PHENYLETHYNYL TERMINATED IMIDE (PETI) COMPOSITES MADE 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 pressures, during high temperature processing, to create fully consolidated composite parts. Recently, NASA Langley has licensed a series of low viscosity Phenyl Ethynyl Terminated Imide, PETI, oligomers that possess a wide processing window to allow for Resin Transfer Molding, RTM, processing. These resins, PETI-8 and PETI-330, demonstrate void fractions of ~1% under elevated pressure consolidation. However, when used with a standardized thermal curing cycle in a High Temperature Vacuum Assisted RTM (HT-VARTM) process, they display undesirable void contents in excess of 7%. It was determined previously that under the thermal cycles used for laminate fabrication, the phenylethynyl endcap underwent degradation leading to volatile evolution. Modifications to the processing cycle used in the laminate fabrication have reduced the void content significantly (typically less than 3%) for carbon fiber biaxially woven fabric. For carbon fiber uniaxial fabric, void contents of less than 2% have been obtained using both PETI-8 and PETI-330. The resins were infused into carbon fiber preforms at 260 ºC and cured between 316 ºC and 371 ºC. Photomicrographs of the panels were taken and void contents were determined by acid digestion. 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.

1 Introduction

Due to their combination of processability and mechanical properties, aromatic polyimides are finding increased use in aerospace applications. Polyimide composites are very attractive for applications that require a high strength to weight ratio and performance at use temperatures above 260 °C. 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), are readily processed into 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 a low melt viscosity and a post cure glass transition temperature (T_g) of 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 designed specifically 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 almost 98% of room temperature open hole compression strength after aging 500 h at 288 °C [3]. LaRC™ PETI-8 is a phenyleethynyl endcapped aromatic polyimide (M_n ~1125 g/mole) based on 3,3',4,4'-biphenyltetracarboxylic dianhydride, a 50:50 molar ratio of 3,4'-oxydianiline and 1,3-bis(3-aminophenoxy) benzene. PETI-8 has a post cure T_g of around 300 °C, and produces acceptable tensile shear strengths and flatwise tensile strengths when processed under vacuum bag pressure only [4]. This eliminates the need for costly autoclave processing. PETI resin composites have been processed using standard and double-vacuum-bag (DVB) processes and 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 twenty years ago for application in commercial and military, ground-based and marine composite structures [6,7] and has shown potential to reduce the manufacturing cost of these structures. The upper tool of the matched metal mold used in RTM is replaced in the VARTM process by formable vacuum bag material. Both transfer of the matrix resin and compaction of the part were achieved using atmospheric pressure. Flow of the resin into the part was improved through the use of a permeable resin distribution medium [8] that induces resin flow through the thickness of the part, reducing fill time. The Seemans Composite Resin Infusion Molding Process (SCRIMP) [8] is a vacuum infusion process using a high-permeability layer to rapidly distribute the resin on the part surface and allows 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 as well and have found applications in the marine industry [11, 12]. However, it should be noted that the focus so far has been on room temperature VARTM.

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, a 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. Here the resins were infused at temperatures above 250 ºC and cured between 316 and 371 ºC. Although the evaluation of these resins has shown that they exhibit the necessary melt flow characteristics for HT-VARTM processing, the resulting laminates have void contents greater than 7% by volume [13, 14]. Recently, researchers at NASA LaRC have reducing the void content to <3%, while still achieving sufficient fiber volume (>58%) [15]. It was determined that the high temperature required for infusion with low pressure consolidation resulted in degradation of some the phenylethynyl groups, forming 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 <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., Anahiem, CA, USA.

2.2 High Temperature VARTM

The HT-VARTM 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. Aluminum (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 perform, 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. 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 the case of the uniweave fibers, twenty and ten layers were used with PETI-8, and only ten layers with PETI-330. Each type of carbon fabric was heat treated with a 1 h hold at 400 °C, prior to infusion, to remove sizing.

Prior experience demonstrated [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 first oven was used to heat the resin pot and the second oven was used to heat the tool and the preform. The connecting tube was.kept at a temperature 2-5 ºC above the infusion temperature. The resin pot was placed in the first oven and heated to the injection temperature under full vacuum. The tool/preform was heated separately in the second oven under full vacuum, to the injection temperature. 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. Vacuum on the pot was then reduced to 50.8 kPa and the connecting valve between the pot and heated 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) was opened to allow the resin to flow until infusion was complete. Depending on the type of carbon fabric used, the infusion time varied. 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. Upon completion of infusion, the inlet valve connecting the pot to the tool was closed 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. 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.

2.3 C-Scan

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 15 MHz Panametrics transducer with 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.

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 concentrated sulfuric acid was added. The beaker was placed on a hot plate and heated until the mixture started to fume, heating 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. The fibers floated to the top of the solution, and the solution appeared clear indicating complete digestion of the matrix. The contents were filtered into preweighed crucibles, washed with ~400 ml distilled water and rinsed with acetone. The crucibles were dried in an oven at 160 °C for 4 h, cooled to room temperature in a dessicator and weighed. Resin and fiber contents and volume fraction of voids were calculated 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 (SBS) according to ASTM D2344 and flexural strength and modulus following ASTM D790. SBS tests were carried out at room temperature and elevated temperatures. 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. The flexural tests were carried out at room temperature, 177 and 288 °C (PETI-330 only) at a crosshead 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 0.89 kN and a span-to-thickness ratio of 8:1.

3 Results and Discussion

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. PETI-8 took less time to infuse due to its lower viscosity at 260 °C. 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. For both resins, the staged cure cycle reduced the void content from ~8% to 3.0 - 3.4% for the 5-HS IM7 fabric [Figure 1]. The second hold at 371 ℃ was 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 enabled additional reaction and consolidation. Unlike PETI-330, holding the sample for an additional 1 h at 371 °C did not reduce the void content of the PETI-8 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.
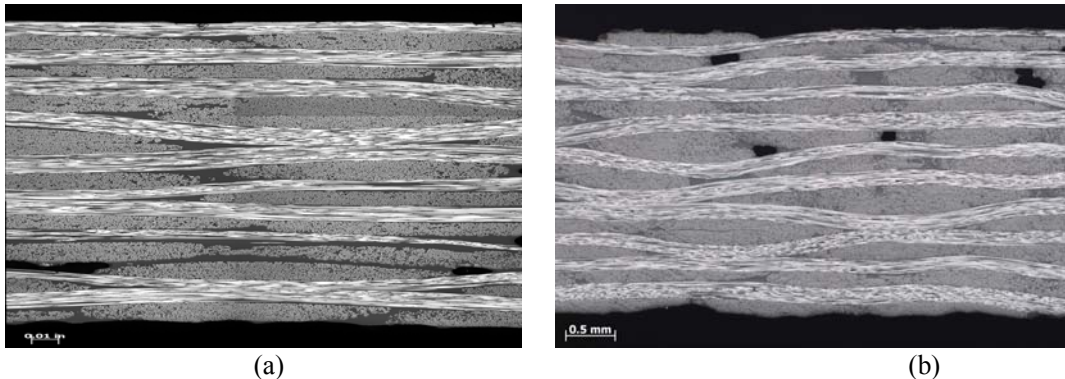

| (a) | (b) |

Figure 1: Photomicrograph of PETI-8/IM7 5HS (a) and PETI-330/IM7 5HS (b) after staged cure cycle.

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 void content. This was based on the use of porous membranes [17] for VARTM, a process carried out at the University of Delaware. A section from this panel is shown on Figure 2(a). The average void content was 3.1%, with two of the four samples having a void content <3%. In the second experiment, an additional layer of breather cloth was used and the 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 low molecular weight residue. The photomicrograph of this sample, Figure 2(b), had an average void content of 2.5%. In the third experiment, the vacuum on the bags was monitored and 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 reduced to 50.8 kPa (15" of Hg) and then increased to 101.6 kPa. This vacuum fluctuation or "bumping" was done twice on the inner bag. The vacuum on the outer bag was then increased to 101.6 kPa, and the normal cure cycle was started. As in the previous two runs, an extra layer of breather cloth was placed above the flow media. A very high quality panel was obtained and Figure 3 shows the photomicrographs from this panel. Samples from this panel had the lowest void content (2.3%)

obtained to date on this type of C-fiber, 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 reduced void content to 2.6% [Figure 4]. However, the "bumping" process did not make any measureable difference (< 0.05%).
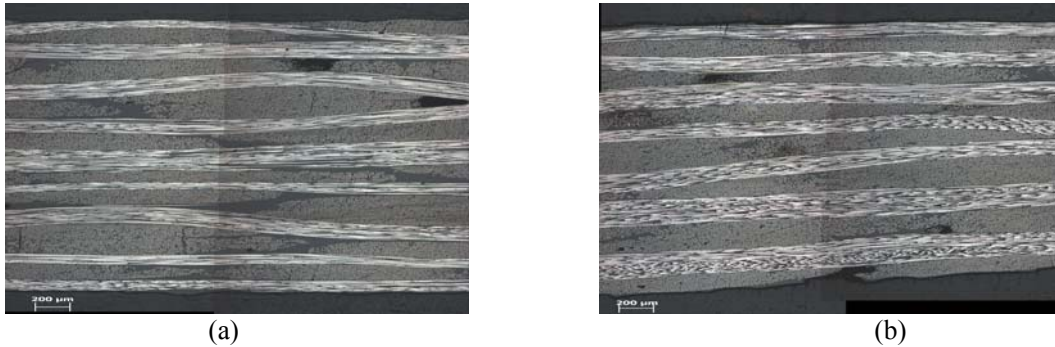

<table>
<tr><td>(a)</td><td>(b)</td></tr>
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Figure 2: Photomicrograph of PETI-330/IM7 5HS; with extra breather cloth (a), heat treatment of C-fibers (b).
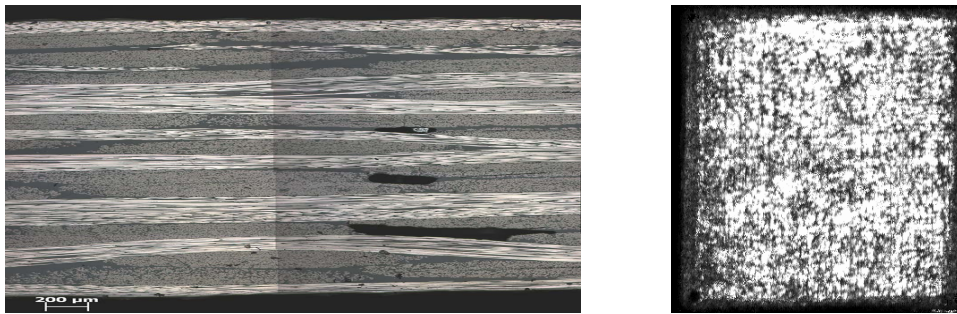

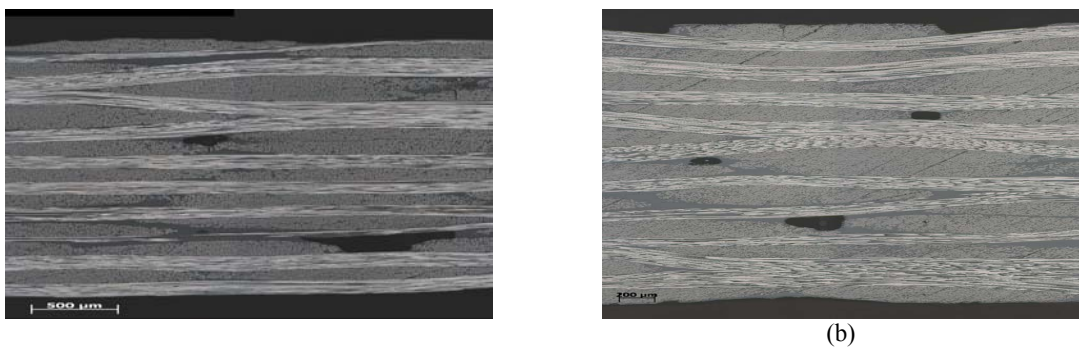Figure 3: Photomicrograph of PETI-330/IM7 5HS panel with 2.3% void content and its corresponding C-scan.

(b)

Figure 4: Photomicrograph of PETI-8/IM7 5HS; 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%.

A significantly lower void content was observed when the uniweave carbon fabric was used. With PETI-8, composite panels containing ten and twenty plies of IM7 unidirectional carbon fabric were made. In each lay-up, the fiber direction corresponded to the resin flow direction. The photomicrographs of these cured samples are shown in Figure 5. The processing cycle involved heat treatment of the carbon fibers and staging the cure cycle for both samples. Here, 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". The various conditions for HT-VARTM processing, the corresponding void contents and

fiber volumes are summarized in Table 1. Evidently, the use of the uniweave fibers achieved the goal of making HT-VARTM composites having <2% voids.

The third set of carbon fibers that were tested was the T650 8HS. With both resins, the composites exhibited a void content slightly >3% and fiber volume >60%.
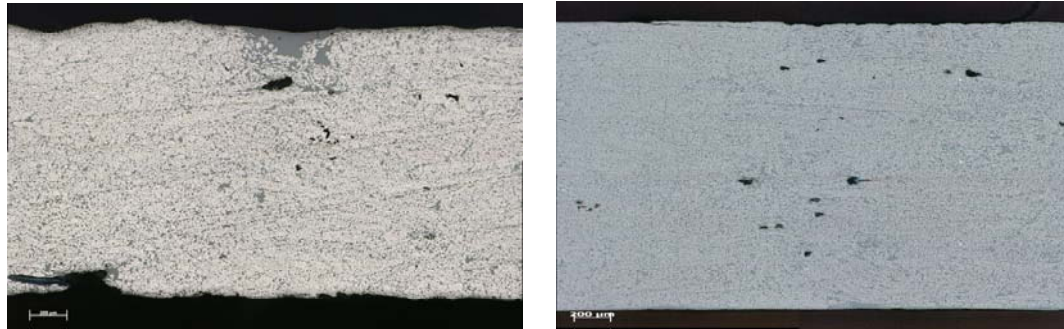
| (a) | (b) |

Figure 5: Photomicrographs of PETI-8/IM7 uniweave fabrics; 10-plies (a) and 20 plies (b).

Table 1: Processing conditions for VARTM of PETI resins.

Resin	C-fabric	*Processing Conditions	Void content, %	Fiber volume, %
PETI-8	IM7 5HS	staged cure cycle	3.0	55.4
PETI-8	IM7 5HS	heat treatment of C-fibers, staged cure cycle	2.6	54.9
PETI-8	IM7 5HS	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 8HS	heat treatment of C-fibers, staged cure cycle	>3%	>60%
PETI-330	IM7 5HS	staged cure cycle	3.4	54.7
PETI-330	IM7 5HS	Extra breather cloth, staged cure cycle	3.1	56.7
PETI-330	IM7 5HS	Extra breather cloth, heat treatment of C-fibers, staged cure cycle	2.5	57.3
PETI-330	IM7 5HS	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**
PETI-330	T650 8HS	Extra breather cloth, heat treatment of C-fibers, staged cure cycle	~3%	>60%

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

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 DVB [5] and commercial grade PETI-5 samples obtained as a part of the High Speed Research (HSR) program at NASA [18]. These composites were fabricated from IM7 unidirectional prepreg tape. The SBS data are shown in Figure 6. The PETI-5 and the PETI-8/DVB tapes had higher strength values as they had higher molecular weights (5,500 g/mol and 2,500 g/mol respectively)

resulting in lower crosslink densities. The PETI-8 used in this study had a molecular weight of 1125 g/mol. At room temperature (RT), the PETI-8/uniweave had higher strength compared to PETI-8/T650 and PETI-8/IM7. However, the uniweave sample had only 68% retention of strength at 177 °C compared to 89% for T650 8HS and 79% for IM7 5HS. The HT-VARTM samples had better strength retention at elevated temperatures than the PETI-5 samples made from commercial grade prepregs.

The RT flexure strength, measured in the fiber direction, showed a lower value for the HT-VARTM PETI-8 when compared to either PETI-8/DVB or PETI-5 unidirectional tape composites [Figure 7]. However, at 177 °C, the HT-VARTM samples showed better strength retention compared to the DVB sample. This reduction in strength of the PETI-8 used in the DVB process may result from a lower T_g (~250 °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 was 81 MPa.
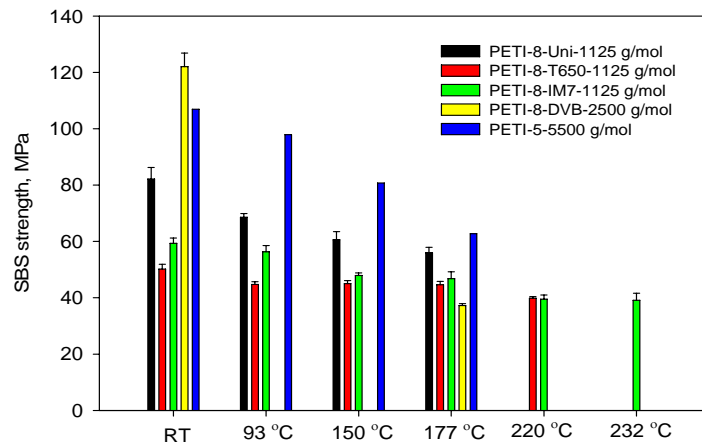

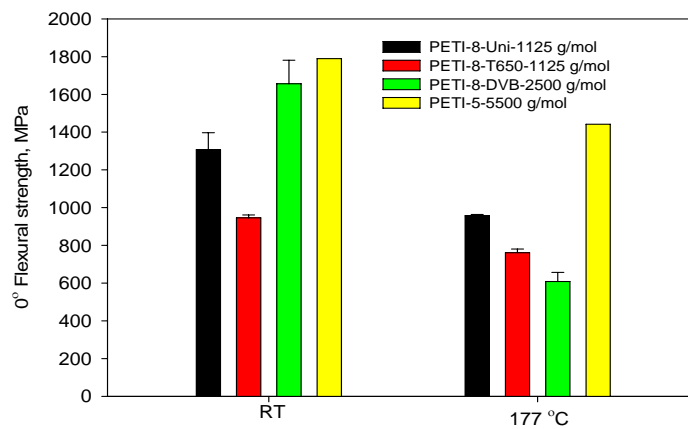Figure 6: SBS strength of PETI-8/C-fabrics; PETI-5 IM7 prepreg data shown for reference.

Figure 7: Flexure strength of PETI-8/C-fabrics; PETI-5 IM7 prepreg data shown for reference.

The SBS data for the PETI-330 samples are shown in Figure 8. The PETI-5 had a higher molecular weight 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 superior strength 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 VARTM also showed a very good

retention of properties, - 87% at 177 °C, 80% at 232 °C and 69% at 288 °C. A previous study with PETI-298 and AS4 fabric found that SBS strength values of the samples processed by RTM or by VARTM were similar at RT and elevated temperatures even though the VARTM sample has a higher void content and a lower fiber volume [13].

Flexure strengths of the PETI-330/IM7 uniweave samples were determined at RT and elevated temperatures. From Figure 9, it can be observed that the samples had good property retention at higher temperatures, – 78% at 177 °C and 61% at 288 °C. This retention was similar to that exhibited by the PETI-5 samples. Transverse flex samples had an average strength of 44.2 MPa with a 77% retention of strength at 288 °C.
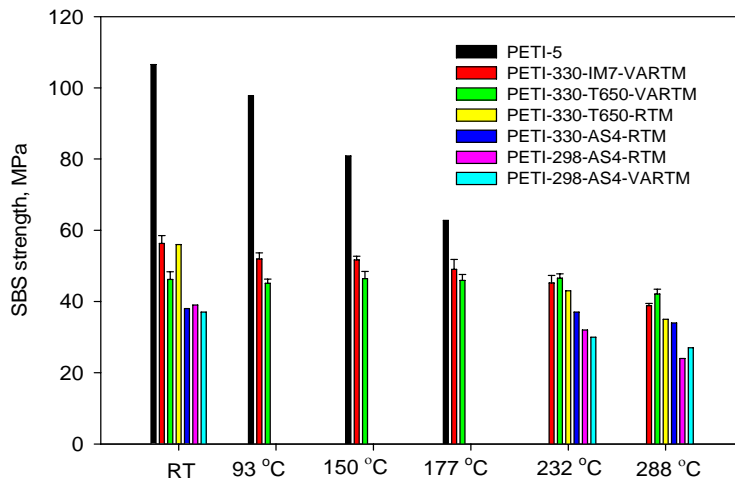

Figure 8: SBS strength of PETI-330/C-fabrics; PETI-5 IM7 prepreg and PETI-298/AS4 data shown for reference.
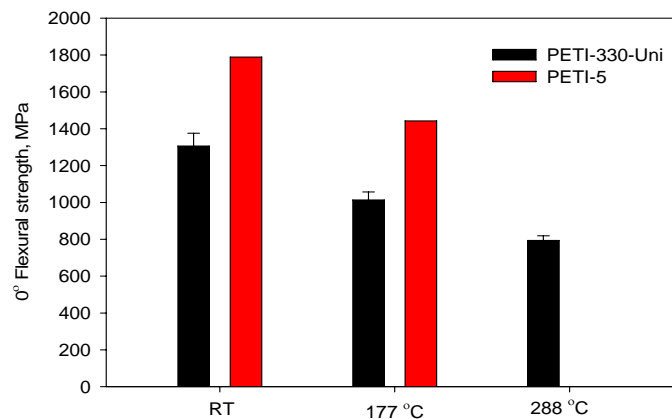

Figure 9: Flexure strength of PETI-330/IM7 uniweave fabric; PETI-5 IM7 prepreg data shown for reference.

4 Summary

One of the toughest challenges in HT-VARTM is the reduction of void content to 2% or less as required for aerospace applications. Prior to this work, conventional HT-VARTM could not fabricate composite panels with less than 2% voids from high temperature polyimide thermoset resins. This countered the observation that high quality panels were made from these same resins using RTM. The

process modifications introduced in this work produced HT-VARTM panels with low void contents. The changes involved curing at a lower temperature 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 uniweave carbon fabric, the void content was lowered to <2%, thereby meeting the requirement for aerospace applications.

Both PETI-8 and PETI-330 composites showed very good retention of SBS and flexure strengths at elevated temperatures. In fact, PETI-330 samples made by HT-VARTM exhibited higher strengths at elevated temperatures than samples made by RTM. Future work will involve the implementation of higher fidelity temperature and pressure controls for the HT-VARTM process followed by additional processing trials.

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