Investigation of Vartm Processing of High Temperature RP-46 Resin System

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INVESTIGATION OF VARTM PROCESSING OF HIGH TEMPERATURE RP-46 RESIN SYSTEM

By

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# TABLE OF CONTENTS

LIST OF TABLES ...........................................................................................................vii
LIST OF FIGURES ........................................................................................................ix
ABSTRACT ......................................................................................................................xiii

## 1 INTRODUCTION ......................................................................................................1
1.1 Problem Statement ..............................................................................................4
1.2 Objectives ........................................................................................................... 7
1.3 Procedure for carrying out the research work ............................................... 9
1.4 Thesis document outline ..................................................................................11

## 2 LITERATURE REVIEW .......................................................................................12
2.1 Literature on Vacuum Assisted Resin Transfer Molding Process .............12
2.2 Issues of Vacuum Assisted Resin Transfer Molding Process .................13
   2.2.1 Dimensional Stability ..............................................................................15
   2.2.2 Voids, Dry-spots and Fiber volume fraction ......................................18
   2.2.3 Surface Quality .....................................................................................20
2.3 Applications of High Temperature Polymer Matrix Composites ..........21
2.4 High Temperature Polymer Matrix Composite Resin Systems ..........27
2.5 Larc™ RP-46 Polyimide ..................................................................................29

## 3 MATERIAL SELECTION ....................................................................................33
3.1 Benchmarking of candidate resin systems ..................................................33
3.2 Resin selection ..................................................................................................37
3.3 Fiber selection ..................................................................................................38
EXPERIMENTAL SETUP AND METHODOLOGY

4.1 Experimental Setup

4.1.1 Vacuum bagging supplies

4.1.2 Heating devices

4.1.3 Miscellaneous supplies

4.2 Manufacturing of the test specimens

4.2.1 Experimental test matrix

4.3 Tool and vacuum bag preparation

4.4 Infusion process

4.5 Cure and post cure cycle

4.6 Summary of operating parameters for VARTM

4.7 VARTM processing data analysis

TEST RESULTS AND ANALYSIS

5.1 Thermal analysis

5.1.1 Dynamic Mechanical Analysis

5.1.1.1 IM7/RP46 baseline Composite

5.1.1.2 VARTM samples

5.1.1.3 Comparison of DMA results

5.1.2 Thermal Mechanical Analysis

5.1.2.1 Neat resin

5.1.2.2 IM7/RP46 baseline Composite

5.1.2.3 VARTM samples

5.1.2.4 Comparison of TMA results
5.1.3 Thermal Gravimetric Analysis.

5.1.3.1 Neat resin

5.1.3.2 IM7/RP46 baseline Composite

5.1.3.3 VARTM samples

5.1.3.4 Comparison of TGA results

5.2 Mechanical Property Testing

5.2.1 Specimen preparation and setup

5.2.2 Tensile Strength

5.2.3 Tensile Modulus

5.2.4 Benchmarking of tensile test results

6 CONCLUSION

6.1 Summary

6.2 Future work

APPENDIX A

REFERENCES

BIOGRAPHICAL SKETCH
LIST OF TABLES

2.1 Cure cycle definitions 16
3.1 Comparison of promising resins 38
4.1 Experimental test matrix for RP-46 processing using VARTM 45
4.2 Viscosity of RP-46 resin when heated to different temperatures 52
4.3 Operating parameter during tool and vacuum bag preparation 60
4.4 Operating parameter during resin infusion process 60
4.5 VARTM processing data 61
4.6 Analysis of vacuum pressure during infusion on $W_f$ and $V_f$ 62
4.7 Infusion times per sample 62
5.1 DMA test results for VARTM flat panel parts 71
5.2 DMA results divided by infusion method 72
5.3 CTE for RP-46 neat resin at different temperatures 78
5.4 CTE for IM7/RP-46 baseline composite at different temperatures 79
5.5 TMA test results for VARTM flat panel parts 80
5.6 TMA results divided by infusion method 81
5.7 TGA test results for VARTM flat panel parts 86
5.8 TGA results divided by infusion method 87
5.9 Tensile test data for eleven laminates 90
<table>
<thead>
<tr>
<th>Section</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.10</td>
<td>Tensile strength data divided by infusion method</td>
<td>92</td>
</tr>
<tr>
<td>5.11</td>
<td>Tensile modulus for eleven laminates</td>
<td>93</td>
</tr>
<tr>
<td>5.12</td>
<td>Tensile modulus data divided by infusion method</td>
<td>95</td>
</tr>
<tr>
<td>5.13</td>
<td>Tensile strength comparison</td>
<td>96</td>
</tr>
</tbody>
</table>
LIST OF FIGURES

1.1 Turbine vane and exhaust nozzle, Integrated High Performance Turbine Engine Technology 3
1.2 Setup of VARTM process 6
1.3 Composite cowl doors for an RB211-524 (Rolls Royce) 8
1.4 Thesis documentation outline 11
2.1 Future of the composites industry 13
2.2 Schematic of VARTM setup 16
2.3 Part measurement points and fiber volume fractions with measured locations (1-3) 17
2.4 Average fiber volume fractions 17
2.5 Plots of unfilled volume fraction versus induction coil voltage for the coil located at (a) 15.24 cm and (b) 5.08 cm from the inlet at a vacuum level of -77 kPa 20
2.6 Composite structural applications in military aircrafts 22
2.7 F-22 Raptor by Lockheed Martin Corporation 23
2.8 Cross beam integral structure (one quadrant) fabricated by VARTM at MSO, Lockheed Martin using BMI resin 24
2.9 Compression strength vs Temperature for VARTMed Hexcel 650 and 651, wet and dry 25
2.10 NASA GLENN Research center, Cleveland, OH 26
2.11 GE–90 HP cooling tube (PMR-15) 27
2.12 Imidization and cross-linking of polyimides 29
4.1 Stages of VARTM processing of RP-46 resin 42
4.2 Initial VARTM sample 43
4.3 Good quality VARTM sample

4.4 Schematic representation of tool and vacuum bag preparation stage

4.5 Vacuum bag preparation

4.6 Tool and vacuum bag setup

4.7 Laminates of glass and EPON 862 epoxy resin

4.8 Resin infusion stage

4.9 Schematic diagram of the infusion stage

4.10 Badly cured flat panel

4.11 Low temperature cure cycle for VARTM processing of RP-46 resin

4.12 DSC curve for the neat resin

4.13 Flat panel made using the lower temperature cure cycle

4.14 Schematic representation of cure and post-cure cycle

5.1 DMA test on the baseline composite (IM7/RP46)

5.2 DMA test results for sample RS #1a and RS #1b

5.3 DMA test results for sample RS #2a and RS #2b

5.4 DMA test results for sample RS #3a and RS #3b

5.5 DMA test results for sample RS #4a and RS #4b

5.6 DMA test results for sample RS #5a and RS #5b

5.7 DMA test results for sample RS #6a and RS #6b

5.8 DMA test results for sample RS #7a and RS #7b

5.9 DMA test results for sample RS #8a and RS #8b
5.10 DMA test results for sample RS #9a and RS #9b
5.11 DMA test results for sample RS #10a and RS #10b
5.12 DMA test results for sample RS #11a and RS #11b
5.13 Part-to-part and sample-to-sample variations in storage modulus at room temperature
5.14 Part-to-part and sample-to-sample variation in storage modulus at 300°C
5.15 eSEM images of samples RS #1, RS #4 and RS #11
5.16 TMA results for RP-46 neat resin
5.17 TMA results for IM7/RP-46 baseline composite
5.18 CTE of the VARTM samples
5.19 Comparison of CTE
5.20 TGA test results for neat resin
5.21 TGA test results for IM7/RP-46 samples
5.22 Weight loss for VARTM samples
5.23 Comparison of weight loss profiles
5.24 Example of tensile test samples
5.25 MTS tensile testing equipment
5.26 Comparison of tensile strength
5.27 Comparison of tensile modulus
5.28 Comparison of tensile strength and modulus
6.1 Overall comparisons of test results
A.1 Wrightlease 5900 T.O.S (WL5900 TOSC)
| A.2 | Teflon-High permeable layer (HPL) | 101 |
| A.3 | Wrightlease 5900 (WL5900) | 102 |
| A.4 | Bleeder lease E | 102 |
| A.5 | Vacuum bag sealant tape (A-800-3G) | 103 |
| A.6 | Pressure sensitive tape | 103 |
| A.7 | Aluminum mold plate | 104 |
| A.8 | Vulcan programmable oven | 104 |
| A.9 | Hot plate | 105 |
| A.10 | Valves, hoses and connectors | 105 |
| A.11 | Pressure gauge | 106 |
| A.12 | Vacuum pump | 106 |
| A.13 | Resin collection area | 107 |
| A.14 | Temperature measuring laser gun | 107 |
| A.15 | Fiber cutter | 108 |
| A.16 | Operator safety equipment | 108 |
ABSTRACT

The advantages of using polymer matrix composites in various applications are very well known throughout many industries. Their introduction and subsequent development since the 1940’s has led to major cost savings due to their lightweight and excellent mechanical properties. Off late, product designers have been taking advantage of improved thermal properties (CTE, thermo-oxidative stability) that these composite materials have to offer. This began with the development of PMR-15, a high temperature polyimide resin back in the 1970’s. The aerospace industry has increasingly turned towards high temperature polymer matrix composites (HTPMC) to replace other heavier materials in engine components thus improving the thrust to weight of the engine. But, PMR-15 has a major drawback related to high safety standards that are needed during processing. The implementation of these controls during processing resulted in huge costs to the industry. This led to the development of a new polyimide high temperature resin system called RP-46 at NASA Langley research center. RP-46 has excellent thermal and mechanical properties comparable to PMR-15 and is safer to handle due to the absence of the lethal MDA monomer, a carcinogen.

This research investigates the issues related to processing of RP-46 resin system using the Vacuum Assisted Resin Transfer Molding (VARTM) process, a cost effective method for manufacturing composite materials. The entire process is setup keeping in consideration the requirement of high temperature environments for processing of RP-46. A number of initial trials helped understand the dynamics of the process and identify critical factors and key parameters. The various laminates that were made were tested for mechanical properties (ASTM D3039 - Tensile strength and modulus) and thermal properties (Dynamic Mechanical Analysis, Thermal Mechanical Analysis, Thermal Gravimetric Analysis) were performed and the results were compared with RP-46 samples made using autoclave processing. Although the VARTM laminates had issues related to void contents and the release of volatiles
during the infusion stage of the process, the VARTM process was found to be feasible to make composites with RP-46.
CHAPTER 1

INTRODUCTION

Since the 1940’s, composite materials have been popular in many areas of aerospace, military and marine industry. These materials can be used as an excellent alternative to the heavy and expensive metals like steel, aluminum and titanium. The past thirty years have introduced structural designers, automakers, aerospace component designers and many others to the world of advanced materials that are lighter, tougher and stronger than traditional materials. This on going revolution in material capabilities has provided a further thrust to discover better materials. A wide variety of them are now available which have excellent properties.

A fiber reinforced composite material is a union of fibers and resin. The fibers have excellent mechanical and the thermal properties but need some mechanism which will enable them to adhere together as one object during exposure to loads [1]. The resin, which is also known as the matrix, provides this mechanism. The fiber is the primary load-carrying member and the matrix keeps the fibers in the location and orientation and acts as the load transfer medium.

Composite materials can be thought of as the adhesion of two raw materials where the whole is greater than the sum. This means that both fibers and matrix retain their physical and chemical identities and yet they produce a combination of properties that cannot be achieved with either of the constituents acting alone. Since, they have been around for quite sometime now, their usage in the industry has been quite widespread.
The winning combination that propels composite materials to new areas is its high strength and lightweight. But, other properties are also gaining importance. Some of them include low coefficient of thermal expansion (CTE) and good vibrational damping. Composite materials provide design flexibility that can significantly decrease the number of parts needed by eliminating a number of fasteners, joints, raw materials and subsequently lesser assembly time [2].

Another advantage of composites is proven resistance to temperature extremes, corrosion and wear, especially in industrial settings. This characteristic can lead to lower product lifecycle costs. In structural aerospace applications, the advantages of adaptable fiber orientation are many. So is the case with consumer products. A composite golf club shafts, can withstand multiple flex, torque, compression and tension forces due to the strength and stiffness properties of boron and carbon fibers.

One of the areas where composite materials are increasingly being used as replacements for conventional materials is in high temperature applications. Ceramic Matrix Composites (CMC) and Metal Matrix Composites (MMC) have become popular in applications that require high-temperature resistance. Their high costs of fabrication have limited their usage. These materials require high temperature cure of 1832° F (1000° C) and special high temperature resistant equipment is needed for fabricating them. The cost of fibers is prohibitive as well. The commonly used glass and carbon fibers cannot be used since their mechanical and thermal properties are not suited for extreme high-temperatures. Silicon carbide is the most economical fiber for the CMC, which costs much more than normal carbon fibers (~ $1250/ Kg). These advanced composites are used primarily in aero-engine components that need materials that can withstand close to 1500° F (815° C) to 2000° F (1093° C) [3].

Intensive research in industry and academia on CMC and MMC systems through the 1980s, supported by major government funded research programs such as “Integrated High Performance Turbine Engine Technology” (IHPTET) in the US, delivered the first MMC and CMC components into demonstrator and development
engines. Figure 1.1 shows aircraft engine components that are made of CMC and MMC.

![Figure 1.1 Turbine vane and exhaust nozzle, Integrated High Performance Turbine Engine Technology (IHPTET)](image)

This research, however, focuses on the needs of the military and the aerospace industry in connection with the use of high temperature polymer matrix composites (HTPMC) for structural and aero-engine components. These HTPMCs were used to harness the potential cost savings and superior specific strength and stiffness. The challenge to realize applications of the advanced composite systems has increased in recent years by the sharper focus on cost reduction. Complete airframes can now be produced in polymer matrices, and they are essential to modern helicopters.
Temperature requirements limit their use in aero-engines, but most of the nacelle of a modern aero gas turbine is a HTPMC, a component that accounts for around 25% of the weight and 20% of the cost of the power plant [4]. Other HTPMC parts in current engines include fan blades, outlet guide vanes, bypass ducts, nose cone spinners, core engine fairings, annulus fillers and variable guide vane rings.

HTPMCs offer great potential to reduce the cost and weight of military turbine engine and airframe components. State-of-the-art HTPMCs, however, lack the durability to meet critical system lifetime performance requirements. Their use has also been limited by their dependence on expensive manufacturing methods [5]. Another factor limiting their widespread use is the availability of resin systems that can satisfy the performance requirements. The fabrication of parts using such resin systems results in lengthy manufacturing processes caused by expensive tooling materials and a need for careful discharge of by-products and solvents.

1.1 Problem statement

In most of the processes employed in major sectors of industry, including material production and processing, power generation, aerospace and military, efficiency increases with increase in process temperature. Hence, in order for the process to be efficient and viable, high temperatures are a necessity. Therefore, it is imperative to develop materials capable of withstanding mechanical, thermal and environmental conditions during high temperature operations [6].

The operating temperature and the duration of the exposure to high temperatures varies widely depending on the function of the part. For example, some components in rockets are subjected to minutes of this temperature range, while certain others have to withstand 100,000 hours, such as in power generating plants. In general, it is a known fact that these high temperature systems have to resist the mechanical and corrosive conditions imposed by the operating environment.

Apart from the need for materials to operate at high temperature there are also requirements of these materials to be low-cost and lightweight. There has been, in the
recent past, a lot of research work with focus on making the fabrication of lightweight high-temperature composite materials more affordable. Although many more materials are available now than a decade ago, the amount of testing and characterization of these materials has been slow. The potential of these advanced composite materials in high temperature applications is still to be completely fathomed. Some of the areas where these materials will play pivotal role are – improving the performance of future military aircraft that require jet engines to have a higher thrust to weight ratio, materials for radiation-hardened electronics and sensors for working at extreme environments on planets, lightweight and affordable propulsion system components and high-performance airframe materials and structures.

High-temperature materials can be defined in several ways, all of which are somewhat arbitrary. Most of the definitions are application based. In this research work, HTPMCs are taken to be those materials that are used specifically for their heat-resisting capabilities and strength at temperatures above 600°F (316°C). There are essentially two categories of HTPMCs based on the glass transition temperature ($T_g$), which is an important material parameter for classification and analysis. The $T_g$ maybe defined as the temperature at which an amorphous polymer changes from a glass into a rubber [7]. Choosing $T_g$ as criterion, the various polymer resin systems are classified as follows.

- 300°F (149°C) to 600°F (316°C) – Materials with $T_g$’s in this range are considered to be suitable for medium temperature applications such as airframes and engine supports
- 600°F (316°C) + – Materials with $T_g$’s in this range can be used to make parts such as guide vanes and fan blades which are subjected to higher temperatures due to their proximity to the core engine systems.
Since, the main purpose of this study is to manufacture high-temperature composite parts at affordable costs, a number of manufacturing processes were considered for the manufacture of high-temperature composite materials. One process, which is known as much for its cost effectiveness as for the quality of parts produced, is the Vacuum Assisted Resin Transfer Molding (VARTM) using the vacuum bag and a resin distribution medium; henceforth, referred to as VARTM.

VARTM is a technique that has drawn more attention in the last decade because the manufacturing costs associated with this process are significantly lower than the traditional pre-preg processes. In fact, VARTM technology requires less expensive equipment and operating conditions while still allowing the manufacture of complex 3-D shaped parts [8,9]. The setup for this process is shown in Figure 1.2.

![Figure 1.2 Setup of VARTM process](image)

In 1970, the NASA Lewis Research Center developed the PMR-15 processable, high temperature polymer resin system for aero-propulsion applications. The use of polymer matrix composites have led to substantial weight savings, thereby leading to improved fuel economy, increased passenger and payload capacity and better maneuverability.
Since the development of PMR-15, a number of polymers have been developed that can be used in the high temperature applications. The main classes of polymers for high temperature applications are – Polyimides, Bis-maleimide (BMI - A type of polyimide), Poly-cyanate and Phenols.

The emphasis in this research work is on Larc RP-46 resin system. This resin was developed as a superior replacement for the existing polyimide resin systems. It has the ability withstand extreme temperature conditions that make it an ideal choice for aerospace, defense, ballistics applications as well as commercial applications, which require extreme heat resistance.

1.2 Objectives

The objective of this research work is to primarily investigate and evaluate the producibility of parts using the RP-46 resin system with the VARTM process. It will also benchmark the RP-46 based PMCs fabricated by VARTM with those fabricated by other processes.

1.2.1 Processability

This focuses on the VARTM process and the ability to produce high quality parts repeatedly. In order to accomplish this, the following issues need to be addressed.

- Attaining reliable and robust bagging and sealing, which is crucial for success and low production costs with VARTM [10]. Since resin is under vacuum during infusion and cure, any air leaking into the resin expands, with serious consequences for part quality.
- Attaining negligible void content – Voids present in the laminate affects the mechanical and thermal properties of the part.
- Attaining good fiber volume fractions – The fiber volume fractions obtained by VARTM is not as high as the autoclave cured laminates.
- Incorporation of a heating mechanism for the setup during infusion, cure and post-cure stage
1.2.2 Performance
The composite part should be able to withstand high temperature service environments (over 600°F) that components of the aero engines are exposed to without any significant changes to their properties. The structure needs to meet or exceed current requirements for strength and stiffness. All these have to be accomplished while keeping the costs at an affordable level.

1.2.3 Benchmarking
After fabrication, the quality of part is measured by comparing it with parts fabricated using the autoclave process. Mechanical, and thermal properties shall be compared.

![Figure 1.3 Composite cowl doors for an RB211-524 (Rolls Royce)](image)

Applications of HTPMCs are uniquely challenging in that they require flexible, low-cost processability of complex parts and materials must demonstrate environmental durability. This study aims to provide a better understanding of the
VARTM process for RP-46 fabrications and the critical detail manufacturing for the process.

1.3 Procedure

1.3.1 Material selection
This research work focuses on low cost manufacturing methods that will significantly reduce the production cost of the components that make use of the RP-46 based resin system.

RP-46 is chosen as the resin system for the high temperature fabrication of aerospace components using the VARTM process. The major factor for this selection is that it has a very high glass transition temperature ($T_g$) compared to the other resin systems such as HEXCEL – 650 (BMI), RS – 9 (Poly-cyanate), CYCOM 520 – 4 RTM (BMI). The carbon fiber is selected for sample parts since they have excellent high temperature capabilities.

1.3.2 Benchmarking of HTPMCs
There are a number of materials that are at the disposal of the researcher depending on the application. However, not all of them can meet the critical requirements of low viscosity (below 1000cp, several hours working time), out-of-autoclave processing and high temperature capabilities. Hence, we look at all the candidate systems that are compatible with VARTM and compare a few system characteristics and properties such as - curing requirements, viscosities, cost, mechanical, thermal, coefficient of thermal expansion, storage and handling.

1.3.3 Selection of the part to be fabricated
This thesis research work will be carried out by initially fabricating flat panels. This stage of the research involves conducting the initial fabrication on the simple shapes, which enables us to study the various issues related to VARTM processing of high
temperature composite materials. These studies will help in transitioning into manufacturing of other shapes such as composite fan blades etc.

1.3.4 Process setup and fabrication of the demonstration part
The steps in fabrication of the parts include tooling setup, lay-up, pre-forming, resin infusion, cure and post-cure. A short series of experiments are to be conducted to exercise the fabrication method, develop an understanding of its behavior.

The test apparatus for the VARTM process is a setup and a number of points related to the process are to be addressed. Some of them are heating of the mold plate for maintaining high temperature across the cross section of the mold and heating of the resin amongst others.

1.3.5 Mechanical testing and thermal studies
The part has to demonstrate high temperature capabilities, good repeatability of responses such as tensile strength and modulus, and environmental compatibility. Dynamic mechanical analysis (DMA) and differential scanning calorimetric (DSC) analysis are performed to study the $T_g$. TGA and TMA are performed as well to understand the weight loss or thermo-oxidative stability and coefficient of thermal expansion of the part.

1.3.6 Evaluation and analysis
In this master’s thesis work, the accomplishment of fabrication of RP-46 based PMC parts using the VARTM process is the main goal. After manufacturing of the flat panels made of RP-46, each of the laminates is studied for fiber volume fraction ($V_f$), weight fraction ($W_f$) and thickness variation analysis. The microstructure characterization of the laminates produced under VARTM is carried out to visually analyze the presence of voids, delaminated layers and other forming discrepancies.
1.4 Thesis document outline

This thesis focuses on the producibility of composite parts using VARTM process that can withstand high temperatures. Chapter 2 looks into the literature review of the intended research work. Chapter 3 reviews the material selection for the resin system and fibers. This chapter determines the viable materials for VARTM processing at high temperature. Chapter 4 discusses the various materials that are used for the final process setup. This chapter elaborates on the different sections of VARTM processing. Chapter 5 reports on the results from the various experiments that are carried out. The conclusions of the research and the proposed future work are discussed in Chapter 7.

![Diagram of thesis structure]

**Figure 1.4** Thesis documentation outline
CHAPTER 2

LITERATURE REVIEW

This chapter discusses the work done previously in VARTM and HTPMCs. It includes sections that review issues with VARTM processing, HTPMC resin systems and applications. It also presents an in depth view on RP-46 structure and processing of polyimides.

2.1 Literature on Vacuum Assisted Resin Transfer Molding (VARTM)

The VARTM process dates back to 1989 when the process was used as a lower cost alternative to autoclaving. Ever since a version of VARTM has been patented by Seeman Composites, whose process came to be called as Seeman Composite Resin Infusion Molding Process (SCRIMP), the composite manufacturing industry has tried hard to incorporate this version of VARTM process for the manufacture of a variety of composite structures. SCRIMP differed from traditional VARTM in that it made use of a distribution medium, which enabled the vacuum within the mold to be used to draw resin into the mold cavity and wet the fibers. SCRIMP was the first process that enabled the use of a distribution media thus saving labor, time and producing better quality of the parts [9].

The VARTM process began as a low cost process, which was primarily catered to the marine industry. Over the years, VARTM has been considered as an efficient manufacturing process. In keeping with this trend of utilizing low cost manufacturing methods for high quality parts, the composite industry’s vision is as shown in Figure 2.1. According to the figure, the marine industry has been using traditional manufacturing techniques like VARTM that is low cost and not very high
quality. But, the aerospace industry requires higher quality parts, which require higher
temperature and pressures to process that results in increasing the cost of producing
the part. In the last few years, the combining of these technologies have occurred due
to considerable progress made in the development of newer materials for the
processes, like the resin systems which previously needed high temperature and
pressure to cure thereby increasing the cost.

![Figure 2.1 Future of the composites industry](image)

As a result of this merging, VARTM is increasingly being used or
experimented with, in the aerospace industry. The following section discusses the
various issues and solutions for VARTM processing.

### 2.2 Issues of VARTM processing

The biggest disadvantage of the VARTM process is low fiber volume fraction when
compared to autoclave processing [8]. This can be attributed to the resin infusion
process, which occurs under vacuum pressure. There are essentially three stages to
fabricate a part using the VARTM process.
1. Experimental setup – mold, vacuum bag and resin preparation
2. Mold filling and fabric wetting by the resin
3. Curing and post curing cycle

Each step in the process directly or indirectly affects the final quality of the part. The experimental setup directly impacts the resin infusion stage, which directly affects the mechanical properties of the composite. The curing and post curing stages control the thermal properties of the part. Darcy’s law provides a mathematical equation to describe the flow of resin during the mold filling stage.

\[ q = -\frac{\rho_o}{\eta} \left( \frac{dp}{dx} \right) \quad \text{Eq - (2.1)} \]

Where, \( q \) = Volumetric flow rate per unit area (m/s) in the x direction
\( \rho_o \) = Permeability (m²)
\( \eta \) = Viscosity (N-s/m²)

\( \left( \frac{dp}{dx} \right) \) = Pressure gradient (N/m³), which is negative in the direction of flow (positive x direction)

It states that the flow velocity is related to the local pressure gradient, the fluid viscosity, and the permeability of the porous medium [10], where the permeability is a measure of the resistance to the flow provided by the preform.

The fiber volume fraction (\( V_f \)) indicates the quality of the part. An optimum \( V_f \) can be attained by having an ideal inlet pressure for the resin, an appropriate setup, resin with viscosity less than 1000 cps, presence of a good resin distribution media (RDM) and good permeability of the preform.

There are a variety of new VARTM supplies that are available, which have improved the mold filling exercise. A number of companies are in the market of making resin distribution mediums that aid in the proper wetting of the fibers. Heating blankets are available that can heat the bag and higher temperatures within
the bag will enable the viscosity to remain at a lower level thereby ensuring smooth flow and prevent the resin from reaching the gel point. Heated lines can be incorporated that maintain the temperature of the system when the fluid travels through the hose. In addition to this, material scientists and engineers have continuously been developing resin systems that can be used with low cost manufacturing methods like VARTM.

2.2.1 Dimensional stability of the laminate

This research focuses on the feasibility of producing aerospace quality parts and a critical issue that affects the ultimate consideration of composites in these applications is the dimensional tolerance of the part. The challenge facing the composites industry today that could determine how widespread the use of low cost methods of manufacturing can become for high temperature applications is the control of part dimensions and an accurate prediction of part-to-part variation. This control can be achieved during the processing cycle-tool preparation, infusion and curing (and post-curing). A good understanding of the factors and the process parameters will help in reducing the dimensional variability between the parts. The part complexity will impact the control of dimension in a big way.

The part infiltration stage or the mold filling stage of the composite processing cycle for VARTM has been the subject of a large number of studies. And these studies have enabled the development of comprehensive analytical and numerical tools for prediction of flow under the bag, part thickness variation and fiber volume fractions. However, the effect of curing on the dimensional stability has not been a discussion of many studies. It is during this stage of processing that residual stresses are induced causing part distortions.

Hubert et al [11] studied the dimensional stability of carbon fiber SAERTEX fabric/SI-ZG-5A epoxy resin C-shaped laminates manufactured by the VARTM process using different cure cycles followed by a similar post-cure cycle. These cure cycles enabled the identification of critical parameters for the VARTM process. Table
2.1 shows the cure cycles used. Cycle B was low temperature cure and Cycle C was a rapid one step cycle with a faster heating rate. Cycle A was the manufacture recommended cycle. Figure 2.3 describes the schematic setup of the process.

**Table 2.1 Cure cycle definitions**

<table>
<thead>
<tr>
<th>Cycle</th>
<th>Definition</th>
</tr>
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</table>
| A     | Heat to 66°C @ 0.56°C/min  
       | Hold 2.5 hours  
       | Heat to 125°C @ 0.56°C/min  
       | Hold 2.5 hours  
       | Cool to RT |
| B     | Heat to 66°C @ 0.56°C/min  
       | Hold 13 hours  
       | Cool to RT |
| C     | Heat to 125°C @ 1.1°C/min  
       | Hold 2.5 hours  
       | Cool to RT |

**Figure 2.2** Schematic of VARTM setup
The results from the processing and subsequent curing of the parts under three different cure cycles indicated that the spring-in was the largest for parts from cycle C and A followed by low temperature cure cycle B. The average fiber volume fraction was 46.9%, 48.0% and 46.5% for parts A, B and C respectively. Part B had the most uniform fiber volume fraction distribution (Figure 2.3 and 2.4) compared to A and C. Part B was also thicker than A and C.

The low spring-in from cycle B can be attributed to the low temperatures change than cycles A and C. The residual stresses caused by the difference between longitudinal and transverse CTEs are greater for parts A and C. However, the $T_g$ for the part from cure-cycle B was the lowest.

![Figure 2.3](image)

**Figure 2.3** Part measurement points and fiber volume fractions with measured locations (1-3)

![Figure 2.4](image)

**Figure 2.4** Average fiber volume fractions
This work highlights the need for understanding the critical curing procedures for processing the composite parts and the identifying optimum parameters to minimize the residual stresses that cause distortions. Hubert proposed the low temperature curing cycle B since it produced a part with the least amount of spring-in.

### 2.2.2 Voids, Dry-spots and Fiber volume fraction

The quality of fiber reinforced composite parts is governed largely by the process parameters and materials used during the manufacturing process. One of the most important quality characteristics of the laminate is the void content in the finished part. The presence of these voids has a significant affect on the mechanical properties, thermal properties, dielectric properties, surface finish etc., of the part.

Voids formation occurs mainly due to variations of permeability in the laminate, out-gassing of dissolved gas in the resin, evaporation of volatile components, shrinkage and leakage in mold or connections that allows air from outside to creep into the setup [12]. Studies have shown that using degassed resin reduces the void content in the laminate and improves part quality. The degassed resin greatly reduces the risk of out-gassing from the resin and it dissolves the bubbles in the resin that are formed during the flow.

Also, for VARTM processing under low absolute pressure of vacuum, it has been found that resins with volatile content expand greatly thereby increasing the void content. Therefore, before infusion, maximum vacuum pressure should be kept to minimize any residual air left inside the tows [8].

The local permeability variations in the preform are inevitable due to the varying levels of compaction under the bag. In such circumstances, real-time flow control of the resin is imperative for preform saturation and void-free fill. Johnson and Pitchumani [10] at the Composites Processing Laboratory in the University of Connecticut have shown induction heating of the preform under the bag to improve the quality of the part. Through this method of heating, the resin viscosity is reduced to counteract preform regions with lower permeability. Their work included
developing a process model for the non-isothermal flow during the VARTM process in the presence of induction heating. They made use of a heterogeneous preform that had known permeability variations to understand the key parameters that were involved in void free fill. Experiments were conducted to validate these process models through which process simulations were developed to deep dive into the effects of processing parameters such as - induction heating location, induction heating power level, and vacuum level on three heterogeneous preform geometries.

The key parameters and their levels in this testing of localized induction heating mechanism for void free processing were -

- Heating coil location: 5.08 cm and 15.24 cm from the line inlet.
- Induction heating power level for the heating coil: Varied between 0 and 200 V.
- Permeability ratio (ratio of high permeability fiber over low permeability fiber in the heterogeneous preform): 10, 14.5, and 18.8, measured at –77 KPa.

The plots in Figure 2.5 (a) and (b) suggest that above an induction heating power level for most of the permeability ratios and coil location, the unfilled volume fractions will be reduced to zero.

The experimental results indicated that the introduction of a heating mechanism at regions of lower permeability of the preform quickened the mold filling exercise and produced complete saturation of the preform (unfilled volume fraction, $V_o = 0\%$).

Since VARTM is conducted under vacuum bag pressure only, it fiber volume fraction is generally not as high as autoclave-cured laminates. But the following aspects mainly determine the feasibility of using VARTM [12]:

1. Geometry of the part
2. Materials used
3. Tooling and materials
4. Injection process
A combination of the correct tooling and injection process, large structures can be produced with one go.

Figure 2.5 Plots of unfilled volume fraction versus induction coil voltage for the coil located at (a) 15.24 cm and (b) 5.08 cm from the inlet at a vacuum level of -77 kPa

2.2.3 Surface quality of the laminate
Besides dimensional stability, another aspect of VARTM produced part that needs some attention is the surface quality. In VARTM processing, the laminate is under the vacuum bag, which is pressed against it under vacuum pressure. Unlike in resin transfer molding process (RTM) where the resin is infused between two molds held under pressure, tight tolerances and excellent surface quality cannot be achieved with the VARTM process. The unevenness of the vacuum bag can be attributed to the
surface of the preform that may be wavy due to presence of excessive resin in certain regions and resin distribution medium. However, a recent study has shown that RTM quality parts can be produced by VARTM processing.

Heider et al [13] tested different types of fibers (orthogonal 3D preform, woven and unidirectional) for their surface roughness using the VARTM process. Their study also benchmarked the surface roughness with the better performing RTM process. In their work, semi-rigid caul plates of different kinds were introduced into the process that reduced the surface roughness of the part.

The surface roughness was evaluated using a high accuracy laser displacement sensor. The unidirectional fibers had the best surface quality using plastic caul plates and RTV60 caul plates from General Electric [13].

2.3 Applications of High Temperature Polymer Matrix Composites

Polymer matrix composites (PMCs) were introduced in aircrafts as early as the 1970’s under the NASA Aircraft Energy Efficiency (ACEE) Program [14]. This program sought to obtain actual flight experiences with composite materials and to compare the long-term durability of flight components to the data obtained from an environmental exposure ground test program [15]. The participants of the ACEE program included aerospace giants such as, Boeing Commercial Airplane Company, Douglas Aircraft Company and Lockheed Corporation. During the earlier years of the program, smaller components that were lightly loaded were designed and introduced into service. Some of these components were – fairing panels, spoilers and upper left rudder of Douglas DC-10 aircraft [15]. As a result of this program, by 1987, 350 composite components had entered into commercial airline flight service. In spite of this, the experience of composites in aerospace applications thus far has been inconsistent. This is mainly because of lack of understanding of failure characteristics, higher costs of processing and unreliability in the estimates of design and development costs of composite structures.
Extensive use of composite materials in commercial aircrafts was started by Airbus which used these materials for a variety of low and medium temperature components like deflector doors, nose wheel doors, flap access doors, pylon fairings, nose radome, cooling air inlet fairings, engine cowling panels, etc. Its wings were 13% composite materials [15].

Besides commercial aircrafts, composite materials have contributed significantly towards increasing the operational efficiency of the military aircrafts. Some of the common parts that are made of HTPMCs are the fuselage, intermediate wing spars, horizontal and vertical stabilizers and wing control surfaces (wing skin) [20].

![Composite structural applications in military aircrafts](image)

**Figure 2.6** Composite structural applications in military aircrafts

Figure 2.6 indicates the growing trend of using PMCs in military aircrafts. It compares the application of composite materials as a percentage of structural weight for a number of aircrafts. Based on the data provided in reference 15, the F-22 Raptor, shown in Figure 2.7 has 24% thermoset composite material and 1% thermoplastic composite material.
There is only a limited amount of research that has been done on HTPMC’s using the VARTM process. One such paper that was written by Thomas et al [8] at Lockheed-Martin Space Systems for the MSO group discusses the use of low cost processing methods for making the equipment section of a missile. The cost of the equipment section in a missile contributes to the majority of the structural cost. This equipment section is shown in Figure 2.8. Their work focused on elimination of labor required to assemble multiple parts into a complex structure. This labor includes such activities as machining individual composite parts, drilling fastener holes, preparing bond lines, bonding parts together and inspection. An integral fabrication strategy, which eliminates the need for a number of parts to be joined together, saves more cost since the parts do not have to be made to high tolerances.
Figure 2.8 Cross beam integral structure (one quadrant) fabricated by VARTM at MSO, Lockheed Martin using BMI resin [8]

The evaluation of four resin systems was carried out which were believed to be VARTMable (resin with viscosity less than 1000 cp, several working hours time, and negligible volatiles). The resins were – two cyanate esters, namely, PT – 30 phenolic triazine and Bryte EX – 1510 and two bismaleimides (BMI’s), namely, Hexcel 650 BMI and Hexcel 651 BMI.

After preliminary studies, the two of the cyanate ester resins were not pursued further because PT – 30 was brittle and EX – 1510 lacked the same mechanical properties as the BMI’s. Hexcel 650 was selected over 651 based primarily on higher compression strength in intermediate temperature regimes. Figure 2.9 compares the compression strength versus temperature for the two BMI’s.
The “cruciform” fiber configurations commercially available were made use of for this complex cross beam structure. The tows on this fabric extended in both orthogonal directions. Z – Stiffeners cross-section was chosen with its lower flange going inwards.

Carbon fiber tooling was used in this demonstration since curing is done at low temperature (cure 140 F and post - cure 392 F) in an oven. The dimensional results obtained as a result of low temperature curing were excellent.

Pre-forming of the dry, continuous fabric was done using a tacky solution, which is prepared by dissolving the base resin in a safe workable solvent. This is sprayed on the tool and on successive fabric layers after they are applied. The resin and the vacuum trap for the exiting resin are kept outside the oven while the entire mold setup is placed within the oven for better temperature control. The use of high-quality tooling materials, careful sealing of any joints between tooling members and
leak checking of the sealed tool itself are crucial for the success of VARTM. Double bagging was used with a breather layer between the two bags that draws any leaking air. This was connected to an independent vacuum line that pulled the air out since any air leaking into the setup affects the part quality. The double bag also helped in maintaining the pressure on the vacuum seals near the resin inlet after infusion since the pressure inside the bag rises to atmospheric pressure. Resin infusion was done under full vacuum to minimize the amount of residual air left inside the tows. The degassing of the resin was done in-situ. These measures resulted in parts with zero void content.

VARTM parts with 58 % fiber volume fractions were produced, while the same fabric in pre-preg/autoclaved laminates was about 60 %. The labor cost saving from adopting the VARTM process for the equipment section was of the order of 75%. The laminates retain useful strength up to 650°F thereby eliminating the need for thermal insulation.

Figures 2.12 and 2.13 show some of the HTPMC’s that are being used in aircrafts and aero-engines - fan blades, outlet guide vanes, bypass ducts, nose cone spinners, core engine fairings, annulus fillers, nacelle and variable guide vane rings. These parts are shown in Figures 2.10 and 2.11.

![Materials and Structures for High Performance](image)

**Figure 2.10** NASA GLENN Research center, Cleveland, OH
2.4 High Temperature Polymer Matrix Composite resin systems

This research effort focuses on the development of composite parts that can withstand high temperatures in aerospace applications using VARTM. Any resin system that is finally considered should have a high $T_g$ and meet certain requirements to be processable (having melt viscosities of less than 500 cps, low volatile emission and shrinkage) by VARTM. At present, there are a number of resin systems with $T_g$ over 550°F. But not all of them can be readily processed by VARTM. This can be attributed to its inherent low processing pressure (max: full vacuum or 14.5 psi), higher viscosity and more restrictive requirements of the resin’s very low volatile content.

The NASA Lewis Research Center developed the first HTPMC resin system called as PMR-15, a polyimide, for aerospace applications in the 1970’s [17]. PMR stands for polymerization by monomeric reactants. Polyimide based resin systems have been known to perform satisfactorily under high temperatures.

Since the development of PMR-15, the market for HTPMC resin system has grown significantly. High temperature polymer matrix composites are continually being utilized or evaluated for a number of aerospace applications by NASA. Aromatic polyimides, in particular, have long been recognized as attractive matrices.
for such applications. The imide group provides a stiff molecular backbone, which
results in a high $T_g$ and excellent chemical resistance and mechanical properties [18].

Studies have shown that PMR-15 has a high $T_g$ of 316$^\circ$C (600$^\circ$F), which
permits its usage in certain components for aerospace engines (the outer bypass duct
for the F-404 engine is made of PMR-15). The advantages of PMR-15 besides its
high $T_g$ are – good thermo-oxidative stability, low cost and easy availability.
However, it has a high level of toxicity mainly due to one of its reactant components,
4, 4’ – methylenedianiline (MDA). PMR-15 also lacks toughness, which is another
disadvantage that has limited its applications [21].

Occupational Safety and Health Administration (OSHA) and Environmental
Protection Agency (EPA) heavily regulates the use of PMR-15 in the workplace due
to of MDA, which is believed to be an animal carcinogen and a suspect human
carcinogen. They require certain engineering controls to be in place, which further
adds to the cost of the aerospace industry to the tune of millions of dollars [17].

A number of resin systems were considered for this thesis research, which will
be discussed in detail in Chapter 3). The resin categories included BMIs, Phenols,
phenylethynyl imides and polyimides whose rheological, thermal and mechanical
properties were examined from the data sheets provided by the manufacturers. The
material selection process resulted in a resin system designated as LARC$^{\text{TM}}$-RP46
[20]. This resin system was developed by NASA Langley Research Center, which has
improved characteristics over its predecessor, PMR-15 resin.

RP-46 resin performs well even at high temperatures. It retains its composite
fracture toughness at elevated temperatures. Autoclave processing is the primary
manufacturing method for RP-46 based composite, which is a costly method. Since,
this resin has good melt viscosities and a 3-day pot life, it should lend itself well to
VARTM processing that saves cost. The success of this research effort will lie in
manufacturing of RP-46 based laminates that have good wetting by the resin, low
void content, easy processability, good mechanical and thermal properties and the
consistency with which these criteria can be achieved.
2.5 LARC™ RP-46 polyimide resin system

Polyimides are a class of polymers that are thermally stable. The complexities involved in polyimide synthesis can be attributed to a variety of monomers that are available and several methodologies that exist for polymer synthesis. Minor variations in the structure can dramatically alter the properties of the polyimide [21].

The most widely practiced procedure in polyimide synthesis is a two-step poly (amic acid) process [21]. This involves reacting a dianhydride and diamine at ambient temperature in a dipolar aprotic solvent to yield appropriate poly(amic) acid, which is then cured into the final polyimide. Solvents are essential to the polyimide synthesis process since most polyimides are insoluble due to their aromatic structures.

In the two step process, the addition of dianhydride to a diamine in a solvent forms the intermediate poly(amic) acid due to the nucleophilic of the amino group on the carbonyl carbon of the anhydride group as shown in Figure 2.12.

![Figure 2.12 Imidization and cross-linking of polyimides [21]](image)

The second step in the two step process is the thermal imidization or curing of the poly(amic) acid to form the polyimide. The achievement of 100% imidization of poly(amic) acid by thermal curing has been subject of many theories which can be divided into two broad classifications [21]:
• Gradual heating to 250-350°C depending on the Tg of the polyimide
• Heating the poly(amic acid) mixture to 100°C and holding for one hour, heating from 100°C to 200°C and holding for one hour, heating from 200°C to 300°C and holding for one hour and slow cooling to room temperature from 300°C.

The imidization process is governed by complex factors that include several side reactions that compete with the propagation reaction and also dynamically changing physical properties such as diffusion rate, chain mobility, solvation and acidity.

RP-46 resin is a PMR based resin system that contains the 3,4’-oxydianiline (3,4’-ODA) instead of the 4,4’-methylenedianiline (MDA) as the diamine in the process. ODA is more appealing than the MDA since it reduces the toxicity of the resin and is more flexible than the MDA [22]. Therefore, its incorporation in the PMR polyimide structure imparts better resin flow, which in turn improves the processability. This also means greater toughness. The anhydride monomers – 5-norbornene-2, 3’-dicarboxylic anhydride (NA, Kodak) and 3,3’, 4,4’-benzophenonetetracarboxylic dianhydride (BTDA, Allco) refluxed in methanol for 2 hours results in 5-norbornene-2, 3-dicarboxylic acid (NE) and dimethyl ester of 3,3’, 4,4’-benzophenonetetracarboxylic (BTDE). Figure 2.15 shows the imidization and the cross-linking process that results in the formation of a polyimide along with the byproducts obtained at different temperatures.

The RP-46 resin that was obtained as samples from Unitech, LLC had 30% methanol solution in the mixture. As mentioned earlier, this solution helps in the formation of BTDE and NE from BTDA and NA. The presence of solvents in the imidization process is essential. It has been reasoned that the presence of solvents reduces the affinity of anhydrides to react with water and other impurities and yields high molecular weight polyimides [21] during the imidization process. Another important reason for using solvents is that while the monomers are all basic, the final product is acidic. The acid-base interaction of the resulting acid with a basic solvent
medium is exothermic and one of the most important driving forces of the reaction. This rate of the reaction will be faster for more basic and polar solvents.

As mentioned earlier, the number of monomers that are available for polyimide synthesis is extremely vast. The scope of this research does not extend to identifying individual reactions at the monomer level including various side reactions and is focused wholly on the higher level propagation reactions namely – imidization and cross-linking. Some of the well-known polyimides in the industry, besides LARC RP-46 are PMR-15, PMR-II-50, VCAP-50 and AF-R-700B.

Pater [22] compared the processing of RP-46 with PMR-15 in his work mentioned in the reference. RP-46 had a larger processing window and exhibited better flow. The results indicated that LARC™ RP-46 is readily processed into a high quality composite with excellent reproducibility.

The $T_g$ for RP-46 from the studies carried out by Pater after post-curing for 4 hours each at 316°C, 343°C and 371°C is 410°C. The post-curing process definitely improves the thermal resistance of the part [22], in other words the $T_g$. Also, the thermo-oxidative stability of the RP-46 is equivalent to PMR-15.

IM-7 Carbon fibers have been used most commonly as reinforcement with RP-46. There is data available in the literature regarding the important mechanical properties of IM-7/RP-46 composite but the one property that is of interest to this research is the tensile strength. The VARTM samples that are prepared will be tested for the tensile strength in accordance with ASTM D-3039 standards and these results will be compared with the data obtained from literature reviews. The tensile strength for this composite is 2623 MPa and a modulus of 155.8 GPa was reported in reference 21. The Unitech, LLC website indicates results of woven carbon T650-35/RP-46 composite tensile test done through ASTM D-3039 as having a strength of 608 MPa. The in-depth analysis and comparison of thermo-oxidative stability using Thermal Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) are done in chapters 4 and 5. The weight loss profile and the heat capacities are discussed in those chapters. However, not a single research paper mentions dynamic
mechanical analysis (DMA) test results for IM-7/RP-46 composites. Therefore, samples of IM-7/RP-46 composite were obtained and DMA tests were performed to obtain the storage modulus and $T_g$. These results are discussed in Chapter 5.
CHAPTER 3

MATERIAL SELECTION

3.1 Benchmarking of candidate resin systems

Since fibers are capable of withstanding very high temperatures, the type of resin system that is used most affects the performance of HTPMCs. After a thorough study of the various resins in the market, a list of candidate thermoset resin systems were considered for VARTM processing. In order to do this, certain criteria for processing requirements were set. The candidate resin systems are the ones that meet those criteria or are close to meeting them.

The standards, not listed in the order of rank are –

- $T_g$ (glass transition temperature) of the neat resin should be over 600°F ($316°C$)
- Viscosity of the neat resin should be less than 1000 cps to enable VARTM processing
- The resin should have a good pot life
- Cost of the resin should be affordable
- Emission of volatiles during processing should be minimal

Although the PMR-15 polyimide high temperature resin system is one of the most widely used resin systems for HTPMC applications, its use requires certain stringent engineering and safety controls due to the presence of MDA. Hence, only the following resin systems were considered as candidates for VARTM processing.
3.1.1 Larc RP-46

NASA Langley Research Center developed this thermoset polyimide and its been marketed by Unitech Corporation, LLC. The properties of this polyimide resin are listed below.

- Less toxic when compared to other HTPMC resin systems such as PMR-15. As a result, it is much easier to process and saves the industry millions of dollars due to lesser regulations and controls at the workplace [23]
- This resin system, when fully cured, creates a finished product that does not absorb moisture or cause micro cracking and blistering. It can be attributed to the resins’ unique patented formulation
- It has a continuous working temperature range between 150°F (65°C) and 675°F (357°C) and it has the ability to withstand extreme temperatures of 1500°F (815°C) for very short durations
- The $T_g$ for this resin is 740°F (393°C)
- Compression molding and filament winding are the primary manufacturing methods
- Current applications that make use of this resin system include – aerospace, defense, missile systems and ballistics, automobile and oil exploration

3.1.2 PETI-330

It is a Phenylethylnyl-imide resin system, developed by NASA along with M & P Technologies in collaboration with Clark Atlanta University (HiPPAC Center).

- The highlight of this resin system is its low melt viscosity, which enables it to be used with non-autoclave processing techniques such as resin transfer molding (RTM) and VARTM
- Advances in the chemistry of this type of polymers have led to high $T_g$ resins that can be processed by infusion methods
• The need for this type of a resin system has been felt since it can deliver comparable results with other PMC resin systems while still being environmentally friendly

• Recent studies have shown that the $T_g$ of PETI-330 is 597°F (314°C) [26]

• This resin system can be used as a part of high temperature composite structures, which operate slightly above 550°F (288°C)

• This resin has shown promising results when used as a PMC material of choice in an airframe in NASA’s Advanced Space Transportation Program (ASTP) that involves the development of hypersonic air breathing vehicles for access to space [24]

3.1.3 HEXCEL-F650

It is one of the most popularly used high temperature resin system, which belongs to the bismaleimide (BMI) group. The F-650 was developed by Hexcel Corporation to overcome various limitations of making complex structures with existing resins and also to enable them to be used at high temperatures.

• It is cost effective resin system, which delivers complex parts with simple fabrications

• There are no by-products during processing and is volatile free

• It also has excellent tack, handling characteristics and straightforward layup

• It has an excellent pot and shelf life and hence can be used in complex applications that take weeks for processing

• The $T_g$ for the F650 is under dry condition is well over 600°F (316°C) [28]

• It is dimensionally stable at greater than 500°F(260°C)

• Excellent rheological properties for processing with liquid molding techniques

• This resin was the choice of the Lockheed-Martin Space Systems, MSO group for manufacturing the equipment section of a missile. This resin was chosen primarily because of its $T_g$ and high compression strength at high
temperatures and even in wet conditions [8]. Their work was discussed in the literature review in Chapter 2

### 3.1.4 DMBZ-15
A NASA Glenn developed and patented high temperature polyimide resin designed for applications requiring short-term exposure to high temperatures.

- DMBZ-15 was developed as an alternative to PMR-15 that contains the toxic MDA. This MDA was substituted by DMBZ
- The $T_g$ of DMBZ-15 is 785°F($418°C$) [25]
- Excellent thermo-oxidative stability. 0.5% weight loss at 550°F($288°C$) for 500 hours
- It is very well suited for applications involving missile components

### 3.1.5 CYCOM\textsuperscript{TM}-5250-4 RTM
Cytec Engineered Materials Inc. based in Anaheim, CA develops the 5250-4. It is a homogenous BMI resin developed specifically for the resin transfer molding process. It is also known to have the highest open-hole compression (OHC) value of any organic matrix resin.

- It has a $T_g$ of 520°F($271°C$) under dry conditions and 405°F($207°C$) in wet condition [26]
- It has a low viscosity during processing and hence is suitable for liquid molding technologies
- It can withstand service temperatures up to 400°F($204°C$); but, its toughness and processability make it ideal for 180°F($82°C$) temperature applications where epoxies are commonly used
- It has a long pot-life of 60 days at room temperatures
- It has wide applications include wing spars, fuselage skins and stiffeners; low operating temperature, critical load bearing components and engine components
3.2 Resin selection

After reviewing all the potential resins that could be used, the best resin system that could be processable by VARTM processing is chosen. The cost of PETI-330 resin system is the major factor for eliminating it. Since this research focuses on materials and processes that are affordable, PETI-330 is not considered further.

The $T_g$ of CYCOM 5250-4 RTM resin system is lower compared to the others. It has a $T_g$ that is less than 600°F (316°C) and hence does not meet the temperature requirement.

DMBZ-15 has a very high $T_g$ of 785°F (414°C). This resin is ideal for applications that require short-term exposure to high temperatures such as missile parts and not for long-term exposures in aerospace components. DMBZ-15 has been manufactured by solvent assisted RTM which is an infusion molding technique pioneered by NASA under the Advanced Subsonic Technology program [27]. Since this resin is ideal for short-temperature bursts, it is not considered further.

The RP-46 polyimide resin system has the best mechanical properties. It also has one of the highest $T_g$ amongst all the systems. This, along with its cost and availability to the researcher makes it an obvious resin to pursue. The RP-46 resin, which contains 30% methanol, has a viscosity of 300 cps when heated to 149°F (65°C) for 30 minutes.

Resins such as HEXCEL F650 and PETI 330 can be used for making composites with VARTM. However, RP-46 has a higher $T_g$ and far superior mechanical properties. There is tremendous cost savings from manufacturing the HTPMC parts using VARTM process and RP-46 resin system. After ascertaining from Unitech Corp LLC that no data for VARTM processing of RP-46 is available, the research progressed with it being the resin of choice. Table 3.1 below shows a comparison of important properties of the resins that were considered. All the costs displayed in table were obtained from the respective companies.
### Table 3.1 Comparison of promising resins

<table>
<thead>
<tr>
<th>Commercial name</th>
<th>RP-46</th>
<th>PETI-330</th>
<th>HEXCEL-F650</th>
<th>DMBZ-15</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type of resin</td>
<td>Polyimide</td>
<td>Phenylethynyl imide</td>
<td>BMI</td>
<td>Polyimide</td>
</tr>
<tr>
<td>Vendor</td>
<td>Unitech Corp</td>
<td>Eikos Inc</td>
<td>Hexcel Corp</td>
<td>Maverick Corp</td>
</tr>
<tr>
<td>Cost</td>
<td>Approx $90 per lb</td>
<td>Approx $1500 per lb</td>
<td>$55 per lb</td>
<td>@ $150-200 per lb</td>
</tr>
<tr>
<td>$T_g$</td>
<td>740°F (393°C)</td>
<td>572°F (300°C) – 662°F (350°C)</td>
<td>600°F (316°C) Dry and 450°F (232°C) Wet</td>
<td>1.740 cps @ 73°F (23°C) using a Brookfield RV-DV-1+ viscometer, 250 ml beaker, spindle #3, 10 RPM</td>
</tr>
<tr>
<td>Viscosity, cps</td>
<td>300 cps @ 149°F (65°C)</td>
<td>Less than 1000 cps @ 530°F (276°C)</td>
<td>Less than 1000 cps @ 306°F (152°C)</td>
<td>Short term exposure (&lt; 1000hrs) – 750°F (398°C), brief exposure to 800°F (426°C)</td>
</tr>
</tbody>
</table>

### 3.3 Fiber selection

Since fibers occupy most of the volume in the laminate, the choice of type of fiber is an important one. Carbon fibers are the most popular fibers in aerospace applications due to their excellent mechanical and thermal properties. Structurally the carbon fibers are a blend of amorphous carbon and graphitic carbon. The graphitic form provides the high tensile modulus [1] which range between 207 GPa to 1035 GPa. Carbon fibers are available commercially in long and continuous tows that range from 1000 to 160,000 parallel filaments used in high performance applications [1]. In this
thesis research, \(0/90^\circ\) plain weave bi-directional continuous carbon fibers are used to provide the load carrying mechanism for the flat panels made with RP-46 resin.
CHAPTER 4

EXPERIMENTAL SETUP AND METHODOLOGY

4.1 Experimental setup

4.1.1 Vacuum bagging materials
All supplies for the VARTM process were procured from Airtech International Inc., a leader in vacuum bagging supplies. The supplies from this vendor are listed below and a brief description is provided.

4.1.1.1 Vacuum bagging film. The high temperature Wrightlease 5900 T.O.S (400% elongation bagging film) is used as the vacuum bagging film (appendix Figure A.1).

4.1.1.2 Teflon High Permeable Layer (Resin distribution medium). The resin distribution medium has proved to be very effective in resin flow under the vacuum bag. It has a square configuration (appendix Figure A.2). At the time of this research work, this product was obtained as samples and not available in the market. Therefore, the product name was not included.

4.1.1.3 Release film. The Wrightlease 5900 high temperature fluorocarbon film with a maximum use temperature of 650°F (343°C) is used as the release film (appendix Figure A.3).

4.1.1.4 Bleeder. The high temperature capable bleeder E is used to impart good surface quality to the parts (appendix Figure A.4).
4.1.1.5 **Vacuum bag sealant tape.** The A-800-3G, a high temperature sealant tape that has good tack. It can withstand a temperature of 800°F (426°C) (appendix Figure A.5).

4.1.1.6 **High-temperature pressure sensitive tape.** Airkap 1 is a polyimide pressure sensitive tape coated with a fully cured silicone adhesive for high temperature lay-ups to 750°F (399°C) (appendix Figure A.6).

4.1.1.7 **Mold plates.** The aluminum mold plates are 1 ft * 1 ft in length and breadth. The thickness of mold is half an inch. The sanding of the mold surface is done to keep it flat without any aberrations (appendix Figure A.7).

4.1.2 **Heating devices**

The heating devices were used for tool and resin preparation before infusion, heating of the mold during infusion, curing of the laminate and for post-curing.

4.1.2.1 **Oven.** A Vulcan programmable furnace is used as the oven capable of reaching temperatures of 932°F (500°C) (appendix Figure A.8).

4.1.2.2 **Hot plate.** The hot plate is capable of reaching temperatures up to 572°F(300°C) (appendix Figure A.9).

4.1.3 **Miscellaneous supplies**

The Vac-Valve 409 SSHTR Stainless Steel is used to connect the bag to the tubing. Brass ball valves (¼” NPT) are used to control the flow of resin. The vacuum pump shown in Figure A.12 in the appendix is used to provide the necessary pressure. The resin trap for the exiting resin is incorporated into the design (appendix Figure A.13).
A temperature laser gun is used to read the temperature on the bag during the curing process. A fiber cutter is employed to cut the carbon fiber plies. Safety gear that is employed during processing is shown in Figure A.16 in the appendix.

The next three sections discuss the procedure followed for making the high temperature composite panels using RP-46 resin. All the tasks needed to make the panels can be broadly categorized into three stages – tool and vacuum bag preparation, infusion process and cure and post-cure cycles.

![Figure 4.1 Stages of VARTM processing of RP-46 resin](image)

**4.2 Manufacturing of test specimens**

A number of smaller parts (88 mm * 76 mm, 6 plies, 0/90 bi-directional carbon fabric) were initially produced to understand the process dynamics of using RP-46 in VARTM manufacturing. These parts had many problems related to processing (Figure 4.2). Some of the major challenges were – long infusion times due to slow flow rate of resin, many dry spots; bad part quality, volatile emission concerns, low intra-laminar strength and undesirable surface quality. These smaller parts aided in understanding of operating parameters.
After successful manufacturing of smaller parts, the research continuously pushed for thicker parts that had more plies (12 plies, 0/90 bi-directional carbon fabric) and then focused on making slightly bigger parts (220 mm * 120 mm, 6 plies, 0/90 bi-directional carbon fabric). These parts had 10 plies and Figure 4.3 shows one of the sample parts made by VARTM.
The core analysis for understanding the feasibility of using VARTM for RP-46 is done using eleven laminates (220 mm * 120 mm, 10 plies, 0/90 bi-directional carbon fabric) that were produced over a period of time. This section further describes the experimental test matrix with operating parameters for each of the eleven samples.

4.2.1 Experimental test matrix

The operating parameter used during the processing of each of the test parts is shown in Table 4.1. This table describes the learning process that was involved in making good quality parts using the VARTM process. The operating conditions were changed depending on the quality of parts obtained from each trial. The laminates were tested for tensile strength, storage modulus and Tg. The storage modulus and tensile strength, which are dependent on the processing conditions, displayed a certain amount of variability between parts. The test results for each of the laminates will be discussed in detail in chapter 5. Section 4.3, 4.4 and 4.5 describe in detail the processing steps and tasks involved for making the laminates.
Table 4.1 Experimental test matrix for RP-46 processing using VARTM

<table>
<thead>
<tr>
<th>Presence of peel plies</th>
<th>1 SIDE</th>
<th>2 SIDES</th>
</tr>
</thead>
<tbody>
<tr>
<td>Presence of Sealant Tapes</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>High Permeable Layer</td>
<td>Not present completely</td>
<td>Present completely</td>
</tr>
<tr>
<td>Vacuum Pressure Post Infusion</td>
<td>15”</td>
<td>10”</td>
</tr>
<tr>
<td>Vacuum Pressure during Infusion</td>
<td>FULL VACUUM</td>
<td>15”</td>
</tr>
<tr>
<td>Sample #</td>
<td>RS #1</td>
<td>RS #2</td>
</tr>
</tbody>
</table>

4.3 Tool and vacuum bag preparation

All the activities in this stage of the VARTM process directly or indirectly affects the infusion stage. The Figure 4.4 shows a schematic diagram of all the tasks in the sequence that they are carried out.
The first step is to cut the carbon fibers using the fiber cutter. The fiber is cut to 22 * 12 cm and care should be taken to prevent fiber pullout during handling of the cut plies. 10 plies are cut for each of the experimental laminates. The aluminum mold surface is first dusted and sanded using a sander for smooth surface quality of the part. Any burrs or surface distortions are eliminated when sanding is done. The gates and vents are which are essentially short metallic pipes and outlet valves are cleaned with acetone and washed thoroughly and if any leftover resin is present from the previous experiment, it is burnt out by placing in an oven at 932°F (500°C) for an hour.
According to the MSDS from Unitech Corporation, the RP-46 should be refrigerated under 40°F (4.44°C) for best results. During the tool preparation phase, the resin in the beaker should be removed from the refrigerator and allowed to warm to the room temperature.

The vacuum bag preparation begins with cutting of the release film to an appropriate size and placing it on the sanded surface of the aluminum mold. The release film aids in the separation of the part from the mold surface after infusion and curing. A layer of peel ply on the tool side of the part was incorporated after the fifth sample due to inconsistent surface quality obtained on the tool side of the part. This could be due to resin flow and high temperature environments that may have caused certain patterns on the surface that were undesirable. After the placement of peel plies, which are also known as bleeders, the part quality was considerably better. The 10 layers of carbon fiber plies are placed on the bleeder as shown in Figure 4.5 (a). Another layer of peel plies are placed on the carbon fiber plies. The peel plies are used for protecting the part’s upper surface from the marks of the high permeable layer, wrinkles caused by the bag and for easy separation of the part from under the vacuum bag. They also impart a good surface quality. Above the peel plies is the high permeable layers. The high permeable layers are an absolute necessity for VARTM processing of RP-46 resin systems. The Teflon coated square configuration high permeable layer (HPL) is placed above the peel ply. The placement of the HPL is shown in the figure 4.5 (c). The first three laminates (RS #1, RS #2 and RS #3) did not have the HPL spread across the entire length of the part since literature review suggested that the HPL be 1 to 2 inches away from the end of the fibers [30]. However, these samples showed poor stiffness and distortions in thickness between the resin inlet side and the vacuum side of the part. Therefore, from the fourth sample onwards, the HPL was spread along the entire length of the part.
In the resin flow zone closer to the inlet or the gate, we place two layers of HPLs. This provides the resin with more space or gaps to move towards the fabric at a faster flow rate and enable complete wetting sooner. The high temperature vacuum bagging sealants are applied around the release film as shown in Figure 4.5 (d).

Figure 4.5 Vacuum bag preparation
The paper on top of the adhesive sealant is removed so that the vacuum bag can be placed on top of the sealant. The gate is placed on the sealant. The gate is a short metal pipe connected to a hose through which the resin enters the mold cavity. The vent, which is connected to the resin collection area, is a vacuum valve. This valve is connected to a ball valve as shown in the Figure 4.5 (e).

This ball valve ensures no air leaks into the cavity when the mold setup is disconnected from the external setup and placed inside the oven during curing. Adequate measures should be taken to ensure that the vacuum bag and the valves are sealed perfectly to ensure no air leaks into the apparatus. Any air leaks could affect the part quality significantly since air bubbles tend to expand rapidly under vacuum. To ensure absolute vacuum within the bag, high-temperature pressure sensitive sealant tapes are used from the fourth sample onwards which proved to be very effective in preventing leaks or gaps.

The resin, which is at room temperature, is placed inside a vacuum oven and degassing at half vacuum is done for an hour to remove any air bubbles present in the resin. The resin is then heated to 149°F (65°C) for 30 minutes to reduce the viscosity.

The mold setup is placed on the hot plate and it is heated to a temperature between 131°F (55°C) and 149°F (65°C) (Figure 4.6). At this time, all the hoses and other connections are made which are necessary to carry out the processing of the laminate. The pressure under the bag is controlled by using a three way connector on the hose that connects the resin collection area and the vacuum pump. The third outlet is connected to the ball valve which is opened and closed to a level at which the desired vacuum pressure can be obtained. The pressure is read using a vacuum pressure gauge connected to the setup.
4.4 Infusion process

The infusion process has undergone constant changes between each trial to obtain optimum material properties from VARTM processing. The earlier trials were done using vegetable oil and EPON 862 epoxy resin system to understand the process kinetics and the dynamics influencing crucial responses such as infusion times, complete wetting of the part, minimal dry spots and good visual surface quality of the part. Figure 4.7 shows the laminates made using 12 plies of bi-directional glass fibers and EPON 862 epoxy resin.

Figure 4.7 Laminates of glass and EPON 862 epoxy resin
The learning curve towards acceptable and consistent processing of RP-46 composites imposed challenges related to resin flow and viscosity, complete wetting of the fiber under the bag, presence of voids in the final laminate and air leaks through the bag. The following processing steps helped to a certain extent to overcome these issues.

- Use of a high permeable layer (HPL) – The HPL was a crucial ingredient for the VARTM processing of RP-46. This allowed the resin to rapidly advance towards the vacuum vent from the gate, which would otherwise take a long time. The HPL is capable of withstanding high temperature and it is made of Teflon. The mesh of the HPL has a square configuration, which is shown in the previous section.

- Change of inlet setting – The resin inlet or gate was a vacuum valve similar to the vacuum vent kept in the vertical direction. But, this setup resulted in resin being blocked below the valve and unable to move. Therefore, experiments were conducted using the horizontal setup and the flow was much better. This maybe due to thickness of the laminates. Our experimental laminates are not thick enough to warrant the use of vacuum valves for resin inlet.

- Heating RP-46 before infusion – The resin under room temperature is extremely viscous and hence not suitable for VARTM processing. For a resin to be VARTMable, it has to have a viscosity of less than 500 cps during infusion. Therefore, rheological study of RP-46 is done to determine the appropriate infusion temperature. The Table 4.2 shows the viscosity of RP-46 when heated to different temperatures. This is done using a Cole-Palmer viscometer.
Table 4.2 Viscosity of RP-46 resin when heated to different temperatures

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Viscosity (cps)</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>1140</td>
</tr>
<tr>
<td>45</td>
<td>739</td>
</tr>
<tr>
<td>48</td>
<td>432</td>
</tr>
<tr>
<td>55-60</td>
<td>450</td>
</tr>
<tr>
<td>~65</td>
<td>350-310</td>
</tr>
<tr>
<td>65-75</td>
<td>300-340</td>
</tr>
</tbody>
</table>

The heating of the resin to 149°F (65 °C) reduces the viscosity to the range of 350-310 cps. At this viscosity, the RP-46 resin can be successfully infused and made to flow through the fibers under the bag.

- Heating the aluminum mold plate between 131°F (55°C) – 149°F (65 °C) – During infusion, the hot resin comes in contact with the cold aluminum mold, which drastically increases the viscosity. This hinders the flow of the resin and proves a major obstacle for complete fiber wetting. The heating of the mold plate to a temperature between 131°F (55°C) – 149°F (65 °C) enables the viscosity to be low enough for infusion. This temperature range was determined after a number of trials.

- Optimum vacuum bag setup – The optimum vacuum bag setup was reached after driving for continuous improvements in the flow of the resin under the bag after successive trials. The vacuum bag setup is significantly related to flow and infusion times of the resin. The optimum vacuum bag setup was discussed in the previous section (section 4.2 - Tool and vacuum bag preparation).

- Controlling infusion pressure – After producing 6 parts at full vacuum, experiments under lower vacuum pressures during infusion were conducted to study the material properties. The results from these tests are discussed in chapter 5.
• Degassing – The presence of air bubbles in the resin during infusion severely affects the quality of the part since air bubbles expand 50 times under vacuum and what are small air bubbles expands rapidly when under the vacuum bag [12].

• Robust bagging – The use of proper sealants that have high temperature capabilities and appropriate bagging techniques reduce or completely eliminate air creeping under the vacuum bag which is an absolute necessity. High temperature pressure sensitive tapes are used to seal the bag and to ensure uniform thickness of the laminate.

The infusion process begins with the application of vacuum pressure under the bag. A schematic diagram of the VARTM processing during the infusion stage is shown in Figure 4.9. Full vacuum is applied under the bag for 30 minutes before infusion. The resin inlet or the gate is sealed. This minimizes any residual air left inside the tows at the start of infusion [8].

**Figure 4.8 Resin infusion stage**
After the bag has been under full vacuum for half an hour, the pressure is varied depending on the desired pressure during infusion using the pressure regulator valve. The inlet hose is dipped into the beaker containing the resin and vacuum pressure pulls the resin from the beaker into the mold setup under the bag. The teflon high permeable layer (HPL) permits the flow of the resin with ease. The resin moves across the length of the setup from inlet to outlet. Complete infusion of the fibers is accomplished anywhere between 1 minute for full vacuum and 5-6 minutes for lower vacuum pressures. After the resin has wet fibers completely, the inlet is kept open for 5 minutes at the same level of pressure. This ensures that the resin have completely wet the fibers. The pressure is then reduced to lower level (15”, 10”, 5” and 2” of Hg) to prevent the vacuum from pulling the resin out of the wet fibers. The inlet is turned
off and the mold plate is heated for curing of the resin with the fibers. During infusion, care should be taken to ensure that the temperature of the mold apparatus is in the range of 131°F (55°C) – 149°F (65 °C).

4.5 Cure and Post-Cure cycles

The thermosetting cure reactions are generally thermally induced, with high performance composites usually associated with high cure temperatures 392°F (200°C) to complete the cure [29]. Understanding the cure behavior of thermosetting system is essential in the development and optimization of composite fabrication process.

According to the manufacturer of RP-46, the laminate should be cured at 617°F (325°C) for an hour until it cools to room temperature and post curing should be done at 662°F (350°C) for a duration of 2 hours. This curing and post curing cycle was used to make some of the initial panels, which resulted in bad surface quality of the part. The Figure 4.10 shows a panel that was made using this curing and post curing schedule. The part surface had a lot of aberrations and roughness.

Figure 4.10 Badly cured flat panel
This surface roughness can be attributed to the faster heating rate during the cure cycle. The remaining experimental runs were conducted at a low curing temperature on a hot plate and then placed inside the oven for completing the cross-linking process. Also, during the post-curing phase, the part is debagged and placed inside the oven as a freestanding part. This strategy improved the surface quality significantly.

The low temperature curing process is divided into two distinct stages based on where the mold is being heated. This cure cycle is described in Figure 4.11. The stage one is carried out on the hot plate itself. Once the resin has wetted the fibers significantly and has flowed through the length and breadth of the laminate, the hot plate on which the mold is placed is heated. The temperature of the hot plate is raised to 257°F (125°C) from 131°F (55°C) - 149°F (65°C) during infusion. The mold temperature is then held at 257°F (125°C) for 15 to 20 minutes.
The temperature is then raised to 392°F (200°C). The mold temperature is held at this point for 15 to 20 minutes followed by a temperature increase to 482°F (250°C) for 15 to 20 minutes. During the entire stage of curing, the vacuum pressure is maintained. This vacuum pressure is essential to pull the methanol and water vapors from under the bag. These vapors are emitted when the temperature is in the range of 257°F (125°C) to 320°F (160°C). This is indicated by the differential scanning calorimetry (DSC) curve of the neat resin shown in Figure 4.12.

The mold setup is then transferred over to the oven from where further curing is done. During transfer of the mold setup, the ball valve is closed to ensure no air creeps into the setup. The connections to the resin collection area are disconnected and the mold with its vacuum bag setup is placed inside the oven.
The cross-linking reactions begin at 482°F (250°C). The temperature is then raised to 617°F (325°C) @ 2°C/min. It is maintained at this level for a duration of one hour which completes the cross linking process. The setup is then allowed to cool to room temperature.

During both these curing stages, safety precautions are taken to avoid exposure to fumes and volatiles exiting the bag. Proper ventilation, respirators, high temperature gloves and worker enclosures were some of the safety measures needed during processing. Methanol and water are the byproducts released during curing. It is also observed that the smoke or fumes emitted during the curing process was considerably lesser when the bagging was leak free. RP-46 is a flammable resin and hence extreme precaution has to be taken before entering this stage of processing.

The post curing process begins with debagging the part and placing it in the oven as a freestanding part. Post curing is done for 4 hours at 662°F (350°C) @ 2°C/min which increases the strength and the Tg of the part. The final part is allowed to cool to room temperature. Figure 4.13 shows a smaller panel made using this cure and post cure cycle.

![Figure 4.13 Flat panel made using the lower temperature cure cycle](image)
Raise temperature to 125°C and hold for 15 mins

Raise temperature to 200°C and hold for 15 mins

Disconnect from setup on top of the hotplate and place inside the oven

Raise temperature to 250°C and hold for 15 mins

Raise temperature to 325°C and hold for 1 hour @ 2 C/min

Allow cooling to room temperature

Remove laminate from the bag

Place the part in the oven for post-curing at 350°C for 4 hrs while heating it at 2 C/min

Allow cooling to room temperature

Figure 4.14 Schematic representation of cure and post-cure cycle
4.6 Summary of operating parameters for VARTM

The experimental runs in this research work are not a part of design of experiments (DOE) study. This is mainly due to constraints in the availability of raw materials (resin, high permeable layers and other raw materials). Hence, a number of smaller parts that would require lesser quantity of resin were produced. In doing so, a fair understanding of the inherent process dynamics was achieved which helped during the manufacturing of larger parts. A summary of the important factors is indicated in the Table 4.3 and 4.4 along with operating parameters.

Table 4.3 Operating parameter during tool and vacuum bag preparation

<table>
<thead>
<tr>
<th>Factors</th>
<th>Parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Degassing time</td>
<td>1 hr</td>
</tr>
<tr>
<td>Degassing pressure</td>
<td>15” of Hg</td>
</tr>
<tr>
<td>Resin temperature</td>
<td>65°C for 30 minutes before infusion</td>
</tr>
<tr>
<td>Mold temperature</td>
<td>55-65°C</td>
</tr>
</tbody>
</table>

Table 4.4 Operating parameter during resin infusion process

<table>
<thead>
<tr>
<th>Factors</th>
<th>Parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pre-infusion pressure</td>
<td>Full vacuum</td>
</tr>
<tr>
<td>Pressure application time</td>
<td>30 minutes</td>
</tr>
<tr>
<td>Infusion pressure</td>
<td>30”, 15”, 10”, 5” of Hg</td>
</tr>
<tr>
<td>Post-infusion pressure</td>
<td>15”, 10”, 5” of Hg</td>
</tr>
</tbody>
</table>
4.7 VARTM processing data analysis

Table 4.5 presents the data collected during the processing of the eleven VARTM samples. Important indicators of the quality of composite laminates are - fiber volume fraction and weight fractions. The fiber volume fraction or $V_f$ is calculated using the Equation 4.1 and the weight fraction is the ratio of weight of the fibers ($W_f$) to the weight of the composite laminate ($W_c$).

$$V_f = \frac{\rho_m W_f}{\rho_f W_f + \rho_m W_m} \quad \text{Eq - (4.1)}$$

Where,

- $\rho_m$ - density of the resin matrix, g/ccm
- $\rho_f$ - density of the fibers, g/ccm
- $W_f$ - weight of the fibers, g
- $W_m$ - weight of the resin matrix, g

Table 4.5 VARTM processing data

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Wt of fibers (Wf)</th>
<th>Wt of laminate (Wc)</th>
<th>Wt of resin in laminate (Wm)</th>
<th>Wt fraction of fibers (%)</th>
<th>Volume fraction of fibers (%) (Vf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS #1</td>
<td>47.75</td>
<td>71.22</td>
<td>23.47</td>
<td>67.0%</td>
<td>61.1%</td>
</tr>
<tr>
<td>RS #2</td>
<td>48.45</td>
<td>78.67</td>
<td>30.22</td>
<td>61.6%</td>
<td>55.3%</td>
</tr>
<tr>
<td>RS #3</td>
<td>49.75</td>
<td>75.35</td>
<td>25.6</td>
<td>66.0%</td>
<td>60.0%</td>
</tr>
<tr>
<td>RS #4</td>
<td>50.13</td>
<td>72.3</td>
<td>22.17</td>
<td>69.3%</td>
<td>63.6%</td>
</tr>
<tr>
<td>RS #5</td>
<td>50.7</td>
<td>73.04</td>
<td>22.34</td>
<td>69.4%</td>
<td>63.6%</td>
</tr>
<tr>
<td>RS #6</td>
<td>50.1</td>
<td>73.3</td>
<td>23.2</td>
<td>68.3%</td>
<td>62.5%</td>
</tr>
<tr>
<td>RS #7</td>
<td>49.65</td>
<td>75.85</td>
<td>26.2</td>
<td>65.5%</td>
<td>59.4%</td>
</tr>
<tr>
<td>RS #8</td>
<td>48.71</td>
<td>73.34</td>
<td>24.63</td>
<td>66.4%</td>
<td>60.4%</td>
</tr>
<tr>
<td>RS #9</td>
<td>47.3</td>
<td>65.84</td>
<td>18.54</td>
<td>71.8%</td>
<td>66.3%</td>
</tr>
<tr>
<td>RS #10</td>
<td>47.27</td>
<td>68.94</td>
<td>21.67</td>
<td>68.6%</td>
<td>62.7%</td>
</tr>
<tr>
<td>RS #11</td>
<td>46.76</td>
<td>65.46</td>
<td>18.7</td>
<td>71.4%</td>
<td>65.9%</td>
</tr>
<tr>
<td>AVG</td>
<td>48.77</td>
<td>72.11</td>
<td>23.34</td>
<td>67.8%</td>
<td>61.9%</td>
</tr>
<tr>
<td>Standard Deviation</td>
<td>1.36</td>
<td>4.07</td>
<td>3.34</td>
<td>0.029</td>
<td>0.031</td>
</tr>
<tr>
<td>Variance</td>
<td>1.86</td>
<td>16.57</td>
<td>11.2</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The density of the matrix, $\rho_m$, is 1.35 g/ccm [22] and the density of the bi-directional 0/90° plain weave carbon fiber, $\rho_f$, is 1.75 g/ccm based on datasheets maintained in records.

The five laminates made using lower vacuum pressures have a higher average $V_f$ than those made under full vacuum as shown in Table 4.6. The standard deviation of composite weight ($W_c$) for low vacuum infused samples is twice that of the full vacuum composites. This could be due to different vacuum infusion pressures used (15", 10" and 5") under the low vacuum category. The average weight fractions of the fibers in laminates produced in these two categories are not very different.

**Table 4.6 Analysis of vacuum pressure during infusion on $W_f$ and $V_f$**

<table>
<thead>
<tr>
<th></th>
<th>Wt of fibers g ($W_f$)</th>
<th>Wt of laminate g ($W_c$)</th>
<th>Wt of resin in laminate g ($W_m$)</th>
<th>Wt fraction of fibers %</th>
<th>Volume fraction of fibers % ($V_f$)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Full Vacuum - Average</strong></td>
<td>49.48</td>
<td>73.98</td>
<td>24.5</td>
<td>66.96%</td>
<td>61.01%</td>
</tr>
<tr>
<td>Full Vacuum - Std deviation</td>
<td>1.13</td>
<td>2.67</td>
<td>3.05</td>
<td>0.02</td>
<td>0.032</td>
</tr>
<tr>
<td>Full Vacuum - Variance</td>
<td>1.28</td>
<td>7.13</td>
<td>9.35</td>
<td>0.00086</td>
<td>0.000987</td>
</tr>
<tr>
<td><strong>Low Vacuum - Average</strong></td>
<td>47.93</td>
<td>69.88</td>
<td>21.98</td>
<td>68.74%</td>
<td>62.94%</td>
</tr>
<tr>
<td>Low Vacuum - Std deviation</td>
<td>1.20</td>
<td>4.59</td>
<td>3.44</td>
<td>0.028</td>
<td>0.0312</td>
</tr>
<tr>
<td>Low Vacuum - Variance</td>
<td>1.44</td>
<td>21.08</td>
<td>11.87</td>
<td>0.00082</td>
<td>0.0009</td>
</tr>
</tbody>
</table>

Table 4.7 provides the infusion time data for the VARTM samples. The samples that are infused under full vacuum took lesser time for complete wetting compared to those samples, which were infused at lower pressures.

**Table 4.7 Infusion times per sample**

<table>
<thead>
<tr>
<th>Sample #</th>
<th>RS #1</th>
<th>RS #2</th>
<th>RS #3</th>
<th>RS #4</th>
<th>RS #5</th>
<th>RS #6</th>
<th>RS #7</th>
<th>RS #8</th>
<th>RS #9</th>
<th>RS #10</th>
<th>RS #11</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min, Secs</td>
<td>1.0</td>
<td>1.5</td>
<td>1.15</td>
<td>0.55</td>
<td>0.44</td>
<td>1.20</td>
<td>1.28</td>
<td>1.31</td>
<td>1.25</td>
<td>2.05</td>
<td></td>
</tr>
</tbody>
</table>
CHAPTER 5

TEST RESULTS AND ANALYSIS

This chapter discusses the results of various tests performed on the 11 laminates that were produced during this thesis work. Dynamic Mechanical Analysis (DMA) for storage modulus and Tg determination, Thermal Mechanical Analysis (TMA) for coefficient of thermal expansion, Thermal Gravimetric Analysis (TGA) for weight loss profile of the composite under high temperature environments are performed. ASTM D-3039 test for determining the tensile strength and modulus of the composite laminate are also carried out. The results obtained from the VARTM samples are compared with those obtained from the neat resin and baseline composite laminate (IM7/RP46) made by autoclave processing. eSEM images of the laminate are taken for visual analysis of fiber wetting and voids.

5.1 Thermal Analysis

5.1.1 Dynamic Mechanical Analysis (DMA)
DMA is performed to measure the viscoelastic properties such as the storage modulus and it also indicates the Tg of composite laminate. DMA is a technique that bridges the disciplines of thermal analysis and rheology [31]. The composite samples are fixed into position in the DMA instrument using the double cantilever fixture and mechanical deformation was applied to the sample via the instrument’s drive motor to obtain a combination of temperature and deformation profile. DMA tests are performed with the DMA-2980 from TA instruments where the sample is heated to
500°C at 5°C/min. The composite samples tested included the IM7/RP-46 baseline composite and eleven VARTM samples.

5.1.1.1 IM7/RP46 baseline Composite. Figure 5.1 depicts the DMA curve obtained for the baseline part.

From the curves, it is observed that the Tg of the composite is 672°F (356°C). The storage modulus is in the region of 50 GPa at room temperature. The descent of the storage modulus curve begins at 581°F (305°C). At this temperature, the modulus is 45 GPa.

5.1.1.2 VARTM samples. This section presents the graphs obtained from DMA tests carried out on eleven samples. Two samples from each laminate are used for testing the repeatability of modulus and Tg.
Figure 5.2 DMA test results for sample RS #1a and RS #1b

Figure 5.3 DMA test results for sample RS #2a and RS #2b
Figure 5.4 DMA test results for sample RS #3a and RS #3b
Figure 5.5 DMA test results for sample RS #4a and RS #4b

Figure 5.6 DMA test results for sample RS #5a and RS #5b
**Figure 5.7** DMA test results for sample RS #6a and RS #6b

**Figure 5.8** DMA test results for sample RS #7a and RS #7b
Figure 5.9 DMA test results for sample RS #8a and RS #8b

Figure 5.10 DMA test results for sample RS #9a and RS #9b

Figure 5.11 DMA test results for sample RS #10a and RS #10b
The experimental test matrix shown in the previous chapter (Experimental Setup and Methodology – chapter 4) is distinctly divided into two, based mainly on the vacuum pressure during infusion. The first six samples were infused at full vacuum and the remaining five at lower vacuum pressures.

The DMA curves for the parts made under full vacuum have large sample-to-sample variation. This trend continues till the fourth part after which better consistency in storage modulus values are observed. The first three samples (RS #1, RS #2 and RS #3) have extremely low modulus with maximum variability.

The sample RS #4a has an extremely high modulus and RS #4b has a poor modulus thus highlighting the inconsistency in the modulus values within the same part under full vacuum infusion. The best results for modulus in terms of value and
repeatability is observed for samples RS #10a and RS #10b. Table 5.1 shows the data from the DMA tests for all the samples.

Table 5.1 DMA test results for VARTM flat panel parts

<table>
<thead>
<tr>
<th>Part #</th>
<th>Vacuum Pressure at infusion</th>
<th>Tg °C</th>
<th>Storage Modulus (E’) GPa</th>
<th>Storage Modulus at 300°C (E’) GPa</th>
<th>Tg °C</th>
<th>Storage Modulus (E’) GPa</th>
<th>Storage Modulus at 300°C (E’) GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS #1</td>
<td>Full Vacuum</td>
<td>363.08</td>
<td>10.50</td>
<td>9.18</td>
<td>387.68</td>
<td>3.82</td>
<td>2.82</td>
</tr>
<tr>
<td>RS #2</td>
<td>Full Vacuum</td>
<td>377.37</td>
<td>4.58</td>
<td>3.29</td>
<td>382.92</td>
<td>2.61</td>
<td>1.76</td>
</tr>
<tr>
<td>RS #3</td>
<td>Full Vacuum</td>
<td>366.36</td>
<td>9.22</td>
<td>7.33</td>
<td>373.40</td>
<td>7.34</td>
<td>5.50</td>
</tr>
<tr>
<td>RS #4</td>
<td>Full Vacuum</td>
<td>364.67</td>
<td>24.98</td>
<td>23.80</td>
<td>359.11</td>
<td>13.30</td>
<td>11.75</td>
</tr>
<tr>
<td>RS #5</td>
<td>Full Vacuum</td>
<td>358.32</td>
<td>19.53</td>
<td>17.64</td>
<td>362.29</td>
<td>21.05</td>
<td>19.19</td>
</tr>
<tr>
<td>RS #6</td>
<td>Full Vacuum</td>
<td>368.64</td>
<td>13.13</td>
<td>11.31</td>
<td>368.64</td>
<td>15.06</td>
<td>12.77</td>
</tr>
<tr>
<td>RS #7</td>
<td>Low Vacuum (15”)</td>
<td>355.15</td>
<td>15.68</td>
<td>13.61</td>
<td>353.15</td>
<td>12.5</td>
<td>10.60</td>
</tr>
<tr>
<td>RS #8</td>
<td>Low Vacuum (10”)</td>
<td>359.11</td>
<td>15.00</td>
<td>12.82</td>
<td>366.26</td>
<td>18.03</td>
<td>16.29</td>
</tr>
<tr>
<td>RS #9</td>
<td>Low Vacuum (10”)</td>
<td>363.08</td>
<td>14.03</td>
<td>12.25</td>
<td>360.73</td>
<td>14.04</td>
<td>11.89</td>
</tr>
<tr>
<td>RS #10</td>
<td>Low Vacuum (10”)</td>
<td>358.32</td>
<td>18.59</td>
<td>17.14</td>
<td>356.73</td>
<td>19.74</td>
<td>17.73</td>
</tr>
<tr>
<td>RS #11</td>
<td>Low Vacuum (5”)</td>
<td>363.88</td>
<td>16.56</td>
<td>14.24</td>
<td>363.88</td>
<td>16.39</td>
<td>14.20</td>
</tr>
</tbody>
</table>

DMA tests indicate that there is not much variability in the Tg of the samples tested. It is in the range of 671°F (355°C) to 734°F (390°C). However, the storage modulus varies significantly between samples of the same part and between parts made under similar processing conditions. There is more consistency in the modulus for the parts made using lower infusion pressures. Figures 5.13 and 5.14 show the variation in the modulus between samples and parts at room temperature and at 300°C.
Table 5.2 DMA results divided by infusion method

<table>
<thead>
<tr>
<th>Storage Modulus Avg Value @ Full Vacuum (GPa)</th>
<th>Storage Modulus std dev @ Full Vacuum</th>
<th>Storage Modulus Variance @ Full Vacuum</th>
</tr>
</thead>
<tbody>
<tr>
<td>12.09</td>
<td>7.14</td>
<td>51.01</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Storage Modulus Avg Value @ Low Vacuum (GPa)</th>
<th>Storage Modulus std dev @ Low Vacuum</th>
<th>Storage Modulus Variance @ Low Vacuum</th>
</tr>
</thead>
<tbody>
<tr>
<td>16.06</td>
<td>2.26</td>
<td>5.14</td>
</tr>
</tbody>
</table>

The average value of storage modulus at room temperature for full vacuum infusion from the 12 samples (6 parts) tested is 12.09 GPa and the standard deviation is 7.14 GPa, while that of low vacuum infusion for the 10 samples (5 parts) tested is 16.06 GPa with standard deviation of 2.27 GPa. This reflects better process control for producing composite parts while using lower infusion pressures than when using higher pressures.
The variation in the dynamic mechanical properties of these VARTM samples can be attributed to the processing conditions that were used to make these parts. Any changes in these conditions affect the molecular weight distribution and microstructure of the samples. The test matrix in chapter 4 discusses the processing parameters used for each of the parts.

Besides processing conditions, any changes in the fiber/resin concentration, presence of voids and dry spots, fiber orientation has a significant impact on the storage modulus [32]. It must be noted that the samples are not cut from the same region of the laminate, which could have a bearing on the DMA properties. Lower vacuum infusion has better and more consistent storage modulus due to better flow of resin and subsequently better wetting of fiber. At full vacuum, although the fibers are wet faster, the vacuum tends to pull the resin out of the fibers after wetting.
Figure 5.14 Part-to-part and sample-to-sample variation in storage modulus at 300°C

The presence of air bubbles under the bag during infusion is much lesser for lower vacuum pressures, which impact the stiffness. Under full vacuum infusion, global wetting may have been reached while local wetting might not have occurred completely. The fiber orientation tends to get distorted when the resin is infused at higher pressures. This has an impact on the modulus. DMA tests require precisely cut samples, which are absolutely parallel and uniform in thickness. The sample preparation also contributes to the variability of the modulus between samples.
The eSEM images along the thickness of the samples under full vacuum infusion (Figure 5.15 (a) and (b)) indicates the presence of voids between the layers. These voids could be the reason for inconsistencies observed in the storage modulus between samples. Figure 5.15 (c) displays the eSEM image of RS #11, which is
produced under low vacuum with no voids, which means that the resin wets the fibers more uniformly and to a higher degree.

The evaporating vapors from methanol and water during processing add to the complexity of controlling the process. These vapors are pulled out of the bag by vacuum and it is observed that the elimination is best achieved by completely reducing the vacuum to the lowest possible level after infusion is complete. This ensures that the resin, whose viscosity is still low enough to be pulled out of the bag during processing, stays within the vacuum bag while the methanol vapors and water exit.

An interesting observation from the DMA curves for tan δ, which is the ratio of loss modulus (E”) and storage modulus (E’) [35], whose peak indicates the Tg of the composite laminates shows double peaks for samples RS #4, RS #5, RS #6 and RS #8. The second peak of all these samples are lower than the first peak except for sample RS #6. From literature review, the reasons behind the presence of separate peaks for tan δ in the DMA curve have been debatable. They vary from experimental factors [36] to movement of Tg of polymeric materials, which were not cured properly during the process. Studies have also shown that the second peak could be due to the existence of a resin region entrapped near the interface that was not cured to the same degree thus having a differing network structure, which caused this higher temperature thermal transition [39]. Xie et al [34] in his research involving the study of Thermal Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) results of IM7/RP-46 pre-pregs discovered the formation of cyclopentadiene between 644°F (340°C) and 752°F (400°C) during curing. The reason for the double peaks in some of the VARTM samples may be related to the inconsistent formation of this compound during curing and cross-linking of the monomers. Further research into the thermal behavior is needed to understand the existence of these double peaks.

It can be concluded from the DMA testing of the samples at lower infusion vacuum pressures help in reducing the variability of storage modulus and produce higher values of modulus on an average. However, it must be noted that the highest
modulus was produced under full vacuum (RS #4a). This clearly indicates lack of process control when infusing under high vacuum pressures. Further experimentation by means of a designed experiment is required to better understand the variability of the storage modulus.

5.1.1.3 Comparison of DMA results. The IM7/RP-46 autoclaved composite has a storage modulus of 50 GPa at room temperature with a Tg of 672°F (356°C). Since this composite is made using uni-directional intermediate modulus carbon fiber, its storage modulus tends to be higher due to fiber orientation. A storage modulus in the range of 25 to 30 GPa at room temperature should be a good target for a bi-directional fiber reinforced composite.

The VARTM samples under low vacuum pressure infusion have an average of 16 GPa for the storage modulus and the Tg is in the range of 671°F (355°C) to 734°F (390°C), which is comparable to the IM7/RP-46 composite. It must be noted that although the modulus is not close to being in the 25 to 30 GPa range, it has improved significantly during the course of experimentation. The variability of the modulus between samples has reduced.

5.1.2 Thermal Mechanical Analysis (TMA)
The TMA test is conducted to determine the co-efficient of linear thermal expansion (CTE) of the samples in the transverse direction using the TMA-2940 from TA instruments. The sample size for the TMA tests is approximately 3 * 3 mm in length and breadth. The sample is heated to 450°C at 20°C and allowed to cool to 40°C to equilibrate. This is done in order to eliminate residual thermal stresses in the sample. The second round of heating involves a ramp rate of 10°C/min to 500°C during which the CTE is measured. TMA test is carried out on the neat resin, IM7/RP-46 baseline composite and the VARTM samples are performed and compared.
5.1.2.1 Neat resin

The CTE for the neat resin in the transverse direction is shown in Table 5.3 and the Figure 5.16 displays the percentage change in sample thickness when the temperature is changed. The thickness of the sample is 0.339 mm.

Table 5.3 CTE for RP-46 neat resin at different temperatures

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>CTE (µm/m°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT – 100</td>
<td>31.53</td>
</tr>
<tr>
<td>100 – 200</td>
<td>48.73</td>
</tr>
<tr>
<td>200 – 300</td>
<td>73.51</td>
</tr>
<tr>
<td>300 – 400</td>
<td>331.27</td>
</tr>
</tbody>
</table>

Figure 5.16 TMA results for RP-46 neat resin
5.1.2.2 IM7/RP-46 baseline composite. The CTE for the baseline composite is shown in Table 5.4 and the Figure 5.17 represents the change in thickness as a function of temperature. The thickness of the sample is 1.3803 mm.

Figure 5.17 TMA results for IM7/RP-46 baseline composite

Table 5.4 CTE for IM7/RP-46 baseline composite at different temperatures

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>CTE (µm/m°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT – 100</td>
<td>-</td>
</tr>
<tr>
<td>100 – 200</td>
<td>25.68</td>
</tr>
<tr>
<td>200 – 300</td>
<td>31.39</td>
</tr>
<tr>
<td>300 – 400</td>
<td>40.11</td>
</tr>
</tbody>
</table>
5.1.2.3 VARTM samples. Eleven VARTM samples are tested to determine their CTE. Table 5.5 shows the CTE for the VARTM samples at different temperatures. The CTE is displayed in \( \mu \text{m/m/}^\circ\text{C} \), thickness in mm and the temperature in \( ^\circ\text{C} \).

Table 5.5 TMA test results for VARTM flat panel parts

<table>
<thead>
<tr>
<th>Sample #</th>
<th>RT – 100</th>
<th>100 – 200</th>
<th>200 – 300</th>
<th>300 – 400</th>
<th>Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS #1</td>
<td>18.07</td>
<td>40.85</td>
<td>59.11</td>
<td>117.42</td>
<td>3.013</td>
</tr>
<tr>
<td>RS #2</td>
<td>11.05</td>
<td>42.19</td>
<td>60.11</td>
<td>122.83</td>
<td>2.979</td>
</tr>
<tr>
<td>RS #3</td>
<td>26.27</td>
<td>34.88</td>
<td>52.47</td>
<td>125.37</td>
<td>2.521</td>
</tr>
<tr>
<td>RS #4</td>
<td>30.97</td>
<td>40.44</td>
<td>70.57</td>
<td>152.56</td>
<td>2.213</td>
</tr>
<tr>
<td>RS #5</td>
<td>27.30</td>
<td>35.63</td>
<td>51.64</td>
<td>108.71</td>
<td>2.848</td>
</tr>
<tr>
<td>RS #6</td>
<td>-2.47</td>
<td>0.80</td>
<td>31.67</td>
<td>132.20</td>
<td>2.630</td>
</tr>
<tr>
<td>RS #7</td>
<td>28.70</td>
<td>37.44</td>
<td>52.79</td>
<td>110.16</td>
<td>2.464</td>
</tr>
<tr>
<td>RS #8</td>
<td>22.53</td>
<td>45.21</td>
<td>68.73</td>
<td>138.23</td>
<td>2.738</td>
</tr>
<tr>
<td>RS #9</td>
<td>29.94</td>
<td>38.82</td>
<td>54.07</td>
<td>117.74</td>
<td>2.374</td>
</tr>
<tr>
<td>RS #10</td>
<td>33.22</td>
<td>41.30</td>
<td>60.93</td>
<td>142.26</td>
<td>1.926</td>
</tr>
<tr>
<td>RS #11</td>
<td>18.46</td>
<td>23.59</td>
<td>45.80</td>
<td>121.94</td>
<td>2.425</td>
</tr>
<tr>
<td>Average</td>
<td>22.19</td>
<td>34.65</td>
<td>55.26</td>
<td>126.31</td>
<td>2.56</td>
</tr>
<tr>
<td>Std Dev</td>
<td>10.52</td>
<td>12.57</td>
<td>10.77</td>
<td>13.69</td>
<td>0.33</td>
</tr>
<tr>
<td>Variance</td>
<td>110.72</td>
<td>157.89</td>
<td>116.07</td>
<td>187.54</td>
<td>0.11</td>
</tr>
</tbody>
</table>
Figure 5.18 CTE of the VARTM samples

Table 5.6 TMA results divided by infusion method

<table>
<thead>
<tr>
<th></th>
<th>100 C</th>
<th>200 C</th>
<th>300 C</th>
<th>400 C</th>
<th>Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Full Vacuum -</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td>18.53</td>
<td>32.47</td>
<td>54.26</td>
<td>126.52</td>
<td>2.70</td>
</tr>
<tr>
<td>Full vacuum - Std deviation</td>
<td>12.56</td>
<td>15.79</td>
<td>12.99</td>
<td>15.00</td>
<td>0.31</td>
</tr>
<tr>
<td>Full vacuum - Variance</td>
<td>157.75</td>
<td>249.40</td>
<td>168.83</td>
<td>225.06</td>
<td>0.09</td>
</tr>
<tr>
<td><strong>Low vacuum -</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td>26.57</td>
<td>37.27</td>
<td>56.46</td>
<td>126.07</td>
<td>2.39</td>
</tr>
<tr>
<td>Low vacuum - Std deviation</td>
<td>5.96</td>
<td>8.19</td>
<td>8.708</td>
<td>13.68</td>
<td>0.29</td>
</tr>
<tr>
<td>Low vacuum - Variance</td>
<td>35.55</td>
<td>67.21</td>
<td>75.83</td>
<td>187.38</td>
<td>0.09</td>
</tr>
</tbody>
</table>
Since, the working temperature of the VARTM composite is around 572 - 608°F (300 - 320°C), we concentrate on the CTE values from room temperature to 572°F (300°C). From Table 5.6, the low vacuum infused samples have a higher CTE than full vacuum samples. However, this large difference is mainly due to the negative CTE obtained for sample RS #6.

The sample RS #6 when heated during the DMA test displayed a unique double peak for tan δ curve where the first Tg peak is shorter than the second peak. The second Tg peak occurs at over 400°C. This sample has inconsistent curing as displayed by the tan δ curve, which could have impacted the thermal linear expansion coefficient.

If the sample RS #6 is omitted due to a special case, the average of the CTE values for full vacuum samples is 22.73 µm/m/°C which is close to the low vacuum average. Also, it should be noted that the variance of the CTE is far higher for the full vacuum samples compared to the low vacuum samples.

**5.1.2.4 Comparison of TMA results.** The CTE’s of the IM7/RP-46 baseline composite and VARTM samples are lower than that of the neat resin sample since the resin without any reinforcement expands linearly at a far greater rate under high temperatures than when fiber reinforcements are present.

At 200°C, the average CTE of the VARTM samples is higher than the baseline composite by 8.97 µm/m/°C and this difference is larger at 300°C where the average CTE of the VARTM sample is 55.26 µm/m/°C and that of the baseline composite is 31.39 µm/m/°C. Therefore, the transverse linear expansion of the VARTM composite sample is higher than the autoclaved composite and the type of infusion carried out does not significantly affect the CTE. However, the variation in CTE is higher when using full vacuum.
5.1.3 Thermal Gravimetric Analysis (TGA)

TGA analysis is done to characterize the weight loss as a function of temperature. It provides data related to thermal degradation of the composite sample when subjected to heat. An important aspect of HTPMCs is its ability to withstand high temperatures without undergoing substantial weight loss.

TGA tests are carried out on neat resin sample, IM7/RP-46 baseline composite sample and the VARTM samples. The test specimens are extremely small with dimensions of 1 * 1 mm in length and breadth respectively. The TGA graphs indicate the percentage weight loss at 200°C and 300°C, and the onset temperature, which is the temperature at which the probe of the equipment begins to drop vertically [33]. Since the onset temperature is much greater than the working temperature, the discussion does not consider the weight loss at onset point. The TGA tests are carried
out using the TGA-Q50 from TA instruments. The samples are heated at a 20°C/min to 900°C.

5.1.3.1 Neat resin. The weight loss profile of the neat resin sample is shown in Figure 5.20. At 200°C, 0.1639 % of weight loss occurs which increases to 0.1993% at 300°C.

![TGA test results for neat resin](image.png)

**Figure 5.20** TGA test results for neat resin

5.1.3.2 IM7/RP-46 baseline composite. Figure 5.21 shows the TGA graph for the IM7/RP-46 baseline composite. At 392°F (200°C), 0.7582 % of weight loss occurs which increases to 0.8418% at 572°F (300°C).
5.1.3.3 VARTM samples. Table 5.7 lists the weight loss percentages for the eleven VARTM samples at 200°C and 300°C. The TGA graphs that are obtained for each sample showed an initial drop in weight, which is typically associated with moisture loss after which the profile plateaus until the polymer degradation temperature is reached.
Table 5.7 TGA test results for VARTM flat panel parts

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Weight loss at 200°C (%)</th>
<th>Weight loss at 300°C (%)</th>
<th>Onset temperature (°C)</th>
<th>% Weight lost between 200 and 300°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS #1</td>
<td>0.9677</td>
<td>1.124</td>
<td>500.01</td>
<td>0.1563</td>
</tr>
<tr>
<td>RS #2</td>
<td>0.7891</td>
<td>0.9111</td>
<td>468.01</td>
<td>0.1220</td>
</tr>
<tr>
<td>RS #3</td>
<td>0.7404</td>
<td>0.8302</td>
<td>495.70</td>
<td>0.0898</td>
</tr>
<tr>
<td>RS #4</td>
<td>0.7257</td>
<td>0.7709</td>
<td>446.51</td>
<td>0.0452</td>
</tr>
<tr>
<td>RS #5</td>
<td>0.5508</td>
<td>0.6079</td>
<td>484.49</td>
<td>0.0571</td>
</tr>
<tr>
<td>RS #6</td>
<td>0.6440</td>
<td>0.7787</td>
<td>509.19</td>
<td>0.1347</td>
</tr>
<tr>
<td>RS #7</td>
<td>0.4321</td>
<td>0.4648</td>
<td>469.25</td>
<td>0.0327</td>
</tr>
<tr>
<td>RS #8</td>
<td>0.2201</td>
<td>0.2602</td>
<td>476.73</td>
<td>0.0401</td>
</tr>
<tr>
<td>RS #9</td>
<td>0.8666</td>
<td>0.8854</td>
<td>456.17</td>
<td>0.0188</td>
</tr>
<tr>
<td>RS #10</td>
<td>0.9127</td>
<td>0.9140</td>
<td>449.54</td>
<td>0.0013</td>
</tr>
<tr>
<td>RS #11</td>
<td>0.7672</td>
<td>0.7993</td>
<td>488.97</td>
<td>0.0321</td>
</tr>
<tr>
<td>VARTM Average</td>
<td>0.6924</td>
<td>0.7588</td>
<td>476.78</td>
<td>0.07</td>
</tr>
<tr>
<td>Std deviation</td>
<td>0.2205</td>
<td>0.2370</td>
<td>20.9111</td>
<td>0.0514</td>
</tr>
<tr>
<td>Variance</td>
<td>0.0486</td>
<td>0.0562</td>
<td>437.2726</td>
<td>0.0026</td>
</tr>
</tbody>
</table>

The RS #1 had the highest drop in weight between 392°F (200°C) and 572°F (300°C) followed by RS #6. RS #6 also had a negative CTE at room temperature as discussed in the section 5.1.2.3. Low vacuum samples also have very low change in weight between 392°F (200°C) and 572°F (300°C) as shown by the last column in Table 5.7. From Table 5.8, although the average weight loss percentage is lower for low vacuum samples at 392°F (200°C) and 572°F (300°C), the variability as indicated by the variance value is higher than full vacuum samples.
Figure 5.22 Weight loss for VARTM samples

Table 5.8 TGA results divided by infusion method

<table>
<thead>
<tr>
<th>Infusion Method</th>
<th>Weight loss at 200°C (%)</th>
<th>Weight loss at 300°C (%)</th>
<th>Onset temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Full Vacuum - Average</td>
<td>0.74</td>
<td>0.84</td>
<td>483.99</td>
</tr>
<tr>
<td>Full vacuum - Std deviation</td>
<td>0.14</td>
<td>0.17</td>
<td>23.19</td>
</tr>
<tr>
<td>Full vacuum - Variance</td>
<td>0.02</td>
<td>0.03</td>
<td>537.83</td>
</tr>
<tr>
<td>Low vacuum - Average</td>
<td>0.64</td>
<td>0.66</td>
<td>468.13</td>
</tr>
<tr>
<td>Low vacuum - Std deviation</td>
<td>0.30</td>
<td>0.29</td>
<td>15.80</td>
</tr>
<tr>
<td>Low vacuum - Variance</td>
<td>0.09</td>
<td>0.08</td>
<td>249.54</td>
</tr>
</tbody>
</table>
5.1.3.4 Comparison of TGA results.

![Graph showing weight loss comparison](image)

**Figure 5.23** Comparison of weight loss profiles

The comparison graph shown in Figure 5.23 indicates that there is not much difference in the weight loss profiles between the baseline composite and the VARTM samples. The average weight loss percentages of samples made under full vacuum are higher when compared to the overall average of the VARTM samples.

5.2 Mechanical Property Testing

5.2.1 Specimen preparation and setup

The samples were subjected to tensile loads under guidelines provided by ASTM standard D3039. Two samples were cut from each laminate (Figure 5.24).
The tensile testing was conducted using an MTS 810 tensile testing machine coupled with the teststar data acquisition and data analysis software package (Figure 5.25).
5.2.2 Tensile Strength

The formula used to calculate the tensile strength is shown in Equation 5.1.

\[ S = \frac{P}{bd} \quad \text{Eq - (5.1)} \]

Where, \( S \) = ultimate tensile strength, MPa
\( P \) = maximum load, N
\( b \) = width, mm
\( d \) = thickness, mm

Table 5.9 shows the tensile strength in MPa for two samples from the same VARTM part.

### Table 5.9 Tensile test data for eleven laminates

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Sample #a (MPa)</th>
<th>Sample #b (MPa)</th>
<th>Average (MPa)</th>
<th>Difference (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS #1</td>
<td>228.21</td>
<td>231.15</td>
<td>229.68</td>
<td>2.9</td>
</tr>
<tr>
<td>RS #2</td>
<td>202.16</td>
<td>246.84</td>
<td>224.50</td>
<td>44.6</td>
</tr>
<tr>
<td>RS #3</td>
<td>322.73</td>
<td>216.32</td>
<td>269.52</td>
<td>106.4</td>
</tr>
<tr>
<td>RS #4</td>
<td>420.91</td>
<td>424.10</td>
<td>422.51</td>
<td>3.1</td>
</tr>
<tr>
<td>RS #5</td>
<td>356.19</td>
<td>396.32</td>
<td>376.25</td>
<td>40.1</td>
</tr>
<tr>
<td>RS #6</td>
<td>376.77</td>
<td>374.47</td>
<td>375.62</td>
<td>2.3</td>
</tr>
<tr>
<td>RS #7</td>
<td>202.28</td>
<td>215.45</td>
<td>208.87</td>
<td>13.1</td>
</tr>
<tr>
<td>RS #8</td>
<td>317.63</td>
<td>241.75</td>
<td>279.69</td>
<td>75.8</td>
</tr>
<tr>
<td>RS #9</td>
<td>296.45</td>
<td>410.32</td>
<td>353.39</td>
<td>113.8</td>
</tr>
<tr>
<td>RS #10</td>
<td>373.25</td>
<td>284.83</td>
<td>329.04</td>
<td>88.4</td>
</tr>
<tr>
<td>RS #11</td>
<td>406.93</td>
<td>380.09</td>
<td>393.51</td>
<td>26.8</td>
</tr>
<tr>
<td>Average  (MPa)</td>
<td></td>
<td></td>
<td>314.78</td>
<td></td>
</tr>
<tr>
<td>Std deviation (MPa)</td>
<td></td>
<td></td>
<td>80.15</td>
<td></td>
</tr>
<tr>
<td>Variance</td>
<td></td>
<td></td>
<td>6425.15</td>
<td></td>
</tr>
</tbody>
</table>
The test specimens under progressive tensile load and deformation failed due to fiber breakage. The tensile samples are brittle and lack toughness, which is a characteristic of carbon fiber based composite. The samples had catastrophic failure during breakage without any warnings.

![Tensile Strength Graph](image)

**Figure 5.26** Comparison of tensile strength

The average tensile strength from eleven VARTM samples is 314 MPa. However, there is huge variability in the tensile test results with standard deviation of 80.15 MPa. Figure 5.26 indicates the variability of tensile strength in the samples of the same laminate and between other laminates tested. The line – variability between samples shown in the figure reflects the difference of the tensile strength between the two samples of the same laminate. It can be seen that the variability is the highest for RS #9 followed by RS #3. Samples RS #8 and RS #10 also have very high variability.
between samples. RS #8, RS #9 and RS#10 were made using a vacuum infusion pressure of 10” of Hg. However, the variation of tensile strength between different laminates is greater for samples processed under full vacuum as indicated by Table 5.10. The average tensile strength for parts under full vacuum is higher than those under low vacuum infusion process.

Table 5.10 Tensile strength data divided by infusion method

<table>
<thead>
<tr>
<th>Infusion Method</th>
<th>Average</th>
<th>Std deviation</th>
<th>Variance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Full Vacuum</td>
<td>316.35</td>
<td>85.54</td>
<td>7318.14</td>
</tr>
<tr>
<td>Low Vacuum</td>
<td>312.90</td>
<td>77.720</td>
<td>6040.42</td>
</tr>
</tbody>
</table>

This indicates that the VARTM samples at low vacuum infusion lack repeatability of tensile strength within the same laminate and to a certain extent reproducibility between separate laminates as well. And high vacuum infusion laminates lack reproducibility of tensile strength between separate parts.

5.2.3 Tensile Modulus

The formula used for calculating the modulus is shown in Equation 5.2.

\[
E = \left( \frac{\Delta P}{\Delta l} \right) \left( \frac{l}{bd} \right)
\]

Eq - (5.2)

Where, \( E = \) modulus of elasticity, MPa

\( \left( \frac{\Delta P}{\Delta l} \right) \) = slope of the plot of load as a function of deformation within the linear portion of the curve
Table 5.11 shows the tensile modulus in GPa for two samples from the same VARTM part.

**Table 5.11 Tensile modulus for eleven laminates**

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Sample #a</th>
<th>Sample #b</th>
<th>Average</th>
<th>Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS #1</td>
<td>49</td>
<td>46.22</td>
<td>47.61</td>
<td>2.78</td>
</tr>
<tr>
<td>RS #2</td>
<td>37.82</td>
<td>27.75</td>
<td>32.785</td>
<td>10.07</td>
</tr>
<tr>
<td>RS #3</td>
<td>68.248</td>
<td>-</td>
<td>68.248</td>
<td>0</td>
</tr>
<tr>
<td>RS #4</td>
<td>71.54</td>
<td>57</td>
<td>64.27</td>
<td>14.54</td>
</tr>
<tr>
<td>RS #5</td>
<td>54.18</td>
<td>54.806</td>
<td>54.493</td>
<td>0.626</td>
</tr>
<tr>
<td>RS #6</td>
<td>63.56</td>
<td>85.49</td>
<td>74.525</td>
<td>21.93</td>
</tr>
<tr>
<td>RS #7</td>
<td>41.72</td>
<td>37.04</td>
<td>39.38</td>
<td>4.68</td>
</tr>
<tr>
<td>RS #8</td>
<td>64.55</td>
<td>83.46</td>
<td>74.005</td>
<td>18.91</td>
</tr>
<tr>
<td>RS #9</td>
<td>98.99</td>
<td>101.106</td>
<td>100.048</td>
<td>2.116</td>
</tr>
<tr>
<td>RS #10</td>
<td>66.37</td>
<td>71.33</td>
<td>68.85</td>
<td>4.96</td>
</tr>
<tr>
<td>RS #11</td>
<td>55.59</td>
<td>64.86</td>
<td>60.225</td>
<td>9.27</td>
</tr>
<tr>
<td><strong>Average (GPa)</strong></td>
<td></td>
<td></td>
<td><strong>61.93</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Std deviation (GPa)</strong></td>
<td></td>
<td></td>
<td><strong>19.43</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Variance</strong></td>
<td></td>
<td></td>
<td><strong>377.60</strong></td>
<td></td>
</tr>
</tbody>
</table>

The tensile modulus is measured for the initial slope of the stress-strain curve at the elastic region where the proportionality of stress-strain is at its maximum. The sample b of RS #3 had inaccurate strain values due to which the modulus could not be reported.
The VARTM samples did not produce modulus values that could be obtained from carbon fibers. The variability between samples and within the same sample is high. Table 5.12 indicates this variability in the variance values obtained for low vacuum infusion pressure samples when compared to the full vacuum pressure samples. Therefore, the type of infusion method does not affect the tensile modulus. This variability may be caused by other factors – vacuum bag setup, post infusion pressure, inconsistent curing that is causing part-to-part variation.
Table 5.12 Tensile modulus data divided by infusion method

<table>
<thead>
<tr>
<th>Infusion Method</th>
<th>Average</th>
<th>Std Deviation</th>
<th>Variance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Full Vacuum</td>
<td>55.96</td>
<td>16.12</td>
<td>259.73</td>
</tr>
<tr>
<td>Low vacuum</td>
<td>68.50</td>
<td>21.43</td>
<td>459.06</td>
</tr>
</tbody>
</table>

Figure 5.28 compares the tensile strength with the modulus for the part. There is no trend that the graph indicates. However, the highest modulus is obtained for sample RS #8 which is processed under low vacuum pressure. The RS #4 which has the highest tensile strength reported a poor modulus.

Figure 5.28 Comparison of tensile strength and modulus
5.2.4 Benchmarking of tensile test results

It is known widely that the tensile strength and modulus of plain-woven fabric reinforced laminate are lower than those of the equivalent cross-ply laminate. This is attributed to the presence of fiber undulation in woven fabrics where the fiber yarns in one direction crisscross the yarn in other direction. The straitening out of these crimped fibers under tensile loading creates high stress in the matrix. As a result micro-cracks are formed in the matrix at relatively low loads. The mechanical handling during the weaving process of woven fibers tends to further reduce the tensile strength.

Table 5.13 Tensile strength comparison

<table>
<thead>
<tr>
<th></th>
<th>MPa</th>
<th>ksi</th>
</tr>
</thead>
<tbody>
<tr>
<td>Woven Carbon/RP-46 with VARTM</td>
<td>314.78</td>
<td>45.65</td>
</tr>
<tr>
<td>IM7/RP-46 baseline composite [23]</td>
<td>2627.04</td>
<td>381</td>
</tr>
<tr>
<td>Woven T650-35/RP-46 [23]</td>
<td>608.14</td>
<td>88.2</td>
</tr>
</tbody>
</table>

Table 5.13 compares the tensile strength obtained for the VARTM samples with those obtained from Unitech Corporation for the autoclaved sample [23]. It clearly indicates that the tensile properties obtained for the VARTM parts are lower than the autoclaved parts for woven fabric as well.

The inconsistencies and the lower values for the VARTM samples can be a result of several experimental factors, which were not considered during the experimentation such as, the heat rate of the hot plate which could not be controlled very accurately thus subjecting the processing to variations during the curing process.

96
which could be the single biggest cause for part-to-part variation. The imidization process is extremely sensitive to heat variations due to several side reactions occurring along with the main curing propagation reaction. Any variation in heat rate occurring during the process would affect the polymerization of monomers to poly(amic) acid and finally to polyimide.

It must also be noted that the quality of the fibers used while making the parts with VARTM might not have been the best due to age, mechanical handling over the years in storage and moisture absorption which definitely affects the final tensile properties of the parts. During manufacturing of carbon fibers, they are subjected to oxidative treatment in air and plasma to modify the surface. They are also sized with organic polymers for better adhesion with the matrix. This deteriorates over a period of time, which could be the case with the fibers used with the VARTM samples.

The experiments that were carried out tried to eliminate variations in factors to discern clear trends in data. However, the curing process may have introduced variability, which could not be controlled. Hence, more experimental runs with controlled curing are needed along with better fibers to determine the exact tensile properties of the laminates under VARTM processing.
CHAPTER 6

CONCLUSION

6.1 Summary

The primary objective of this research thesis was to demonstrate the processability of VARTM parts using the RP-46 polyimide resin system. Processability implies, identification of appropriate vacuum bagging materials and heating devices, demonstrating the usability of the resin with the process, understanding process capability, identifying key processing parameters for improvement in important characteristics such as resin flow, wetting and fiber weight and volume fraction. The learning curve from initial trials, which produced laminates of poor quality in terms of dry spots and surface distortions to VARTM parts with good surface quality provided a challenging experience. The focus was clearly on producing good parts consistently, which is again a part of processability.

Once it was clearly established that RP-46 could be used with VARTM, the thesis work moved towards manufacturing of flat panel composites using different manufacturing methods. The changes in the operating parameters were done based on data collected from tests performed on the previous samples. Since, this thesis report is only a the preliminary study of the use of RP-46 with low-cost VARTM, a detailed and more sophisticated design of experiments (DOE) was not carried out. However, the results from various tests reported in this report gives a clear indication to what the significant factors might be and their levels.

The major issue with the VARTM processed part is the presence of voids in the structure and poor wetting of the fiber on the surface which could be a result of
exiting methanol and water vapors. However, it must be stated that the quality of the laminates was significantly better when infused under lower vacuum. This can be substantiated by the DMA test results, which indicate lesser variability within samples. This is due to uniform flow of resin under the bag, wetting all areas of the laminate unlike full vacuum infusion. The variability between parts made with lower vacuum pressures is small compared to the high vacuum infusion category. Lower vacuum pressures result in better infusion thus producing good viscoelastic properties as measure by the DMA tests. The Tg of the VARTM samples obtained is comparable to the IM7/RP-46 baseline composite. Summary of the test results shown in Figure 6.1 (weight loss indicated in secondary axis) clearly indicates the need for a properly designed experiment to be carried to measure the variation sources for CTE, weight loss profiles and tensile strength, since the experiments produced equal variation in samples for low and full vacuum.

Figure 6.1 Overall comparisons of test results
6.2 Future work

A very important part of establishing the parameters for any process is to carry out a design of experiments study, which would identify the critical factors that affect particular responses. This should be a part of future work in this area if RP-46 is to be used with VARTM on a larger scale. Moreover, this kind of a study would enable the researcher to deep dive into the cause and effects of particular changes to parameters and link them with specific responses.

The chemistry of polyimides is complex with a number of monomers available for polymerization under heat. The imidization reaction involves a number of changes in the resin chemistry, which leads to the emission of methanol and water vapors. These emissions could cause voids on the surface of the laminates. A new process setup, which can enable easier removal of these vapors without affecting the part, should be investigated. The incorporation of an additional vacuum line that is activated during the vapor emission could be a solution.

The study carried out in this thesis involved making the laminates under one common curing strategy. The Tg of the composite gives an indication to the level of curing. The Tg of the VARTM samples is in the region of 680°F (360°C), which is still slightly lower than the true potential of the resin. Alternate curing strategies must be investigated for optimum Tg. Also, further research into the occurrence of double peaks in the DMA curves for tan δ should be investigated.

Finally, the use of RP-46 with VARTM will depend mainly on the ability to demonstrate repeatability in process and obtain consistency in stiffness, dimensional stability under elevated temperatures, thermo-oxidative stability and mechanical strength. The baseline for further improvement has been set in this thesis report.
APPENDIX A

Vacuum bagging supplies

Figure A.1 Wrightlease 5900 T.O.S (WL5900 TOSC)

Figure A.2 Teflon High permeable layer (HPL)
Figure A.3 Wrightlease 5900 (WL5900)

Figure A.4 Bleeder Lease E
Figure A.5 Vacuum bag sealant tape (A-800-3G)

Figure A.6 Pressure sensitive tape (AIRKAP 1)
Figure A.7 Aluminum mold plate

Heating devices

Figure A.8 Vulcan programmable oven
Figure A.9 Hot plate

Miscellaneous supplies

Figure A.10 Valves, hoses and connectors
Figure A.11 Pressure gauge

Figure A.12 Vacuum pump
Figure A.13 Resin collection area

Figure A.14 Temperature measuring laser gun
Figure A.15 Fiber cutter

Figure A.16 Operator safety equipment
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BIOGRAPHICAL SKETCH

Thammiah Prasad is currently a master’s student in Industrial Engineering at FAMU-FSU College of Engineering and expecting to graduate in April of 2004. He was born in August of 1978, to Mr. and Mrs. Prasad Nanaiah. He received his Bachelor’s of Engineering in Industrial Engineering in August 2000 from Bangalore University at Bangalore, India. His research interests include composites manufacturing, production management and quality. In the immediate future, he is looking forward to pursuing a career in the field of Industrial Engineering.