Epoxy/Glass and Polyimide (LaRC™ PETI-8) /Carbon Fiber Metal Laminates Made By The VARTM Process*

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ABSTRACT

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. Polyimide composites are very attractive for applications that require a high strength to weight ratio and thermal stability. Vacuum assisted resin transfer molding (VARTM) has shown the potential to reduce the manufacturing cost of composite structures. Fiber metal laminates (FML) made via this process with aluminum, glass fabric, and epoxy resins have been previously fabricated at LaRC. In this work, the VARTM process has been refined for epoxy/glass FMLs and extended to the fabrication of FMLs with titanium/carbon fabric layers and a polyimide system developed at NASA, LARC™ PETI-8. Resin flow pathways were introduced into the titanium foils to aid the infiltration of the polyimide resin. Injection temperatures in the range of 250-280°C were required to achieve the necessary VARTM viscosities (<10 Poise). Laminate quality and initial mechanical properties will be presented.

1. INTRODUCTION

1.1 Fiber Metal Laminates

Fiber metal laminates (FML) are multicomponent materials utilizing metals, fibers and matrix resins. Typical FML are prepared by stacking alternating layers of metal foils and fiber/matrix resin prepreg followed by consolidation in a press or autoclave. FMLs consisting of aluminum sheets and aramid fiber/epoxy prepreg were first developed by Vogelsang et al. at the Technische Universiteit Delft together with ALCOA in the 1980s [1] and are known as ARALL (Aramid Reinforced ALuminum Laminate). GLARE (GLAss REinforced FML), which replaces aramid fibers with glass fibers, was introduced in 1991. Fiber metal laminates combine some of the best properties of the metal and the composite making them suitable for aerospace applications. For example, and as shown in Figure 1, GLARE is used in the fuselage of the Airbus A380 and is being evaluated for use as blast resistant cargo containers due to the unique combination of properties.

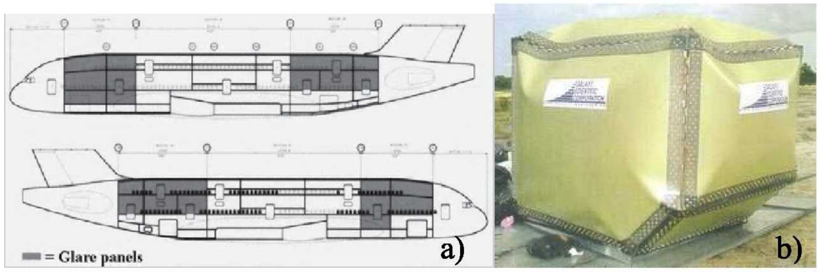

Figure 1. Current uses of GLARE: a) Airbus A380, b) cargo containers

1.2 Vacuum Assisted Resin Transfer Molding of FML

GLARE is expensive to produce and part size is limited due to the required prepreg and use of an autoclave in consolidation. Traditionally, composites have also been fabricated by similar methods. A more cost effective process for preparation of composites has been developed [2, 3] which uses liquid resin infused into dry fabric layers by vacuum pressure only to produce high quality materials. This process, know as vacuum assisted resin transfer molding (VARTM), utilizes a flow distribution media to allow the resin to proceed rapidly on the surface over the length of the part followed by the slower infusion through the thickness of the part, thereby decreasing infusion times. Figure 2 illustrates a typical VARTM set-up.

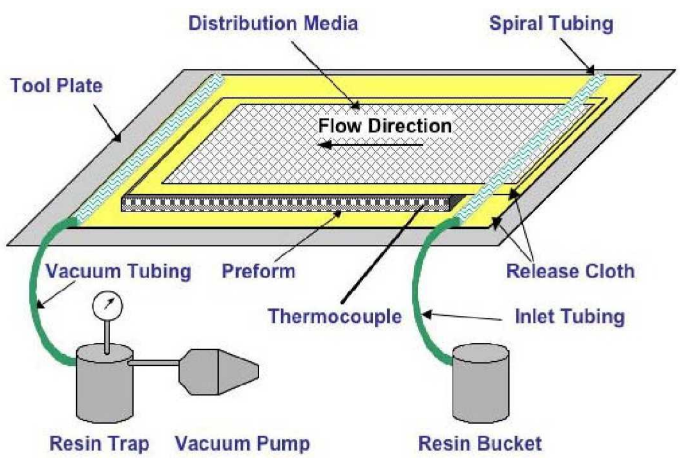

Figure 2. Illustration of a VARTM set-up.

NASA LaRC has developed a VARTM process for FML which utilizes flow pathways (perforations) into metal layers to allow for through the thickness resin infusion [4-6]. The materials produced by this process will be referred to as VARTMFML.

1.3 High Temperature VARTM

Polyimide (PI) composites are used in the aerospace industry due to their high strength to weight ratio and excellent thermal stability. Researchers at LaRC have developed several PIs from various aromatic diamines and dianhydrides that can be melt processed into coatings, adhesives, composites and films without the use of an autoclave. Controlled molecular weight imide oligomers containing phenylethynyl groups [phenylethynyl terminated imide (PETI), e.g. PETI-

8, PETI-330] have exhibited exceptional processability during fabrication of neat resin moldings, bonded panels and composites. LaRC™ PETI-330 was designed specifically for resin transfer molding (RTM) and resin infusion (RI) processing and laminates exhibited excellent properties [7-9]. LaRC™ PETI-8 also produced excellent mechanical properties when processed with vacuum bag pressure only [10] as well as using standard and double-vacuum-bag processes [11].

The CAPRI VARTM [3] process has been extended to composite panel fabrication with various LaRC PIs (PETI-330, PETI-8) at high temperatures, henceforth referred to as HT-VARTM. In this case, the resins are infused at temperatures above 250 ºC and cured above 316 ºC. In HT-VARTM, resin flow lines, tools, sealants and bagging materials must be able to tolerate the high temperature processing cycle. Recent work [12] has demonstrated the reduction of the void contents in composite parts to less then 2% with fiber volumes greater than 58% by controlling process variables.

In the current work, Al/Glass VARTMFML have been further optimized and characterized. Also the HT-VARTM process has been utilized to fabricate Titanium/Carbon/Polyimide VARTMFML.

2. EXPERIMENTAL

2.1 Materials

Two resin systems were used for the VARTM processing trials. A two part epoxy system, SC-85, was supplied by Applied Poleramic, Inc., Benica, CA, USA and PETI-8 polyimide powder was purchased from Imitec Inc., Schenectady, NY, USA.

Treated and primed 2024-T3 Aluminum 0.381 mm thick sheets were obtained from Delft University of Technology, Delft, Netherlands and used as received except for a solvent wipe prior to lay up. Titanium 6-4 0.406 mm thick sheets were purchased from National Specialty Alloys, Buford, GA, USA and were surface treated utilizing the Pasa-Jell™ 107 process [PRC-DeSoto International, Inc., Glendale, CA, USA] prior to lay up.

Eight-harness satin weave S-glass fabric was purchased from US Composites, West Palm Beach, FL, USA and IM7-6K unidirectional woven fabric (GP sizing, 160 gsm, Sticky String 450 1/0 fill fiber) was obtained from Textile Products, Inc., Anaheim, CA, USA.

2.2 Mechanical Testing

Fatigue testing performed at NASA LaRC utilized an Instron 100 kN test stand.

2.3 Fiber Metal Laminate VARTM

The processing of VARTMFML involved alternately stacking layers of a metal foil containing resin flow pathways (perforations) and fabric. The preform was then infused with a resin via a VARTM process as shown in Figure 3.

The insertion of the resin flow pathways was accomplished by simply drilling with a # 78 (0.41 mm / 0.016 in diameter) or a #67 (0.81 mm / 0.032 in diameter) drill bit using a Dremel tool mounted on a portable, tabletop press. Two hole spacing patterns, large (2.54 cm distance between holes, offset) and small (1.27 cm distance between holes, offset), were utilized in this work and are illustrated in Figure 4.

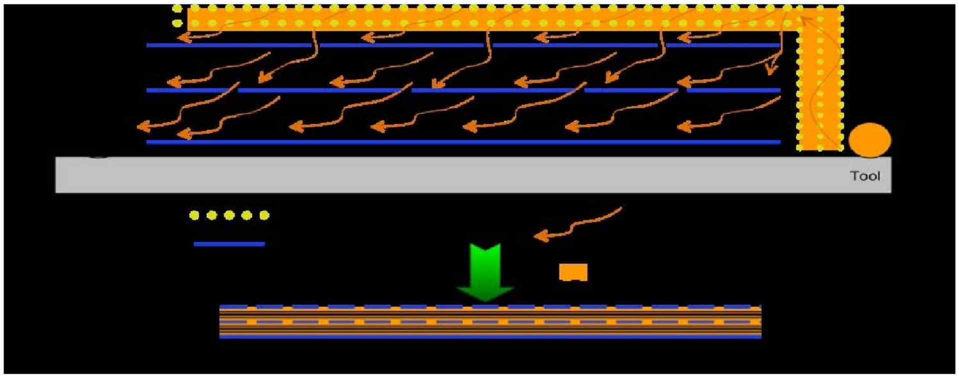

Figure 3. Illustration of the resin flow during VARTM of VARTMFMLs.

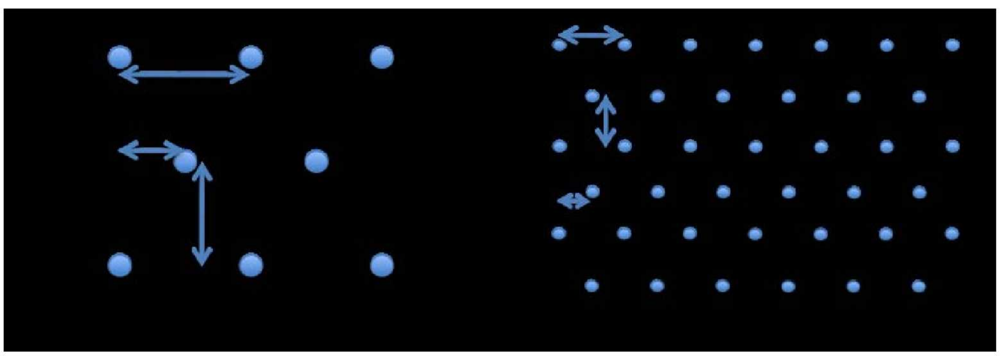

Figure 4. Schematic of hole-spacing patterns.

In order to experimentally verify the effect of hole size and pattern, four panels with various hole sizes and patterns were infiltrated simultaneously using the set up shown in Figure 5. The simultaneous infiltration was utilized to eliminate any potential deviations from cure conditions. Panels with no holes, 0.41 mm holes/ 2.54 cm spacing, 0.83 mm holes/ 2.54 cm spacing and 0.41 mm holes/ 1.27 cm spacing were infused with SC-85. All panels were Al/ 8-HS Glass fabric FML with a lay up of [Al/0°/90°/Al/0°/90°/Al/0°/90°/Al/0°/90°/Al].

Based on processing trials and fatigue data for both metal foils and FML (Section 3), Al/ Glass/ SC-85 epoxy panels were infiltrated utilizing an updated lay-up technique (Section 3.1.1) and CAPRI. Two forty centimeter by forty centimeter Al/8-HS Glass fabric FMLs [Al/0°/90°/Al/0°/90°/Al/0°/90°/Al/0°/90°/Al] were fabricated. One panel consisted of Al foils with the 1.27 cm spacing and 0.83 mm holes and the other panel consisted of Al foils with the 1.27 cm spacing and the smaller 0.41 mm holes. These panels were machined into fatigue coupons and tested in tension-tension fatigue.

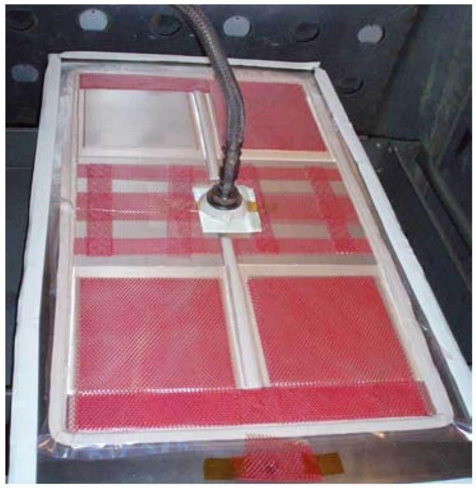

Figure 5. Photograph of infusion of multiple panels with various hole sizes and patterns.

2.4 High Temperature VARTM

The HT-VARTM set-up utilized in this work is shown in Figure 6. Polyimide bagging material and high temperature sealant were used to seal both an inner bag that contained a VARTMFML preform, five layers of aluminum (Al) screen flow media, Release Ease™ fabric, a breather material, and an outer bag for redundancy should a leak occur in the inner bag after infiltration. The tool was placed in an air circulating oven and heated to 260 °C. Vacuum (101.6 kPa or 30" of Hg) was pulled on both inner and outer bags and the resin pot as the resin was heated in order to degas the resin and remove air from the preform. Two ovens connected to each other by a heated tube were used. The resin pot was placed in the first oven and heated to the injection temperature under full vacuum. The tool, also under full vacuum, was heated separately in the second oven to the injection temperature. Upon reaching the infusion temperature, the resin was degassed for an additional 5 minutes, vacuum on the pot was reduced to 50.8 kPa and the connecting valve between the pot and heating tube opened to allow for resin flow until infusion was complete. The connecting tube was a 0.64 cm diameter stainless steel (SS) tube encased in a 1.27 cm diameter tube around which a heating coil was wrapped. It 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.

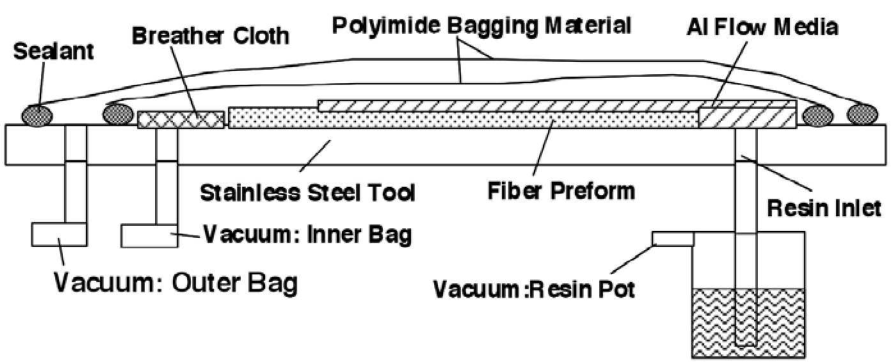

Figure 6: Schematic of HT-VARTM set up.

Using an improved VARTMFML lay-up in combination with the HT-VARTM process, titanium/ IM7 Uniweave/ PETI-8 FML were fabricated. An initial 15 cm x 15 cm panel with a simple 5/4 layup [Ti/0°/Ti/0°/Ti/0°/Ti/0°/Ti] was infiltrated utilizing the 1.27 cm spacing and 0.83 mm hole pattern. For this panel, 0.127 mm thick Ti-15-3-3-3 foils that had been surface treated by the Pasa-Jell™ 107 process were utilized. The cured panel was machined into test coupons and tested for flexural properties.

A larger HT-VARTMFML 20 cm x 27 cm panel with the smaller 1.27 cm spacing pattern and smaller 0.41 mm holes was also infiltrated. The bagged Ti/ IM7-6k preform with Al screen media prior to infusion is shown in Figure 7. A panel lay-up of $[Ti/90°/Ti/0°_2/Ti/0°]_S$ was chosen to compare to previous NASA open hole fatigue data on autoclaved Ti/PMC hybrid laminates panels generated during the High Speed Research Program in 1998. [13] For this panel, 0.406 mm thick Ti-6-4 sheets that had been surface treated by the Pasa-Jell™ 107 process were utilized.

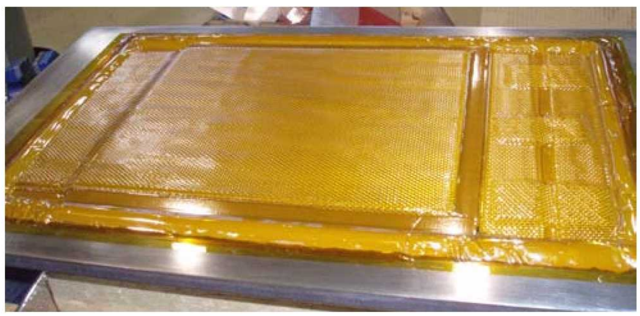

Figure 7. Photograph of HT-VARTM set-up of 20 cm x 27 cm PETI-8/ IM7/ Ti VARTMFML.

2.5 Flow Visualizations

The flow visualization fixture of the VARTMFML process (Figure 8) located at Michigan State University was used to observe the resin infiltration process. It is identical to the VARTM fixture currently used to manufacture FML's except for the changes noted below which were necessary to observe the fluid (oil with a low viscosity similar to VARTM epoxies) during infiltration. A clear, scratch resistant, polycarbonate tool plate was used in place of the metal tool plate. The dimensions of the tool were 0.914 m long by 0.508 m wide. Three 0.95 cm diameter holes were drilled and tapped into the plate for the resin inlet and vacuum connections. The resin inlet tube is shown on the right and the vacuum outlet tube is shown on the left.

Clear plastic acetate film, 0.381 mm thick, was used in place of the metal sheet. Resin pathways were machined into the acetate sheets to permit resin flow through the sheets during processing. The remaining components and all dimensions of the visualization fixture are identical to the VARTM fixture. Mounted below the polycarbonate tool was a mirror which was used to observe the resin flow along the bottom surface of the preform. Use of the mirror allows the video camera to simultaneously record the flow fronts on both the top and bottom surfaces of the preform.

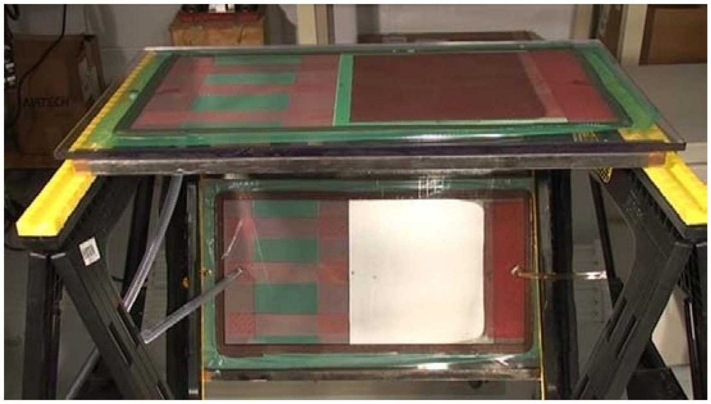

Figure 8. Flow visualization fixture.

3. RESULTS AND DISCUSSION

3.1 Aluminum / Glass FML

3.1.1 VARTM Infiltrations

Four panels with various hole sizes and patterns were infiltrated simultaneously using the set up shown in Figure 5. The resulting panels had dry spots in the center as race tracking occurred. This problem, which had occurred in previous panel infiltrations [14-15], prompted a change in lay-up procedures that were established and validated with model simulations and flow visualization experiments. (Section 3.1.2) [16-17] Instead of using excess fabric on the edges of the panels which allowed for the resin to racetrack around the panel, further infiltrations utilized fabrics cut to the size of the foils with the preform placed adjacent to the edge of the infiltration set-up, thereby eliminating the race tracking effect. Al/ Glass/ SC-85 epoxy panels were successfully infiltrated utilizing this updated lay-up technique and CAPRI. Two 40 cm x 40 cm Al/ 8-HS Glass fabric FMLs [Al/0°/90°/Al/0°/90°/Al/0°/90°/Al/0°/90°/Al] were fabricated and tested in fatigue. The panels fabricated as shown in Figure 5 were also machined into fatigue specimens and tested to further understand the effect of hole pattern and size on fatigue.

3.1.2 Flow Visualizations

Hybrid preforms with different flow pathway hole diameters and patterns were successfully infiltrated using the VARTM process and the flow visualization fixture shown in Figure 8. The glass fabrics in each of the hybrid preforms appeared to be completely wet-out with fluid and no significant dry spots were visually detected. As expected, the flow pathway hole diameter did not have a significant affect on the flow into the distribution media or the top surface of the hybrid preform. Total wet-out of the distribution media was achieved in about 30 seconds for all cases.

The flow patterns, at the bottom surface of the glass fabric next to the tool plate clearly showed that the flow path hole diameter had a significant effect on both the basic shape of the pattern and the amount of time required to completely wet-out the hybrid preform. As the flow path hole diameter increases, filling of the hybrid preform becomes dominated by transverse flow through the acetate films and glass fabric. This results in a significant reduction in total fill time. As shown in Figures 9 and 10, increasing the diameter of the holes in the acetate films from 0.41 mm to 0.83 mm, results in a decrease in the total fill time from about 30 minutes to 8.5 minutes.

Reducing the spacing between the 0.41 mm holes also showed similar results. For the acetate films with the 0.41 mm hole diameter, reducing the distance between holes from the 2.54 cm hole spacing to the 1.27 cm hole spacing improved flow times as shown in Figure 11. Therefore, it appears that by either increasing the hole size or increasing the number of holes provides enough flow and reduces flow time enough to eliminate the potential of poor panel quality. The guidance from these experiments along with the fatigue results for the various hole sizes and patterns (Section 3.1.4) have helped establish a preferred hole size and pattern.

3.1.3 Flow Modeling

A simulation model of the VARTMFML process was developed using the computational fluid dynamics code FLUENT. A volume of fluid (VOF) multiphase model was adopted to model the filling process. The numerical scheme ensured first order accuracy in time interpolation and second order accuracy in space interpolation. Beneath the photographs of the flow patterns (Figures 9-11) are the results of the FLUENT VARTM simulation at the corresponding times. The scale bar beneath the simulation results represents the volume fraction of fluid in the hybrid preform. The color red represents a volume fraction of fluid of 1, or fully saturated. The color blue represents a volume fraction of fluid of 0, or completely dry.

Overall, the model was able to capture the basic shapes of the flow patterns (Figures 9-11). However, the absolute times do not correspond well due to the use of literature values for the permeabilities of the glass fabric and distribution media. A new FLUENT model is currently being developed which gives a better description of the flow pathways in the acetate films.

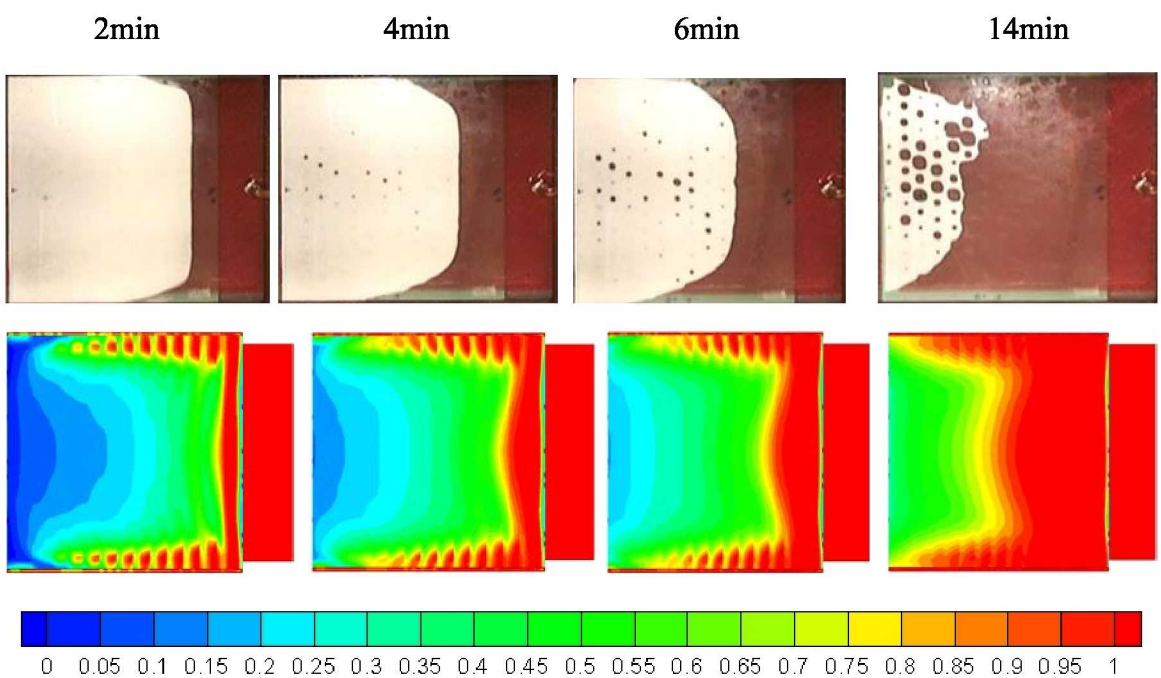

Figure 9. Flow patterns on the bottom surface of the hybrid preform with a flow pathway hole diameter of 0.41 mm and hole spacing of 2.54 cm. (infusion photographs: top, model flow front predictions: bottom)

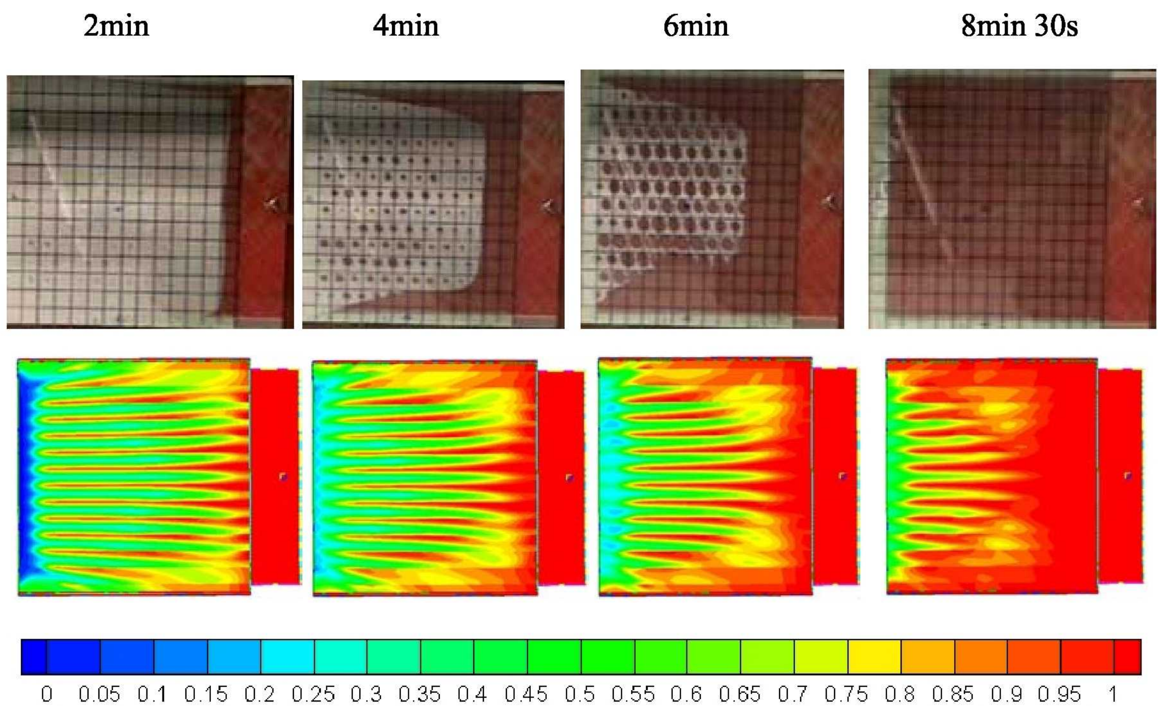

Figure 10. Flow patterns on the bottom surface of the hybrid preform with a flow pathway hole diameter of 0.83 mm and hole spacing of 2.54 cm. (infusion photographs: top, model flow front predictions: bottom)

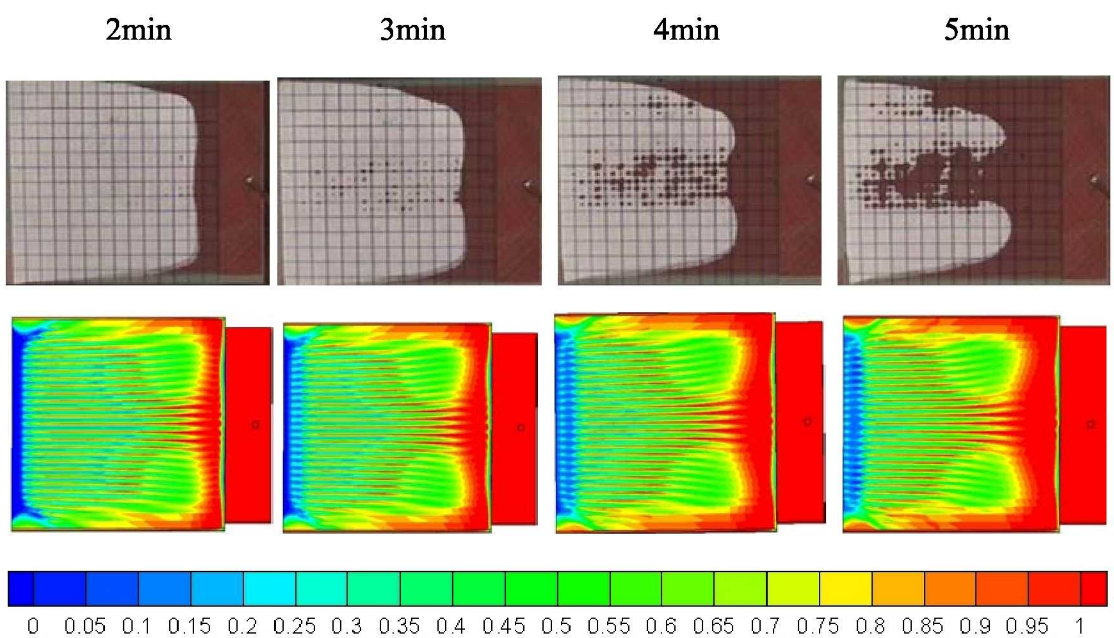

Figure 11. Flow patterns on the bottom surface of the hybrid preform with a flow pathway hole diameter of 0.41 mm and hole spacing of 1.27 cm. (infusion photographs: top, model flow front predictions: bottom)

3.1.4 Foil Fatigue Testing

Metal foils that were machined and drilled to the dimensions shown in Figure 12 were tested in fatigue to the same maximum stress that the metal layers were subjected to in previous laminate testing. [14-15] The results for the aluminum samples are shown in Table 1. As expected, the presence of the holes resulted in a reduction in fatigue properties of the aluminum foil. The baseline fatigue life of just over 20 thousand cycles was reduced to just under 10 thousand with the smaller 0.41 mm holes and to just over 5 thousand with the larger 0.82 mm holes. However, the hole spacing did not significantly affect the fatigue properties. Since the smaller spacing pattern did not further reduce the fatigue properties and improves the flow characteristics of the infiltration (Section 3.1.2), this hole pattern appears to be the better choice.

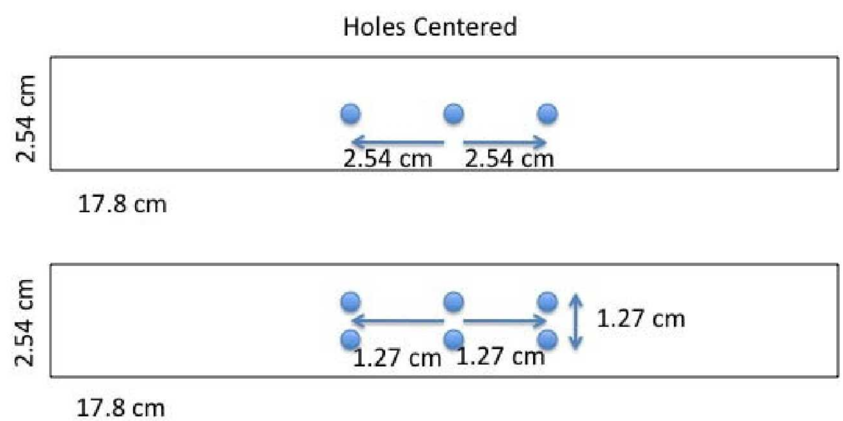

Figure 12. Schematic of foil fatigue specimens.

Table 1. Tension-tension fatigue data of aluminum foils with various holes sizes and patterns.

Cycles to Failure @ 241 MPa (35 ksi), R=0.1, 5Hz	**No Holes Straight**[1]	**No Holes Dogbone**	**0.41 mm Holes, 2.54 cm Spacing, Centered**[2]	**0.41 mm Holes, 1.27 cm Spacing, Centered**[2]	**0.81 mm Holes, 2.54 cm Spacing, Centered**[2]	**0.81mm Holes, 1.27 cm Spacing, Centered**[2]
Average of 5 Specimens	20983	20551	9801	9645	5230	5879
Standard Deviation	1485	4679	2200	5857	2320	1674

[1]Failed in grips
[2]Shown in Figure 12

3.1.5 VARTM Panel Fatigue Testing

Initial fatigue data obtained on VARTMFML at Georgia Institute of Technology is presented in Figure 13. [14-15] Initial FML fabricated with treated Al foils that were 0.304 mm thick and are labeled as thin while later batches of treated Al foil utilized were 0.381 mm thick and labeled as thick. Also shown for comparison are the results of various versions of GLARE products. Since the thin and thick specimens had the same layup, the thin specimen results in a larger percentage of composite material thus showing an improved fatigue life when compared to the thick specimens at the same stress. All the VARTMFML panels were fabricated to simulate the GLARE-3 lay-up, [Al/0°/90°/Al/0°/90°/Al/0°/90°/Al/0°/90°/Al] and utilized the 0.81 mm diameter holes and 2.54 cm spacing. Specimens were taken from well wetted out portions of panels only.

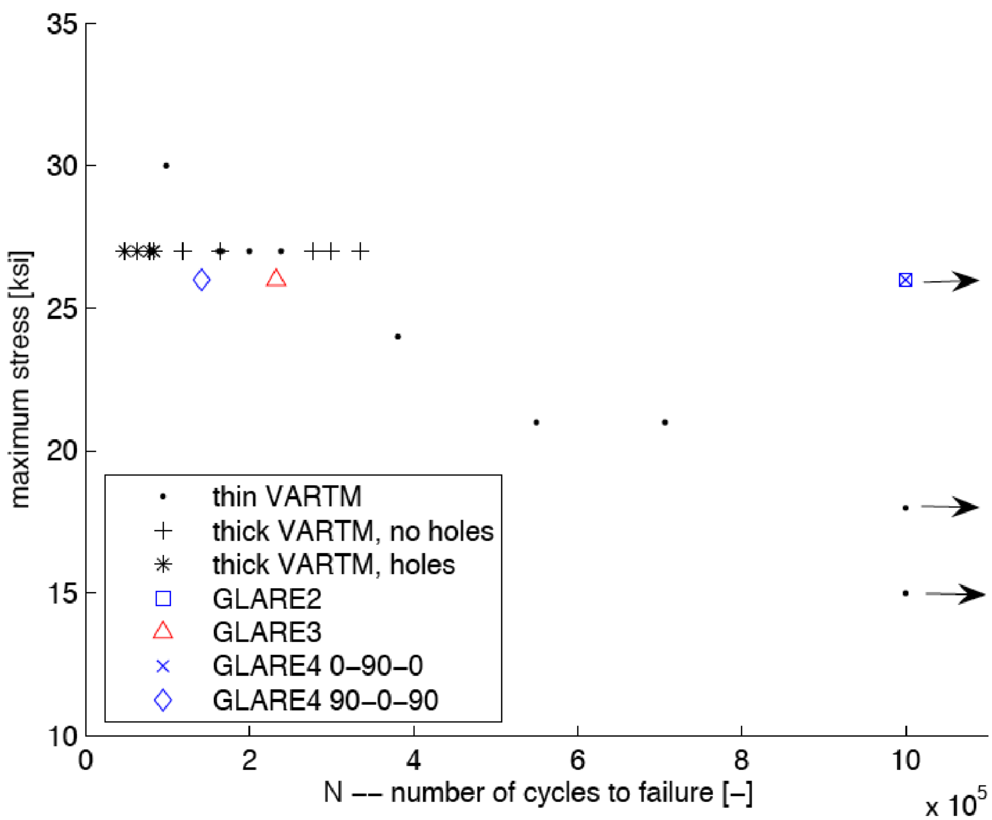

Figure 13. Initial tension- tension fatigue results. (R=0.1, 10Hz) (1ksi= 6.9 MPa)

Panels fabricated simultaneously as described in Section 3.1.1 were tested in fatigue and the data is shown in Figure 14. The data for the three panels showed significant scatter due to the poor quality of the panels. However, it is clear from this data that the edge specimens, which were of higher quality due to the race tracking that occurred during the infiltrations, were the better performing specimens and fell well within the stress concentration factor, Kt=2 and Kt=4, data of 2024 aluminum. [18] (Kt is a measure of the effect of geometry on stress at a notch. A Kt=1 specimen is designed to give pristine material behavior with no significant stress concentrations.

A hole in a plate generally has a stress concentration of Kt=3 if it is not too close to the edge of the material and is not large relative to specimen dimensions.) The Kt =2 and Kt =4 curves for notched aluminum provide data for comparison to that of the FML fabricated herein. Therefore, the better quality samples appear to provide a reasonable fatigue response as compared to notched aluminum. The smaller pattern spacing and the larger holes allows for better wet out and quality while the smaller holes should provide improved fatigue life compared to the larger holes.

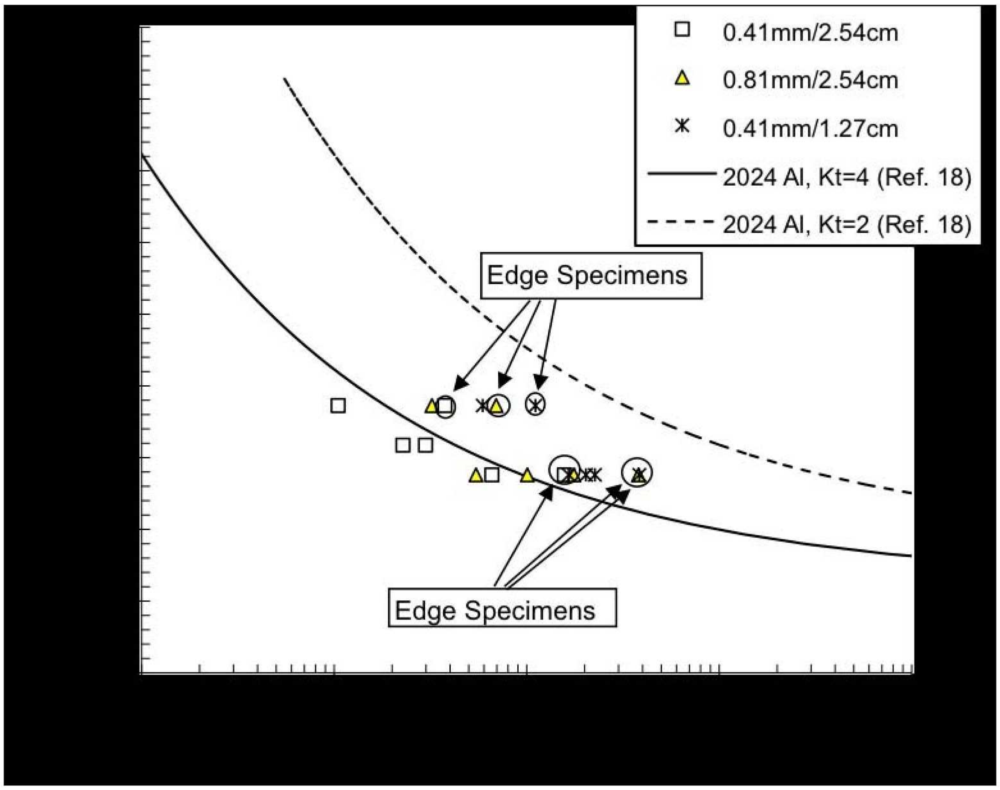

Figure 14. Tension-tension fatigue data of panels fabricated simultaneously with varying flow pathway sizes and pattern. (R=0.1, 10Hz)

The larger panels (40 cm x 40 cm) fabricated as described in Section 2.3 with the smaller hole pattern with both the larger holes and smaller holes were tested in fatigue and the data is shown in Figure 15. The data is compared to the results shown in Figure 14 with the poor quality specimen data removed. (Average values are shown for clarity.) The high quality panels with the larger holes and small spacing (0.81mm/ 1.27cm) and with the small holes and small spacing (0.41mm/ 1.27cm-2), resulted in improved fatigue properties compared to the previous panels especially at the lower stress levels. Since these panels were fabricated with thicker Al foils, the percentage of composite is lower compared to the initial data obtained at Georgia Tech from Reference 14 and 15. It is evident from the data that the smaller holes resulted in better fatigue life, as expected, while providing adequate flow to produce quality panels.

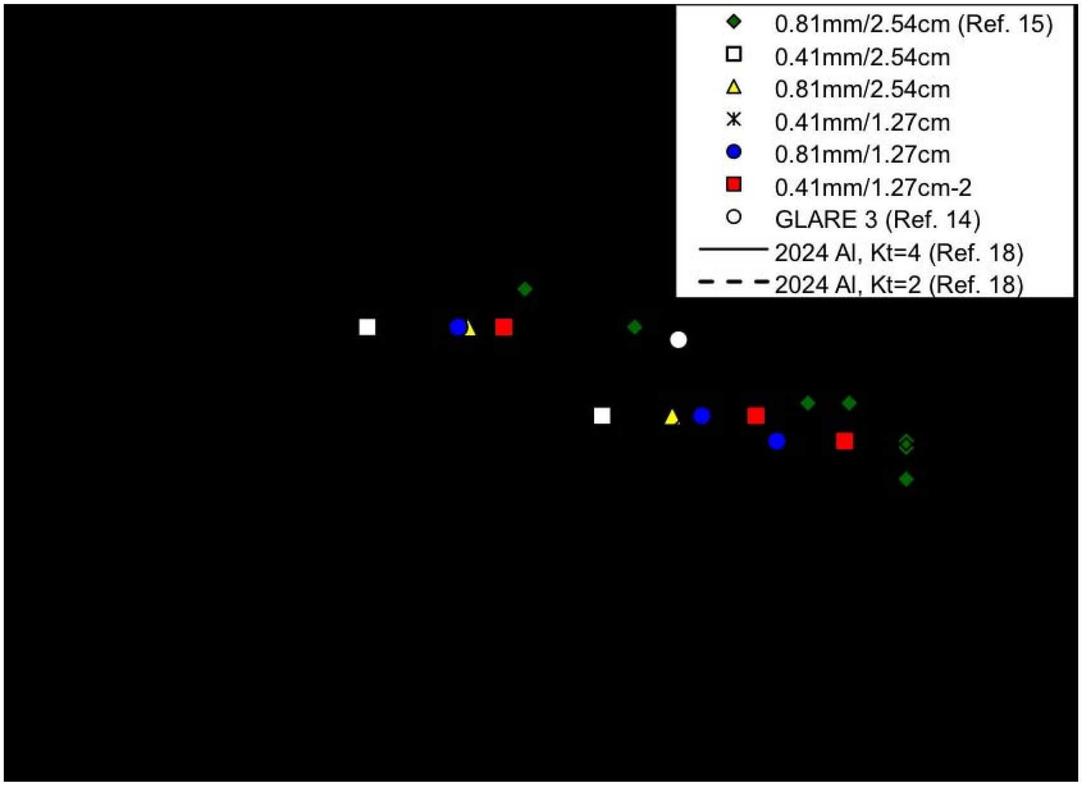

Figure 15. Tension-tension fatigue data of panels fabricated with varying flow pathway sizes and pattern. (R=0.1, 10Hz)

3.2 Titanium / Carbon FML

3.2.1 High Temperature VARTM

The resultant panel from the initial Ti/ IM7/ PETI-8 infiltration had flexural strengths (ASTM D-790) of 1.4±0.12 GPa at RT and 1.05±0.05 GPa at 177°C. Although the panel appeared to wet out well, photomicrographs (Figure 16) indicated porosity between tows in the fabric layers. This porosity problem has been observed in HT-VARTM composites as well. [12] Although this porosity issue has been essentially corrected in carbon fiber composites, it is apparent that further refinement of the processing conditions for HT-VARTMFML will be required.

Figure 16. Photomicrograph of PETI-8/ IM7/ Ti HT-VARTM Panel. (polished edge)

The larger HT-VARTMFML 20 cm x 27 cm panel (Figure 7), however, did not fully infuse from the Al porous flow medium into the Ti/IM7-6k uniweave preform. The similarities in flow observed in the epoxy panels with both the large and small holes does not appear to translate to the polyimide resin system despite similar viscosities at infusion. Future work will attempt to understand the issues associated with HT-VARTMFML by developing a more accurate flow model of the process with a specific PETI-8 resin model.

3.2.2 Titanium Fatigue Testing

Titanium metal foils that were machined and drilled to the dimensions shown in Figure 12 were tested in fatigue to a maximum stress of 345 MPa. This stress level was chosen to provide a large fatigue life with a practical testing time frame. The results of the titanium samples are shown in Table 2. For the Ti foils, only the 0.41 mm holes were utilized. The presence of the smaller spacing pattern with the small holes (Figure 10) had no effect on the ultimate tensile properties, 1001±5 versus 977± 25 MPa. All of the Ti foil fatigue specimens failed in the test grips and not at the holes. Within the large scatter of the data, neither hole pattern significantly reduced the fatigue life of the Ti foil. It should be noted, however, that these specimens were sheared to size and were not tabbed. Better quality specimens may differentiate any potential hole effects more clearly.

Table 2. Tension-tension fatigue data of titanium foils with various holes sizes and patterns.

Cycles to Failure @ 345 MPa (50 ksi), R=0.1, 5Hz	No Holes[1]	0.41 mm Holes, 2.54 cm Spacing, Centered[1,2]	0.41 mm Holes, 1.27 cm Spacing, Centered[1,2]
Average of 5 Specimens	73405	55511	47355
Standard Deviation	17079	14091	10467

[1]Failed in grips [2]Shown in Figure 12

4. CONCLUSIONS

Epoxy/Glass/Al VARTMFML has been successfully refined to consistently produce high quality panels that result in good mechanical properties. Based on process modeling and experimental data, the resin pathway size and distribution can be selected to produce quality FML with minimal reduction in fatigue properties. A refined process technique has been developed which provides a good combination of processing characteristics and mechanical properties. Further optimization, especially in how the resin pathways are introduced (i.e. improving hole edge quality), could further improve the process and resultant properties. However, this work has demonstrated the feasibility of fabricating FML by a VARTM infusion process.

This work has also demonstrated the extension from low temperature epoxy resin systems to high-temperature use polyimide systems. The NASA developed LARC™ PETI-8 resin system was successfully infused via VARTM into a titanium foil/IM7 FML. Although further enhancements of the processing conditions and evaluation of the resultant mechanical properties are still required, this work has demonstrated the potential of the VARTMFML process to successfully fabricate high-temperature fiber metal laminates.

5. REFERENCES

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