High Temperature VARTM of Phenylethynyl Terminated Imides (PETI) Resins

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Abstract

Fabrication of composite structures using vacuum assisted resin transfer molding (VARTM) is generally more affordable than conventional autoclave techniques. Recent efforts have focused on adapting VARTM for the fabrication of high temperature composites. Due to their low melt viscosity and long melt stability, certain phenylethynyl terminated imides (PETI) can be processed into composites using high temperature VARTM (HT-VARTM). However, one of the disadvantages of the current HT-VARTM resin systems has been the high porosity of the resultant composites. For aerospace applications a void fraction of less than 2% is desired. In the current study, two PETI resins, LARC™ PETI-330 and LARC™ PETI-8 have been used to fabricate test specimens using HT-VARTM. The resins were infused into carbon fiber preforms at 260 ºC and cured between 316 ºC and 371 ºC. Modifications to the thermal cycle used in the laminate fabrication have reduced the void content significantly (typically ≤ 3%) for carbon fiber biaxially woven fabric. Photomicrographs of the panels were taken and void contents were determined by acid digestion. For carbon fiber uniaxial fabric, void contents of less than 2% have been obtained using both PETI-8 and PETI-330. Mechanical properties of the panels were determined at both room and elevated temperatures. These include short beam shear and flexure tests. The results of this work are presented herein.

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1. Introduction

Due to their excellent physical and mechanical properties, aromatic polyimides are finding use in aerospace applications. Polyimide composites are very attractive for applications that require a high strength to weight ratio and excellent thermal stability. Recent work at NASA Langley Research Center (LaRC) has concentrated on developing new polyimide resin systems for advanced aerospace applications that can be processed without the use of an autoclave. Researchers have developed several polyimides from various aromatic diamines and dianhydrides that can be melt processed into coatings, adhesives, composites and films. Controlled molecular weight imide oligomers containing phenylethynyl groups [phenylethynyl terminated imide (PETI), e.g. PETI-8, PETI-330] exhibit exceptional processability during fabrication of neat resin moldings, bonded panels and composites. LaRC™ PETI-330 is a low molecular weight imide oligomer (number average molecular weight ($M_{n)}$ ~1250 g/mole with end caps) with a low, stable melt viscosity and a glass transition temperature (T_g) of around 330 °C after curing for 1-2 h at 371 °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 designed specifically for resin transfer molding (RTM) and resin infusion (RI) processing and has been used for making composites by RTM and RI. PETI-330 laminates exhibit excellent thermal and mechanical properties [1,2]. PETI-330 laminates have exhibited excellent retention of room temperature open hole compression strength and short beam shear strength after aging 1000 hr at 288 °C [3]. LaRC™ PETI-8 is an aromatic polyimide (M_n ~1125 g/mole with end caps) based on 3,3',4,4'-biphenyltetracarboxylic dianhydride, a 50:50 molar ratio of 3,4'-oxydianiline and 1,3-bis(3-aminophenoxy) benzene, with 4-phenylethynylphthalic anhydride as the endcapping agent. PETI-8 has a T_g of around 300 °C after curing for 1 h at 371 °C and produces excellent tensile shear strengths and flatwise tensile strengths when processed under vacuum bag pressure only [4], eliminating the need for costly autoclave processing. Composites have been processed using standard and double-vacuum-bag processes and mechanical properties including short beam shear strength, flexural strength and modulus have been evaluated at various temperatures [5].

Vacuum assisted resin transfer molding (VARTM) process was developed as a variation of RTM over ten years ago for application in commercial and military, ground-based and marine composite structures [6,7]. The upper tool of the matched metal mold used in RTM is replaced in the VARTM process by a formable vacuum bag material. Both transfer of the matrix resin and compaction of the part are achieved using atmospheric pressure alone. Flow of the resin into the part is improved through the use of a resin distribution medium [8]. The highly-permeable medium induces resin flow through the thickness of the part, reducing fill times. VARTM has shown potential to reduce the manufacturing cost of composite structures. In VARTM, the fibrous preform is infiltrated on a rigid tool surface contained beneath a flexible vacuum bag. Both resin injection and fiber compaction are achieved under pressures of 101.3 KPa or less. 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% [9]. VARTM using vinyl ester resins have traditionally yielded composites with low void contents as well and have found applications in the marine industry for making yacht hulls [10] and for rotorline wind turbine blades [11]. However, it should be noted that the focus so far has been on VARTM at room temperature. The Seemans Resin Infusion Molding Process (SCRIMP), patented by TPI Composites [8], is a vacuum infusion process using a high-permeability layer to rapidly distribute the resin on the part surface and then allow through-thickness penetration. The Controlled Atmospheric Resin Infusion Process (CAPRI) patented by The Boeing Company [12], is a SCRIMP variation where vacuum debulking and a reduced pressure difference is used to minimize thickness gradients and resin bleeding.

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 VARTM at *high temperatures*, hence forth referred to as HT-VARTM. In this case the resins are infused at temperatures above 250 °C, and cured between 316 °C and 371 °C. In HT-VARTM, resin flow lines, tools, sealants and bagging materials must be able to tolerate the high temperature processing cycle. Preliminary evaluation of these resins has shown that they exhibit the necessary melt flow characteristics for HT-VARTM processing, but the laminates typically have void contents greater than 7% by volume [13, 14]. In the past few years, researchers at NASA LaRC have been successful in reducing the void content in composite parts to less than 3% while still achieving sufficient fiber volume (>58%) [15]. Initial work focused on identifying the source of the volatiles leading to void formation. It was determined that due to the high temperature required for infusion and the low pressure, a small amount of degradation of the phenylethynyl groups was occurring leading to volatile by-products. By adjusting the processing cycle, void content was reduced to routinely achieve ≤3%.

This paper focuses on the HT-VARTM processing trials carried out under several conditions by control of the process variables in an effort to reduce voids to less than 2%. In an attempt to further reduce porosity, optimization of the cure cycle by introducing higher fidelity control of the temperature and pressure is underway.

2. Experimental

2.1 Materials

Two 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.

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., CA, USA.

2.2 High Temperature VARTM

The HT-VARTM set-up utilized in this work is shown in Figure 1. A 1.27 cm thick steel plate was utilized as a tool. Three holes were drilled and tapped into the plate to provide one resin inlet and two vacuum outlets. Aluminum (Al) screen material was utilized as the flow medium. Polyimide bagging material and high temperature sealant were used to seal both an inner bag that contained the appropriate number of layers of carbon fiber perform, five layers of Al screen flow media, Release Ease™ fabric, a breather material, and an outer bag that provided redundancy should a leak occur in the inner bag after infiltration. For IM7 biaxial fibers, ten layers were used with both resins whereas for the T650, ten layers were used with PETI-8 and eight layers with PETI-330. In case of the uniweave fibers, twenty and ten layers were used with PETI-8 and only ten layers with PETI-330.

It was demonstrated earlier [15] that the process worked best using a two-oven set-up where the two ovens were connected to each other by a heated tube. The resin pot was placed in the first oven and heated to the injection temperature under full vacuum. The tool was heated separately in the second oven under full vacuum, to the injection temperature. Upon reaching the infusion temperature, the resin was degassed for 5 minutes, vacuum on the pot was reduced to 50.8 kPa and the connecting valve between the pot and heating tube was opened to allow the resin to flow until infusion was complete. The connecting tube comprised of a 0.64 cm (¼") diameter stainless steel tube encased in a 1.27 cm (½") diameter tube around which a heating coil was wrapped. The connecting tube was kept at a temperature 2-5 ºC above the infusion temperature. Once infusion was completed, the connecting valve was shut off and the cure cycle was started.

2.3 C-Scan

C-scan of the composite panels were carried out using a 3 axis (x, y and z) Ultrasonic Scanner from SONIX Advanced Acoustic Solutions with a WIN IC (C-Scan) Version 4.1.0k software. A Panametrics transducer of 15 MHz/0.635 cm (0.25") diameter and 3.175 cm (1.25") focal length was used. 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. The C-scan mode, however, has limitations because it provides only planar information and cannot display the depth of flaws in the thickness direction.

2.4 Acid Digestion

Acid digestion of cured composites was carried out following ASTM D3131. Each specimen was weighed to the nearest 0.0001 g and placed into a 100-ml beaker and 30 ml sulfuric acid was added. The beaker was placed on a hot plate and heated until the mixture started to fume; heating was continued for 5 h. The beaker was then removed from the hot plate and 30 ml of 30% hydrogen peroxide was added down the side of the beaker to oxidize the matrix. The solution was allowed to cool; at this point the fibers floated to the top of the solution and the solution appeared clear. If the matrix is not completely digested, the solution may be filtered and reintroduced into the beaker to repeat the digestion procedure. Otherwise, the contents were filtered into preweighed crucibles, washed with ~400 ml distilled water and rinsed with acetone to remove all moisture. The crucibles were dried in an oven at 160 °C for 4 h, cooled to room temperature in a dessicator and weighed. Equations were used to calculate resin and fiber contents and volume fraction of voids using the obtained weights. Calculations were based on a 1.77 g/cc fiber density and a 1.31 g/cc resin density.

2.5 Composite Mechanical Properties

Mechanical properties of the composites were determined by short beam shear strength (SBS) following ASTM D2344 and flexural strength and modulus following ASTM D790. SBS tests were carried out at room temperature and several elevated temperatures. A Sintech 2W mechanical testing machine with a 1000 lb load cell and a heating chamber (Thermcraft) was used. The test speed was 1.27 mm/min. The flexural tests were carried out at room temperature, 177 °C and 288 °C (PETI-330 only) at a test speed of 0.76 mm/min using the same load cell and heating chamber. The transverse flexural tests (carried out perpendicular to the fiber direction) followed the method discussed by Adams et. al. [16] and used a load cell of 200 lb and a span-to-thickness ratio of 8:1.

3. Results and Discussion

Based on previous work at NASA [15] and in order to have better control of the processing parameters, the two-oven set up was used. Each type of carbon fabric was heat treated with a 1 h hold at 400 °C prior to infusion to remove sizing. Both PETI-8 and PETI-330 were heated to the infusion temperature of 260 ºC under vacuum and further degassed at that temperature for 5 mins. Depending on the type of carbon fabric used, the infusion time varied. Compared to PETI-330, PETI-8 always took less time to infuse. The reason for this was the lower viscosity of the PETI-8 at 260 °C. For PETI-330, the infusion time was typically 20 mins for the IM7 biaxial, and about 1 h for both the T650 and the uniweave. However, all samples were typically allowed to infuse for up to 2 h after the start of infusion to ensure that the resin had flowed through the thickness of the panel. PETI-8 panels typically wet out the preforms in 10 to 20 mins, but were allowed to continue to infuse for up to an hour. Upon completion of infusion, the inlet valve connecting the pot to the tool was shut off and the cure cycle started. A staged cure cycle was used for both resins. For PETI-330, the cure cycle involved taking the panel to 310 ºC and holding for 8 h. After that it was taken to 371 ºC and held for another 1 h before being cooled down to room temperature. The second hold at 371 ºC is significant for PETI-330 since the resin has a cured T_g of 330 °C and taking it to a temperature above its T_g allows the polymer chains to remain mobile which in turn enables additional reaction and consolidation. In the case of PETI-8, 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. Unlike for PETI-330, holding the sample for an additional 1 h at 371 °C did not reduce the void content of the sample. Since the T_g of PETI-8 is lower than the cure temperature of 316 °C the polymer chains remain mobile allowing full reaction and consolidation. For both resins, the staged cure cycle reduced the void content to 3.0 - 3.4% for the 5-HS IM7 fabric [Figure 2].

Several other approaches were investigated in an attempt to further reduce the void content. All these experiments were carried out with the IM7 biaxial fiber. In the first experiment, an additional layer of breather cloth was placed above the Al screen flow media in an effort to reduce the voids. This was based somewhat on the use of porous membranes [17] for VARTM, a process carried out at the University of Delaware. Figure 3(a) shows a section from this panel. The average void content was 3.1% with two of the four samples having a void content <3%. In the second experiment, in addition to the extra layer of breather cloth, the carbon fibers were heat treated (tool with C-fibers taken to 400 °C, held for 1 h and cooled down to 260 °C) to remove the fiber sizing and any low molecular weight residue that may have remained behind on the fibers during manufacture. Figure 3(b) shows the photomicrograph of this sample and the average void content was 2.5%. In the third experiment the vacuum on the bags was adjusted. Upon completion of infusion of the resin at 260 °C, the vacuum on the outer bag was removed and the vacuum on the inner bag was brought down to 50.8 kPa (15" of Hg) and taken back to 101.6 kPa. This vacuum fluctuation or "bumping" was done twice on the inner bag. The vacuum on the outer bag was then brought back to 101.6 kPa and the normal cure cycle was started. As in the previous two runs, here too, an extra layer of breather cloth was placed above the flow media.

A very high quality panel was obtained and Figure 4 shows the photomicrographs from this panel. Samples from this panel gave the lowest void content obtained to date – 2.3% with one of the four samples having a void content <2.0%. For PETI-8, heat treatment of the carbon fiber and the staged cure cycle brought the void content down to 2.6% [Figure 5]. However, the "bumping" process did not make any significant difference.

A significant reduction in the void content was observed when the uniweave carbon fabric was used. Using the PETI-8 resin, composite panels containing ten and twenty plies of IM7 unidirectional carbon fabric were made. In each case, the lay-up was such that the fiber direction corresponded to the resin flow direction. Figure 6 shows the photomicrographs of these samples. The processing cycle involved heat treatment of the carbon fibers as well as staging the cure cycle and for both samples, ***a void content of <2% was obtained.*** In the case of PETI-330, owing to its higher viscosity, only the composite with ten plies could be processed. For the sample with "bumping" the void content was 1.3% and 0.9% with no "bumping". Table 1 summarizes the various conditions for VARTM and the corresponding void contents and fiber volumes. Evidently, use of the uniweave fibers has achieved the goal of making VARTM composites having <2% voids.

The third set of carbon fibers that is currently being tested is the T650. A run with PETI-8 has been successfully completed while one with the PETI-330 is pending. With PETI-8 the composites exhibited a void content above 3% and fiber volume over 60%.

Specimens used for mechanical testing were prepared following the standards mentioned in section 2.5. The SBS tests were carried out over several temperatures. For PETI-8 samples, SBS was also carried out on the uniweave composite containing 20 plies. The data was compared to PETI-8 samples that were obtained by double vacuum bag (DVB) [5] and PETI-5 samples obtained as a part of the High Speed Research (HSR) program at NASA [18]. Figure 7 shows the SBS data with the error bars representing the standard deviation. It should be noted that the PETI-5 and PETI-8 used for DVB had higher molecular weights (5,500 g/mol and 2,500 g/mol respectively) than the PETI-8 used in this study (1125 g/mol). At room temperature (RT), the PETI-8/uniweave had higher strength compared to PETI-8/T650 or PETI-8/IM7. However, the uniweave sample had only 68% retention of strength at 177 °C compared to 89% for T650 and 79% for IM7. The PETI-5 and the PETI-8/DVB had higher strength values mainly because they had higher molecular weights and lower crosslink densities. However, the VARTM samples from this study had better retention of strength at elevated temperatures.

The RT flexure strength, measured in the fiber direction, showed a lower value for the PETI-8/uniweave when compared to either PETI-8/DVB or PETI-5 [Figure 8]. However, at 177 °C, the VARTM sample showed 73% retention of strength compared to 37% for the DVB sample. This reduction in strength may be due to the fact that the PETI-8 used in the DVB process had a lower T_g (<200 °C) compared to the PETI-8 used for the current study. The flexure strength was also measured in direction perpendicular to the fiber orientation and the RT strength was 81 MPa.

Figure 9 denotes the SBS data for the PETI-330 samples. The data for PETI-5 has been included for reference purpose only. It should be noted that the PETI-5 had a higher molecular weight than the PETI-330 and hence its strength values were higher. However, at higher temperatures, the PETI-330/IM7 processed by VARTM showed a very good retention of properties – 87% at 177 °C, 80% at 232 °C and 69% at 288 °C. It has been seen in a previous study with PETI-298 and AS 4 fibers [13] that SBS strength values of the samples at RT and at elevated temperatures are similar when processed by RTM or by VARTM even though the VARTM sample has a higher void content and a lower fiber volume. PETI-330 has been processed by RTM to yield composites that have shown an excellent retention of strength at elevated temperatures [3]. For the RTM studies T650 and AS4 carbon fibers were used. The current focus has been primarily with IM7 but future processing trials will involve T650 which will then allow a direct comparison between the two processes.

Flexure strengths of the PETI-330/uniweave samples were determined at RT and elevated temperatures. From Figure 10, it can be observed that the samples had good retention of properties at higher temperatures – 78% at 177 °C and 61% at 288 °C. This retention is similar to that exhibited by the PETI-5 samples. Transverse flex samples showed an average strength of 44.2 MPa with a 77% retention of strength at 288 °C.

4. Summary

One of the toughest challenges faced in HT-VARTM is the reduction of void content to 2% or less required for aerospace applications. Prior to this work, it had not been possible to fabricate composite panels with less than 2% voids from high temperature polyimide resins by conventional HT-VARTM in spite of the fact that these resins have been successfully fabricated into high quality panels using RI or RTM. This work has successfully processed HT-VARTM panels with low void contents by various process modifications. These involved curing at a lower temperature but for a longer period of time, and staging the cure cycle. With biaxial carbon fabric, composites with void content <3% have been routinely fabricated. With the uniweave carbon fabric, the void content was lowered to <2%, thereby meeting the requirement for aerospace applications.

Mechanical properties of the panels that include SBS and flexure tests were determined at both room and elevated temperatures. For both PETI-8 and PETI-330, the composites showed very good retention of mechanical properties at elevated temperatures. Future work will involve implementation of higher fidelity temperature and pressure controls for the HT-VARTM process followed by additional processing trials, as well as comparing mechanical properties of samples with those obtained from other processes like RTM.

5. Acknowledgement

The authors thank James M. Baughman of Lockheed Martin for the photomicrographs, Janice Y. Smith of NASA LaRC for mechanical testing and Joseph G. O'Donnell of NASA LaRC for cutting the panels. The authors also appreciate the valuable discussions and feedback they received from Dr. Joseph G. Smith, Jr. of NASA LaRC.

6. Figures

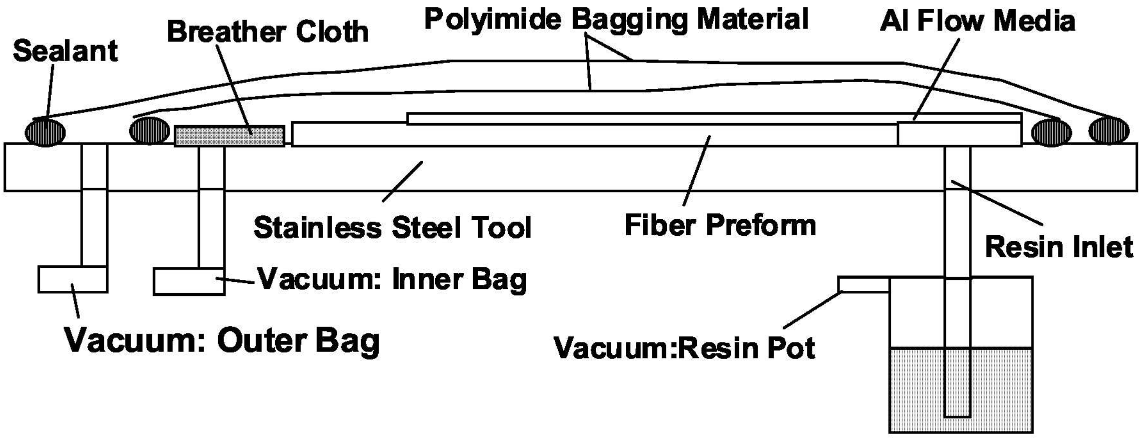
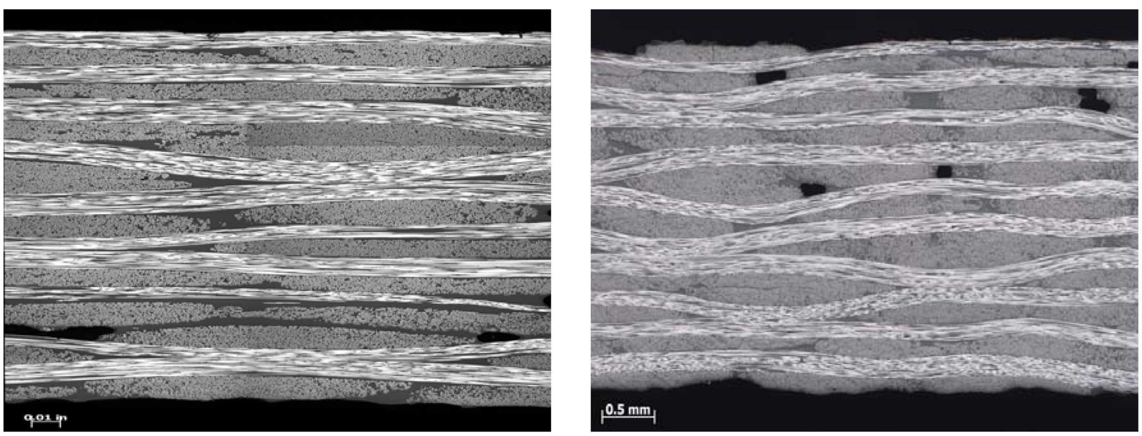
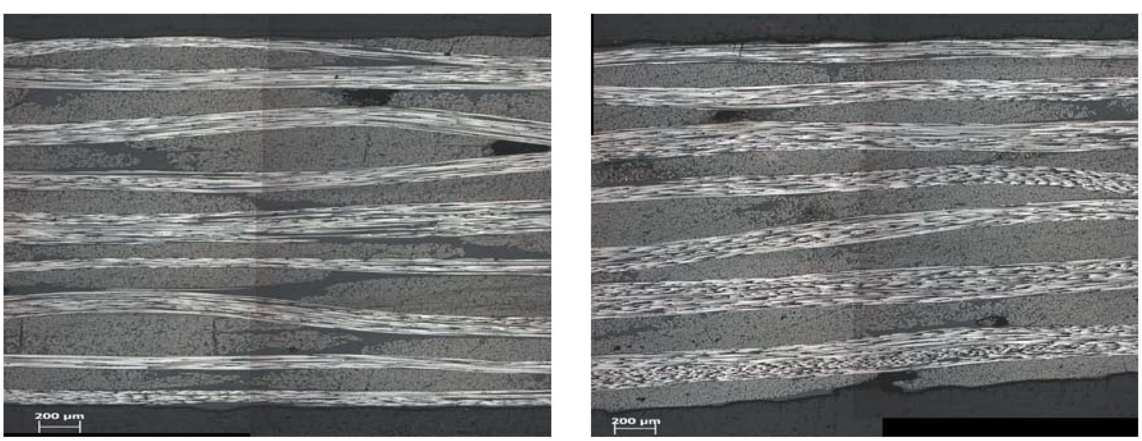


Figure 1: Schematic of HT-VARTM set up



(a) (b)

Figure 2: Photomicrograph of PETI-8(a) and PETI-330 (b) after staged cure cycle



(a) (b)

Figure 3: Photomicrograph of PETI-330; with extra breather cloth (a), heat treatment of C-fibers (b)

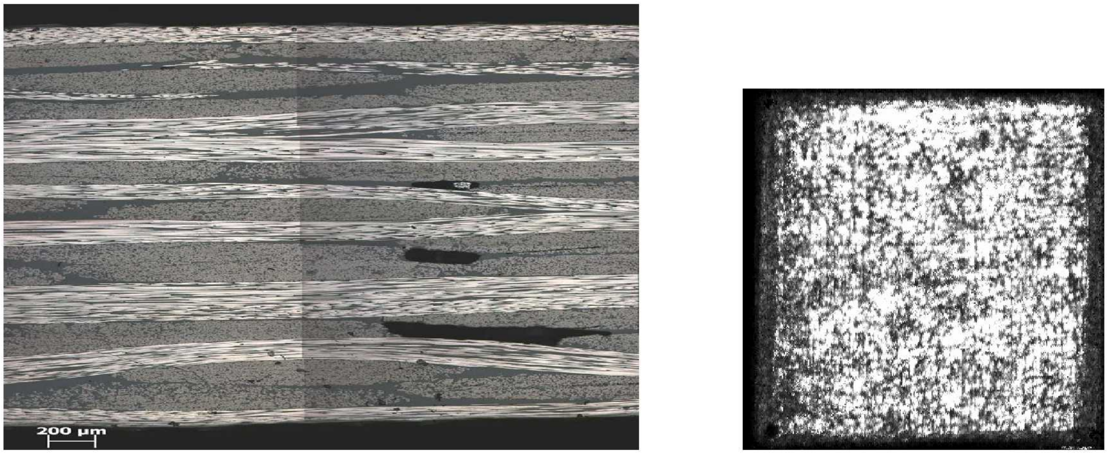
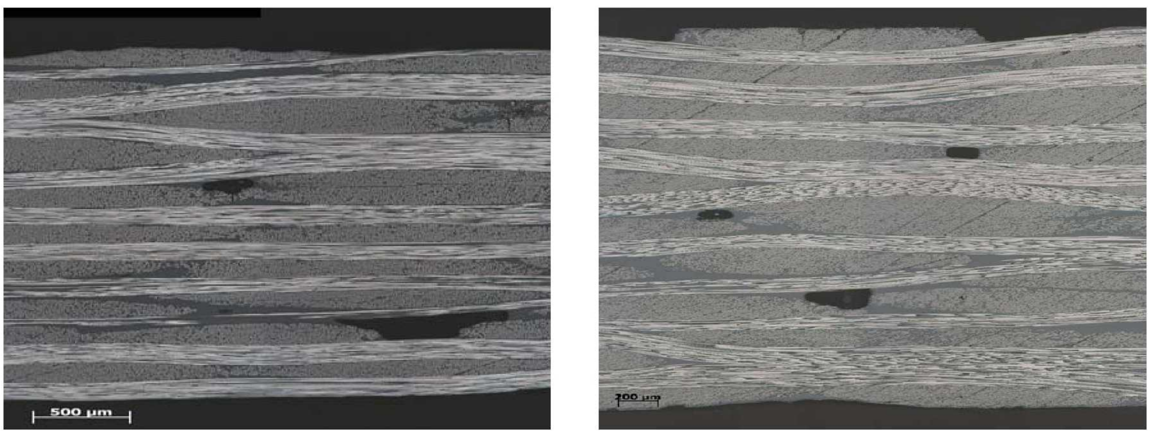


Figure 4: Photomicrograph of PETI-330 composite panel with 2.3% void content and its corresponding C-scan



(a) (b)

Figure 5: Photomicrograph of PETI-8; without “bumping” (a), and with “bumping” (b). Both have undergone heat treatment of C-fibers and staged cure cycle with void contents of 2.6%

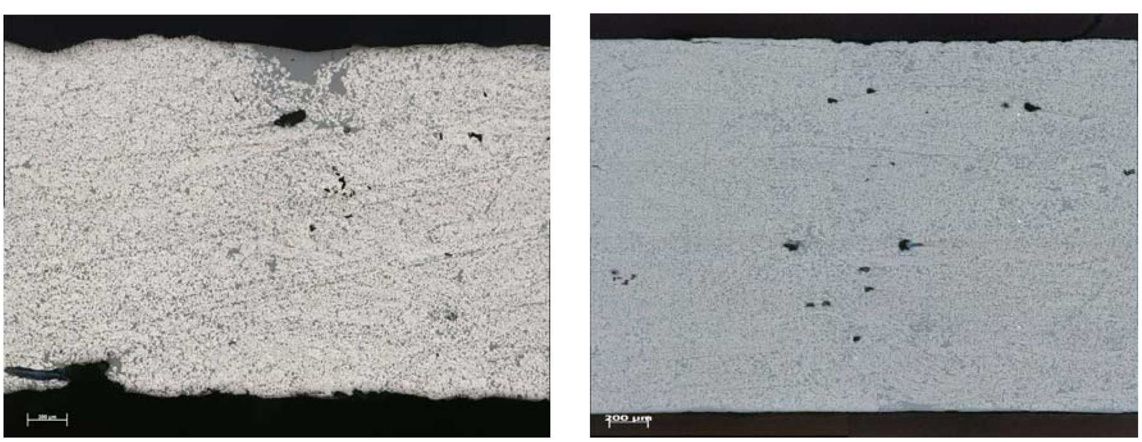


Figure 6: Photomicrographs of PETI-8/uniweave fabrics; 10-plies (a) and 20 plies (b)

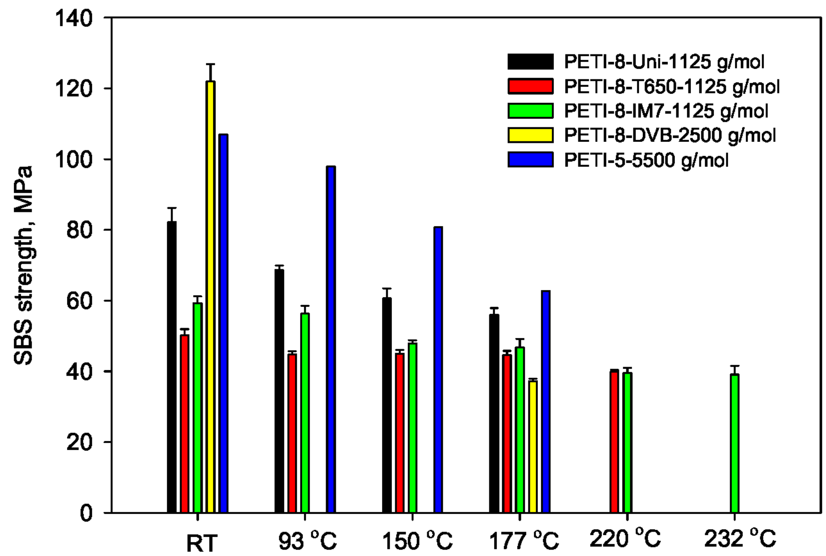


Figure 7: SBS strength of PETI-8 with various C-fabrics; PETI-5 data shown for reference

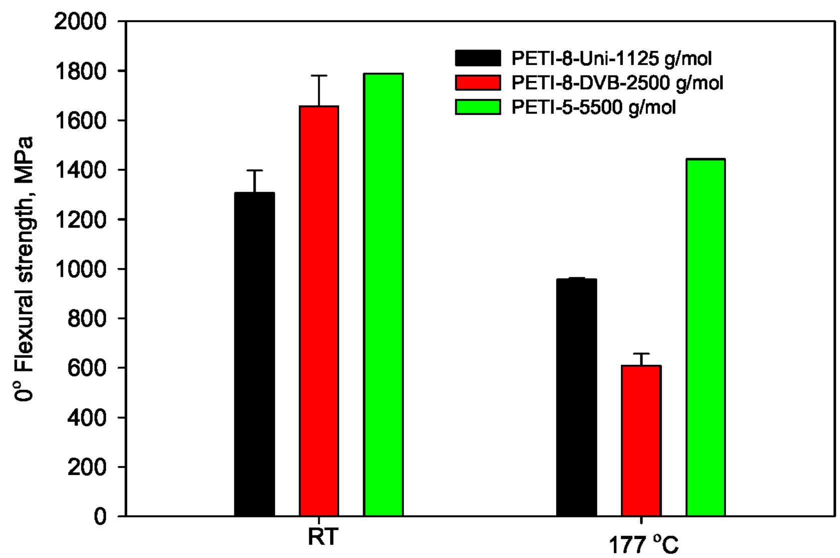


Figure 8: Flexure strength of PETI-8/uniweave C-fabrics; PETI-5 data shown for reference

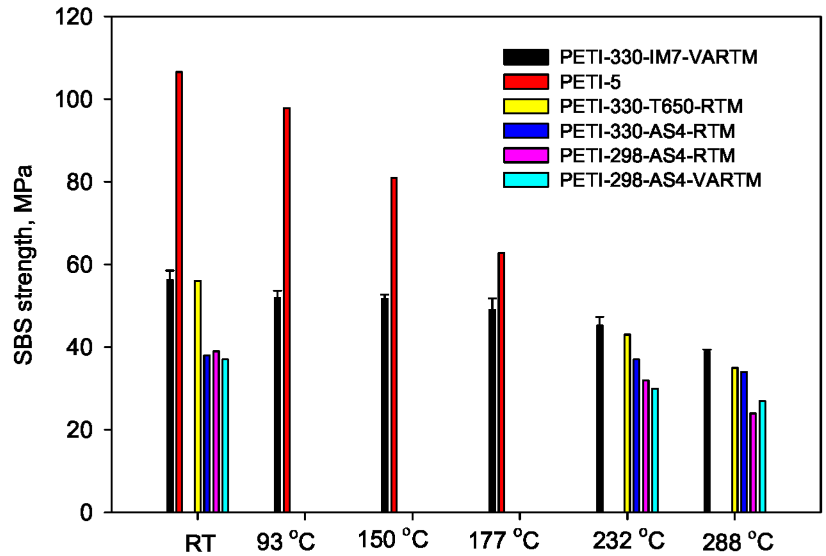


Figure 9: SBS strength of PETI-330 with various C-fabrics; PETI-5 data shown for reference

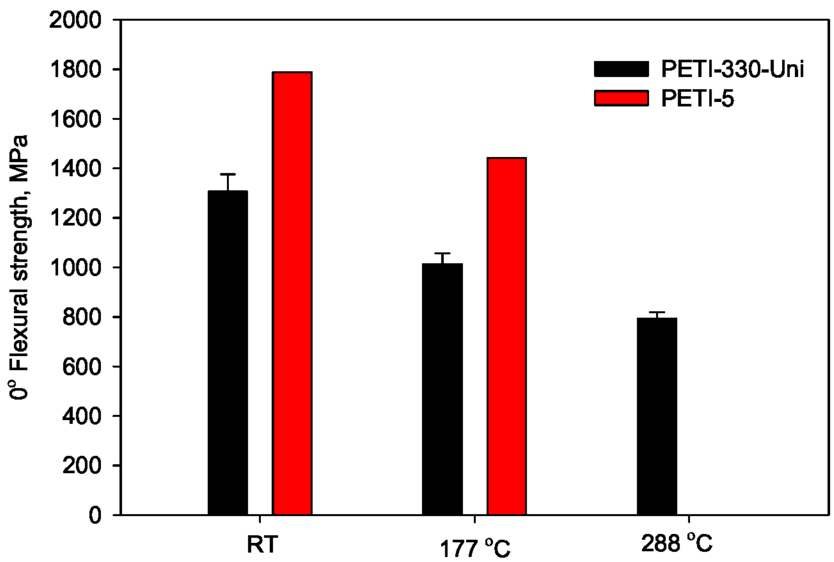


Figure 10: Flexure strength of PETI-330/uniweave C-fabrics; PETI-5 data shown for reference

Table 1: Processing conditions for VARTM of PETI resins

Resin	**C-fabric**	**Processing Conditions**	**Void content, %**	**Fiber volume, %**
PETI-8	IM7	staged cure cycle	3.0	55.4
PETI-8	IM7	heat treatment of C-fibers, staged cure cycle	2.6	54.9
PETI-8	IM7	heat treatment of C-fibers, "bumping", staged cure cycle	2.6	55.4
PETI-8	IM7-uni	heat treatment of C-fibers $[0]_{20}$ staged cure cycle	**1.4**	**59.1**
PETI-8	IM7-uni	heat treatment of C-fibers $[0]_{10}$ staged cure cycle	**1.1**	**61.2**
PETI-8	T650	heat treatment of C-fibers, staged cure cycle	>3%	>60%
PETI-330	IM7	staged cure cycle	3.4	54.7
PETI-330	IM7	Extra breather cloth, staged cure cycle	3.1	56.7
PETI-330	IM7	Extra breather cloth, heat treatment of C-fibers, staged cure cycle	2.5	57.3
PETI-330	IM7	Extra breather cloth, "Bumping" @ 260°C, staged cure cycle	2.3	54.7
PETI-330	IM7-uni	Extra breather cloth, heat treatment of C-fibers $[0]_{10}$, staged cure cycle	**0.9**	**60.6**
PETI-330	IM7-uni	Extra breather cloth, heat treatment of C-fibers $[0]_{10}$, "Bumping", staged cure cycle	**1.3**	**59.8**

* Pot vacuum and tool vacuum were 50.8 kPa and 101.6 kPa respectively for all runs

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