Development of a High-Temperature Vacuum Assisted Resin Transfer Molding Testbed for Aerospace Grade Composites

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Submitted by

Dr. Ben Wang

Florida Advanced Center of Composite Technologies (FAC^2T)

Florida A &M University-Florida State University College of Engineering

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1. Introduction

Composite materials are lightweight and strong, providing a winning combination that propels composites to new areas of respect in the manufacturing industry. Manufacturers are attempting to find more applications where composites can be used. For instance, composite materials are being looked at for use as replacements for conventional materials in high-temperature applications. Traditionally, hand lay-up prepreg/autoclave techniques have been used to fabricate polymeric composites for applications that require high-temperature resistance. Yet, high manufacturing costs have limited their usage. In recent years, vacuum assisted resin transfer molding (VARTM) has proven to be an affordable process for manufacturing large composite structures, when compared to conventional RTM and hand lay-up prepreg/autoclave techniques. Manufacturing high-quality, large composite parts for high temperature applications using VARTM technique will significantly reduce costs due to low cost tooling, flexible part integration and larger part dimension.

Traditionally, VARTM has been used for making composites for normal temperature applications. A thorough understanding of material and processing behaviors is a prerequisite for successfully applying a high temperature (HT)-VARTM process. Processing high temperature (≥350°F) composites using VARTM faces some technical challenges due to its inherent low processing pressure (maximum full vacuum or 14.7 psi), the high viscosity of popular high-temperature resin matrices (such as bismaleimides), and the processing difficulty of eliminating solvents during processing (for PMR type polyimide such as PMR-15). Furthermore, high processing temperatures, low processing pressures, potential preform spring-backs and large part sizes may lead to large dimension variations. Tight dimension tolerances are critical for producing net-shaped parts, eliminating second processing and achieving affordable aerospace applications.

Connell *et al.* prepared and characterized high performance imide resins with a combination of properties that are particularly useful for the fabrication of composite

parts via RTM and/or resin infusion (here called VARTM) [3]. Thereafter Connell and Criss *et al.* fabricated carbon fiber reinforced composites via RTM processes using recently invented imide resins PETI-298, PETI-330 and PETI-375 and determined their thermal and mechanical properties [3-8]. It was found that these imide resins show the combination of processability for RTM and high performance.

The goal of the HT-VARTM research is to develop a testbed that incorporates in-situ optical thickness monitoring, vacuum and temperature control to demonstrate the processing capability of manufacturing dimensionally stable composites of high temperature vacuum assisted resin transfer molding (HT-VARTM) process.

This report details the research efforts by the Florida Advanced Center for Composite Technologies (FACCT) research team at the Florida A&M University-Florida State University College of Engineering (FAMU-FSU CoE) in developing the HT-VARTM process and the testbed.

1.1 Vacuum Assisted Resin Transfer Molding (VARTM) Overview

VARTM is a low cost processing method by which composite materials are processed, and in which a vacuum is the only driving force for liquid resin transfer, and also provides the consolidation pressure for the entire part. Recently, VARTM has shown great potential in its low cost and environment friendly characteristics, especially when fabricating large-scale structures used in ship/boat and automobile industry.

The general sequence of events that comprises VARTM is illustrated in Figure 1.1-1. One general set-up idea is that resin is infused into a center point in the laminate. Resin is drawn through the fabric preform via vacuum pressure. Generally, only a single-sided mold is used, with a flexible vacuum bag on the topside of the part. Typically a matched tooling system is used, which allows integrated structures to be formed. The flow medium is placed on top of the part to direct the resin flow and increase flow rate. Peel ply must be used to tear the flow medium and vacuum bag apart and obtain parts

with good surface finishs. The final arrangement of materials should look similar to Figure 1.1-2.

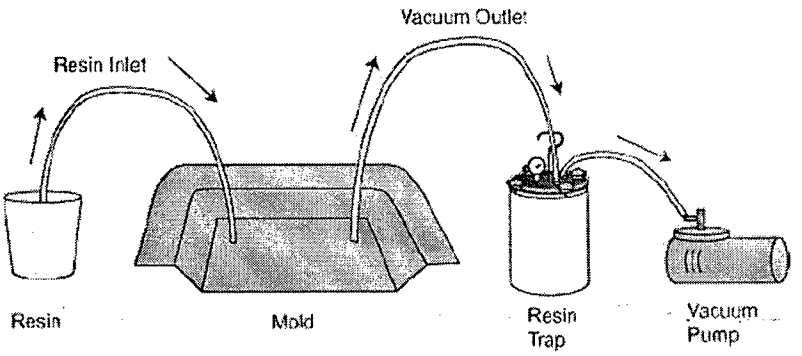


Figure 1.1-1 General sequence of events in VARTM

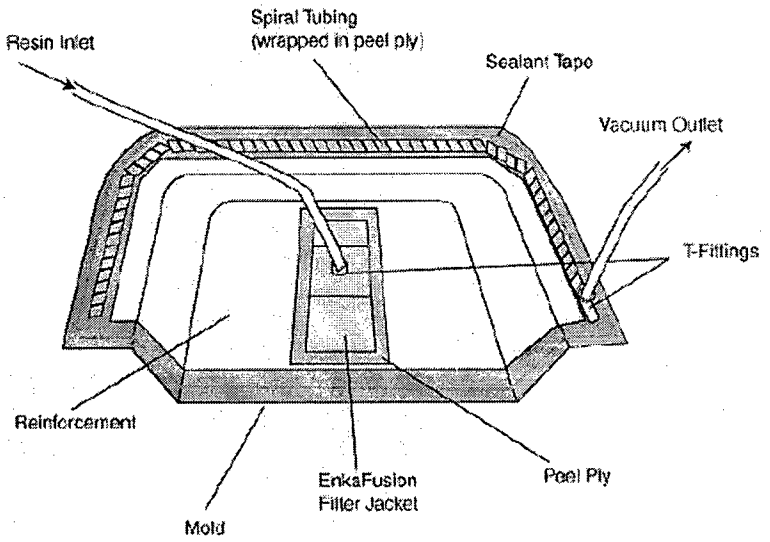


Figure 1.1-2 Typical set-up of VARTM

Compared to other composite processes, VARTM has many advantages such as low processing pressure (14.7 psi or less), suitability for large and/or integrated structures fabrication, only using single-sided molds, and lower cost compared to prepreg/autoclave technique or RTM.

1.2 Research Approach

The objective of the project is to develop a HT-VARTM testbed to demonstrate the processing capacity of manufacturing dimensionally stable composites. The testbed features a programmable vacuum and temperature control and an in-situ part dimension measurement. Figure 1.2-1 shows a schematic of the HT-VARTM testbed.

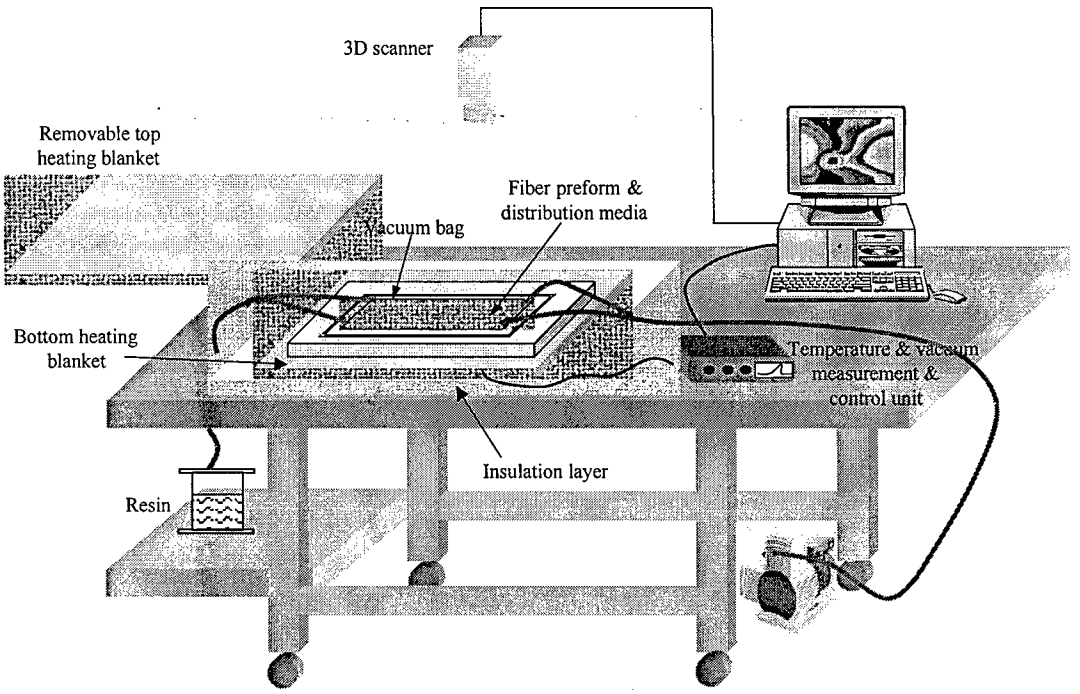


Figure 1.3-1 Schematic of the proposed HT-VARTM testbed

The testbed integrates in-situ monitoring of dimensional variation along the thickness direction, on-line monitoring and control of vacuum level and temperature, and liquid resin infusing and curing. A 3D scanner is used to monitor any dimension variations. Flexible heating blankets raise the mold temperature to help wet out the fiber preform by resin and cure the part. A control system can monitor and control the vacuum provided by a pump. The system can also control the temperature of the whole set-up to

facilitate the resin flow and cure cycle. The data and information of dimensional variation is stored in the computer during the process. The heating and vacuum histories are recorded during the process.

The technical activity for this project consists of four tasks: (1) Material selection and analysis, (2) HT-VARTM testbed development, (3) HT-VARTM flow and curing experiments, and (4) Testing and ccharacterization. The following sections discuss the details of the four tasks performed in this project.

2. Task I: Materials Selection

2.1 Fiber System

Originally IM-7 carbon fiber was selected to be the reinforcing materials. However, an out-gassing issue was discovered by AFRL scientists when processing above 450°F. Out-gassing from the fiber may lead to significant micro-cracks and porosity in the composites. The research team switched to the T650/35 8HS fabric purchased from Fabric Development Inc. Figure 2.1-1 shows the T650/35 8HS fabric, and Figure 2.1-2 shows the magnified view of the T650/35 8HS fabric.

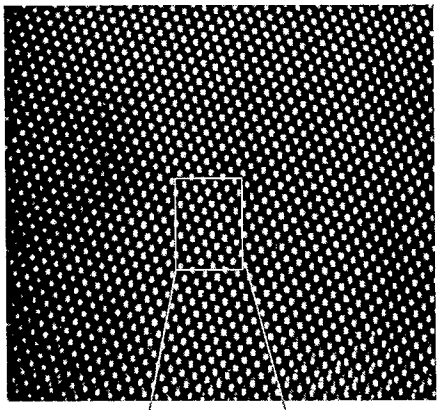


Figure 2.1-1 T650-35 8HS fabric

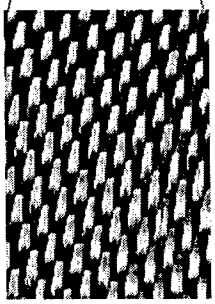


Figure 2.1-2 Magnified view of T650-35 8HS

T650-35 fibers are continuous, no-twist carbon filaments made from PAN precursor, surface treated to improve handling characteristics and structural properties. The filament count was 3000 filaments/tow. The typical tensile modulus was 35×10^6 psi. Typical tensile strength was 650,000 psi. Table 2.1-1 provides the material identification and average properties.

Table 2.1-1 Material identification and average properties of carbon fabric T650/35

Yarn Type	T650/35 3K 309 NT
Weave	8 Harness Satin
Weight (Oz./Sq. Yd)	11.0
Thickness (inches)	0.024

In order to evaluate the ease-of-flow during infusion process, the research team at the Florida Advanced Center for Composite Technologies (FACCT) at the FAMU-FSU College of Engineering performed the permeability test on the T650-35 fabric using linear injection. The permeability of T650-35 fabric was measured to be 1.29×10^{-10} m^{-2}. Results shown in Figure 2.1-3 indicate the suitability of T650-35 carbon fabrics to VARTM processing.

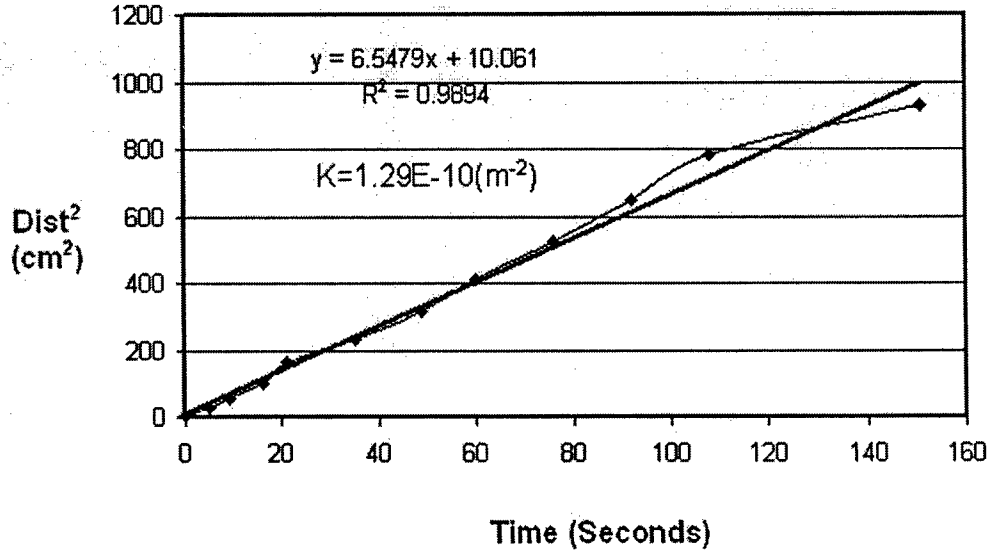


Figure 2.1-3 Permeability test results of T650-35 fabric

2.2 Resin System

High performance/high temperature composites are potentially useful on advanced aerospace vehicles in structural applications and as aircraft engine components, such as inlet frames and compressor vanes. Work on high temperature transfer molding resins was initiated in the late 1990s as part of NASA's High Speed Civil Transport Program [9-11]. At that time, the thermal performance requirement for structural composites was excellent retention of un-aged mechanical properties after 60,000 hours at 177°C. Several materials were developed that exhibited cured glass transition temperatures in the 230-250°C range and an acceptable combination of processability (e.g. low and stable melt viscosity), thermal and mechanical performance. Although different chemistries were investigated, the resin chemistry that exhibited the best combination of processability, performance, cost and practicality was based on phenylethynyl terminated imide oligomers (PETI). However, when the HSCT program ended in 1999, there were no applications for these materials. Recently, interest has been expressed by the aerospace industry in high temperature matrix resins that are processable by methods requiring low melt viscosity and have no volatiles [8].

Currently, the work has focused on increasing the use temperature to >300°C by increasing the cured glass transition temperature without sacrificing processability or toughness. PETI-330 was developed at NASA Langley Research Center and has shown potential in composite applications requiring high temperature performance combined with the ability to be readily processed without the use of autoclave or complex cure or post-cure cycle. This material is particularly useful for the fabrication of high-temperature structures for jet-engine components, structural components on high-speed aircraft, spacecraft, and missiles. The following sections will discuss the characteristics and status, synthesis and chemistry of PETI-330, and its processing, physical and composite properties. PETI-330 was purchased from UBE America, Inc.

2.2.1 Characteristics

Compared to typical and outstanding PMR polyimide PMR-15, PETI-330 demonstrates superior characteristics in regard not only to mechanical and ageing properties of composite with carbon reinforcement, but also to the processing suitability to liquid composite molding techniques. Figure 2.2.1-1 shows the PETI-330 powder resin.

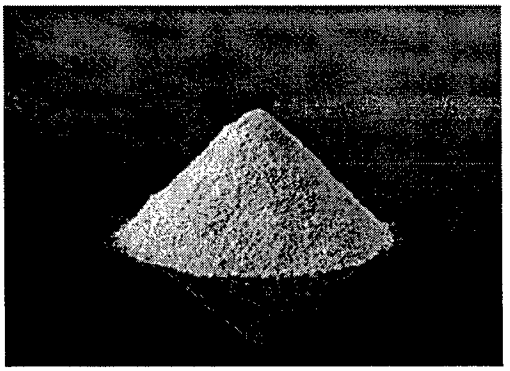


Figure 2.2.1-1 PETI-330 powder resin

The unique characteristics include: (1) major advancement in materials technology combining unprecedented processing characteristics with long term (1,000 hours) performance at 300°C, (2) the combination of easy processing for composite fabrication combined with high temperature performance and toughness, (3) low and stable melt viscosity highly suitable to VARTM, (3) simple and efficient one hour full cure process, (4) high glass transition temperature 330°C/626°F, (5) solvent free (no volatiles), and (6) non toxic.

2.2.2 Synthesis of PETI-330

Using the methods outlined by Connell, *et al.* in [3], PETI-330 was synthesized following the procedures below.

Researchers placed 1,3,4-APB (374 g, 1.28 moles), 1,3-PDA (138 g, 1.28 moles) and NMP (750 g) into a 7 L reaction kettle equipped with a mechanical stirrer, thermometer and nitrogeninlet/outlet. The mixture was stirred for ~1 hour to dissolve the diamines. A slurry of a-BPDA (399 g, 1.36 mole) and PEPA (595 g, 2.4 moles) in NMP (1000 g) was added. Additional NMP (1765 g) was used to rinse all of the anhydrides, resulting in a 30% solids (w/w) mixture. Upon addition of the anhydride/NMP slurry, the reaction temperature increased to ~75°C. The mixture was stirred for ~4 h (all solids had not dissolved at this point). Toluene was added (300 mL) and the reaction kettle was fitted with a Dean Stark trap and reflux condenser. The mixture was heated to reflux (~185°C) and refluxed overnight. The next day, toluene was removed from the system via the Dean Stark trap (the reaction temp eventually reached ~205°C during the toluene removal) and the reaction solution was allowed to cool to ~75°C. The warm solution was filtered through a coarse porosity, sintered glass funnel and poured into water in a blender. The solid was isolated by filtration, washed in warm water three successive times. The solid was air-dried overnight at room temperature and subsequently dried in a forced air oven at 135°C for ~24 h (until constant weight was achieved) to give a quantitative yield of yellow powder (1415 g, 100%).

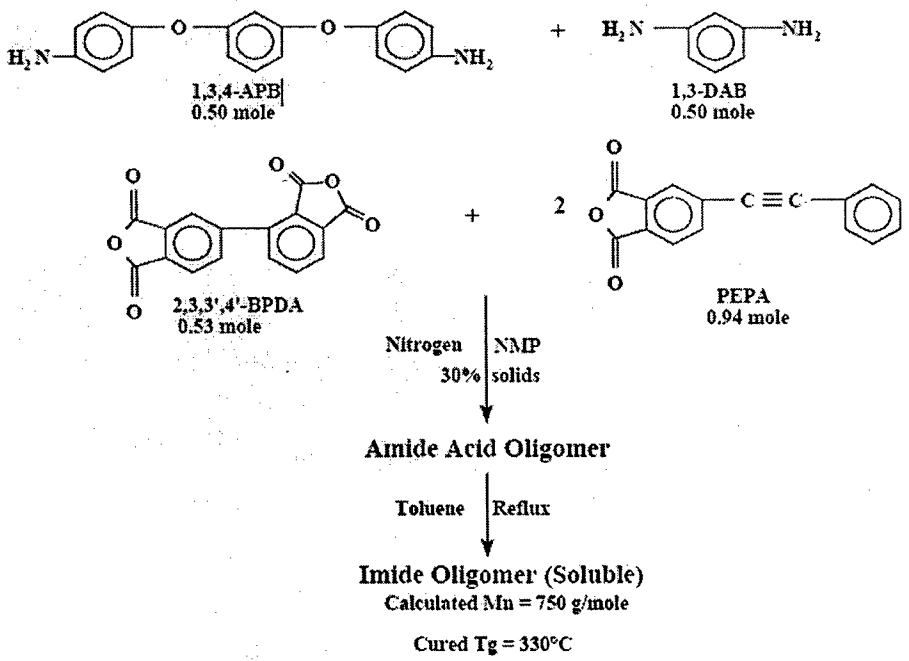


Figure3.2.2-2: Synthesis of PETI-330

2.2.3 Rheology

Dynamic rheological properties were measured using PETI-330 discs compression molded. The test chamber of the rheometer was at room temperature prior to specimen introduction. The specimen was heated from 23° to 280°C at a heating rate of 4°C/min and held for 2 hours to assess melt stability. The sample was then heated to 371°C at the same heating rate and held for 0.5 h in air. The result, as is shown in Figure 2.2.3-1, was initially complex melt viscosities (η^*) after 2 hrs at 280°C. From Figure 2.2.3-1, PETI-330 showed a low viscosity when heated to 280°C and remained stable as long as 2 hours when maintained at this temperature. While at the process of being heated to 371°C, the resin viscosity initially decreased, showing the effect of increasing temperature on the mixture of compounds, but then increased very quickly to >1000 Pa•s due to the increasing rate of polymerizations as temperature increased.

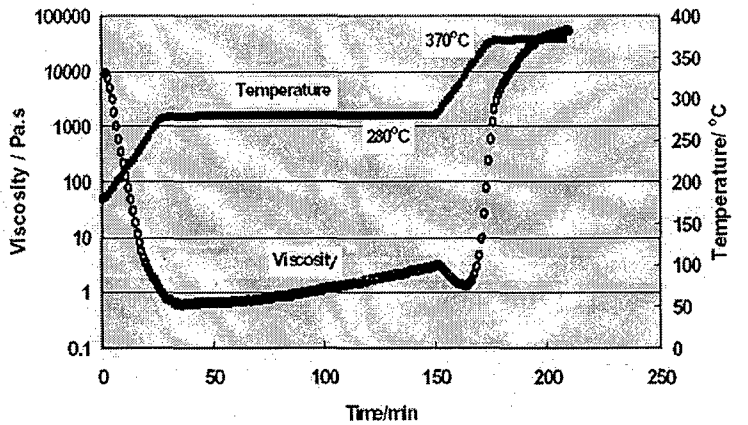


Figure 2.2.3-1. Melt viscosity vs. temperature curve of PETI-330
(Courtesy of Dr. Connell at NASA LaRC)

To verify the rheological properties of PETI-330, the research team at FACCT also performed dynamic rheological tests using similar test sample sizes and under the same conditions as research conducted at NASA LaRC. Figure 2.2.3-2 shows the melt

viscosity vs. temperature of PETI-330. Results shown in Figure 2.2.3-1 and 2.2.3-2 verify the VARTM processability of PETI-330.

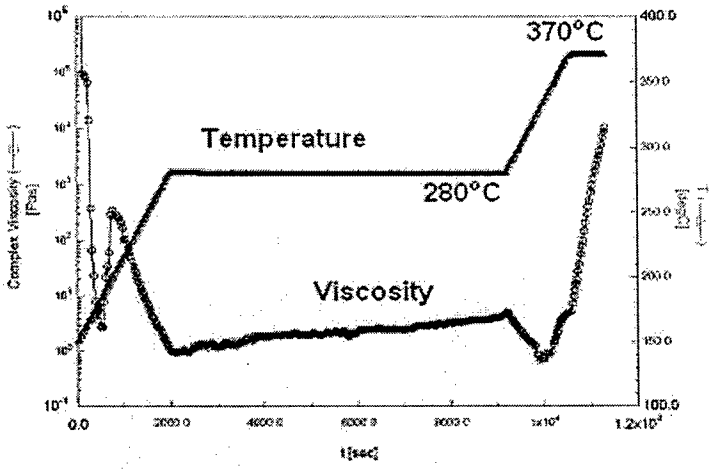


Figure 2.2.3-2. Melt viscosity vs. temperature curve of PETI-330
(*Measured by FAMU-FSU College of Engineering Research Team*)

2.2.4 Thermal Properties of PETI-330

Connell *et al.* characterized thermal properties of PETI-330 in Ref [3]. The number 330 refers to the cured T_g of the neat resin. The T_g was obtained from neat resin powder cured for 1 h at 371°C in an aluminum pan in a DSC cell. The experiment was conducted at a heating rate of 20°C /min, and the T_g was reported at the mid-point of the inflection of the ΔH versus temperature curve. Under these conditions the baseline deflection associated with the T_g can span a temperature range of 15–25°C. Deviations in the reported cured resin T_g values are expected when different measurement techniques and different heating rates are used. The characterization of the cured resin T_g was determined by three different techniques, which are presented in Table 2.2.4-1. In some cases, the uncured neat resin powders exhibited transient crystalline melting transitions on initial heat-up by DSC. These transitions occurred at temperatures well below the cure temperature and cannot be recovered by annealing. For the TMA and DMTA

experiments, the neat resin, cured for 1 h at 371 °C, was obtained directly from the RTM tool after laminate fabrication.

Table 2.2.4-1 Characterization of neat resin [3]

Resin	T_g, °C by DSC[a]	T_g, °C by TMA[b,c]	T_g °C by DMTA[b,d]
PETI-330	330	313	326

NOTES

[a] Obtained on neat resin powders after 1 hour at 371 °C in an aluminum pan at a heating rate 20 °C/min.

[b] Cured resin obtained from inside RTM tool, cured for 1 hour at 371 °C

[c] TMA heating rate 10 °C/min

[d] DMTA heating rate 10 °C/min, frequency 0.1–10 Hertz

2.2.5 Mechanical Characterization of Laminates

Connell *et al.* provided the mechanical properties of PETI-298, PETI-330 and BMI-5270 on T650-35 fabric [3]. The laminates were made with eight plies of unsized T650-35 8HS carbon fabric with a $(0/90)_{4S}$ lay-up (quasi-isotropic). Fabric sizing was removed by heating the fabric at 400°C for 2 hours under a vacuum prior to insertion in the tool. The PETI powder was charged in the resin chamber, heated to 280°C and de-gassed prior to injection into the tool. Laminate fabrication involved injecting the molten resin at ~280°C into the preheated tool followed by a cure at 371 °C for 1 h under ~1.4 MPa hydrostatic pressure.

The mechanical properties of PETI-298, PETI-330, and BMI-5270 on T650-35 fabric are presented in Figures 2.2.5-1. The properties of the BMI-5270, a RTM processable matrix resin from Cytec Engineered Materials, are included for comparison purposes.

The open hole compression (OHC) strengths and moduli at room temperature and 288°C are presented in Figures 2.2.5-1 and 2.2.5-2, respectively. The room temperature OHC strengths of all three were comparable. When tested at 288°C, the PETI-330/T650-35 laminates exhibited the highest OHC strength, followed by PETI-298 and the BMI-5270. When tested at 288°C, the PETI-330 exhibited a retention of ~74% of room temperature OHC strength, followed by PETI-298 (~69%) and then BMI-5270 (~60%). The retention of OHC modulus at 288 °C was higher for PETI-298 (91% of RT modulus) and PETI-330/T650-35 (92% of RT modulus) laminates as compared to the BMI-5270/T650 (75% of RT modulus) laminates.

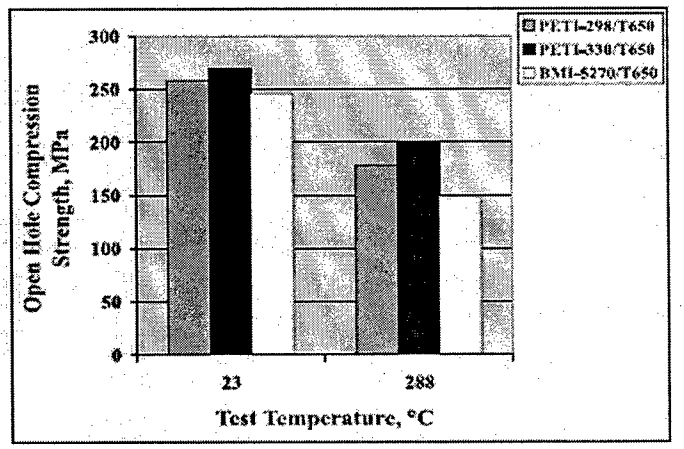


Figure 2.2.5-1 Open hole compression strength of T650-35 laminates at 23 and 288°C [3]

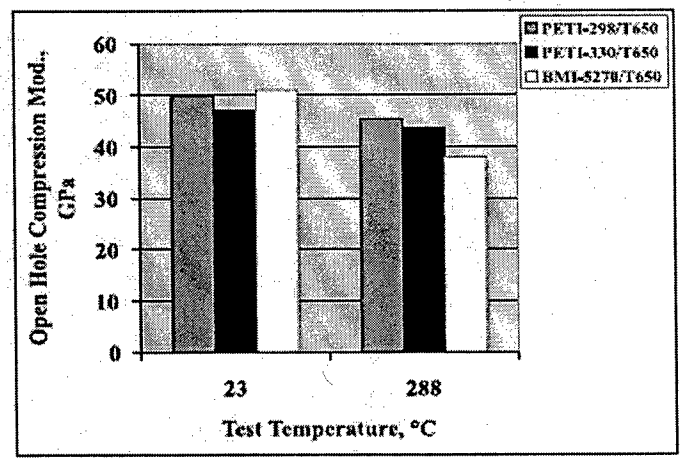


Figure 2.2.5-2 Open hole modulus of T650-35 laminates at 23 and 288°C [3]

The SBS strengths as a function of temperature are presented in Figure 2.2.5-3. The SBS strengths of PETI-330/T650 and the BMI-5270/T650-35 were compared. As expected, the PETI-330/T650-35 exhibited significantly higher SBS strengths at RT and elevated temperature. These specimens retained 62% of their RT SBS strength at 288°C. The RT UNC strengths for PETI-298, PETI-330 and BMI-5270 T650-35 laminates were 457, 520 and 356 MPa, respectively.

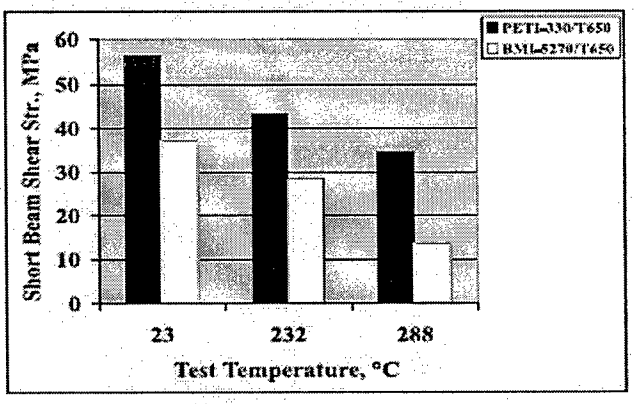


Figure 2.2.5-3 Short beam shear strength of T650-35 laminates measured at 23, 232 and 288 °C [3]

The effect of isothermal aging at 288°C in air on OHC properties and SBS strengths were investigated. The effect on RT OHC strength and modulus of PETI-298/T650-35 and PETI-330/T650-35 are presented in Figures 2.2.5.4 and 2.2.5.5, respectively. For comparison purposes, BMI-5270/AS-4 laminate properties are included in the graphs. The effects of aging on OHC strength are comparable for both PETI-298 and PETI-330 T650-35 laminates with both retaining about ~78% of their unaged OHC strength after 1000 hours.

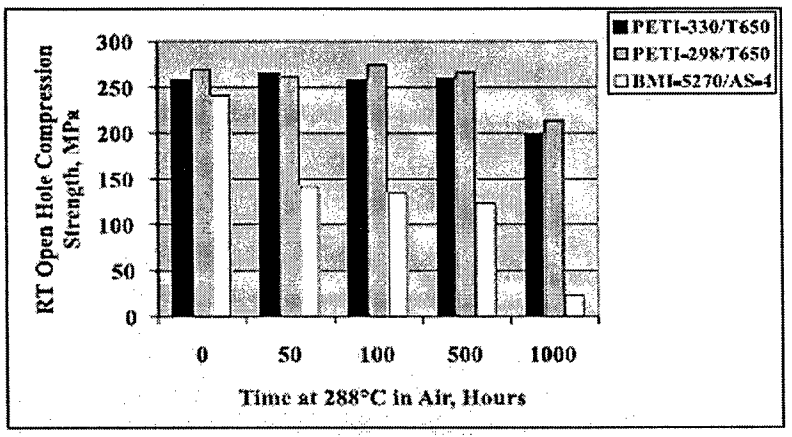


Figure 2.2.5-4 Effect of isothermal aging at 288 °C in air on OHC strength [3]

The effects of isothermal aging on the OHC modulus (Figure 2.2.5.5) were minimal with PETI-298 and PETI-330 T650-35 laminates retaining ~85% and ~92%, respectively, of their unaged moduli after 1000 hours at 288°C in air. The effects of isothermal aging on the SBS strength are presented in Figure 2.2.5.6. In this case, PETI-298/T650-35 specimens were not available for aging, thus the data for PETI-298/AS-4 SBS specimens are included. Aging at 288°C in air, the PETI-330/T650-35 specimens exhibited a retention of ~93% and ~75% of unaged RT SBS strength after 500 and 1000 h, respectively. In contrast, after aging at 288°C in air, the BMI-5270/T650 specimens retained ~73% and 40% of unaged RT SBS strength after 500 and 1000 h, respectively [3].

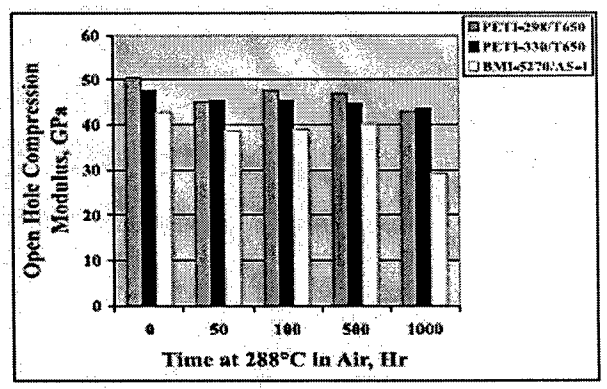


Figure 2.2.5-5 Effect of isothermal aging at 288 °C in air on OHC modulus [3]

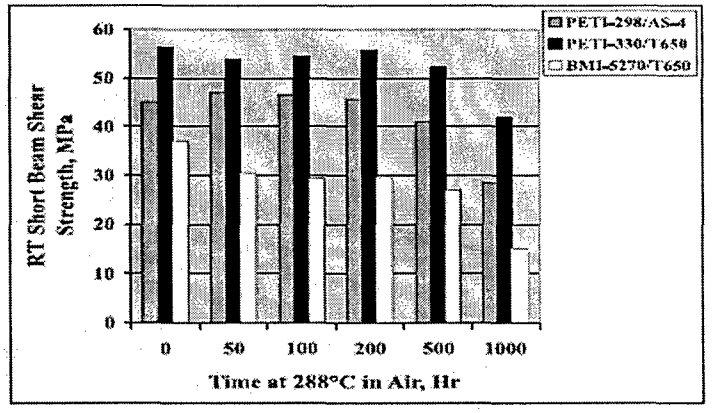


Figure 2.2.5-6 Effect of isothermal aging at 288 °C in air on short beam shear strength measured at room temperature [3]

3. Task II: HT-VARTM Testbed Development

3.1 System Components

Researchers at FACCT constructed an HT-VARTM testbed, which is shown in. Figure 3.1-1. The testbed has the following capabilities:

- Fabricates parts up to 2 ft by 2 ft,
- High temperature processing up to 593°C (1100°F),
- Programmable controls of the heat and vacuum during processing, and
- In-situ 3D part dimension measurement.

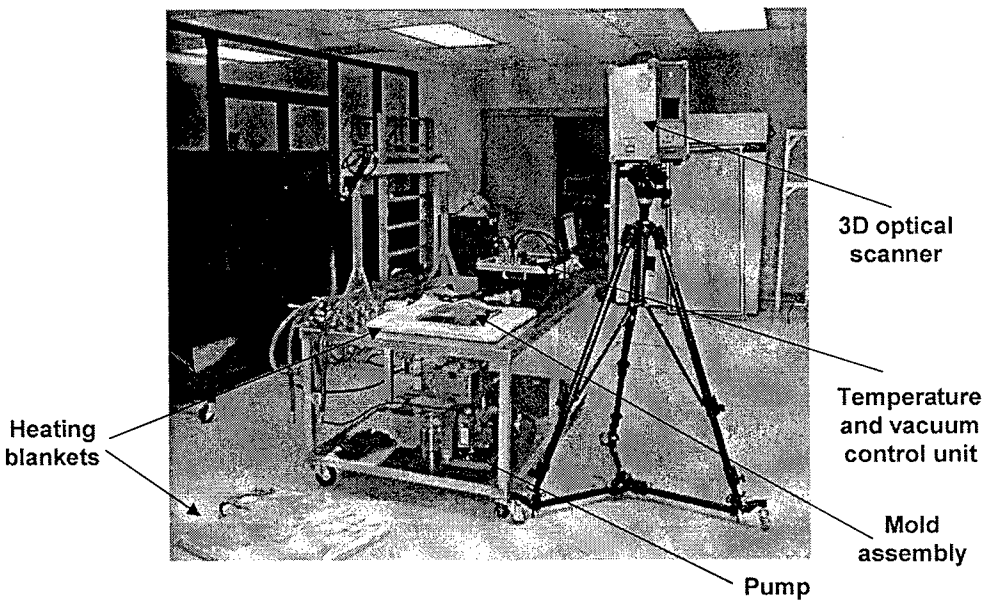


Figure 3.1-1 HT-VARTM Testbed

To achieve the above capabilities, the following components were obtained:

1) 3D optical scanner - VIVID 910, shown in Figure 3.1-2, was purchased from Konica Minolta Corporation. VIVID 910 has the specifications listed in Table 3.1-1.

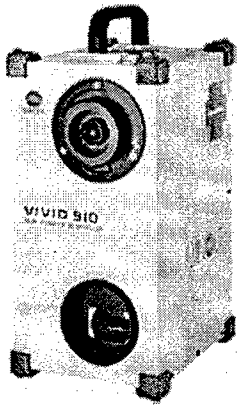


Figure 3.1-2 VIVID 910

Table 3.1-1 Specifications of VIVID 910 Scanner

Specification	VIVID 910 fw
Range Resolution (Z-Depth)	0.336 mm at highest resolution (600 mm to object).
Resolution (X&Y)	1.12 mm at highest resolution
Data Points per Scan	76,800
Scan Time	0.3 Seconds
Optics	8 mm Wide-Angle Lens
View Finder	5.7" Color LCD Display
Distance to Object	0.6 m to 2.0 m
Field of View (FOV per scan)	From 360mm X 270mm up to 900mm X 1200 mm
Color Resolution (per scan)	640 X 480 X 24 bits color depth
Safety	Class 1 Laser (FDA); Eye Safe.
Power	100 – 240 VAC; 50 – 60 Hz; Auto Switching

2) Temperature and vacuum control system - ACR-II Hot Bonders were purchased from BH Thermal Corporation. An ACR-II Hot Bonder is shown in Figure 3.1-3. ACR-II Hot Bonders control the heat and vacuum for on-the-spot composite and metal bond repairs. Packaged in an easy-to-carry case, the hot bonder can hold the heating blankets, vacuum hoses, and accessories. ACR-II Hot Bonders offer

cutting-edge technology like a USB data port for easy data transfer and easy-to-navigate software on a full-color touch screen.

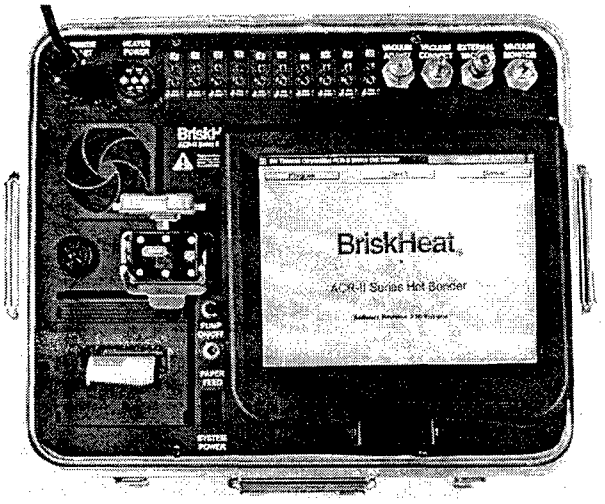


Figure 3.1-3 ACR-II Hot Bonders

ACR-II Hot Bonders has the following capabilities:

- Accepts either standard or mini J-type thermocouple connectors
- 10 thermocouple sensors per zone
- Single or dual zone models
- Portable, easy-to-carry case holds your heat curing blankets, vacuum hoses, and accessories
- Universal voltage: 90-264VAC
- 1400°F (760°C) maximum temperature control
- Models designed for hazardous / flight line environments
- Programmable to either English or Metric units
- Cure recipes can be edited on hot bonder or on included BriskHeat® Recipe Data Editor for Windows® software

ACR-II Hot Bonders has the following specifications:

i. General

- 10.4" (264mm) touch screen with easy-to-use interface
- USB port for data transfer (USB flash disk included)
- Input ground fault interrupter breaker protected
- Audible and visual alarms for high and low temperature / vacuum limits
- Digital data logger: prints and records real-time status of cure including program parameters

ii. Temperature Control

- Cure up to 1400°F (760°C)
- 10 thermocouple sensor inputs per zone
- Accepts either standard or mini J-type thermocouple connectors

High temperature cloth composite heat curing blankets – FGH and SXH Series was also purchased from BH Thermal Corporation. Figure 3.1-4 shows SXH Heat Curing Blanket. Their product highlights are: (1) designed for use with the newer high temperature thermoplastic and polyimide composite materials, (2) highly flexible up to a 1" radius or 2" X 2", (3) Compatible with ACR-II Series Hot Bonder. Its specifications are listed in Table 3.1-2.

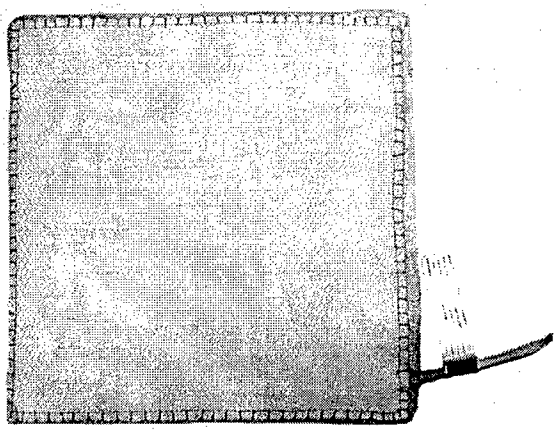


Figure 3.1-4 SXH Heat Curing Blanket

Table 3.1-2 Specifications of Heat Curing Blankets: FGH and SXH

Specification	FGH series	SXH series
Structure	Heating element and a 1" (25 mm) layer of high-density fiber glass is covered in an abrasion resistant fiber cloth (FGH) or SAMOX® cloth (SXH)	
Maximum exposure temperature	800 °F (425 °C)	1100 °F (593 °C)
Power density	7 watts / in^2 (0.011 watts/mm^2)	13 watts / in^2 (0.020 watts/mm^2)
Dielectric strength	Over 2000 volts	
Power cord	6 ft (1.8 m) with choice of power plug	

3) Vacuum pump – RobinAir 15600 was purchased from www.tequipment.net. Figure 3.1-5 shows the RobinAir 15600 vacuum pump. Its specifications are listed in Table 3.1-3.

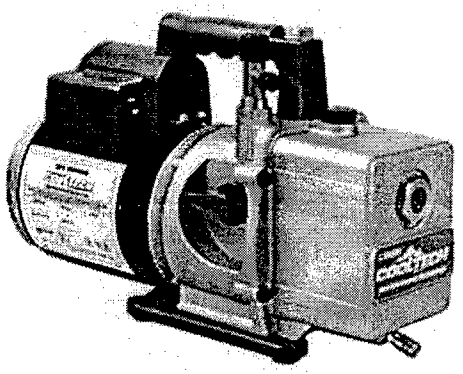


Figure 3.1-5 RobinAir 15600 Vacuum Pump

Table 3.1-3 Specifications of RobinAir 15600 Vacuum Pump

Specification	
Free Air Displacement	6 CFM
Number of Stages	Two
Factory Micron Rating	20 microns
Intake Fitting	1/4" MFL and 1/2" MFL Tee
Oil Capacity	15 oz. (445 ml)
Motor Size	1/2 HP
Voltage	110-115V/220-250V, 50/60 Hz, 142 l/m at 50 Hz
Weight	27 lbs (12 kg)

4) FACCT researchers assembled the testbed stand. Figure 3.1-6 shows the scheme of HT-VARTM testbed stand with a 3' X 4' working space.

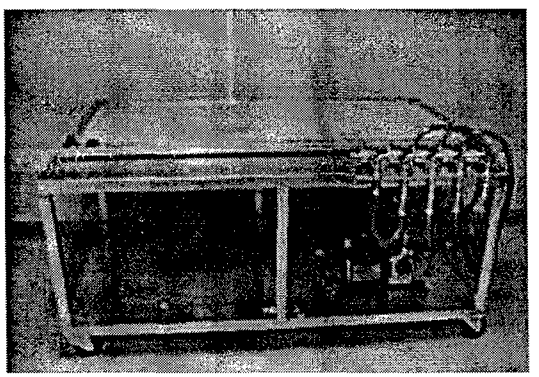


Figure 3.1-6 HT-VARTM Testbed stand

3.2 Testing of HT-VARTM Testbed

The HT-VARTM Testbed was used on VARTM processing of Epon 862/T650-35 composite. Figure 3.2-1 shows the VARTM set-up for Epon862/T650-35 processing. Figure 3.2-2 shows the whole experiment set-up.

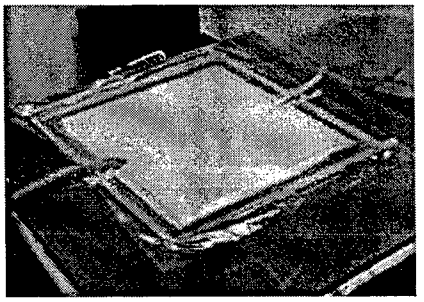


Figure 3.2-1 VARTM Set-up of Epon/T650-35

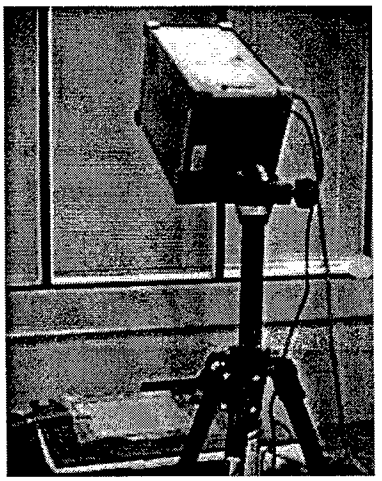


Figure 3.2-2 The Testbed testing set-up

Figure 3.2-3 shows the temperature profile for Epon862/T650-35 infusing and curing. The mold set-up was heated to 140°F, held for 30 minutes to let resin infuse and wet-out the carbon fabric, within one hour heated to 350°F to cure the Epon 862 resin, then cooled to room temperature. The pictures by the VIVID 910 are shown in Figure 3.2-4. Results processed by software PET show the bag assembly thickness variation

between the resin inlet point and vacuum outlet point right after infusion (before curing) was calculated to be 38%. Figure 3.2-5 shows the thickness measurement results of the cured part. Thickness variation of the cured Epon862/T650-35 was calculated to be 6.4%. Figure 3.2-6 shows the cured test panel processed and scanned by HT-VARTM testbed.

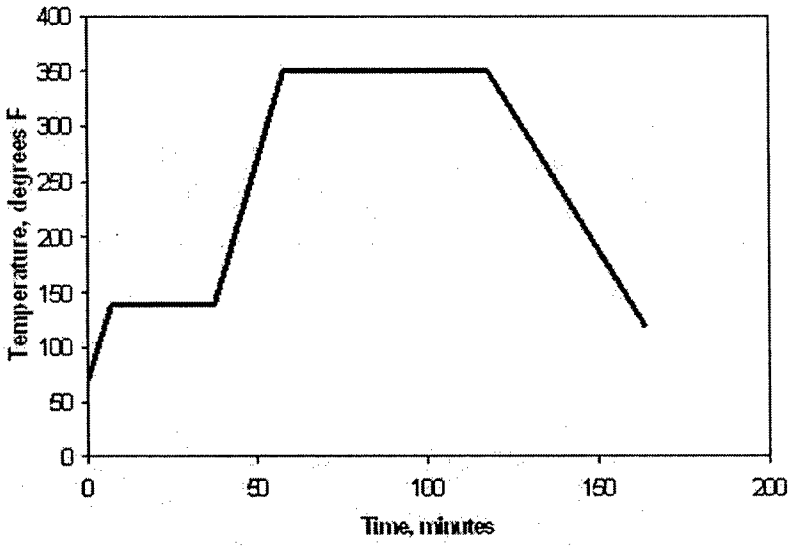
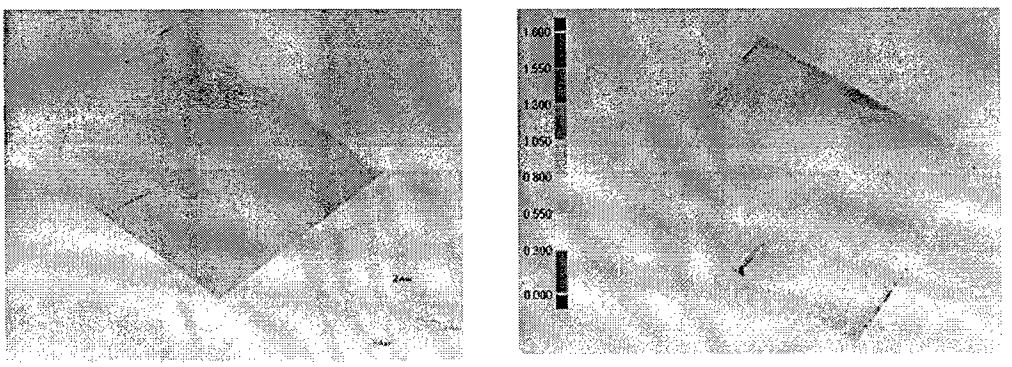


Figure 3.2-3 Temperature profile of Epon 862/T650-35 composite



Before infusion Immediately after infusion

Figure 3.2-4 3D Scanned Bag Assembly Thickness Variation

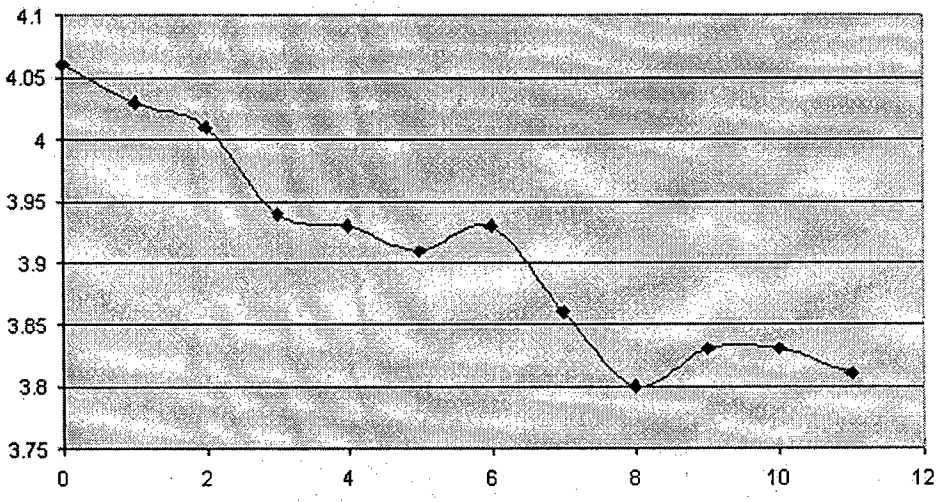


Figure 3.2-5 Thickness measurement results of cured Epon862/T650-35

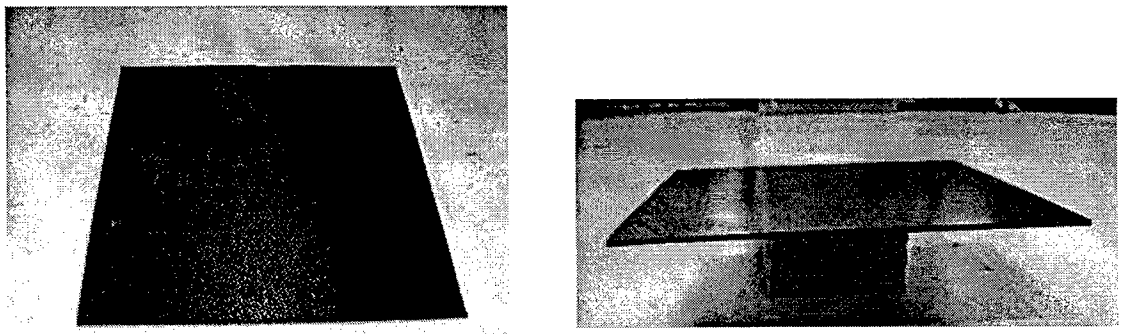


Figure 3.2-6 Cured test panel of Epon862/T650-35 (dimension 12" X 12", fiber volume fraction 47.3%)

4. Task III: HT-VARTM Infusion and Curing Experiments

Unlike normal or room temperature VARTM processing, HT-VARTM is performed at elevated temperature (> 280°C) under which the resin matrix PETI-330 becomes molten and is able to flow. Several methods were proposed to realize non-autoclave and low-cost processing of high temperature resistant composites. Two methods were developed using the same tool plate to heat the resin matrix and facilitate resin flow through fiber fabric on the tool plate: (1) In-Plane Flow Method, and (2) Through-Thickness Flow Method. The Heater Assisted Resin Flow method was another method used to heat the resin separately and then infuse the resin and wet out the fiber fabric in the mold cavity.

4.1 In-Plane Flow Method

Unlike ordinary VARTM processing, composite structures of aerospace grade quality were fabricated in four steps: (1) mold preparation and resin placement, (2) preform lay-up and vacuum bagging, (3) resin flow, and (4) curing process.

4.1.1 Mold Preparation and Resin Placement

In-plane flow is generally used to form flat laminates or structures with simple shapes. A flat mold or slightly curved mold is required. The mold is cleaned, and the surface is rubbed with a mold release agent. In this test, the liquid release agent Release-All-50 from Airtech International was used. The mold was heated to 125°C and held for 30 minutes to gain high temperature release ability. When the mold cooled, a rectangle was formed with three laterals with the high temperature sealant tape AVBS 750 from Airtech International. A specific quantity of PETI-330 was placed onto the mold surface within the rectangle. The powder resin was flattened using a stirring stick.

4.1.2 Preform Lay-up and Vacuum Bagging

At least one layer of reinforcement fiber mat or other woven fabric was cut and then placed near the resin area. In the test case, the preform was made of T650-35 fabric. The preform was surrounded by the sealant tape AVBS 750. One layer of release fabric, Bleeder Lease E from Airtech International, was placed on top of the preform and resin area to provide release and assist in the flow effect. On the opposite side of the preform was placed at least one layer of resin absorber and breather, Airweave UHT 800 from Airtech International, or an ordinary glass fiber cloth. The resin breather directs resin flow, absorb excess resin, and distribute vacuum pressure inside the vacuum bag. A plastic vacuum bag, Thermalimide from Airtech International, was sealed by AVBS 750 to form the vacuum bagging. A metal vacuum valve was installed on the vacuum bag and connected to metal hose to apply vacuum onto the bag. The vacuum hose leads the excess resin to a resin trap, called resin reservoir, thus controlling the ability of the fiber volume fraction of the composite parts fabricated by this processing technique. Figure 4.1.2-1 shows the set-up for resin and preform placement. Vacuum was applied to the bag from a vacuum pump, and a good seal was achieved by checking leakages and careful adjusting by hand. Figure 4.1.2-2 shows the vacuum bagging of this assembly.

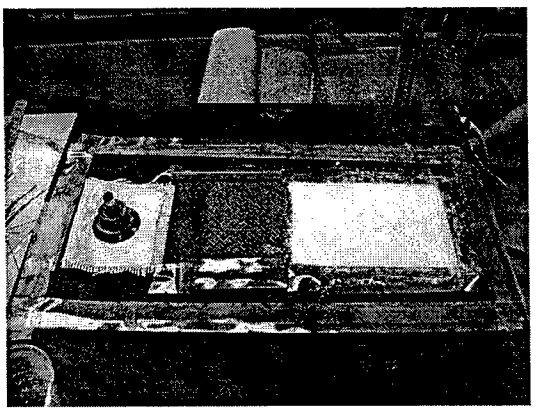


Figure 4.1.2-1 Resin and preform placement for in-plane flow

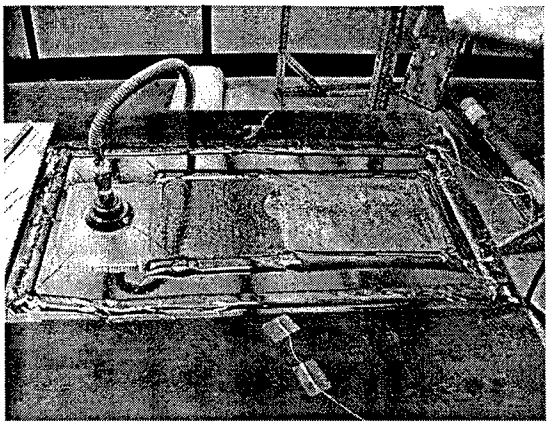


Figure 4.1.2-2. Vacuum bagging for in-plane flow

4.1.3 Resin Infusion

The whole mold assembly was heated with heating blankets and controlled by ACR-II Hot Bonders. The temperature within the bag was sensed by imbedding J-type thermocouples in the bag. For resin system PETI-330, the temperature was elevated to 280°C at a heating rate of 3~5°C per minute. To improve heating uniformity and reduce heat loss, the mold and fiber/resin vacuum bagged assembly was covered with another piece of the heating blanket. The blanket on top of the mold provided faster heating and uniform temperature distribution between the upper and bottom of the composite structure. Resin melting is shown in Figure 4.1.3-1. One picture shown in Figure 4.1.3-1 was taken during the molten PETI-330 flowing and impregnating the preform under a full vacuum. Within the soak time of 20-40 minutes for our experiment, the molten resin permeated through the preform and the bleeding material. Excess resin flowed from the bleeding media material to a resin collector.

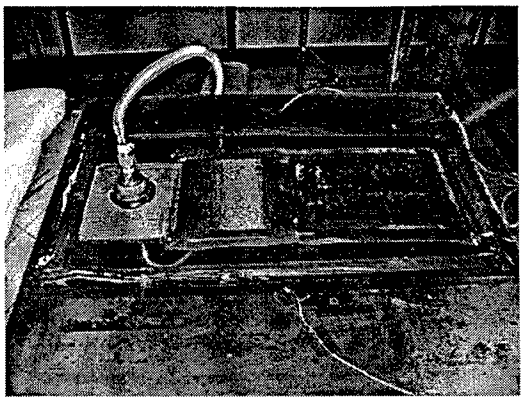


Figure 4.1.3-1 Resin melting during HT-VARTM

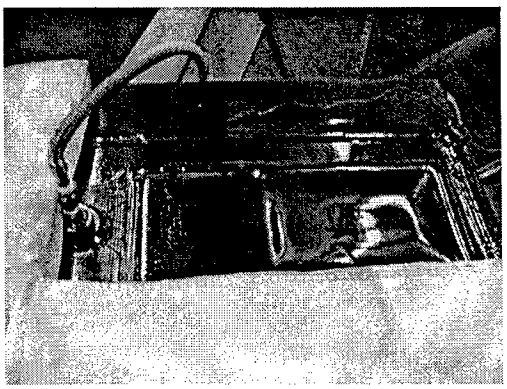


Figure 4.1.3-2 In-plane resin flow during HT-VARTM

4.1.4 Curing Process

When the soak time was finishedd and PETI-330 had wetted the preform completely, the mold temperature was raised to 371°C and held for one hour while maintinaing a full vacuum onto the bag. When the curing process was complete, the mold was cooled and the vacuum stopped, and the part was removed from mold. Figure 4.1.4-1 shows the appearing of the bottom face of composite flat panels fabricated by this method.

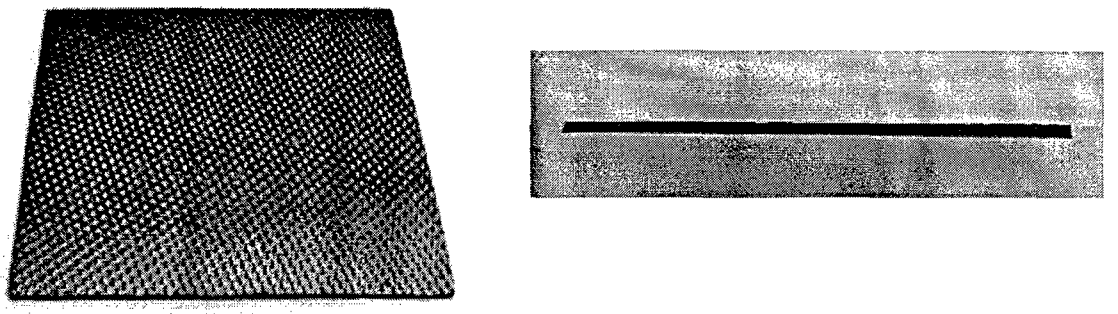


Figure 4.1.4-1 Flat panel fabricated by in-plane flow (dimensions: 8"X 8" X1/8", fiber volume fraction: 59.6%)

4.2 Through-Thickness Flow Method

The advantage of through-thickness flow is that it contributes to a rapid good wetting of fabric. A uniform and thin resin film is placed directly in mold cavity. Through-thickness flow method can be used to manufacturing flat laminates or structures of simple shape.

4.2.1 Mold Preparation and Resin Film Compression

In this method, a closed mold with a female mold and a male mold is needed. The mold cavity is cleaned and uniformly sprayed with a mold release agent onto the mold surface. Figure 4.2.1-1 shows a cleaned female mold with rectangle cavity feature. After applying with mold release agent, the mold was placed into an oven or put in a heating blanket and heated to 125°C and held for 30 minutes to obtain the high temperature release ability as mentioned in in-plane flow method section.

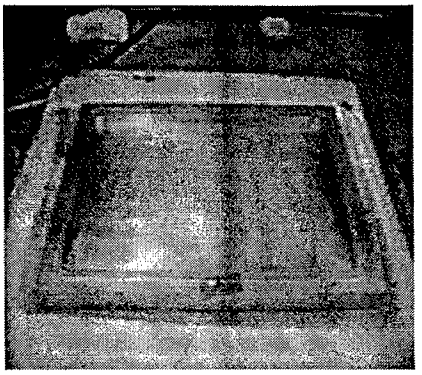


Figure 4.2.1-1 Female mold in through-thickness flow method

When the mold cavity cooled, a specific quantity of resin was placed into the mold cavity. The resin was flattened using a wooden stick. The mold cavity was then loaded into a specially designed press machine to make a thin resin film. Figure 4.2.1-2 shows the press apparatus used. A male mold was installed on the upper plate that was designed to fit the female mold and adjust the gap between the male and female mold. Most air trapped in the bulk of resin powder was squeezed out by hand. Figure 4.2.1-3 shows how the resin film was compressed. The compressed resin film produced is shown in Figure 4.2.1-4.

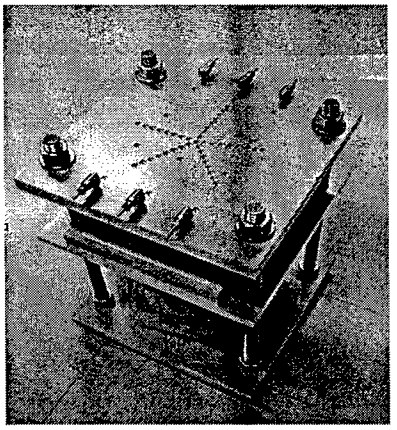


Figure 4.2.1-2 Press apparatus used for resin film compression

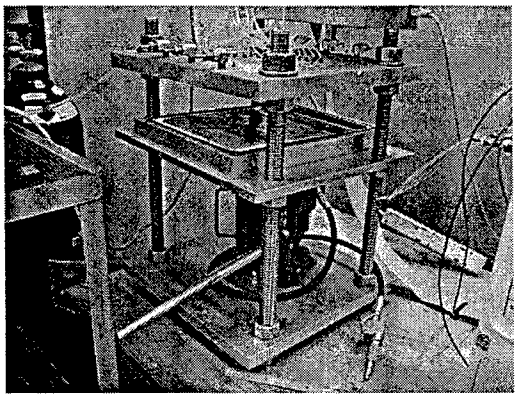


Figure 4.2.1-3 Resin film compression

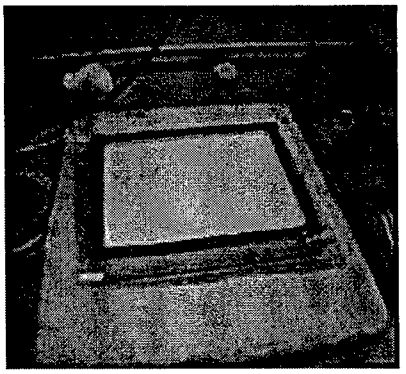


Figure 4.2.1-4 Resin film in through-thickness flow method

4.2.2 Preform Lay-Up and Vacuum Bagging

Eight plies of T650-35 fabric were cut and placed on top of the resin film. One layer of release fabric was placed to cover the reinforcing fabric to provide release ability. Then one layer of resin absorbing and breathing material, Airweave UHT 800, was placed to cover the resin film. Another piece of UHT 800 of a smaller size was placed on top to help cushion the weight of metal valve. Airweave UHT 800 is made of glass fiber and functions to distribute pressure inside the vacuum bag, to absorb and bleed resin from the fabric preform, and to relieve or eliminate the print-through on the upper surface of the final composite component. A high temperature stable plastic vacuum bag, Thermalimide, was used to package the resin and fabrics sealed by high temperature

sealant tap at edges on the female mold. A vacuum valve made of metal was installed on the vacuum bag and connected to fit hose. The vacuum hose can lead the excess resin to a resin trap or resin reservoir, thus providing the ability of controlling the fiber volume fraction of the composite parts fabricated by this processing technique. Figure 4.2.2-1 shows the vacuum bagging assembly.

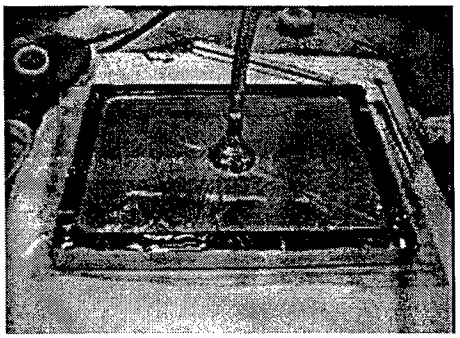


Figure 4.2.2-1 Vacuum bagging assembly in through-thickness flow

4.2.3 Resin Heating and Infusion

Using a heating blanket under the mold set-up, PETI-330 and the whole assembly were heated to 280°C at a heating rate of 3~5°C per minute. With a soak time of 30 minutes at 280°C, the molten resin flowed through the whole fabric preform along vertical (part thickness) direction under a full vacuum, then flowed through the bleeding fabric material. With resin flowing transversely from bottom to top through the reinforcing fabric and distribution media material, the fabrics impregnated with molten resin slowly decreased. Excess resin flowed into the resin reservoir via the valve and hole. Some air trapped with the resin flowed through the bleeding fabrics and finally entered the resin reservoir. Figure 4.2.3.1 shows the through-thickness flow pattern.

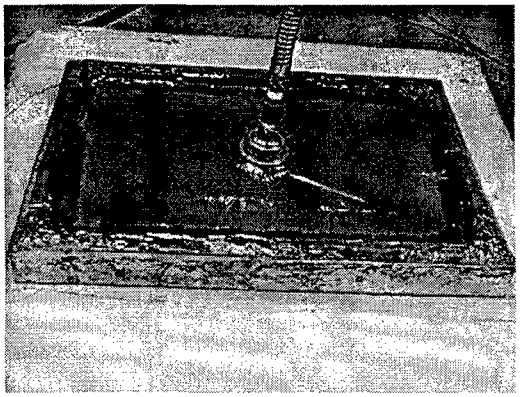


Figure 4.2.3.1 Through-thickness flow

4.2.4 Curing Process

The temperature was raised to 371°C and held for one hour to cure PETI-330 using the same heating blankets. When the temperature of the mold cooled to room temperature, the vacuum was stopped and the composite panel was removed from the mold. Figure 4.2.4-1 shows the upper and bottom views of the demolded flat panels. Print-through resulting from the metal valve on the upper face was still present.

Top surface

Bottom surface

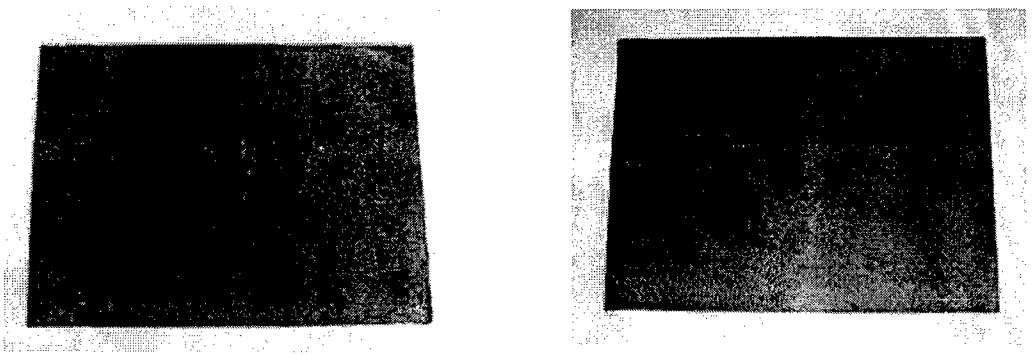


Figure 4.2.4-1 Flat panel made via through-thickness flow method (dimensions: 10"x 14"x 1/8", fiber volume fraction: 59.6%)

4.3 Heater Assisted Resin Flow Method

To produce parts of complex shapes and gain flexibilities of part size, the idea of heating the resin in heating chamber separately was attempted to produce a curved part. A heating chamber was designed to be compatible with the temperature and vacuum control system ACR-II Hot Bonders. A heating program was used to control the heating and melting of the PETI-330 resin in the heating chamber. The heater is shown in Figure 4.3-1. A cylindrical tube that is surrounded with heating elements is supported by a support, and the heating elements are connected to a control box on the same support. Figure 4.3-2 shows the upper view of the resin heater.

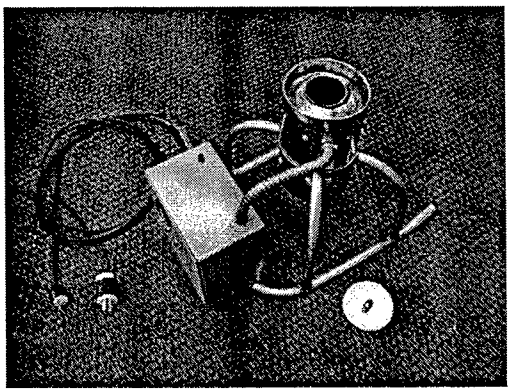


Figure 4.3-1 High temperature resin heater

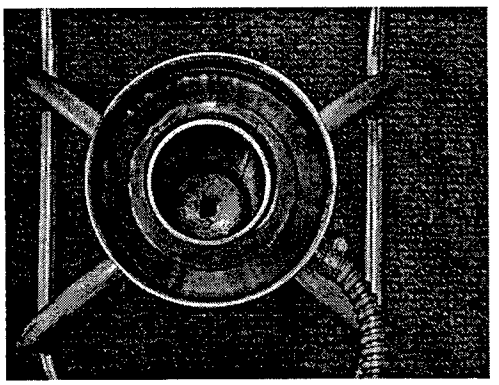


Figure 4.3-2 Top view of the resin heater

4.3.1 Mold Preparation

The male mold as shown in Figure 4.3.1-1 was cleaned and treated with release agent as described in sections of in-plane flow and through-thickness methods.

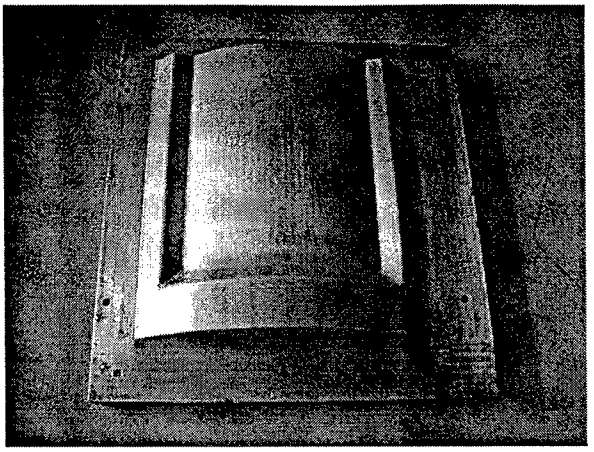


Figure 4.3.1-1 Curved male mold

4.3.2 Preform Lay-Up and Vacuum Bagging

Eight plies of T650-35 fabric were cut and prepared in advance and placed on top of the resin film. One layer of release fabric, Bleeder Lease E covered the preform to provide release ability. A thin long tip of breather material, Airweave UHT 800, was placed at the left and right sides of the carbon preform to direct resin flow from the heater, and bleed excess resin from the vacuum bag. A vacuum bag, Thermalimide, covered the preform and was sealed via AVBS 750. The heater was connected to the male mold using metal hoses and fittings. A metal hose was used to apply vacuum. The hose helped lead the excess resin to a resin trap. The hose was closed between the heater and the mold, and then a full vacuum was applied onto the bag assembly. Figure 4.3.2-1 shows the HT-VARTM system.

4.3.3 Resin Infusion

The resin heater was connected to ACR-II Hot Bonders via a control cord. The Hot Bonders system can control the heating of resin heater and the heating blanket via two programs running at Zone 1 and Zone 2, respectively. The heater raised the temperature to 290°C at 5°C/min and then held for 1-2 hours, which is sufficient to finish the infusion process.

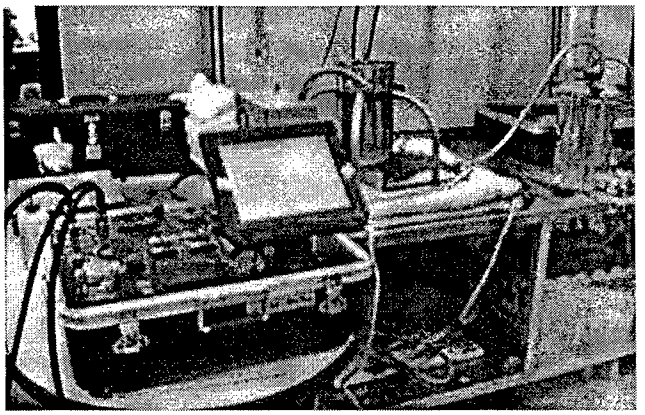


Figure 4.3.2-1 Vacuum bagging assembly for heater assisted resin flow method

The mold was heated by the heating blanket to 280°C at 3-5°C/min. The starting time was adjusted for the resin heater and the heating blanket according to the real temperature-increasing rate of the resin and the mold so as the resin became molten when the mold reached 280°C. When the resin became liquid, the hose between the heater and the mold was opened and resin flowed into the fabric. After 40 minutes, the PETI-330 had completely wetted out the T650-35 fabrics. The hose was shut off and the mold was heated again to a higher temperature for the curing process. Figure 4.3.3-1 shows the whole assembly during resin infusion and curing processes.

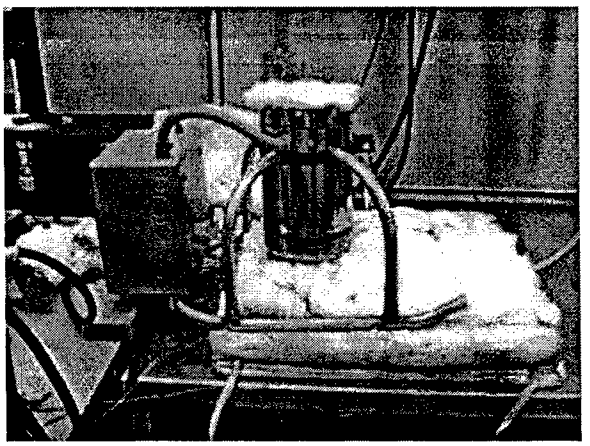
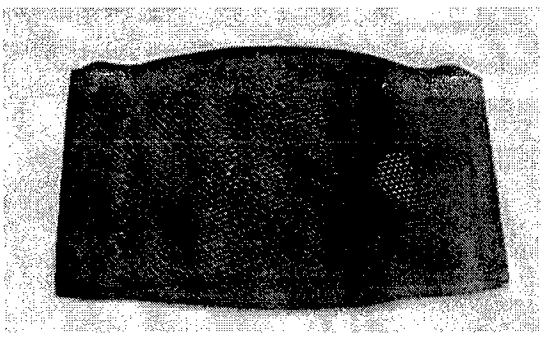


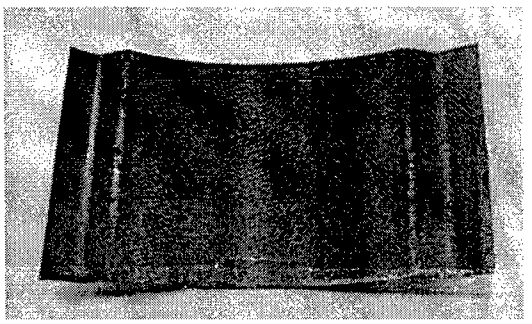
Figure 4.3.3-1 Infusion and curing of heater assisted resin flow method

4.3.4 Curing Process

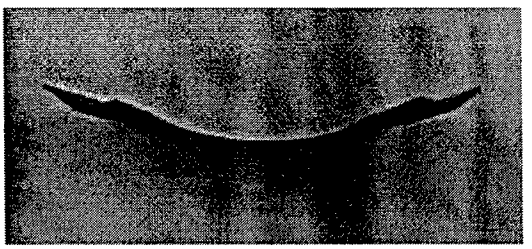
As described in the In-Plane Flow and Through-Thickness Flow sections, the curing process was finished by heating to 371°C and holding for one hour. The mold was cooled and the part was removed. Figure 4.3.4-1 shows the part made by heater assisted resin flow method. There was a 1" x 1" dry spot on the upper surface. Further research is continuing to solve this problem.



Top surface



Bottom surface



Side view

Figure 4.3.4-1 Curved part made by the heater assisted resin flow method (dimensions: 9" x 5" x 1/8")

5. Task IV: Testing and Characterization

5.1 Mechanical Testing

Four plies of T650-35 8 HS fabric were used to produce laminates with PETI-330 by In-Plane Flow Method. The test panel had a fiber volume fraction of 57.6%. Tensile tests were performed at room temperature according to ASTM.D3039. Figure 5.1-1 shows the tensile strength testing results. The mean tensile strength measured was 834.4 MPa. It was reported that the tensile strength of PETI-330/T800H was 968 MPa. Figure 5.1-2 shows the tensile modulus testing results of PETI-330/T650-35. The mean tensile modulus was obtained as 43.6 GPa.

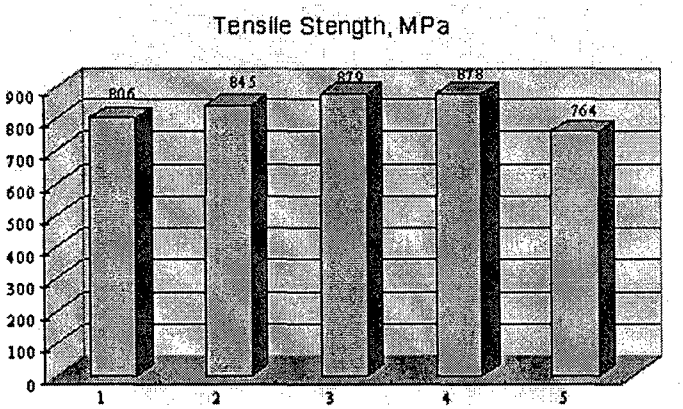


Figure 5.1-1 Tensile strength testing results of PETI-330/T650

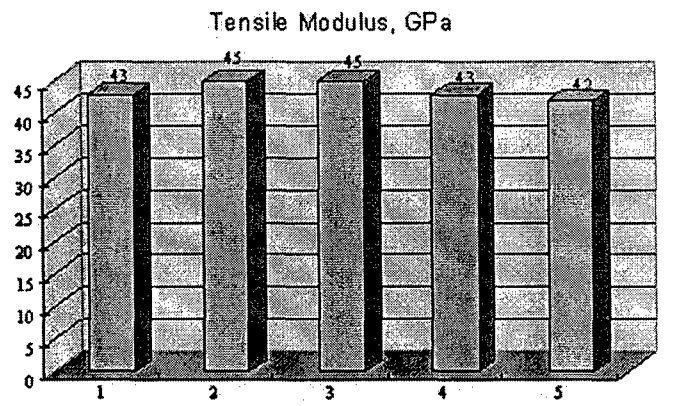


Figure 5.1-2 Tensile modulus testing results of PETI-330/T650

Short beam shear strength was tested at room temperature according to ASTM.D2344. Figure 5.1-3 shows the open hole shear strength testing results. The mean short beam shear strength of the six samples was 43 MPa. This was ~77% of the short beam shear strength of the same laminate made by RTM (using a injection pressure of 2.75 MPa [8]).

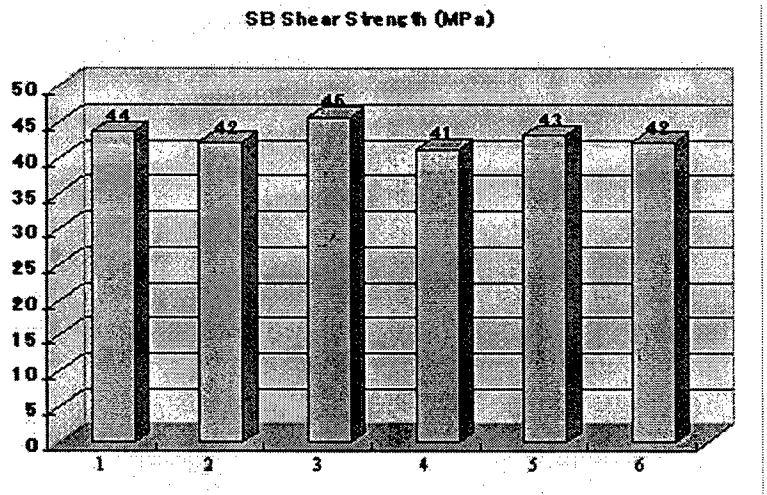


Figure 5.1-3 Short beam shear strength testing results of PETI-330/T650

5.2 Fiber/Resin Interfacial Bonding

After tensile testing, the fracture cross-section surfaces of the tensile samples were analyzed by microscope. Figure 5.2-1 shows a typical fracture behavior for tensile testing. Most fracture points resulted from fiber breaks. However, some fibers were pulled out after tensile fracture. This indicates that fiber/matrix interfacial bonding was not ideal and needs to be further improved.

5.3 Void Content

The void contents of composite panels were tested by acid digestion using sulfuric acid and hydrogen peroxide according to ASTM.D3171-99. For parts processed via in-plane flow method the void contents were measured to be 9~13%. The primary reason for

such high void contents is believed to be due to the formation and/or existence of low molecular weight constituents in PETI-330 oligomers. During the temperature ramp-up and curing process, some of these compounds are released under the negative pressure produced by the high vacuum. It was testified by the thermal gravity analysis (TGA) testing of neat PETI-330 resin, which showed that there has been 2% weight loss until it was heated to 338°C.

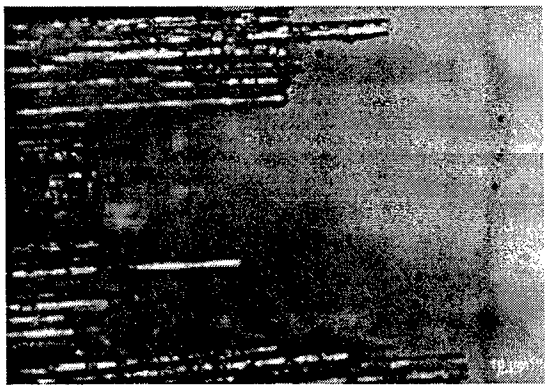
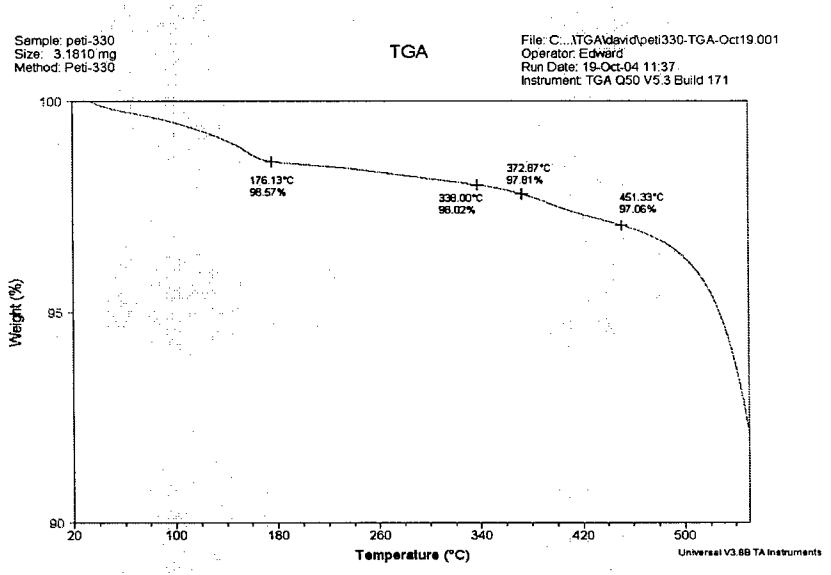


Figure 5.2-1 Tensile fracture behavior of PETI-330/T650-35



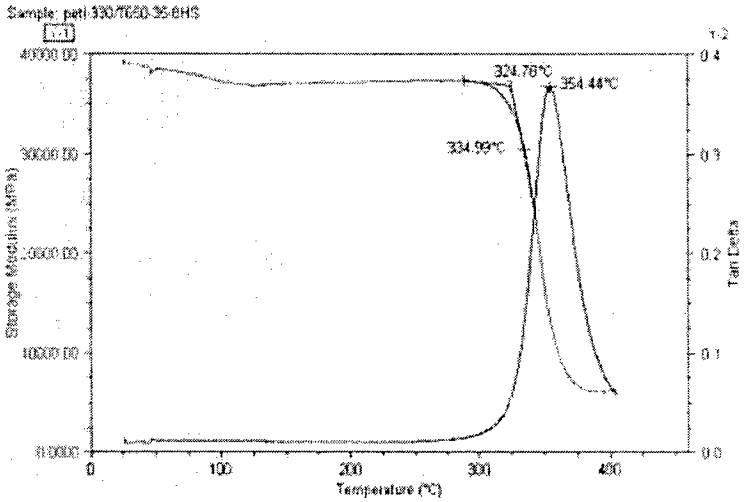
Note

Heating rate: 20°C/min

Figure 5.3-1 TGA test results of neat PETI-330 resin

5.4 Thermal Property

Dynamic mechanical analysis (DMA) testing was performed on the PETI-330/T650-35 laminates made by In-Plane Flow Method. Figure 5.3-1 shows the DMA test results. A storage modulus of ~38 GPa was gained, and the T_g from DMA can be 324, 335, or 354°C, according to different judging standards.



Note

Mode: three-point bending;

Ramp rate: 5C/min;

Figure 5.4-1 DMA test results of PETI-330/T650-35

5.5 Dimensional Variation Measurement

The nature of the VARTM process and high processing temperatures cause dimension variations, which is a challenging issue in the development of HT-VARTM. Figure 5.5-1 shows the dimensional variation measurement of the flat panel shown in Figure 4.2.4-1. It is believed that the dimensional variation results from the unbalanced relaxation of stress is due to the high-temperature curing process and the coefficient of thermal expansion (CTE) mismatch between fiber reinforcement and resin matrix. The flatness of this panel was measured to be 1.187 mm.

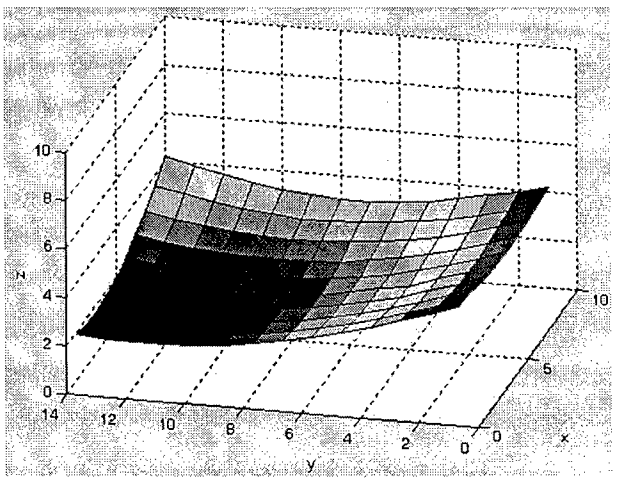


Figure 5.5-1 Dimensional variation measurement of PETI-330/T650-35 flat panel made with the through-thickness flow method

5.6 HT-VARTM Processing Issues and Future Directions

5.6.1 Mechanical Properties Improvement

PETI-330/T650-35 test panels fabricated by HT-VARTM show 23% lower short beam shear strength compared to those by RTM. Since short beam shear strength tests reflect mainly the interfacial bonding between the resin and fibers, further research needs to be conducted to improve the composite quality and the mechanical properties of composites parts fabricated by HT-VARTM.

5.6.2 Interfacial Bonding Improvement

As is seen from the microscopic behavior of the tensile fracture in Figure 5.2-1, there exists fiber pull-out after tensile fracture, which indicates that the interface between resin and fiber did not achieve the outstanding properties of PETI-330. Further improvements are required in future research by facilitating resin flow through fibers and the wetting of fibers.

5.6.3 Large and Complex-Shaped Part Fabrication

Since heat assisted resin flow method is more suitable to HT-VARTM processing of large parts and parts of complex shape, further research will address the hose connection between heater and mold, eliminating dry spots by facilitating resin flow, and achieving complete wetting of the preform and quality composite parts.

5.6.4 Void Content Reduction

The void contents of testing panel made via the in-plane flow method were measured to be in the range of 9% to 13%, which is high for advanced composite applications. Further research will include the characterization of the void contents of parts prepared by through thickness flow and heater assisted flow method, experimental and kinetic studies on the reactions taking place during temperature ramp up and curing process of PETI-330, and processing parameters optimization.

5.6.5 Dimensional Variation Reduction

The FACCT research team has conducted preliminary investigations on using nanotubes to tailor the coefficient of thermal expansion (CTE) of resin so as to eliminate the cure stresses that result from the mismatches of CTE between the resin and fiber reinforcement and high cure temperature. FACCT has investigated the effects of adding nanotubes to epoxy by Molecular Dynamics (MD) simulation and experiments. Figure 5.4.2-1 shows MD model of adding SWNT into epoxy resin. Figure 5.4.2-2 shows that for Epon 862 resin, more than 30% CTE reduction was achieved by adding 1wt% SWNTs.

In the future work, the research team will incorporate carbon nanotubes in PETI-330/carbon fiber composites during HT-VARTM processing. The SWNTs will be mixed with the resin, and then infused into the fiber preform. The team will investigate different mixing methods of direct mixing, co-extrusion, and in-situ polymerization, along with the

dispersion, characterizing rheology properties changes, measuring the CTE reduction, and characterizing the final composite part properties.

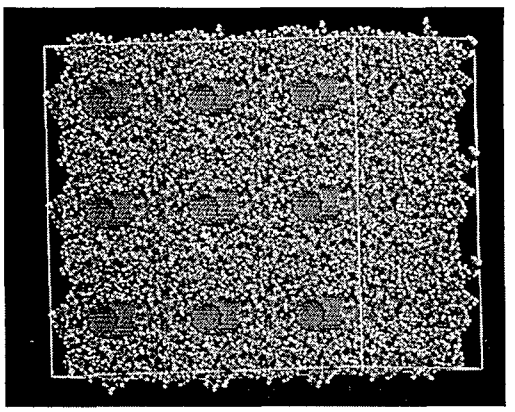


Figure 5.4.2-1 MD simulation model of adding SWNTs into Epon 862

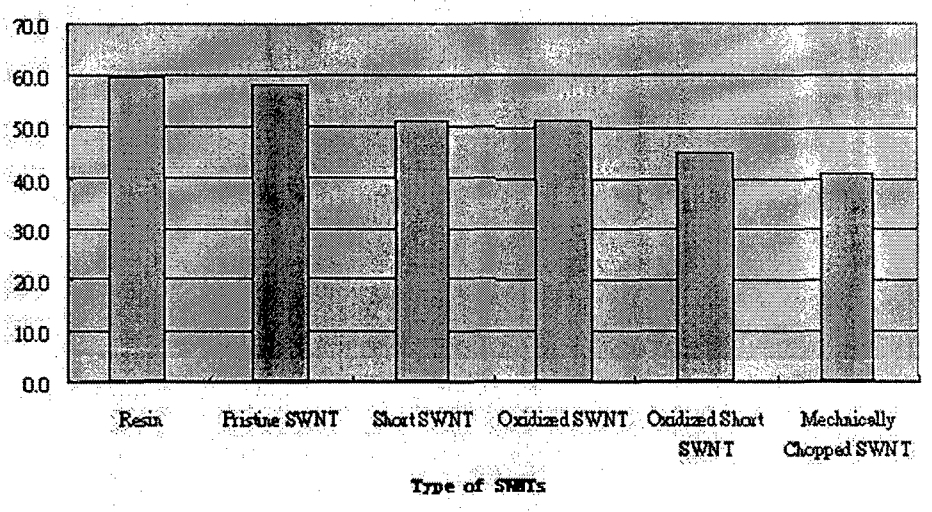


Figure 5.4.2-2 CTE reduction of Epon 862 by adding 1wt% SWNTs

6. Conclusions

In this project, a HT-VARTM testbed was developed for producing high temperature composite structures. Results indicate that the HT-VARTM is feasible for manufacturing composites that has a T_g of ~330 and use temperatures of >300 °C. The following conclusions can be drawn from the study:

- PETI-330 has a combination of VARTM processability and high temperature mechanical properties.
- In-plane resin flow method is effective in making flat panels via HT-VARTM.
- Through-thickness resin flow method is faster and can be used to make flat and simple curved parts via HT-VARTM.
- Heater assisted resin flow method is suitable for producing larger parts and those with complex shape.
- The mechanical properties of the test panels are comparable with those made by RTM and prepreg processes. With future improvements in processing, it is believed that the mechanical properties of composites made by HT-VARTM will be closer to those achieved by other processes.
- Void contents of the test panels are higher than the required for aerospace applications. The main reason is that the resin system may contain some low molecular weight constituents. Extra care must be exercised in the temperature ramp-up and curing processes to minimize the void content.
- Future research will be directed in improving interfacial bonding thus mechanical properties, reducing void contents, reducing dimensional variations, and testing the capabilities of HT-VARTM to make large and complex shaped parts.

7. Acknowledgement

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Ben Wang

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Florida Advanced Center for Composite Technologies
Department of Industrial and Manufacturing Engineering
FAMU–FSU College of Engineering
2525 Pottsdamer Street
Tallahassee, FL 32310

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