

# Processing and Characterization of PETI Composites Fabricated by High Temperature VARTM

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## Abstract

The use of composites as primary structures on aerospace vehicles has increased dramatically over the past decade. As these advanced structures increase in size and complexity, their production costs have grown significantly. A major contributor to these manufacturing costs is the requirement of elevated processing pressures, during the thermal cure, to create fully consolidated composites. For certain composite parts, high temperature vacuum assisted resin transfer molding (HT-VARTM) can offer reduced fabrication costs compared to conventional autoclave techniques. The process has been successfully used with phenylethynyl terminated imide (PETI) resins developed by NASA LaRC. In the current study, two PETI resins, LARC™ PETI-330 and LARC™ PETI-8 have been used to make test specimens using HT-VARTM. Based on previous work at NASA LaRC, larger panels with a quasi-isotropic lay-up were fabricated. The resultant composite specimens exhibited void contents <3% by volume depending on the type of carbon fabric preform used. Mechanical properties of the panels were determined at both room and elevated temperatures. These included open-hole compressive (OHC) and short beam shear (SBS) properties. Limited process modeling efforts were carried out including infusion times, composite panel size limitations and fabric permeability characterization. Work has also been carried out to develop new PETI based resins specifically geared towards HT-VARTM. The results of this work are presented herein.

Keywords: polyimides, phenylethynyl terminated imides, VARTM, mechanical properties

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## 1. Introduction

Polyimide composites are very attractive for applications requiring a high strength to weight ratio and performance at use temperatures above 204 °C. The combination of thermal and mechanical properties, along with improvements in processability allow aromatic polyimides to find increased use in aerospace applications. Recent work at NASA Langley Research Center (LaRC) has concentrated on developing new polyimide resin systems for advanced aerospace applications that can be processed out of the autoclave. Using controlled molecular weight imide oligomers containing phenylethynyl endcaps, phenylethynyl terminated imide (PETI) resins, are readily processed into neat resin moldings, bonded panels and composites. LaRC™ PETI-330 is a low molecular weight imide oligomer (calculated number average molecular weight ( $M_n$ ) ~1290 g/mole) that exhibits a low melt viscosity and a post cure glass transition temperature ( $T_g$ ) around 330 °C. It was prepared using 2,3,3',4'-biphenyltetracarboxylic dianhydride, 1,3-bis(4-aminophenoxy)benzene and 1,3-phenylenediamine and endcapped with phenylethynylphthalic anhydride. The resin was specifically designed for making composites using resin transfer molding (RTM) and resin infusion (RI) processing. PETI-330 laminates exhibit good mechanical properties up to 288 °C [1,2] and have retained approximately 98% of room temperature open hole compression (OHC) strength after aging 500 h at 288 °C [3]. LaRC™ PETI-8 is a phenylethynyl endcapped aromatic polyimide ( $M_n$  ~1125 g/mole) based on 3,3',4,4'-biphenyltetracarboxylic dianhydride, and a 50:50 molar ratio of 3,4'-oxydianiline and 1,3-bis(3-aminophenoxy) benzene. PETI-8 has a post cure  $T_g$  around 300 °C, and produces good tensile shear strengths and flatwise tensile strengths when processed under vacuum bag pressure only [4] thus eliminating the need for costly autoclave processing. PETI-8 resin composites have been processed using standard and double-vacuum-bag (DVB) processes. The resulting mechanical properties including short beam shear (SBS) strength, flexural strength and modulus have been evaluated at various temperatures [5].

The vacuum assisted resin transfer molding (VARTM) process was developed as a variation of RTM over twenty years ago as a commercial and military process for ground-based and marine composite structures, [6,7] and has shown potential to reduce manufacturing costs. The Seemans Composite Resin Infusion Molding Process (SCRIMP) [8] is a vacuum infusion process using a high-permeability layer to rapidly distribute the resin across the part surface increasing through-thickness penetration. The Controlled Atmospheric Resin Infusion Process (CAPRI) patented by The Boeing Company [9], is a SCRIMP variation where vacuum debulking and a reduced pressure difference is used to minimize thickness gradients and resin bleeding. Studies have demonstrated the feasibility of the VARTM process for fabrication of void free structures utilizing epoxy resin systems with fiber volume fractions approaching 60% [10]. VARTM using vinyl ester resins has traditionally yielded composites with low void contents and has found applications in the marine industry [11,12]. However, it should be noted that the focus has been on room temperature VARTM process.

The CAPRI VARTM process has been extended to the fabrication of composite panels from polyimide systems developed at NASA LaRC. Work has focused on processing various LaRC polyimides (i.e. PETI-330, PETI-8) by employing a high temperature VARTM process referred to as HT-VARTM. In HT-VARTM, resin flow lines, tools, sealants and bagging materials must be able to tolerate the high temperature processing cycle. Although the evaluation of these resins has shown that they exhibit the necessary melt flow characteristics for HT-VARTM processing,

the resulting laminates had void contents greater than 7% by volume [13,14]. It was determined that the high temperature required for processing resulted in degradation of some of the phenylethynyl groups, forming volatile by-products during low pressure consolidation. By adjusting the processing cycle the void content was reduced to <3%, while still achieving sufficient fiber volume (>58%) [15,16].

This paper focuses on the HT-VARTM processing trials using a quasi isotropic lay-up. Some limited computational work, based on experimental inputs, was performed to simulate the fabric permeability towards resin viscosity to help establish initial processing parameters. This enabled a better understanding of the process requirements that lead to the subsequent generation of larger composite panels. Work is being carried out to generate an extensive database of mechanical properties that include SBS and OHC. Research is also underway to synthesize new imide based resins that will be suitable specifically for HT-VARTM.

## 2. Experimental

### 2.1 Materials

Three PETI resins were used for the HT-VARTM processing trials. PETI-8 was purchased from Imitec Inc., Schenectady, NY, USA and PETI-330 from Ube Chemicals Ltd, Japan. The third resin, a new material designated PETI-9 was synthesized specifically for HT-VARTM with a  $T_g$  similar to that of the autoclave processable LARC™ PETI-5. This material was scaled up and purchased from Imitec Inc. The following chemicals were obtained from the indicated sources and used without further purification: 1,3-bis(3-aminophenoxy)benzene (1,3,3-APB, Mitsui Toatsu, m.p. 107-109 °C), 1,3-bis(4-aminophenoxy)benzene (1,3,4-APB, Chriskev, m.p. 112-115 °C), 1,3-diaminobenzene (m-PD, Sigma-Aldrich, m.p. 66 °C), 2,3,3',4'-biphenyltetracarboxylic dianhydride (a-BPDA, Ube Industries Inc., m.p. 197 °C), 4-phenylethynylphthalic anhydride (PEPA, Imitec, Inc., m.p. 152 °C) and N-methyl-2-pyrrolidinone (NMP, Fluka Chemical Co.). All other chemicals were used as received without further purification. Three types of carbon fiber fabrics were used for this work: IM7-6K 5-harness satin woven fabric (GP sizing, 280 gsm), T650-35-3K 8-harness satin woven fabric (309 sizing, 366 gsm), and IM7-6K unidirectionally woven fabric (GP sizing, 160 gsm, Sticky String 450 1/0 fill fiber). All fabrics were obtained from Textile Products, Inc., Anaheim, CA, USA.

### 2.2 New Resin Synthesis

A new PETI resin, based on a modification to the currently used PETI-8, was synthesized (Figure 1). The resin, designated as PETI-9, is a phenylethynyl endcapped aromatic polyimide ( $M_n \sim 1125$  g/mole) based on 3,3',4,4'-biphenyltetracarboxylic dianhydride and a 25:75 molar ratio of 3,4'-oxydianiline and 1,3-bis(3-aminophenoxy) benzene.

In-house work has focused on the synthesis of new resins based on PETI-330. PETI oligomers were prepared by the reaction of a-BPDA with the appropriate quantity of aromatic diamines and endcapped with PEPA. The oligomers were prepared by initially dissolving the aromatic diamine(s) in NMP at room temperature under nitrogen. The appropriate quantities of a-BPDA and PEPA were subsequently added in one portion as a slurry in NMP. The reactants were allowed to stir for ~20 h at ambient temperature under nitrogen. Imide oligomers were prepared directly from the amide acid solutions by azeotropic distillation with toluene using a Dean-Stark

trap to affect cyclodehydration. Powders were isolated by addition of the reaction mixture to water followed by washing in warm water. The powders were then dried to constant weight with yields >95%.

### **2.3 Resin characterization**

Differential scanning calorimetry (DSC) was performed on a Setaram DSC 131 at a heating rate of 20°C/min with the  $T_g$  taken at the inflection point of the  $\Delta H$  versus temperature curve. The cured  $T_g$  was determined by heating the sample to 371°C in an aluminum pan and holding for 1 h. Dynamic rheological measurements were obtained using an Advanced Rheometric Expansion System (ARES) Parallel Plate Rheometer from Rheometrics, Inc. The measurements were carried out under nitrogen atmosphere in an oscillatory shear mode using parallel plate geometry (25 mm diameter) at a heating rate of 4 °C/min from 100 to 371 °C at a fixed angular frequency of 10 rad/s.

### **2.4 Simulation**

Flow simulation and preform characterization was conducted at the University of Delaware – Center for Composite Materials (UD-CCM). The permeability of the three types of carbon fabric and the distribution medium (DM) was determined. The in-plane permeability of the fabric was evaluated using standard flow experiments where the fabric layers were placed under the bag and the time versus flow location was recorded via an automated camera system. 1-D Darcy's law was used to calculate the permeability. For the out-of-plane permeability of the fabric and in-plane permeability of the DM, a technique of Gokce et. al [17] was utilized. Here, both flow surfaces (DM and tool side) were recorded and the flow data was matched to a finite element simulation. The data reduction provided the effective permeability of the materials, while considering all the process effects, such as nesting of the fabric into the DM and compaction changes. In addition, lead length (the difference between the saturated flow distance on the DM and tool surface) of the non-saturated flow and non-uniform fill through the thickness of the fabric could be observed.

The permeability of the carbon fabrics and the DM was also investigated at Michigan State University (MSU). The in-plane and through thickness permeabilities of each carbon fiber preform and the aluminum (Al) distribution medium were measured. The approach that was used initially compacted the sample to a known thickness or fiber volume fraction. A fluid of known viscosity and flow rate was then passed through the sample using either an in-plane or through thickness fixture [18]. The corresponding pressure drop was recorded and the permeability calculated using the 1-D form of Darcy's Law. The experiments were repeated for different volume fractions between 35 and 60 percent and a curve of permeability versus fiber volume fraction was constructed. Since the materials are orthotropic, three experiments were required for each material to complete the characterization. These include in-plane x-direction, in-plane y-direction and through thickness z-direction. Two samples for each measurement were run. If there was significant difference between the two tests, a third sample was run. The fluid used in the tests was SAE 40W motor oil.

### **2.5 High Temperature VARTM**

The HT-VARTM set-up utilized in this work is shown in Figure 2. The set-up included a 1.27 cm thick steel plate as the tool. Three holes were drilled and tapped into the plate to provide one

resin inlet and two vacuum outlets. Al screen material was utilized as the flow medium. Polyimide bagging material (Thermalimide™, Airtech) and high temperature sealant were used to seal an inner bag that contained the appropriate number of layers of carbon fiber preform, five layers of Al screen flow media, Release Ease™ fabric, and a breather material. An additional outer bag provided redundancy against leaks in the inner bag after infiltration. During the fabrication of larger 33 cm x 33 cm panels, a quasi isotropic lay-up of 8 plies with  $[\pm 45/(0/90)/\pm 45/(0/90)]_s$  orientation was used for IM7-6K 5-harness and the T650-35-3K 8-harness carbon fiber fabrics. In the case of the uniweave fabric, a quasi isotropic lay-up  $\{[-45/0/45/90]_{2s}\}$  of 16 plies was used. Prior to infusion, each type of carbon fabric was heat treated at 400 °C for 1 h to remove sizing.

Prior experience demonstrated [15] that the process worked best using a two-oven set-up where a heated tube connected the two ovens to each other. All PETI resins were heated to the infusion temperature of 260 °C under vacuum and further degassed at that temperature for 5 mins. Vacuum on the pot was then reduced to 50.8 kPa and the connecting valve between the pot and heated tube opened to allow the resin to flow until infusion was complete. Depending on the type of carbon fabric, the infusion time varied. However, all samples were typically allowed to infuse for up to 2 h or longer after the start of infusion to ensure that the resin had flowed through the thickness of the panel. A staged cure cycle was used for all resins. For PETI-330, the cure cycle involved heating the panel to 310 °C and holding for 8 h. After that it was taken to 371 °C and held for another 1 h before being cooled down to room temperature. In the case of PETI-8 and PETI-9, the samples underwent a 2 h hold at 290 °C, another 2 h hold at 300°C followed by an 8 h hold at 316 °C.

## 2.6 Composite characterization

C-scan inspections of the composite panels were carried out using a 3 axis (x, y and z) Ultrasonic Scanner from SONIX Advanced Acoustic Solutions with WIN IC (C-Scan) Version 4.1.0k software. A conventional ultrasonic pulse-echo C-scan method was used for detecting and characterizing defects in composites with a gain set to about 54 dB.

Acid digestion of cured composites was carried out following ASTM D3131. Calculations were based on a 1.77 g/cc fiber density and a 1.31 g/cc resin density [15].

## 2.7 Composite Mechanical Properties

Mechanical properties of the composites tested were SBS according to ASTM D2344 and OHC according to ASTM D6484. Both SBS and OHC tests were performed at room temperature and elevated temperatures. For SBS, a Sintech 2W mechanical testing machine with a 4.45 kN load cell and a heating chamber (Thermcraft) was used. The crosshead speed was 1.27 mm/min (0.5 in/min). For OHC, an Instron test stand with a 88.96 kN (20 kip) load cell was used. The speed of testing was 0.127 cm/min (0.05 in/min) while the loading and unloading was done at 0.254 cm/min (0.1 in/min).

# 3. Results and Discussion

## 3.1 Resin properties

The newly synthesized PETI-9 had a  $T_g$  of 265 °C, as determined by DSC. A 4 h hold of PETI-9 was carried at the infusion temperature of 260 °C in the parallel plate rheometer and the complex

melt viscosity ( $\eta^*$ ) was between 1 and 2 Poise. This value is lower than that of PETI-8 (~5 Poise) and PETI-330 (~20 Poise) and hence the resin offers the ability to manufacture larger and thicker parts.

While it is possible to process PETI-330 by VARTM, there was a desire to further reduce the  $\eta^*$  of the material and expand its processing window. The first and most obvious method to achieve this was by reduction of the  $M_n$  of the material which typically results in improved melt flow. PETI-330 with  $M_n \sim 1290$  g/mol (Sample III, Table 1) was prepared in-house so as to have a direct comparison between each material. The calculated  $M_n$  was reduced by 25 g/mol increments so as to limit the effect of  $M_n$  reduction upon the mechanical properties of the material while decreasing the  $\eta^*$ . The same molar proportion of m-PD to 1,3,4-APB was used in the synthesis as described for PETI-330 at  $M_n \sim 1290$  g/mol while the molar amounts of a-BPDA and PEPA were varied based on the calculated  $M_n$ . Since the  $\eta^*$  appeared to be dependent upon the molar quantity of a-BPDA, materials with  $M_n > 1290$  g/mol (Samples I and II, Table 1) were prepared. The  $\eta^*$  for these materials showed that the calculated  $M_n$  effect overshadowed the  $\eta^*$  effect offered by a-BPDA even though these materials had increased molar quantities of the monomer. Besides the dependence upon molecular weight, the melt viscosity can also be affected by the molecular weight distribution. For the materials discussed here the molecular weight distributions have not yet been determined, so a definitive assessment cannot be made at this time.

The second method to reduce the  $\eta^*$  was through the incorporation of a flexible diamine. An all meta isomer of 1,3,4-APB (i.e. 1,3,3-APB) was incorporated in PETI-330 at 12.5 mol % increments with a commensurable reduction of 1,3,4-APB that maintains the overall 50 mol % quantity of APB in the PETI material. The calculated  $M_n$  for all materials was maintained at  $\sim 1290$  g/mol. The results of 1,3,3-APB incorporation with respect to  $T_g$  and  $\eta^*$  are shown in Table 2. As expected, the cured  $T_g$  decreased with an increasing amount of 1,3,3-APB. The expected  $\eta^*$  trend however was not observed regardless of temperature. For increasing mol % of 1,3,3-APB, the trend was observed to be sinusoidal with the lowest minimum observed for a 50:50 ratio (B) of the two 1,3-APB monomers.

### 3.2 Simulation

The results obtained from UD-CCM's investigation of the permeability of the three types of carbon fabric and the DM are presented in Figures 3-5. The lead length increased significantly for the uniweave compared to the other fabrics, reflecting the lower out-of-plane permeability of the uniweave system. In addition, race-tracking along the stitches holding the uniweave tows allowed for preferential flow paths through the thickness potentially resulting in dry areas (see Figure 3). The effective permeability values calculated from the flow experiments are shown on Figure 4. The DM permeability is 4-5 orders of magnitude larger compared to the out-of-plane permeability of the fabric. Five layers of DM were stacked beneath the bagging structure increasing the effective permeability and thickness.

UD-CCM used the obtained permeability values in their Liquid Injection Molding Simulation (LIMS) software and ran simulations of the total infusion length and the lead length. Calculations showed that the DM permeability significantly affected both maximum infusion length as well as lead length. Total infusion length increased with an increase in DM

permeability while also increasing lead length and potential for dry spot development. The most significant information obtained from the simulation work related the viscosity of the resin and the permeability of the carbon fabric to the thickness of the infused part (Figure 5). As evident from the Figure, the out-of-plane flow time should be significantly shorter than the gel time to allow larger panel fabrication. Otherwise, the fabric thickness becomes a major limitation during the infusion process for resins with higher viscosities.

The permeability results from MSU for 5 layers of T650-35-3K 8-harness satin are shown in Figure 6. The symbols represent the measured permeabilities. As expected, the through thickness permeability is about two orders of magnitude lower than the in-plane values. The data were fit to a power law mathematical equation for use in the MSU-VARTM simulation model:

$$S = d(V_f)^e \quad (1)$$

where  $S$  is the permeability,  $V_f$  is the fiber volume fraction,  $d$  and  $e$  are constants.

For 10 layers of IM7-uniweave, at low fiber volume fractions, the in-plane permeabilities are similar. However, as the fiber volume fraction increases, the permeability along the fibers,  $S_{xx}$ , becomes much lower than the permeability normal to the fibers,  $S_{yy}$ . This is most likely due to the fibers tows nesting and the bundles compacting under higher compaction loads. As expected, the through thickness permeability is about three orders of magnitude significantly lower than the in-plane values. The low transverse permeability is most likely due to the fact that resin flow is predominately through the nested and highly compacted tow bundles. The data were fit to the power law mathematical equation in Equation (1) for use in the VARTM simulation model and the constants given in Table 3.

Based on the simulation data, it was decided to infuse larger 33 cm x 33 cm panels. It was expected that with PETI-8, infusion of larger and thicker panels would not be an issue. However, the viscosity of PETI-330 is almost an order of magnitude higher than that of PETI-8 at the infusion temperature. As evident from Figure 5, this would, essentially, limit the part size and thickness of the composite.

### 3.3 HT-VARTM

Based on the simulation data from the University of Delaware and MSU, larger panels (33 cm x 33 cm) were processed using HT-VARTM. For these runs, a larger connecting tube comprising a 1.27 cm ( $\frac{1}{2}$ " ) diameter stainless steel tube encased in a 1.91 cm ( $\frac{3}{4}$ " ) diameter tube (around which a heating coil was wrapped) was used that improved the flow of resin into the tool significantly. All three resins, PETI-8, PETI-9 and PETI-330 were used for making these panels following a quasi isotropic lay-up. For the two biaxial fabrics, IM7 and T650, the scale-up did not have any issues. However, with PETI-330 and the IM7-uni fabric, there was incomplete infusion with 16 plies. Hence, for this particular sample, it was decided to infuse only 8 plies. For every cycle, the infusion was carried out at 260 °C for 2 to 2½ h. All carbon fibers were preheat treated (tool with C-fibers taken to 400 °C, held for 1 h and cooled down to 260 °C) to remove sizing and any volatile residue. A staged curing cycle, as described in previous work [16], was also used. Figure 7 shows the photomicrographs of two of these large panels. Table 4

discusses the processing conditions as well as void contents of the 33 cm x 33 cm panels made by HT-VARTM. Although the composite area and volume have quadrupled, the void contents still averaged just under 3.5%.

### **3.4 Mechanical properties**

The SBS data for the PETI-330 samples are shown in Figure 8. The data was compared to commercial grade PETI-5 samples obtained as a part of the High Speed Research program at NASA [19]. These composites were fabricated from unidirectional IM7 prepreg tape and the PETI-5 had a higher  $M_n$  (~5500 g/mol) than the PETI-330. Thus, its strength values were higher. PETI-330 has been processed by RTM to yield composites that have shown an excellent retention of strength at elevated temperatures [3]. In this study, the PETI-330/T650 8HS samples obtained by HT-VARTM had a lower strength at RT when compared to the RTM samples. However, the HT-VARTM samples exhibited higher strengths at elevated temperatures with over 90% retention at 288 °C even though these samples had a higher void content. The PETI-330/IM7 5HS processed by HT-VARTM also showed very good retention of properties, 87% at 177 °C, 80% at 232 °C and 69% at 288 °C and a similar reversal of strength at elevated temperatures when compared to the RTM samples. A previous study with PETI-298 and AS4 fabric found that SBS strength values of the samples processed by RTM or by HT-VARTM were similar at RT and elevated temperatures even though the HT-VARTM sample had a higher void content and a lower fiber volume [13]. The SBS data for PETI-8 specimens have been discussed previously [16] and future work will evaluate the strengths of the PETI-9 specimens.

Defects can occur naturally during composite fabrication, and damage can be induced by impacts during service. Compression loading can be particularly susceptible to defects, due to the tendency for local delaminations to induce buckling failures. For that reason, it was desirable to induce an easily reproducible defect and this led to the tensile or compressive test of a specimen containing a carefully drilled hole. The OHC method provided a controlled simulation of a natural defect in a composite structure, and serve as a method for testing fastener holes. Table 5 denotes the OHC strength of the VARTM samples at room temperature. At the time of this writing, OHC properties at elevated temperatures are in progress. The OHC strength of PETI-330/T650 made by RTM was reported as 270 MPa at 23 °C [3]. For the VARTM samples, the value was lower by 20 MPa. The PETI-8/T650 and PETI-9/T650 composites were similar to those of PETI-330, but produced significantly higher OHC strengths when fabricated with IM7 biaxial fabrics. Upon completion of testing at elevated temperatures, select samples will be aged at elevated temperatures (177 °C for PETI-8 and 9 and 288 °C for PETI-330) for 1000 hours and the OHC strength of these aged samples will be determined at room temperatures.

## **4. Summary**

Experimental work was carried out at both UD-CCM and MSU to determine the permeabilities of the different carbon fabrics and simulation work from both universities was used to guide the NASA HT-VARTM experiments. Based on the results of the modeling, larger panels (33 cm x 33 cm) were fabricated and specimens obtained for OHC. Void contents of the larger panels were slightly higher than previous smaller panels but still averaged just below 3.5%. A new resin, PETI-9, was developed specifically for the HT-VARTM process and the composite panels



exhibited promising results. Work is also ongoing to develop new resins based on PETI-330 that would exhibit lower melt viscosities at the infusion temperature without compromising the  $T_g$ .

### Acknowledgement

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### 5. Figures and Tables

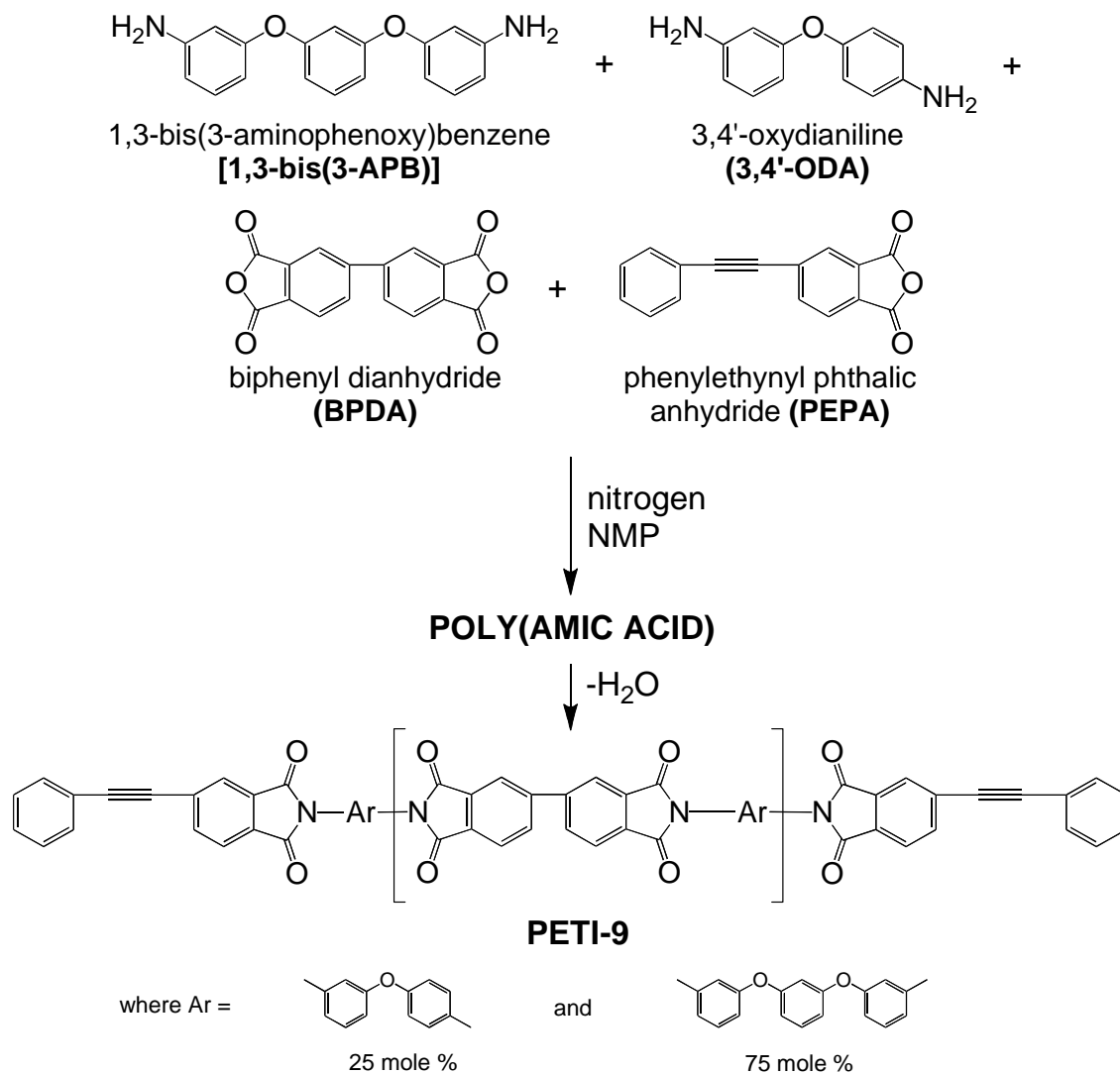


Figure 1: Synthesis of LaRC™ PETI-9

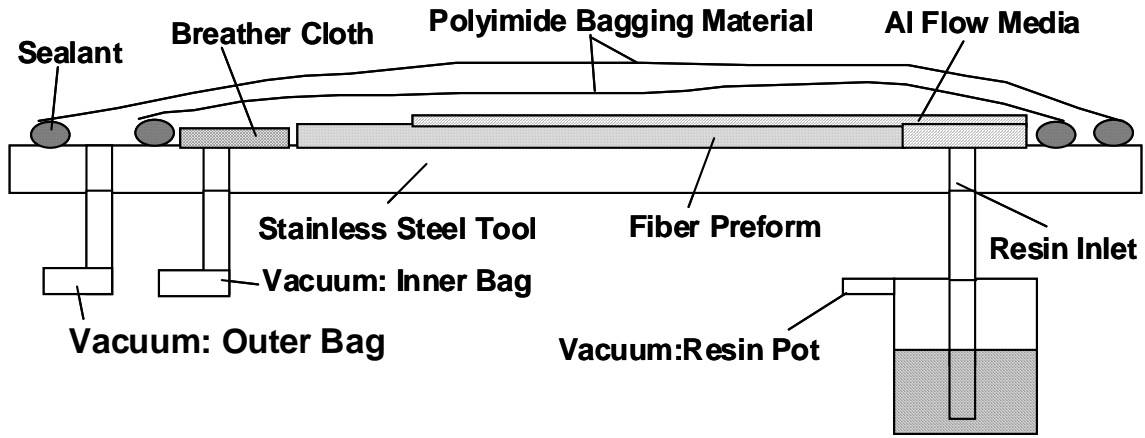


Figure 2: Schematic of HT-VARTM set up

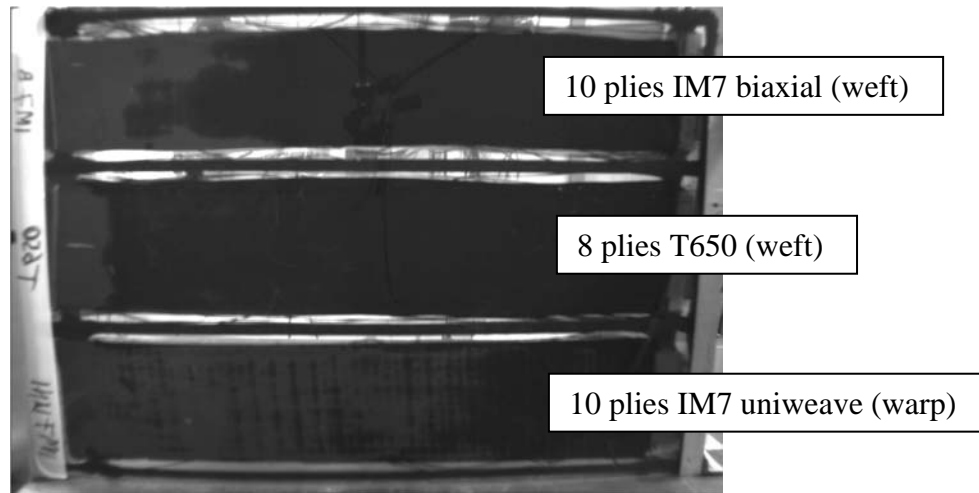


Figure 3: Image of the tool side showing significant differences in lead length and preferential flow through the stitches of the uni-fabric

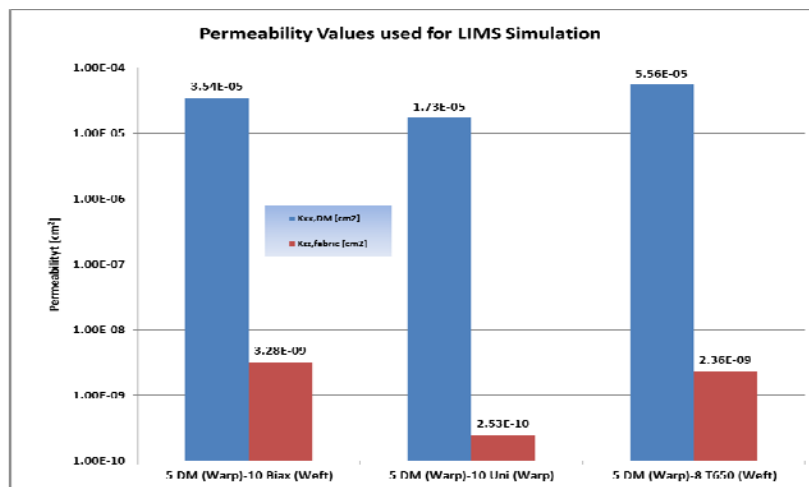


Figure 4: Permeability values obtained by UD flow experiments used in the LIMS simulation

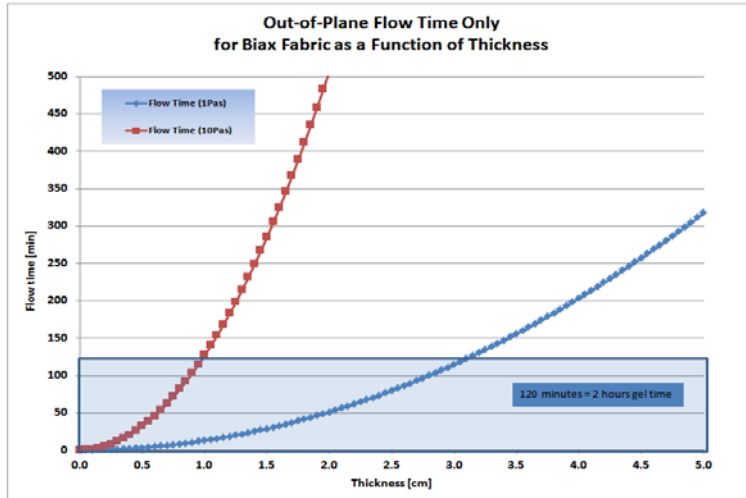


Figure 5: Effect of fabric thickness on infusion length (UD simulation)

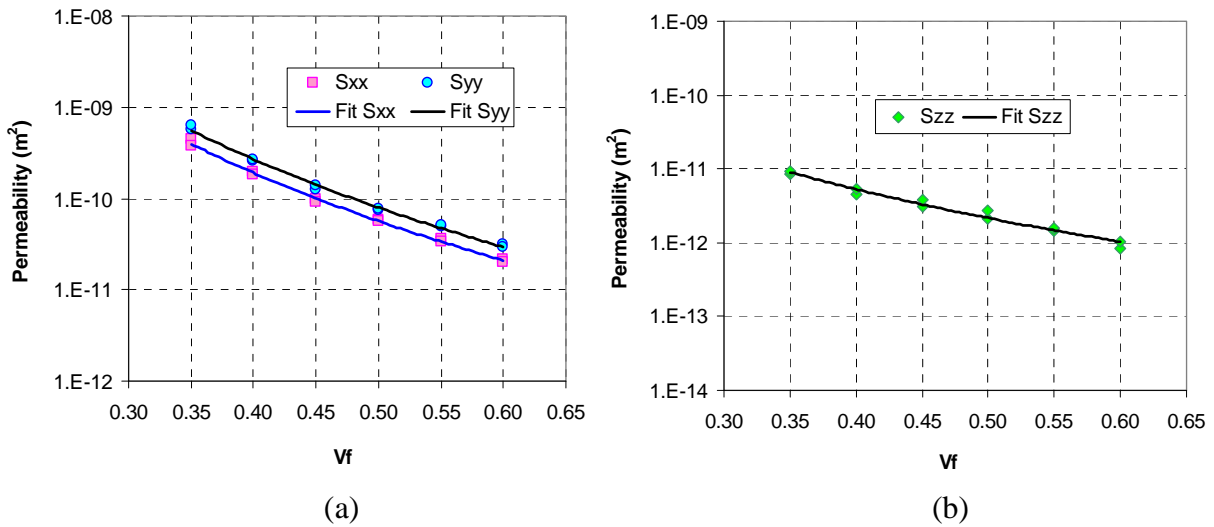
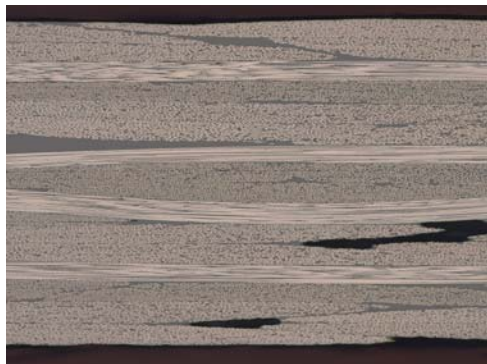
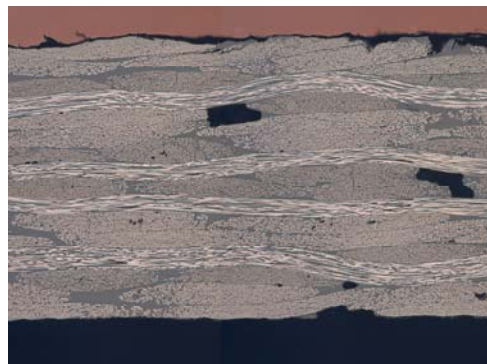


Figure 6: In-plane (a) & transverse (b) permeability versus fiber volume fraction for 5 layers of T650-35-3K 8-harness satin fabric. Symbols represent data. Solid line is best fit to MSU data.



(a)



(b)

Figure 7: Photomicrographs of PETI-9/T650 (a) and PETI-330/IM7 (b)

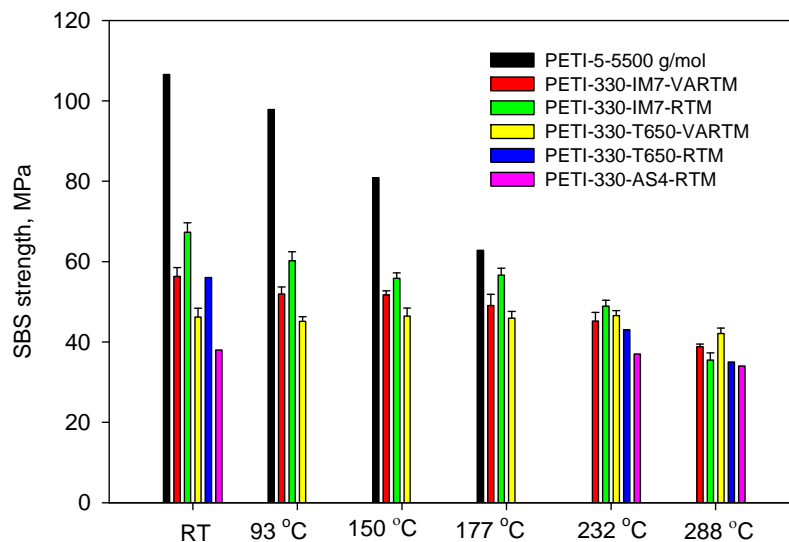


Figure 8: SBS strength of PETI-330/C-fabrics; PETI-5 IM7 prepreg data shown for reference

Table 1. PETI-330 Molecular weight effect

| Sample         | Calc'd $M_n$ , g/mol | $T_g$ , °C | $\eta^*$ at 260 °C, Poise | $\eta^*$ at 280 °C, Poise |
|----------------|----------------------|------------|---------------------------|---------------------------|
| I              | 1390                 | 309        | 109.4 ± 3.5               | 41.0 ± 1.3                |
| II             | 1340                 | 316        | 54.5 ± 2.0                | 16.3 ± 2.3                |
| III (PETI-330) | 1290                 | 327        | 34.9 ± 0.6                | 14.3 ± 0.2                |
| IV             | 1365                 | 333        | 25.0 ± 1.3                | 9.8 ± 0.4                 |
| V              | 1240                 | 338        | 33.0 ± 2.5                | 15.1 ± 1.2                |
| VI             | 1215                 | 340        | 43.0 ± 0.4                | 21.5 ± 1.1                |
| VII            | 1190                 | 343        | 14.0 ± 1.6                | 7.1 ± 1.1                 |

Table 2. Effect of 1,3-Bis(3-Aminophenoxy)Benzene

| Sample   | 1,3,3-APB:1,3,4-APB:mPD mol % | $T_g$ , °C | $\eta^*$ at 260 °C, Poise | $\eta^*$ at 280 °C, Poise |
|----------|-------------------------------|------------|---------------------------|---------------------------|
| PETI-330 | 0:50:50                       | 327        | 34.9 ± 0.6                | 14 ± 0.2                  |
| A        | 12.5:37.5:50                  | 320        | 42.9 ± 0.9                | 17.5 ± 1.2                |
| B        | 25:25:50                      | 306        | 28 ± 0.8                  | 10 ± 0.4                  |
| C        | 37.5:12.5:50                  | 301        | 34 ± 2.8                  | 13.5 ± 1.0                |
| D        | 50:0:50                       | 294        | 48 ± 12.7                 | 28.5 ± 1.1                |

Table 3: Permeability constants for MSU flow model

| <i>Sample</i> | <i>Sxx</i> |          | <i>Syy</i> |          | <i>Szz</i> |          |
|---------------|------------|----------|------------|----------|------------|----------|
|               | <i>d</i>   | <i>e</i> | <i>d</i>   | <i>e</i> | <i>d</i>   | <i>e</i> |
| T650          | 1.30e-12   | -5.45    | 1.81e-12   | -5.46    | 1.32e-13   | -4.03    |
| IM7-uni       | 4.73e-12   | -3.53    | 3.53e-11   | -1.67    | 5.08e-15   | -3.07    |

Table 4: Processing conditions for VARTM of PETI resins for 33 cm x 33 cm panels:

| <b>Resin</b> | <b>C-fabric</b> | <b>Processing Conditions</b>   | <b>Void content, %</b> | <b>Fiber volume, %</b> |
|--------------|-----------------|--|------------------------|------------------------|
| PETI-8       | T650            | $[\pm 45/(0/90)/\pm 45/(0/90)]_s$<br>heat treatment of C-fibers, staged cure cycle | 3.63                   | 63.3                   |
| PETI-8       | IM7             | $[\pm 45/(0/90)/\pm 45/(0/90)]_s$<br>heat treatment of C-fibers, staged cure cycle | 3.46                   | 57.8                   |
| PETI-8       | IM7-uni         | $[-45/0/45/90]_{2s}$<br>heat treatment of C-fibers, staged cure cycle              | 3.58                   | 57.5                   |
|              |                 |  |                        |                        |
| PETI-9       | T650            | $[\pm 45/(0/90)/\pm 45/(0/90)]_s$<br>heat treatment of C-fibers, staged cure cycle | 3.63                   | 63.7                   |
| PETI-9       | IM7             | $[\pm 45/(0/90)/\pm 45/(0/90)]_s$<br>heat treatment of C-fibers, staged cure cycle | 2.68                   | 58.7                   |
|              |                 |  |                        |                        |
| PETI-330     | T650            | $[\pm 45/(0/90)/\pm 45/(0/90)]_s$<br>heat treatment of C-fibers, staged cure cycle | 3.28                   | 62.7                   |
| PETI-330     | IM7             | $[\pm 45/(0/90)/\pm 45/(0/90)]_s$<br>heat treatment of C-fibers, staged cure cycle | 2.95                   | 57.5                   |
| PETI-330     | IM7-uni         | $[-45/0/45/90]_{2s}$<br>heat treatment of C-fibers, staged cure cycle              | 3.79                   | 53.5                   |

Table 5: RT Open hole compression strength of various HT-VARTM composites

| <b>Resin</b> | <b>C-fabric</b>   | <b>OHC strength, MPa</b> | <b>Standard deviation</b> |
|--------------|-------------------|--------------------------|---------------------------|
| PETI-330     | T650              | 249.97                   | 9.36                      |
|              | IM7               | 218.05                   | 6.73                      |
|              | IM7-uni (8 plies) | 216.14                   | 8.41                      |
| PETI-8       | T650              | 257.68                   | 2.97                      |
|              | IM7               | 264.51                   | 7.30                      |
|              | IM7-uni           | 245.15                   | 9.17                      |
| PETI-9       | T650              | 258.34                   | 3.73                      |
|              | IM7               | 251.20                   | 10.34                     |
|              | IM7-uni           | TBD                      | TBD                       |

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