static test data to creep rupture using time-temperature superposition. In this study, short term creep tests were used to validate the model developed by using CSR tests. Unidirectional E-glass/VE composites were analyzed using this methodology.

6.2.1 Flexural Creep Master Curve @RT, Dry Conditions

Figures 6.1 to 6.7 shows how the static three point bending test at varying temperatures and dry conditions can be used to create a creep master curve at room temperature and dry conditions. This master curve was then verified with the actual creep data.

Figure 6.1 provides longitudinal flexural strength under Constant Strain Rate (CSR) with time to failure which was obtained by the three point bending static tests at varying temperatures and dry conditions (section 5.2.1.1.1). Flexural strength for unidirectional samples was determined over a temperature range of 20⁰C to 140⁰C at 30⁰C intervals. Figure 6.1, therefore, shows the relationship between longitudinal flexural CSR strength and the logarithm of real time to failure at different temperatures. It can be seen from the graph that time to failure for the flexural test decreased with increase in the temperature. This is because with increase in temperature the strength of polymer matrix decreases. It is caused due to polymer softening as the temperature approaches T_g.

Figure 6.1 Relationship between log (flexural strength) and logarithmic of real time to failure under dry conditions at range of temperature (20-140⁰C).

To obtain a flexural creep master curve at room temperature and dry conditions, time-temperature superposition principle was used. To apply time temperature superposition principle, shift factors are needed in both horizontal and vertical directions. The shift factors were obtained from US Army through Kazak Composites Co. The shift factors were obtained based on creep data of pure vinyl ester resin. Since creep is primarily a matrix property, because creep response is dominated by matrix with brittle fiber effect the shift factors for neat resin can be employed to its composite.

The shift factors are given by the expression:

$$\log aT_0(T) = \log t - \log t' \quad (6.1)$$

$$\log bT_0(T) = \log D_c(t, T) - \log D_c(t', T) \quad (6.2)$$

where aT_0 is the horizontal shift factor, bT_0 is the vertical shift factor, t is the time, t' is the reduced time, D_c is the creep compliance and T is temperature.

We can use the shift factors related to the neat vinyl ester resin to determine the flexural CSR strength master curve. The data obtained for the shift factors was available only till 100°C. Since the flexural strength was tested till 140°C, therefore, the shift factors were to needed till 140°C. The shift factors generally follow a bilinear trend; hence, a bilinear extrapolation was done to obtain shift factors at 110°C and 140°C. Table 6.1 shows the shift factors at various temperatures under dry conditions using 20°C as reference temperature.

Table 6.1 Time-temperature shift factors for dry conditions at reference temperature of 20⁰C

Temperature (⁰ C)	log(aT ₀)	log(bT ₀)
20	0	0
40	-1.2	0.01
50	-1.7	0.013
60	-2.5	0.017
70	-4	0.02
80	-5	0.02
90	-6.45	0.02
100	-7.3	0.01
110	-8.665	0.01
140	-12.28	0.01

Figure 6.2 is a plot of horizontal shift factor versus temperature which was obtained from equation 6.1. It can be seen that it follows a bilinear trend and hence a bilinear extrapolation is a valid assumption to determine the shift factors at 110⁰C and 140⁰C. The equation seen in the figure represents the second linear part of the shift factors from which the required shift factors were obtained; since 110⁰C and 140⁰C lie on the second linear part of the curve.

Figure 6.2 Bilinear extrapolation curve of shift factor versus temperature at dry conditions

To obtain a master curve at room temperature and dry conditions for the constant strain rate, time-temperature superposition was used using the shift factors as shown in Table 6.1. The points shown in Figure 6.1 were shifted both horizontally and vertically using the shift factors with 20⁰C as the reference temperature to obtain a master curve at 20⁰C and dry conditions. In Figure 6.3, logarithm of flexural CSR strength is related to logarithm of reduced time to failure t^* s under dry conditions; this may also be called as CSR master curve at dry conditions and at 20⁰C, since the shift factors were obtained at dry conditions and with a reference temperature of 20⁰C.

Figure 6.3 Master curve of flexural CSR strength with reduced time at RT and dry conditions.

According to Miyano's ATM approach (Miyano et. al., 2006), the creep master curve can be obtained from the CSR strength master curve by the relationship shown below:

Figure: 6.4 Shifting property of constant strain rate (CSR) strength master curve to creep rupture strength master curve

Now using the above relation, n was calculated using the linear slope of the flexural CSR master curve which is given by $1/(1+1/n)$. According to Miyano, if the CSR strength master curve is horizontally shifted to the left by the factor of $\log(1/n+2)$ we obtain a creep rupture master curve for that particular material at that condition. For the current case, to determine the creep master curve for flexural strength under dry conditions at room temperature, the flexural CSR strength master curve was shifted by a factor of 1.08. The slope of the linear part of the CSR master curve was 0.0907 and $1/n$ was calculated as 10.02. Figure 6.5 shows the flexural Creep strength master curve obtained by shifting the CSR strength master curve by a factor of 1.08.

Figure 6.5 Master curve of flexural CSR and Creep strength with reduced time to failure.

Hence, creep master curve was developed using the static tests or constant strain rate tests which are short term tests and can be performed easily in a laboratory avoiding the need for the cumbersome creep tests. This flexural creep master curve is valid for 20⁰C and dry conditions; hence, the strength corresponding to time on the creep master curve is the

time for creep rupture when the sample is subjected to that particular strength. To validate this model short term creep tests were done which are described in Chapter 5. The short term creep data at room temperature and dry conditions were plotted along the creep master curve as shown in Figure 6.5.

Figure 6.6 Verification of flexural creep strength master curve using experimental creep time to failure data at 20⁰C and dry conditions

As can be seen from Figure 6.6, the experimental creep data for 52%, 54% and 60% loading at room temperature and dry conditions fall well on the creep master curve. Hence, the creep master curves offers a potential to be used in determining the time to creep rupture at room temperature and dry conditions, with a known loading level. Accelerated Testing Methodology was successful in determining the time to failure due to creep rupture using the creep master curve drawn by using static tests data. Similarly, we can use the static data to determine the creep strength master curve for different environmental conditions and at different loading directions as discussed in the sub-sections below.

6.2.2 Flexural Creep Master Curve @80°C @Dry Conditions

Similar approach, as used in section 6.2.1, to prepare creep master curve at room temperature and dry conditions has been used to prepare master curves at higher temperatures. To prepare master curve at 80°C and dry conditions, instead of considering room temperature as reference, we consider 80°C as the reference temperature. Figure 6.7 shows the plot of flexural CSR strength with time to failure of CSR tests over a temperature range of 80°C to 140°C with an interval of 30°C.

Figure 6.7 Relationship between log (flexural strength) and logarithmic of real time to failure under dry conditions at range of temperature (80-140°C).

To obtain a CSR master curve we use time-temperature superposition and shift the points, using horizontal and vertical shift factors. The shift factors for dry conditions are shown in Table 6.1. To prepare creep master curve at 80°C, we take 80°C as the reference temperature and calculate the shift factors; here the shift factors at reference temperatures

are considered as zero and the other shift factors are calculated relative to the reference shift factor at 80°C. The shift factors for 80°C and dry conditions are shown in Table 6.2.

Table 6.2 Time-temperature shift factors for dry conditions at reference temperature of 80°C

Temperature (°C)	$\log(aT_0)$	$\log(bT_0)$
80	0	0
90	-1.45	0.02
100	-2.3	0.01
110	-3.66	0.01
140	-7.28	0.01

Using these shift factors, the points in Figure 6.7 are shifted horizontally and vertically to obtain a CSR master curve. The CSR master curve for 80°C and dry conditions is shown in Figure 6.8. This flexural CSR strength master curve is at reduced time under dry conditions at 80°C.

Figure 6.8 Master curve of flexural CSR strength with reduced time at 80⁰C and dry conditions.

The slope of the curve in Figure 6.8 is 0.1795 which is equal to $1/(1+1/n)$ as discussed in the previous section. Hence, in this case at 80⁰C and dry conditions we get $1/n = 4.571$, which is used to calculate the shift factor $\log(1/n+2)$ to transform flexural CSR master curve to creep rupture master curve. The shift factor to obtain the creep master curve at 80⁰C and dry conditions was found to be 0.8176. Figure 6.9 shows the flexural creep strength master curve obtained by shifting the CSR strength master curve. This master curve is for 80⁰C and dry conditions.

Figure 6.9 Master curve of flexural CSR and Creep strength with reduced time to failure.

This flexural creep master curve is valid for 80⁰C and dry conditions. To verify the master curve short term creep tests were done at 80⁰C and dry conditions. Creep strength master curve was then plotted along with short term creep tests as shown in Figure 6.10.

Figure 6.10 Verification of flexural Creep Strength master curve using experimental creep time to failure data at 80⁰C and dry conditions

As can be seen from figure 6.10, the experimental creep data for 62.6%, 67.8% and 78% loading at 80⁰C and dry conditions fell on the creep master curve. Hence the creep master curves can be used to determine the time to failure due to creep rupture at 80⁰C under dry conditions, with a known loading level.

6.2.3 Flexural Creep Master Curve @RT @Wet Conditions

To prepare a flexural creep master curve at room temperature and wet conditions, the constant strain rate bending tests done under wet conditions under different temperatures were employed. The reference temperature was taken as 20⁰C for this case. Figure 6.11 shows the plot of flexural CSR strength with time to failure for the temperature range of 20⁰C to 140⁰C under wet conditions.

Figure 6.11 Relationship between log (flexural strength) and logarithmic of real time to failure under wet conditions at range of temperature (20-140⁰C).

Since the shift factors vary with the dry to wet conditions, to use time temperature superposition, the shift factors for pure vinyl ester resin under wet conditions were obtained from US Army through Kazak Composites Co. The reference temperature used for these shift factors was 20⁰C and the shift factors were obtained from 20-80⁰C. Table 6.3 lists the horizontal (log aT₀) and vertical (log bT₀) shift factors utilized for time-temperature superposition.

Table 6.3 Time-Temperature shift factors for wet conditions at reference temperature of 20⁰C

Temperature (⁰ C)	log(aT ₀)	log(bT ₀)
20	0	0
40	-1.5	-0.015
50	-2.0	-0.02
60	-2.5	-0.024
70	-4.2	-0.026
80	-5	-0.05
110	-8.9	-0.01
140	-12.65	-0.01

Since the data for the shift factors was available only from 20-80⁰C, the shift factors at 110⁰C and 140⁰C were obtained using the bilinear extrapolation of the horizontal shift factor shown as shown in Figure 6.12. The equation shown in the Figure 6.12 corresponds to the second linear region of the curve and was used to determine the shift factors at 110⁰C and 140⁰C.

Figure 6.12 Bilinear extrapolation curve of shift factor versus Temperature at wet conditions

Now, using the time-temperature superposition principle, the points shown in Figure 6.11 were shifted both horizontally and vertically to obtain a flexural CSR strength master curve. Figure 6.13 shows the relationship of logarithm of flexural CSR strength with reduced time to failure under wet conditions.

Figure 6.13 Master curve of flexural CSR strength with reduced time ($t's$) at RT and wet conditions.

The slope of the linear part of the curve as shown in Figure 6.13 was found to be 0.1142 and $1/n$ was calculated as 7.756 as the slope according to Miyano is given by $1/(1+1/n)$. The shift factor used to transform the CSR strength master curve to the creep master curve was found to be 0.989. Figure 6.14 shows flexural creep strength master for wet conditions obtained by shifting the CSR strength master curve horizontally by the factor of 0.989.

Figure 6.14 Master curve of flexural CSR and Creep strength with reduced time to failure under wet conditions and RT.

Therefore, the creep master curve was obtained for wet conditions and room temperature using Accelerated Testing Method. To verify the master curve, short term creep tests were done at room temperature and wet conditions to determine the time to creep rupture. As can be seen from figure 6.15, the two creep tests with 57% and 60% loading done at room temperature and wet conditions are plotted alongside the creep master curve. It can be seen in Figure 6.15 that the 60% loading creep test fits perfectly on the creep master curve, but the error in case of 57% loading creep test is due to the fact that the static tests used to make the creep master curve were conditioned for 24 hours. Hence, the moisture diffusion was not 100%, leading to the small error seen in case of 57% loading creep test.

Figure 6.15 Verification of flexural Creep Strength master curve using experimental creep time to failure data at 20⁰C and wet conditions

6.2.4 Flexural Creep Master Curve @80⁰C @Wet Conditions

To prepare a creep master curve at 80⁰C and wet conditions, we consider 80⁰C as the reference temperature. Figure 6.16 shows the plot of flexural CSR strength with time over a temperature range of 80⁰C to 140⁰C under wet conditions.

Figure 6.16 Relationship between log (flexural strength) and logarithmic of real time to failure under wet conditions at range of temperature (80-140⁰C).

To obtain a CSR master curve we use time-temperature superposition and shift the points using horizontal and vertical shift factors. The shift factors for wet conditions are shown in table 6.3. To prepare creep master curve at 80⁰C, we take 80⁰C as the reference temperature and calculate the shift factors. The shift factors for 80⁰C and wet conditions are shown in Table 6.4.

Table 6.4 Time-Temperature shift factors for wet conditions at reference temperature of 80⁰C

Temperature (⁰ C)	log(aT ₀)	log(bT ₀)
80	0	0
110	-3.9	-0.01
140	-7.65	-0.01

Using these shift factors, the points in the Figure 6.16 are shifted horizontally and vertically to obtain a CSR master curve. The CSR master curve for 80⁰C and wet conditions is shown in Figure 6.17. This flexural CSR strength master curve is at reduced time under dry conditions at 80⁰C.

Figure 6.17 Master curve of flexural CSR strength with reduced time (t's) at 80⁰C and wet conditions.

The slope of the curve in Figure 6.17 is 0.1414 which is equal to $1/(1+1/n)$, as is discussed in section 6.2.1. Hence, in this case at 80⁰C and wet conditions we get $1/n = 6.072$, which is used to calculate the shift factor $\log(1/n+2)$ to transform flexural CSR master curve to creep master curve. The shift factor to obtain the creep master curve at 80⁰C and dry conditions, was found to be 0.906. Figure 6.18 shows the flexural creep strength master curve obtained by shifting the CSR strength master curve, This master curve was developed for 80⁰C and wet conditions.

Figure 6.18 Master curve of flexural CSR and Creep strength with reduced time to failure under wet conditions and 80⁰C.

This flexural creep master curve is valid for 80⁰C and wet conditions. To verify the master curve, short term creep tests were done at 80⁰C and wet conditions. Creep strength master curve was then plotted with these short term creep tests as shown in Figure 6.19.

Figure 6.19 Verification of flexural Creep Strength master curve using experimental creep time to failure data at 80⁰C and wet conditions

As can be seen from Figure 6.19, the experimental creep data for 63.4%, 67.9% and 74.2% loading at 80⁰C and wet conditions fell on the creep master curve. There is a little error in case of 74.2% loading which is due to incomplete moisture diffusion in case of static tests as they were conditioned only for 24 hrs. Hence, the creep master curves can be used to determine the time to failure due to creep rupture at 80⁰C and wet conditions with a known loading level.

6.2.5 Flexural Creep Master Curve @RT @50% R.H.

To prepare a flexural creep master curve at room temperature and dry+wet conditions (50% R.H.), the constant strain rate bending tests done under wet conditions and different temperatures were used. The reference temperature was taken as 20⁰C for this case. Figure 6.20 shows the plot of flexural CSR strength with time to failure for the temperature range of 20⁰C to 140⁰C under wet conditions.

Figure 6.20 Relationship between log (flexural strength) and logarithmic of real time to failure under dry+wet conditions at range of temperature (20-140⁰C).

Since the shift factors vary with the humidity conditions, to use time temperature superposition, the shift factors for pure vinyl ester resin under dry+wet conditions were obtained from US Army through Kazak Composites Co. The reference temperature used for these shift factors was 20⁰C and the shift factors were obtained from 20-100⁰C. Table 6.5 below lists the horizontal (log aT₀) and vertical (log bT₀) shift factors utilized for time temperature superposition.

Table 6.5 Time-Temperature shift factors for dry + wet conditions at reference temperature of 20⁰C

Temperature (⁰ C)	log(aT ₀)	log(bT ₀)
20	0	0
40	-1.2	0.01
50	-1.7	0.013
60	-2.5	0.017
70	-4.0	0.02
80	-5	0.02
90	-6.45	0.02
100	-7.1	0.01
110	-8.505	0.01
140	-12	0.01

Since the data for the shift factors was available only from 20-100⁰C, the shift factors at 110⁰C and 140⁰C were obtained using the bilinear extrapolation of the horizontal shift factor as shown in Figure 6.21. The equation shown in the Figure 6.21 corresponds to the second linear region of the curve and was used to determine the shift factors at 110⁰C and 140⁰C.

Figure 6.21 Bilinear extrapolation curve of shift factor versus Temperature at dry+wet conditions

Using the time-temperature superposition principle, the points shown in Figure 6.20 were shifted both horizontally and vertically to obtain a flexural CSR strength master curve. Figure 6.22 shows the relationship of logarithm of flexural CSR strength with reduced time to failure under wet conditions.

Figure 6.22 Master curve of flexural CSR strength with reduced time ($t's$) at RT and dry+wet conditions.

The slope of the linear part of the curve shown in Figure 6.3 was found to be 0.134 and $1/n$ was calculated as 6.462 as the slope is given by $1/(1+1/n)$. The shift factor used to transform the CSR strength master curve to the creep master curve was found to be 0.927. Figure 6.23 shows flexural creep strength master for wet conditions obtained by shifting the CSR strength master curve horizontally by the factor of 0.927.

Figure 6.23 Master curve of flexural CSR and Creep strength with reduced time to failure under dry+wet conditions and RT.

6.2.6 Tensile Creep Master Curves

As discussed in the above sections about flexural creep master curves at different conditions (dry, wet and dry+wet), a similar study was done to prepare tensile creep master curves at these three conditions at room temperature using the static or CSR tensile data. To prepare these master curves transverse tensile tests were done at various temperature and different moisture conditions as discussed in chapter 5.

6.2.6.1 Dry conditions

To prepare a creep master curve at dry conditions for transverse tension test, transverse tensile CSR strength was plotted with time over a range of 20-110⁰C for dry conditions as shown in Figure 6.24. These points were shifted using shift factors as given in Table 6.1 both horizontally and vertically to obtain a tensile CSR strength master curve at dry conditions with reduced time. The slope of the curve was found to be 0.029 and the shift factor used to convert CSR master curve to tensile creep strength master curve by shifting the CSR master curve horizontally was found to be 1.55. Figure 6.25 shows the transverse tension CSR master curve along with transverse tension creep master curve.

6.2.6.2 Wet Conditions

Similarly, Figure 6.26 shows transverse tensile CSR strength for wet conditions. These points were then shifted using shift factors as in table 6.3 to prepare the CSR master curve at wet conditions. The slope of the curve was 0.0253 and the shift factor to horizontally shift the CSR master curve to creep master curve was found to be 1.607. Figure 6.27 shows the transverse tensile CSR master curve along with tensile creep master curve for wet conditions.

6.2.6.3 Dry+Wet Conditions

Similarly, Figure 6.28 shows transverse tensile CSR strength for dry+wet conditions. These points were then shifted using shift factors as in table 6.5 to prepare the CSR master curve at dry+wet conditions. The slope of the curve was 0.0154 and the shift factor to horizontally shift the CSR master curve to creep master curve was found to be 1.819. Figure 6.29 shows the transverse tensile CSR master curve along with tensile creep master curve for dry+wet conditions.

Figure 6.24 Relationship between log (tensile strength) and logarithmic of real time to failure under dry conditions at range of temperature (20-110°C).

Figure 6.25 Master curve of tensile CSR and Creep strength with reduced time to failure under dry conditions and RT.

Figure 6.26 Relationship between log (tensile strength) and logarithmic of real time to failure under wet conditions at range of temperature (20-80°C).

Figure 6.27 Master curve of tensile CSR and Creep strength with reduced time to failure under wet conditions and RT.

Figure 6.28 Relationship between log (tensile strength) and logarithmic of real time to failure under dry+wet conditions at range of temperature (20-80°C).

Figure 6.29 Master curve of tensile CSR and Creep strength with reduced time to failure under dry+wet conditions and RT.

6.2.7 Longitudinal Compression Creep master curves

As discussed in the above sections about flexural creep master curves at different conditions(dry, wet and dry+wet), a similar study was done to prepare longitudinal compressive creep master curves under these three conditions at room temperature. To prepare these master curves, longitudinal compression tests were done at various temperature and different moisture conditions as discussed in chapter 5.

6.2.7.1 Dry conditions

To prepare a creep master curve at dry conditions for longitudinal compression test, longitudinal compressive CSR strength was plotted with time over a range of 20-140⁰C for dry conditions as shown in Figure 6.30. These points were shifted using shift factors as given in Table 6.1 both horizontally and vertically to obtain a longitudinal compressive CSR strength master curve at dry conditions with reduced time. The slope of the curve was found to be 0.0961 and the shift factor used to convert CSR master curve to compressive creep strength master curve by shifting the CSR master curve horizontally was found to be 1.057. Figure 6.31 shows the longitudinal compressive CSR master curve along with longitudinal compressive creep master curve.

6.2.7.2 Wet Conditions

Similarly, Figure 6.32 shows longitudinal compressive CSR strength for wet conditions. These points were then shifted by using shift factors as in Table 6.3 to prepare the CSR master curve at wet conditions. The slope of the curve was 0.1315 and the shift factor to horizontally shift the CSR master curve to creep master curve was found to be 0.934. Figure 6.33 shows the longitudinal compressive CSR master curve along with longitudinal compressive creep master curve for wet conditions.

6.2.7.3 Dry+Wet Conditions

Similarly, Figure 6.34 shows longitudinal compressive CSR strength for dry+wet conditions. These points were then shifted by using shift factors as in Table 6.5 to prepare the CSR master curve at dry+wet conditions. The slope of the curve was 0.141 and the shift factor to horizontally shift the CSR master curve to creep master curve was found to be 0.9104. Figure 6.35 shows the longitudinal compressive CSR master curve along with longitudinal compressive master curve for dry+wet conditions.

Figure 6.30 Relationship between log (long. compressive strength) and logarithmic of real time to failure under dry conditions at range of temperature (20-140⁰C).

Figure 6.31 Master curve of longitudinal compressive CSR and Creep strength with reduced time to failure under dry conditions and RT.

Figure 6.32 Relationship between log (long. compressive strength) and logarithmic of real time to failure under wet conditions at range of temperature (20-140°C).

Figure 6.33 Master curve of longitudinal compressive CSR and Creep strength with reduced time to failure under wet conditions and RT.

Figure 6.34 Relationship between log (long. compressive strength) and log of real time to failure under dry+wet conditions at range of temperatures (20-140°C).

Figure 6.35 Master curve of longitudinal compressive CSR and Creep strength with reduced time to failure under dry+wet conditions and RT.

6.2.8 Transverse Compression Creep master curves

As discussed in the above sections about flexural creep master curves at different conditions(dry, wet and dry+wet), a similar study was done to prepare transverse compressive creep master curves under these three conditions at room temperature. To prepare these master curves transverse compression tests were done at various temperature and different moisture conditions as discussed in chapter 5.

Dry conditions

To prepare a creep master curve at dry conditions for transverse compression test transverse compressive CSR strength was plotted with time over a range of 20-140⁰C for dry conditions as shown in Figure 6.36. These points were shifted by using shift factors as given in Table 6.1, both horizontally and vertically to obtain a transverse compressive CSR strength master curve at dry conditions with reduced time. The slope of the curve was found to be 0.1097 and the shift factor used to convert CSR master curve to compressive creep strength master curve by shifting the CSR master curve horizontally was found to be 1.005. Figure 6.37 shows the transverse compressive CSR master curve along with transverse compressive creep master curve.

Wet Conditions

Similarly, Figure 6.38 shows transverse compressive CSR strength for wet conditions. These points were then shifted by using shift factors as in Table 6.3, to prepare the CSR master curve at wet conditions. The slope of the curve was 0.127 and the shift factor to horizontally shift the CSR master curve to creep master curve was found to be 0.948. Figure 6.39 shows the transverse compressive CSR master curve along with transverse compressive creep master curve for wet conditions.

Dry+Wet Conditions

Similarly, Figure 6.40 shows transverse compressive CSR strength for dry+wet conditions. These points were then shifted by using shift factors as in Table 6.5, to prepare the CSR master curve at dry+wet conditions. The slope of the curve was 0.15 and the shift factor to horizontally shift the CSR master curve to creep master curve was found to be 0.88. Figure 6.35 shows the transverse compressive CSR master curve along with transverse compressive master curve for dry+wet conditions.

Figure 6.36 Relationship between log (trans. compressive strength) and logarithmic of real time to failure under dry conditions at range of temperatures (20-140°C).

Figure 6.37 Master curve of transverse compressive CSR and Creep strength with reduced time to failure under dry conditions and RT.

Figure 6.38 Relationship between log (trans. compressive strength) and logarithmic of real time to failure under wet conditions at range of temperature (20-140⁰C).

Figure 6.39 Master curve of transverse compressive CSR and Creep strength with reduced time to failure under wet conditions and RT.

Figure 6.40 Relationship between log (trans. compressive strength) and log of real time to failure under dry+wet conditions at range of temperature (20-140⁰C).

Figure 6.41 Master curve of transverse compressive CSR and Creep strength with reduced time to failure under dry+wet conditions and RT.

6.3 Findley's Power Law Model

This section attempts to determine the time dependent behavior of polymer composites under sustained loads. The time dependent behavior was evaluated using Findley's Power law and was studied for the pultruded tri-axial E-glass/PU samples. The creep deformation with time was collected for these samples. Test procedure is given in Chapter 4 and the results of these are described in chapter 5.

Findley's Power law model was initially developed by Findley in 1944 to study the viscoelastic properties of polymers and this model has been successfully used to model creep properties of polymer composites for the past decade. As it is generally considered that the creep behavior is primarily matrix driven and the fibers provide negligible assistance to creep deformation. Hence, recognizing that creep as primarily matrix driven it Findley's power law which was first developed for thermoplastics can provide a good approximation of the creep study or viscoelastic property of the polymer composite. The Findley power law is usually given as:

$$\varepsilon(t) = \varepsilon_0 + mt^n \quad (6.3)$$

Where: $\varepsilon(t)$ = total time dependent strain

ε_0 = stress dependent initial elastic strain

m = stress dependent and temperature dependent coefficient

n = stress independent and temperature dependent material constant

t = time after loading

Constants m and n needed to formulate the power law were determined using the creep data, rearranging Equation 6.3 and taking logarithm on both sides we get:

$$\log(\varepsilon(t) - \varepsilon_0) = \log(m) + n \log(t) \quad (6.4)$$

Now to obtain m and n , by plotting Equation 6.4 which will yield a straight line, then vertical intercept at time $t = 1$ hr will give m and the slope of the line will give the material constant n . m and n for half inch and quarter inch thick samples of tri-axial

polyurethane pultruded composites along with verification of the model is shown in sections 6.3.1 and 6.3.2.

6.3.1 Half Inch Thick Tri-axial Pultruded Samples

As discussed in Chapter 5, half inch thick samples were subjected to creep with 4 different loading levels. Two of these tests were short term tests at 80% and 65% loading. The 80% loading sample failed in 1.2 hours and the 65% loading sample failed in 92.85 days. The deflections versus time data for different loading levels are shown in Chapter 5.

Since these tests were conducted only for a short interval of time, change in strain with time was determined using the Findley's power law as described in Equation 6.3. To use Findley's power law, it was necessary to determine the constants m and n as seen in Equation 6.3. To determine these constants, a log-log plot was plotted with change in strain versus time. Figure 6.42 shows the log-log graph where the intercept at $y=0$ or $t=1$ hour gives the constant m and the slope of the curve gives the constant n .

Figure 6.42 log-log plots to evaluate Findley's constants m and n for different loading levels

These constants m and n for the four different loadings as calculated from Figure 6.42 are shown in Table 6.6. It can be seen that the value of m increases with increase in %loading, verifying the fact that m is a stress dependent constant. Also, n almost remains constant proving the fact that n is a material property and does not vary with stress. The initial creep strain like the constant m also increases with increase in the loading levels. The 80% loading resulted in tertiary creep faster than the time needed to monitor secondary creep trends. Since Findley's power law can only model secondary creep behavior of composites, the m and n values for the 80% loading were abrupt and are listed only for referencing.

Table 6.6 Findley's constant m and n as calculated from equation 6.4

% Loading of rupture	ϵ_0 ($\mu\epsilon$)	m ($\mu\epsilon$)	n
35	7171	230.14	0.209
50	14765	506.52	0.199
65	16640	708.27	0.176
80	25125	2349.09*	0.774*

* listed only for reference (inaccurate data)

The Findley's model generated by these constants (where, $n= 0.2$) was plotted along with the experimental creep data as shown in Figure 6.43. The Findley's power law model proved to be a good approximation of the creep behavior for the pultruded E-glass/Poly urethane composites at room temperature. Hence, Findley's power law can be successfully used to model creep for lower loading levels, since the tertiary creep stage approaches faster in case of high loading levels.

Figure 6.43 Verification of Findley's power law model on experimental creep strain data.

Figure 6.44 shows the creep strain data extrapolated over a service life of 50 years. As discussed previously, the 65% loading and 80% loading samples failed, but the Figure 6.44 still shows the creep strain over a 50 year life for 65% loading for reference purpose. Since 65% loading is on the higher end, the sample was not able to sustain the load for more than 92 days. Findley's law predicted the secondary creep well, but does not predict the tertiary creep. Hence, it does not determine in the time to failure which occurs in the tertiary creep region, but after the sample reaches this tertiary regime service life of the sample is very low. Hence, it has no considerable meaning in the design view point, therefore, this regime must be neglected. The 65% loading level in the Figure 6.44 is just for comparison with 35% and 50% loading. Table 6.7 shows the increase in strain over a service life of 50 years. It can be seen that the increase in strain was approximately constant for all the three loading levels. Table 6.8 shows the comparison between the predicted strains over a period of 50 years to the strain to failure during the short term testing. As can be seen in the Table 6.8, the strain for 35% and 50% approaches

approximately half of the strain to failure in the short term static tests. However, with higher stress levels the total strain can easily approach the strain to failure of the short term tests. These predictions are valid only for this particular material and to study other pultruded composites further research is needed.

Figure 6.44 Creep strain prediction using Findley's power law over a 50 year period

Table 6.7 Predictive increase in creep strain over a 50 year service life for different loading levels

loading	35%	50%	65% (failed in 92days)
Time (years)	$\frac{\varepsilon(t) - \varepsilon_0}{\varepsilon_0}$		
1	21.8	20.8	21.0
5	30.6	28.7	27.9
10	35.4	33.0	31.5
25	42.9	39.6	37.1
50	49.7	45.5	41.8

**Table 6.8 Comparison of creep strain over a 50 year period
with short term (static test) strain to failure**

	% loading	Strain($\mu\varepsilon$)
Average Short Term Strain to failure	N.A.	35516
Creep strain (50 years)	35	10738
	50	21483
	65 (failed in 92 days)	23607

6.3.2 Quarter Inch Thick Tri-axial pultruded samples

As discussed in the previous section for the half inch thick E-glass/PU samples, quarter inch thick samples were analyzed using Findley's power law for their creep responses. The details about the experiments and deflection versus time curves are shown in Chapter 5. These samples were also subjected to four different loading levels of 80%, 65%, 50% and 35%. Two of these tests were long term with low loading levels of 50% and 35% whereas the other two loading levels of 80% and 65% were short term creep tests.

The creep data from quarter inch thick samples were used to determine change in strain under constant loading by using Findley's power law. Figure 6.45 shows the log-log plot to evaluate the constants m and n as given in Equation 6.3. The vertical intercept at $y=0$ or $t=1$ hr gives the stress dependent constant m and the slope of the log-log plot gives the material constant n .

Figure 6.45 log-log plots to evaluate Findley's constants m and n for different loading levels

The constants m and n for all four loading levels calculated from Figure 6.45 are shown in Table 6.9. It can be seen that stress dependent constant m increased with increase in loading which was also observed in the half inch thick samples. Also, similar trend was observed for initial strain, which increased with increase in loading. The constant n which is material constant remains approximately constant, the value of n for quarter inch thick samples can also be compared to the value constant n from the half inch thick samples. It is observed the value of n remains almost constant for both half inch and quarter inch thick samples at all different loadings, confirming the fact that n is a material constant as the matrix used in both half and quarter inch thick samples was same, and as was discussed earlier that the creep is a property of matrix and that the creep due to fibers can be neglected.

The 80% loading in case of quarter inch thick samples shows abrupt values for constants m and n . As discussed for the half inch thick samples, the tertiary creep approaches faster at higher loading levels and Findley's law cannot determine the tertiary creep regime.

Table 6.9 Findley's constant m and n as calculated from equation 6.4

% Loading	ϵ_0 ($\mu\epsilon$)	m ($\mu\epsilon$)	n
35	10218	716.6	0.203
50	15468	841.9	0.197
65	18750	996.8	0.196
80	22125	1721.8*	0.295*

*listed only for reference (inaccurate data)

The Findley's model produced by these constants (where, $n= 0.2$) was plotted along with the experimental creep data as shown in figure 6.6. The Findley's power law model proved to be a good approximation of the creep behavior for these composites at room temperature.

Figure 6.46 Verification of Findley's power law model on actual creep strain data.

Hence, the Findley's law given in Equation 6.3 can be successfully used to estimate the creep strain over a possible service life of the material as used in constructions. Figure 6.47 shows the creep strain data extrapolated over a service life of 50 years for the quarter inch thick samples. Table 6.10 shows the increase in strain over a service life of 50 years. It was observed that the rate of increase in strain was high in case of 35% loading and almost similar for 50% and 65% loading. Table 6.11 shows the comparison between the predicted strains over a period of 50 years to the strain to failure during the short term testing. As can be seen in the Table 6.8, the total strain for 35% and 50% approaches approximately half of the strain to failure in the short term static tests. However, with higher stress levels the total strain can easily approach the strain to failure of the short term tests.

Figure 6.47 Creep strain prediction using Findley's power law over a 50 year period

Table 6.10 Increase in creep strain over a 50 year service life for different loading levels

	35% loading	50% loading	65% loading
Time (years)	$\frac{\epsilon(t) - \epsilon_0}{\epsilon_0}$		
1	44.2	32.5	31.5
5	61.4	44.6	43.2
10	70.6	51.2	49.5
25	85.1	61.3	59.2
50	97.9	70.3	67.8

**Table 6.11 Comparison of creep strain over a 50 year period
with short term (static test) strain to failure**

	% loading	Strain($\mu\epsilon$)
Average Short Term Strain to failure	N.A.	40029
Creep strain (50 years)	35	20229
	50	26348
	65	31465

CHAPTER 7

CONCLUSIONS AND RECOMMENDATIONS

In this study, three different composite materials were evaluated namely 1) pultruded unidirectional E-glass/Vinyl ester composite, 2) compressed Quasi-Isotropic E-glass/Vinyl ester composite and 3) pultruded tri-axial E-glass/Polyurethane composite. These composites were tested for their static properties (stress and stiffness) and also for their viscoelastic properties through Creep tests under constant load. Two models namely Accelerated Testing Methodology (ATM) and Findley's Power law model were used to determine the creep rupture and strain response. It was found that ATM was successful in predicting the creep rupture properties of a composite using the static tests data, while Findley's power law model was successful in predicting the secondary creep behavior and determining change of strain with time under constant load. The more specific conclusions as a result of present experimental and theoretical studies are stated in the sections below.

7.1 Unidirectional Pultruded E-glass/VE composites

These composites were evaluated for their static and creep properties. Three point bending, longitudinal compression, transverse compression and transverse tension static tests were conducted at five temperatures (20⁰C, 50⁰C, 80⁰C, 110⁰C and 140⁰C) with three humidity variations (dry, wet and 50%R.H.). Short term creep tests were conducted at 1) dry conditions at room temperature, 2) dry conditions at 80⁰C, 3) wet conditions at room temperature and 4) wet conditions at 80⁰C. The conclusions of these results are discussed in the following sections.

7.1.1 Static Properties

- A reduction of 45-55% in strength was observed as the temperature was increased from 20⁰C to 80⁰C for all tests which was relatively small as compared to 85-95% reduction from 80-140⁰C, this was due to the fact that the polymer matrix reached its T_g causing the matrix to soften.

- A reduction of 20-30% in strength was observed as the humidity was changed from dry to 50% R.H. condition at all temperatures; as the moisture results in the development of residual stresses and plasticizes the resin.
- A reduction of 30-40% in strength was observed as the humidity was changed from dry to wet conditions; the immersion of water might have caused redistribution of stresses between glass fibers and polymer matrix leading to decrease in strength.
- Effect of humidity on strength was more significant at lower temperatures.
- At higher temperatures, effect of temperature on strength was more significant than humidity.

7.1.2 Creep properties

- As the loading level decreases, the secondary creep phase increases.
- It was observed that at room temperature and dry conditions, these samples showed a threshold value of 50%, above which the tertiary creep phase approaches faster or narrows the secondary phase.
- Rate of creep increased rapidly with increase in moisture diffusion and at higher temperatures.
- At elevated temperatures, the pattern of response for creep essentially retained but the range of limited creep becomes narrower.
- At higher stresses the creep rupture is attributed to slip between the fiber and matrix.

7.2 Tri-axial Pultruded E-glass/Polyurethane composites

Half inch thick and quarter inch thick composite laminates with same fiber orientation were evaluated for their flexural properties and creep properties at ambient conditions. Creep tests were conducted at four different loading levels (80%, 65%, 50% and 35%) for both set of samples.

7.2.1 Static Properties

- The bending strength to failure for the half inch thick samples was 84.29 ksi and the modulus was 2.66 msi.
- The bending strength to failure of quarter inch thick samples was 58.5 ksi and the modulus was 1.73 msi.

7.2.2 Creep Properties

- Polyurethane samples performed better when compared to vinyl ester laminates. This was due to more ductility in case of polyurethane which provided a better stress distribution.
- The decrease in loading level increased the time to failure and the range of secondary creep phase.
- Higher loading of 80% was susceptible for creep rupture or failure.

7.3 Theoretical Models

Two models were used to study the creep properties. ATM (Miyano et al.) was used to determine the creep rupture properties of the Unidirectional E-glass/VE composites and Quasi-Isotropic E-glass/VE composite; whereas Findley's Power law model was used to determine the change strain with time.

7.3.1 Accelerated Testing Methodology (ATM)

- Accelerated Testing Methodology proved to be a good approximation to determine the time to failure or creep rupture time.
- ATM used the static tests to determine the creep rupture properties and hence preventing the need for cumbersome and time consuming creep tests.
- It closely predicted the time to failure at dry conditions at both room temperature and 80⁰C. It was verified using the experimental creep to failure time.
- ATM showed some errors for wet conditions both at room temperature and 80⁰C, which was attributed to the fact that the static tests were conditioned only for 24 hours, hence, not allowing 100% sorption of moisture.

Findley's Power Law

- Findley's Power law was successful in determining the strains at secondary creep phase and was verified using the experimental creep data.
- The value of constant n was measured to be around 0.20 for both thickness samples and it was verified that n is a material constant and does not depend on stress.
- The constant m increased with increase in the loading level, hence, proving that m is a stress dependent constant.
- 35% and 50% of both the samples reached strains which was almost half of the short term strain to failure in 50 years as calculated by the model.

7.4 Recommendations

- Mechanical properties of FRP composites depend on fiber-matrix adhesion and fiber content. Hence, an attempt should be made to increase the fiber matrix adhesion using SEM to determine the adhesion.
- For using Miyano's ATM approach the coupons must have approx 100% absorption to predict the creep rupture accurately for long term creep tests under wet conditions.
- Elevated temperatures should be used for long term creep tests and Findley's constant should be determined to verify the temperature dependent nature of constant m .
- The moisture and temperature effects should be studied on a creep test as a combination which is seen in a service life of a structure.
- Creep in polyurethanes composite was much less when compared to vinyl ester composite; hence, the effect of temperature and moisture should be studied on polyurethane composites.

APPENDIX A

MODULUS FOR SHORT TERM TESTS ON UNIDIRECTIONAL E-GLASS/VE COMPOSITES

Test	Conditioning	Temperature (°C)	Average Modulus (msi)
Transverse Tension	Dry	20	2.09
		50	1.357
		80	0.961
		110	0.176
	50% R.H.	20	1.599
		50	1.17
		80	1.05
		110	0.082
	Wet	20	1.509
		50	1.338
		80	0.996
		110	0.544

Test	Conditioning	Temperature (°C)	Average Modulus (msi)
Longitudinal Compression	Dry	20	6.450
		50	4.722
		80	4.866
		110	4.818
	50% R.H.	20	6.676
		50	5.402
		80	6.876
		110	5.524
	Wet	20	6.162
		50	7.663
		80	6.508

Test	Conditioning	Temperature (°C)	Average Modulus (msi)
Transverse Compression	Dry	20	1.445
		50	1.077
		80	0.891
		110	0.250
	50% R.H.	20	1.303
		50	0.718
		80	0.661
		110	1.581
	Wet	20	1.729
		50	0.650
		80	0.463
		110	0.053

Test	Conditioning	Temperature (°C)	Average Modulus (msi)
Three Point Bending	Dry	20	6.45
		50	6.17
		80	6.59
		110	4.99
		140	2.57
	50% R.H.	20	6.07
		50	6.13
		80	6.27
		110	5.16
		140	2.14
	Wet	20	6.00
		50	6.04
		80	5.32
		110	2.32
		140	1.47

APPENDIX B

TENSILE CREEP TESTS CONDUCTED ON UNIDIRECTION E-GLASS/VE COMPOSITES

Specimen dimensions: 0.75 inch width x 0.135 inch thickness x 12 inch length

Trial No. 1: Specimen was first wrapped with 3 layers of biaxial fabric on each end of the test specimen using vinyl ester and then bonded with metal end tabs after full cure. Two holes were drilled across the entire stack (end tab, wrap, specimen, wrap and end tab) in order to attach to the loading frame via two bolts. The load was applied through Dywidag rod. The max load was around 2000 lb and the failure occurred at the wrap bond line and also through shear out of unidirectional laminate.

Specimen and load frame

**Specimen and load frame installed
between I-beam**

Load is being applied

Showing shear out failure

Trial No. 2: Specimen was bonded with two metal end tabs and then attached to the loading frame via two bolts on far end of metal tabs (no holes on the test specimen). The load was applied through Dywidag rod. The max load was noted to be ~3200 lb and the failure occurred with the test specimen fiber peeling.

Trial No. 3: Specimen was first wrapped with 3 layers of biaxial fabric on each end of the test specimen using epoxy and no metal end tabs were bonded. After full cure, two holes were drilled across the specimen in order to attach to the loading frame. The load was applied thru Dywidag rod. The max load was noted to be ~2000 lb and the failure occurred with shear out of unidirectional laminate.

Trial No. 4: Specimen was bonded with two metal end tabs and then attached to the loading frame via two bolts on far end of metal tabs (no holes on the test specimen). The load was applied thru Dywidag bar. The max load was noted to be ~5700 lb and the failure occurred with fiber peeling from specimen.

Trial No. 5: Specimen was first wrapped with 6 layers of biaxial fabric on each end of the specimen using epoxy resin before placing metal end tabs and then attached to the loading frame through metal tabs via two bolts (no holes on the test specimen). The load was applied thru Dywidag rod. The max load was noted to be ~2000 lb and the failure occurred with end tab bond line. A crack was noted that was created while drilling holes.

Trial No. 6: Specimen was first wrapped with 6 layers of biaxial fabric using epoxy resin before placing metal end tabs and then two holes were drilled across the entire stack (end tab, wrap, specimen, wrap and end tab) in order to attach to the loading frame via two bolts. The load was applied through Dywidag rod. The max load was ~5200 lb and the failure occurred with wrap bond line and shear out of the unidirectional laminate specimen.

Trial No. 7: Specimen was bonded with two metal end tabs and then attached to the loading frame via two bolts on far end of metal tabs (no holes on specimen). The load was applied through hydraulic ram. The max load was noted to be ~3300 lb and cohesive failure occurred with test specimen fiber peeling.

Horizontal placement

Hydraulic ram is used to apply tension to avoid torsion while loading.

Close view of failure occurring within minutes at 3500 lbs

Cohesive bond failure of a specimen

APPENDIX C

MIYANO'S ATM APPROACH TO PREPARE TENSILE CREEP MASTER CURVES FOR QUASI-ISOTROPIC SAMPLES WITH HOLES

Relationship between log (tensile strength) and logarithmic of real time to failure under dry conditions at a temperature range of 20-110⁰C for Quasi-Isotropic samples with holes

As discussed in section 6.2.1 about flexural creep master curves in dry condition, a similar study was done to prepare tensile creep master curve at room temperature. To prepare these master curves, tension tests were done at various temperatures (20-110⁰C). To prepare a creep master curve at dry conditions for tension tests, tensile CSR strength was plotted with time over a range of 20-110⁰C for dry conditions. These points were shifted using shift factors as given in Table 6.1 both horizontally and vertically to obtain a tensile CSR strength master curve at dry conditions with reduced time. The slope of the curve was found to be 0.109 and the shift factor used to convert CSR master curve to

tensile creep strength master curve by shifting the CSR master curve horizontally was found to be 1.04. The creep master curve was verified using experimental creep time to failure, section 5.3.2 shows the result for the two creep tests.

Master curve of tensile CSR strength with reduced time at RT and dry conditions

Verification of tensile creep strength master curve using experimental creep time to failure at 20⁰C and dry conditions

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