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CURE CYCLE DEVELOPMENT AND QUALIFICATION FOR THICK-SECTION COMPOSITES

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ABSTRACT

The kinetics of thermoset resin cure are multifaceted, with flow and wet-out being dependent on viscosity, devolatilization being a function of partial pressures, and crosslinking being dependent on temperature. A unique cure recipe must be developed to address and control each factor simultaneously. In the case of thick-section composites, an uncontrolled exotherm could cause the panel to cure from the inside out, causing severe process-induced residual stresses. To identify and control the peak heat generation from the exothermic crosslinking reaction, differential scanning calorimetry (DSC) was conducted for different candidate cure schedules. Resin rheology data and dynamic mechanical analysis (DMA) results were used to confirm a viable resin viscosity profile for each cure schedule. These experiments showed which isothermal holds and ramp rates best served to decrease the exothermic peak as well as when to apply pressure and vent the applied vacuum. From these data, a cure cycle was developed and applied to the material system. During cure, embedded thermocouples were used to monitor heat generation and drive cure temperature ramps and dwells. Ultrasonic testing and visual inspection by microscopy revealed good compaction and < 1 % porosity for two different composite panels with the same resin system. DSC of post-cured samples of each panel indicated a high degree of cure throughout the thickness of the panels, further qualifying the proven-in process.

1. INTRODUCTION

Typical fiber reinforced components are thin-walled structures with a layup consisting of 4-12 plies. When analyzing thin components, a 2D stress state, or plane stress, can be assumed due to the exceptionally small thickness-to-span ratio and may therefore be analyzed using composite laminate theory (CLT). CLT requires only four independent elastic material properties to construct the compliance matrix which relates stress to strain: E_1, E_2, G_{12}, and ν_{12}. These four material properties are obtained using well-established test methods. These properties are also readily available from most material manufacturers and are generally simple to confirm.

Thick section composites (TSCs) are defined in the Composite Material Handbook (CMH) [1] as laminates which exhibit a three-dimensional state of stress in their large thickness-to-span ratios, material constituents, lamination scheme, processing, and service loading. A significant degree of three-dimensional stress is defined as one which contributes to failure, excessive delamination, or vibration in the component.

By definition, TSCs have a non-negligible 3D state of stress. Thus the plane stress assumption would not be valid and CLT may not be applied. In order to analyze the 3D stress state of a TSC, the entire material compliance matrix must be built, which requires 9 independent elastic material

properties. The test methods for obtaining the 5 additional elastic material properties required to construct the 3D compliance matrix are not well-established [1]. What little experimental data does exist is typically specific to the programs for which they were used.

Testing for these through-thickness properties requires careful fabrication of test specimens which are representative of the final component. However, TSCs are inherently difficult to manufacture due to the high likelihood of process-induced stresses being introduced to the material [1]. Mitigation of these effects could require special resins, processing, tooling, and cure cycles.

The non-trivial nature of TSC manufacture warrants further study. The purpose of this study is to establish a complete fabrication and qualification procedure for a TSC test panel. Panel fabrication includes cure cycle development, as well as layup, bagging, and instrumentation methodology. A systematic method of determining an appropriate cure cycle is developed using DSC and DMA on uncured samples of prepreg material. The cure cycle and layup techniques used to create the TSC panels were evaluated using ultrasonic scanning, microscopy, resin digestion for quality assurance. Suggestions for future TSC panel fabrication are derived from the results of this study.

2. CURE CYCLE DEVELOPMENT

The cure cycle for a composite component should be optimized based on both the material properties and the component geometry as cure behavior varies significantly with composite thickness [2]. The manufacturer-provided cure cycle for a material is typically developed for thin laminates [3]. However, TSCs are rarely addressed in the manufacturer's recommendations and the definition of the cure cycle is often developed uniquely through iteration and in-situ temperature monitoring. This method of cure cycle development can be time-prohibitive and can waste significant amounts of material, especially in the case of TSCs. However, characterization of the prepreg behavior using DSC and DMA can be used to optimize a cure cycle.

Two different fiber reinforcements, one carbon fiber and one fiberglass, with the same polymer matrix were studied and manufactured. Both prepregs were woven into an 8-harness satin (8HS). The manufacturer-recommended cure cycle for the materials consisted of a 2.71 ºC/min (5 ºF/min) ramp up to 177 ºC (350 ºF), a 1 hour isothermal hold at 177 ºC, then a 2.71 ºC/min ramp down. The manufacturer made no recommendations for a pressure profile.

In thin laminates, it is simple to achieve an even curing profile since the temperature gradient through a thin geometry is relatively small. The geometry of TSCs causes a significant temperature gradient through the thickness during cure [2] which can be difficult to predict [4]. The manufacturer's cure cycle for the materials being studied was not designed to accommodate that gradient and is therefore not suited to cure TSCs. In this case, the aggressive 2.71 ºC/min ramp may heat a TSC too quickly could exasperate its temperature gradient.

2.1 Temperature Profile Development

According to multiple studies as well as manufacturer recommendations, the cure cycle for a TSC should heat the component slowly with slow ramps [4, 5] and additional preliminary isothermal holds [6] to allow the entire component to reach the desired temperature at once. This allows the resin system to reach its minimum viscosity point at the same time throughout the volume of the panel, which promotes lamination, fiber wet-out, and void reduction.

Slow and even temperature application is also important with regard to the chemical reaction that thermoset resins undergo during cure. The even temperature distribution in a thin laminate allows the crosslinking reaction to occur simultaneously throughout the component, which causes it to solidify uniformly. In a thick component, the significant temperature gradient causes adjacent areas of the laminate to cure at different rates. As one area cures, it solidifies, which constrains all adjacent areas during cure. The strain compounds and creates a process-induced stress field inside of the laminate as it cures [5]. Process-induced stresses can cause a test specimen to fail prematurely or to exhibit uncharacteristic properties or failure modes.

The exothermic nature of the crosslinking reaction presents another challenge in the cure of TSCs. As a thin component cures, the generated heat is dissipated directly to the environment. However, even if a TSC is heated evenly and begins to cure at the same time throughout the volume, the heat being generated at the center of the component can only be dissipated to adjacent areas of the component [7]. The excess heat buildup in the center of the component causes the resin to cure faster, creating an inside-out cure profile [5]. This profile is a common source of process-induced stress in TSC parts. Thus, in order to produce a part with minimal process-induced stress, it is necessary to mitigate the exotherm of the curing resin.

2.1.1 Exotherm Mitigation

In order to control the exothermic crosslinking reaction of the thermoset resin, it is first necessary to quantify the amount of heat generated during cure. Differential scanning calorimetry (DSC) was used to measure the heat output of uncured samples of material as they were subjected to different cure cycles.

According to Mettler-Toledo, "[d]ifferential scanning calorimetry (DSC) measures the difference between the heat flows from the sample and reference sides of a sensor as a function of temperature or time." [8] In this way, DSC can measure the amount of heat output by the curing process of a sample of material. By normalizing the heat output by the mass of the sample, it is possible to directly compare each sample's reaction to each temperature profile.

According to the DSC results from samples of each material, the crosslinking reaction began during the initial 2.71 ºC/min ramp up to the cure temperature of 177 ºC. Crosslinking initiated at approximately 101.7 ºC (215 ºF) and peaked at 140 ºC (284 ºF). At their exothermic peak, the duplicate fiberglass samples output an average normalized heat of 135 mW/g. A portion of the crosslinking reaction also occurred during the isothermal hold at cure temperature. The carbon fiber samples reacted similarly.

In an effort to reduce the exothermic peak during cure, a preliminary 1 hour isothermal hold was added to the beginning of the cure cycle. To capture the full range of reactions to thermal input, the isothermal hold temperature and the ramp between the first and second isothermal holds were varied systematically. As the crosslinking reaction of the resin appeared to occur near 102 ºC, temperatures of 93.3 ºC (200 ºF), 101.7 ºC (215 ºF), and 110 ºC (230 ºF) were chosen. Based on previous studies on the effects of ramp rate on cure behavior [4, 6], ramp rates of 0.56 ºC/min (1 ºF/min), 1.67 ºC/min (3 ºF/min), and 2.71 ºC/min (5 ºF/min) were chosen. The matrix of test runs is shown in Table 1. To ensure repeatability, each cycle was run with duplicate samples of each material system.

Table 1. Cure cycles employed to test for thermal reaction from resin.

Cycle ID	Isothermal Temp. (ºC)	Ramp Rate (ºC/min)
Manu	N/A	2.71
A	93.3	0.56
B	93.3	1.67
C	93.3	2.71
D	101.7	0.56
E	101.7	1.67
F	101.7	2.71
G	110	0.56
H	110	1.67
I	110	2.71

The peak heat of each reaction was extracted from the DSC plots of each cure cycle. The duplicate average peak heat for each cycle is shown in Figure 1.

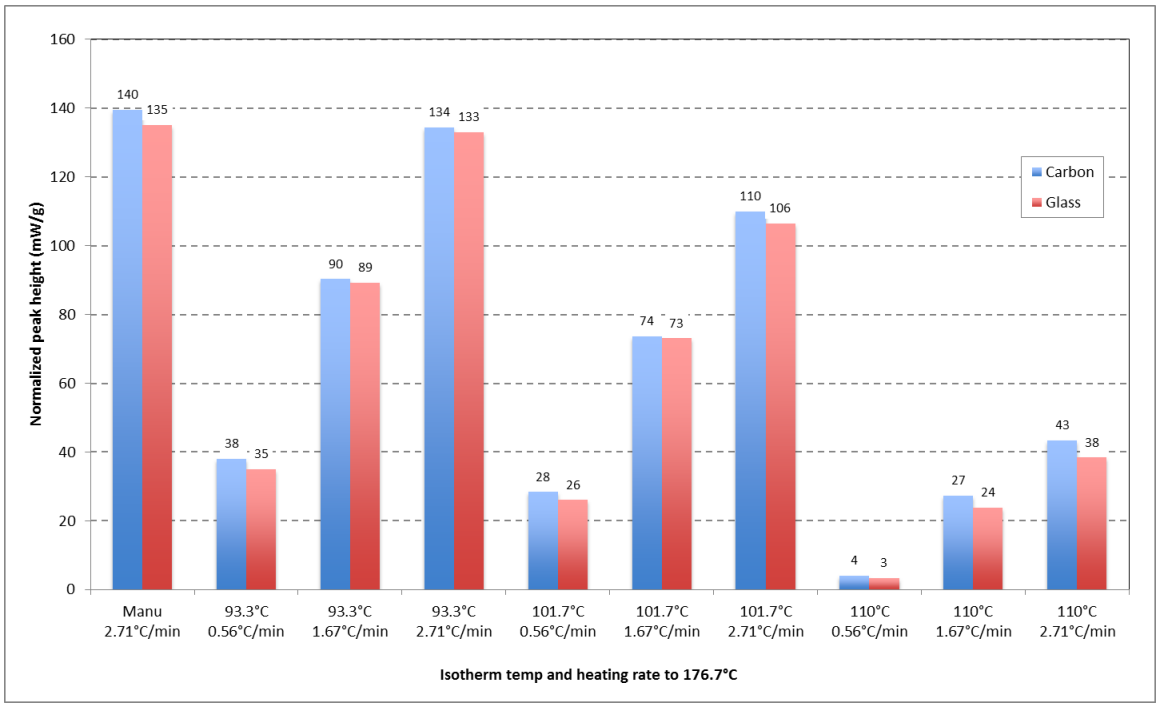

Figure 1. Summary of peak heat values obtained using DSC for carbon and glass.

From these data, it was clear that a higher isothermal hold and a lower ramp rate would minimize the peak heat generated by the material during cure. A higher isothermal hold allowed most of the crosslinking reaction to occur before the final cure temperature was reached. A slow temperature ramp caused the curing reaction to occur more slowly than a faster ramp. The fiber reinforcement material did not seem to affect the peak heat response significantly.

2.1.2 Resin Minimum Viscosity

The temperature profile of a cure cycle also affected the viscosity profile of the resin system in a composite. As previously mentioned, it was necessary to find the minimum viscosity point of the resin in order to produce a high quality panel. Thus the temperature profile must both control the exotherm and induce a low minimum viscosity in the resin.

Dynamic mechanical thermal analysis (DMTA) was used to quantify the viscosity of the resin as a function of time and temperature. "In a dynamic mechanical rheological test, an oscillating strain (sinusoidal or other waveform) is applied to a sample and the resulting stress developed in the sample is measured. The output signals are analyzed and, using established mathematical methods, the rheological parameters are computed." [9]

For each material and each potential cure cycle, two specimens, cut into 1 in round samples, were tested. In order to minimize the amount of time required to perform the DMTA, the three cure cycles which produced the most severe reaction in DSC (Cycles C, E, and G) were tested. In all cases, the resin solidified before reaching the final 177 °C cure temperature. However, the viscosity profile of each test case varied significantly.

For Cycle C, the resin viscosity dropped during the initial 2.71 ºC/min ramp up to 93.3 ºC, then stayed relatively constant through the hold. Then, through the 2.71 ºC/min ramp up to 177 ºC, the viscosity first dropped significantly, then increased as the crosslinking reaction began.

During the higher 101.7 ºC isothermal hold of Cycle E, the resin viscosity increased significantly and started higher for the 1.67 ºC/min ramp up to 177 ºC. At the start of the ramp, the viscosity dropped slightly before the resin solidified.

By the end of the 177 ºC isothermal hold of Cycle G, the resin was almost completely cured. The resin curing finished as soon as the slow 0.56 ºF/min ramp began.

From these data, it was unclear whether the large decrease in viscosity at the beginning of the ramp was caused by the low 93.3 °C isothermal hold or the high 2.71 °C/min ramp. Therefore, additional testing was required to ascertain the relationship between isothermal temperature, ramp rate, and minimum viscosity point. These additional tests led to the development of the final cure cycle.

2.1.3 Final Temperature Profile

In order to satisfy both the exotherm mitigation and minimum viscosity requirements, a compromise between the results of the DSC and DMTA testing was needed. The benefits of the 93.3 °C isothermal hold, from a viscosity standpoint, and the 110 °C isothermal hold, from an exotherm mitigation standpoint, led to the consideration of a two stage cure cycle.

This cycle would have isothermal holds at 93.3 °C and 110 °C prior to the final cure temperature to take advantage of the benefits of each. The ramp rate between the isothermal holds was varied to better understand the effects of ramp rate on minimum viscosity. The following cure schedules were run in the DSC to evaluate the heat output of each:

Table 2. Two-stage cure cycles evaluated in the DSC.

Cycle ID	J	K
Ramp #1	2.71 °C/min	2.71 °C/min
Hold #1 (duration)	93.3 °C (30 min)	93.3 °C (30 min)
Ramp #2	0.56 °C/min	1.67 °C/min
Hold #2 (duration)	110 °C (30 min)	110 °C (30 min)
Ramp #3	0.56 °C/min	0.56 °C/min
Final Hold (duration)	177 °C (30 min)	177 °C (30 min)

The lengths of the isothermal holds were decreased in order to decrease the testing time, as the samples were small enough to cure more quickly in the thermal chamber. As with the previously tested cure cycles, the bulk of the heat output occurred during the ramp from 110 °C to the final cure temperature of 177 °C. The additional isothermal hold had little effect on the exothermic behavior of the resin and the peak exotherm remained low.

Cycles J and K were also tested using DMTA to determine the effect of ramp rate on the viscosity of the resin. From these results, it was clear that a faster ramp between the 93.3 °C and 100 °C isothermal holds "triggered" a lower minimum viscosity point. A 2.71 °C/min ramp was not investigated due to the likely negative effects on the exothermic behavior. Cycle K was selected as the final temperature profile.

2.2 Pressure and Vacuum Profile Development

The primary functions of pressure and vacuum application are consolidation and void reduction. The pressure and vacuum profiles of a cure cycle are optimized based on the viscosity profile of the resin system. As a polymer resin is heated and before it begins the crosslinking reaction, its viscosity tends to decrease at a rate dependent upon the temperature profile of the cure. With appropriate pressure and vacuum application, voids which form during the manufacturing process may be transported out of the laminate. The viscosity profile of the resin helps to determine the amount of pressure to apply to the system during cure. The minimum viscosity point of the resin helps to determine the timing of both pressure and vacuum application.

2.2.1 Resin Flow and Material Wet-out

The overall strength of a composite depends heavily on the bond between the polymer matrix and the fiber reinforcement. According to 3M, wetting-out means "means the adhesive flows and covers a surface to maximize the contact area and the attractive forces between the adhesive and bonding surface" [10]. In the case of fiber-epoxy composites, the epoxy acts as an adhesive between the reinforcement fibers, so full wet-out entails full contact between the fibers and the resin. If the matrix does not fully wet-out the fiber reinforcement, the composite's full strength becomes a limited by individual constituent strengths rather than the combined strength. Therefore, material wet-out is very important when considering a cure cycle. To achieve full wet-out (fully resin-impregnated fiber), the resin must be able to flow across and between plies. The best way to ensure good resin flow is to apply pressure when the resin reaches its minimum viscosity [11]. Thus, pressure was applied during the 1.67 ºC/min ramp between the 93.3 ºC and 110 ºC isothermal holds. The magnitude of the pressure was driven by devolatilization.

2.2.2 Devolatilization

Voids in composite materials may be caused by air pockets trapped between plies, moisture absorbed into the resin, or solvents trapped in the resin. Voids which are cured into a composite laminate serve as a nucleation point for cracks. They may also occupy space between fibers and resin, preventing full wet-out in that area. Thus the minimization of voids is critical to manufacturing high quality laminates.

Both vacuum and pressure play a key role in the removal of voids from a composite. During vacuum application, solvent and water molecules in the resin may be pulled out of solution into gaseous form and existing air pockets may increase in size [12]. Once these voids nucleate, they tend to flow with the resin toward the vacuum port through bleeder paths between fibers and into the bleeder material. As previously discussed, pressure is applied at the resin's minimum viscosity point to promote resin flow. This pressure application aids in the transport of voids out of the laminate by causing greater resin flow. Once the resin begins to gel, the pressure then serves to press any remaining voids in the resin back into solution. Voids are dissolved when their internal pressure is overcome by the hydrostatic pressure in the resin [12]. Thus, the pressure applied to the panel must be greater than the void internal pressure.

Existing research on void dissolution suggested a minimum pressure of 414 kPa (60 psi) to overcome the internal pressure of water vapor voids at the final temperature of 177 ºC [12]. Previous work with the prepreg material also suggested that an autoclave pressure of 414 kPa promoted adequate resin flow during cure. Therefore, at the minimum viscosity point of the material, the autoclave was pressurized to 414 kPa. When the internal pressure reached 138 kPa (20 psi), vacuum was vented.

2.3 Final Cure Cycle

The combination of the determined temperature and pressure profiles yielded the cure cycle shown in Figure 2. The initial 93.3 ºC isothermal hold would allow the material to reach a uniform temperature throughout its volume before triggering the resin's minimum viscosity point with the 1.67 ºC/min ramp up to the second isothermal hold. This 110 ºC hold would allow the material to begin its crosslinking process without causing the exothermic reaction to run away. The final 0.56 ºC/min ramp up to the final 177 ºC cure temperature would cause the panel to heat slowly and evenly while curing, also preventing excessive exothermic heating within the panel.

The final cure cycle gave a peak heat of 14 mW/g and a minimum resin viscosity of 14×10^3 Pa·s for CFRP. For GFRP, the peak heat and minimum viscosity were 11 mW/g and 16×10^3 Pa·s, respectively. The final cure cycle would take 5.75 hours to complete, over twice the time of the manufacturer-recommended cure cycle (2.6 hours). However, manufacturer's cure cycle had an average peak heat of approximately 136 mW/g. Thus the final cure cycle decreased the peak exothermic heat of the material by 91 %.

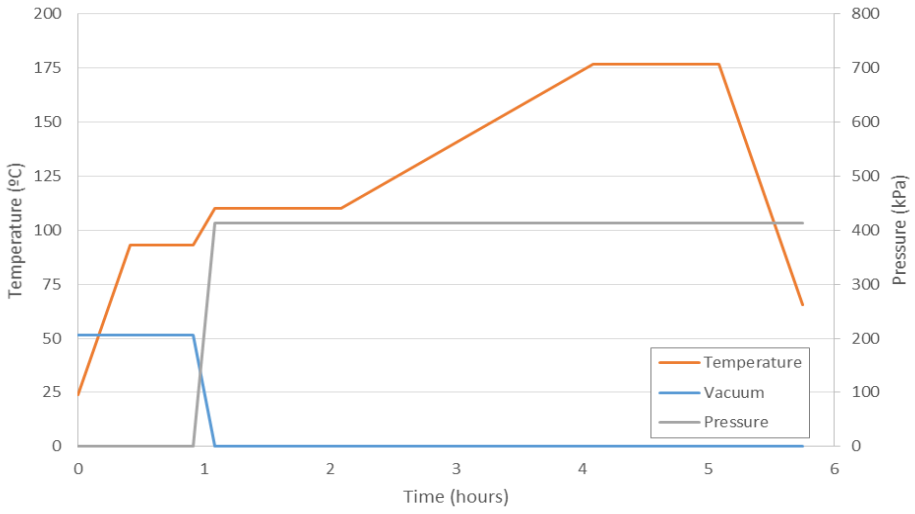

Figure 2. Final cure cycle temperature and pressure profiles.

3. PANEL FABRICATION

Hand layup of TSCs is a unique challenge, as traditional hand layup techniques are designed for thin laminates. While the theoretical calculation of per-ply thickness may still be employed, it may not fully account for the compounding consolidation of many plies. Additionally, the vacuum bagging method of consolidation during cure must be redesigned to maintain the panel's exceptional thickness and overall geometry.

3.1 Laminate Layup

The TSCs evaluated in this study were designed to be 2.54 cm (1 in) thick. Existing cured laminate thickness data were extrapolated to determine the number of plies necessary to create a panel with the desired thickness. These values were confirmed by calculating the theoretical cured ply thickness [13].

The primary concern during layup of any composite part is introducing voids between plies. This is of even greater concern when working with TSCs due to the number of plies per part. With so many layers, it is much more difficult to compact the plies to remove interlaminar air pockets. These pockets can be minimized by debulking as often as every 5 plies [14].

An important part of curing the final laminate is ensuring that the vacuum bag adequately supports and compresses the material into the final desired dimensions. This required a custom vacuum bag configuration with stiff, adjustable dams to support the significant thickness of the laminate, prevent panel deformation, and control resin bleed. Multiple sources [2, 15]successfully implemented a tall, stiff dam for the manufacture of TSCs. In this case, the chosen dam material was angle aluminum stock, which was slotted and bolted to the tool plate. The adjustability of the aluminum dams allowed for edge compression of the laminate to the final desired dimensions. String bleeder was installed at the edges of the laminate to create an outward bleed path. An upper caul plate was used to achieve a good surface finish on both sides for accurate measurement. The instrumented tooling setup prior to bagging is shown in Figure 3.

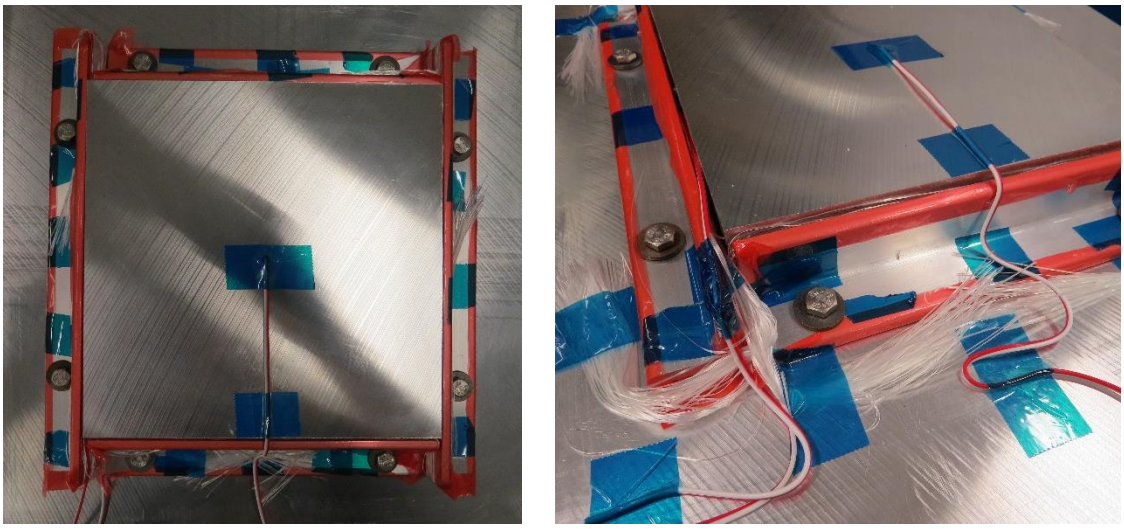

Figure 3. Overhead and detail view of tooling and instrumentation for TSC.

In order to monitor the temperature gradient inside the panel, thermocouples were inserted into the top and bottom of one edge of the panel, as well as into the very center of the panel. The central thermocouple would also allow for monitoring of a runaway exotherm, which was predicted to begin in the center of the panel's volume [5].

3.2 Autoclave Processing

Autoclave curing was chosen to manufacture the TSC panels due to the high level of control over applied temperature and pressure as well as for the ability to monitor internal panel temperature during cure. All elements of the cure cycle were driven by the three embedded thermocouples, including autoclave temperature, vacuum application, and pressure application. An emergency autoclave cooling cycle was programmed into the controller to mitigate excessive heat generated by the exotherm during cure, but was not used. The autoclave cure parameter values during cure are shown in Figure 4.

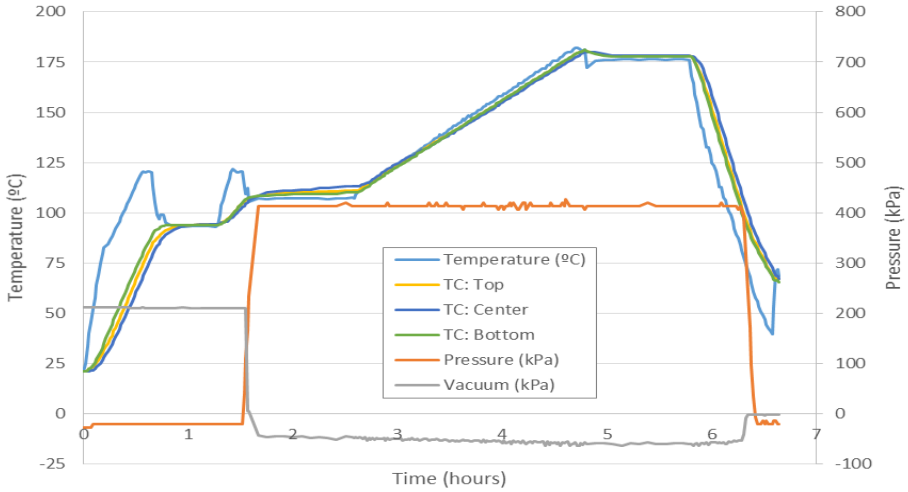

Figure 4. Temperature and pressure values of autoclave and panel during cure.

Inspection of the completed panels revealed that the thermocouples had served as an additional bleed path for the resin in both panels, causing both excessive and uneven bleeding in the panels. Further evaluation was necessary to determine the effect on the final dimensions. Upon initial debagging, the panels appeared well-consolidated and had good surface finishes.

4. QUALITY EVALUATION

After the panels were cured, a number of non-destructive and destructive evaluation techniques were employed to determine the quality of the cure cycle. Both the carbon fiber and fiberglass panel were subjected to test methods which checked the panels' geometry, level of porosity, and degree of cure.

4.1 Final Geometry

The original goal was to fabricate panels which were 2.54 cm thick. After debagging, the panels' thicknesses were measured in an 8-by-8 grid across their surfaces. The dimensions are shown in Figure 5. Red areas represent thicker sections while green areas represent thin sections. White dimensions indicate a geometric process-induced anomaly. It should be noted that the thermocouples of the carbon fiber panel were distributed along the upper edge while the thermocouples of the fiberglass were concentrated in the upper left corner. This supported the hypothesis that the thermocouples for each panel served as bleed paths. The final dimensions also suggest that the panels were over-bled.

2.343	2.343	2.343	2.352	2.356	2.379	2.361	2.364	2.386
2.357	2.356	2.359	2.363	2.368	2.371	2.374	2.377	2.383
2.368	2.368	2.372	2.377	2.380	2.382	2.386	2.390	2.396
2.384	2.385	2.388	2.392	2.393	2.397	2.398	2.402	2.408
2.397	2.400	2.401	2.405		2.409	2.411	2.413	2.421
2.412	2.414	2.415	2.419	2.421	2.422	2.424	2.428	2.433
2.426	2.427	2.430	2.432	2.434	2.436	2.438	2.442	2.445
2.440	2.441	2.442	2.446	2.448	2.450	2.451	2.454	2.457
2.454	2.452	2.455	2.458	2.459	2.460	2.462	2.465	2.465

2.266	2.273	2.276	2.281	2.291	2.303	2.300	2.305	2.313
2.281	2.280	2.285	2.289	2.295	2.300	2.307	2.312	2.319
2.287	2.291	2.295	2.301	2.306	2.311	2.317	2.322	2.327
2.303	2.305	2.308	2.313	2.318	2.322	2.329	2.333	2.291
2.313	2.315	2.317	2.324		2.332	2.340	2.345	2.347
2.325	2.327	2.330	2.335	2.336	2.344	2.350	2.354	2.357
2.346	2.344	2.347	2.350	2.353	2.359	2.360	2.362	2.363
2.353	2.356	2.360	2.364	2.367	2.370	2.371	2.375	2.376
2.361	2.363	2.365	2.369	2.385	2.377	2.381	2.388	2.390

Figure 5. Final thicknesses of the carbon fiber (left) and fiberglass (right) panels.

4.2 Ultrasonic Scanning

Ultrasonic scanning in a full-immersion ultrasonic tank was utilized to obtain an initial estimate of any major internal irregularities. The images would indicate large areas of porosity as well as potential process-induced defects. Ultrasonic images from both panels are shown in Figure 6.

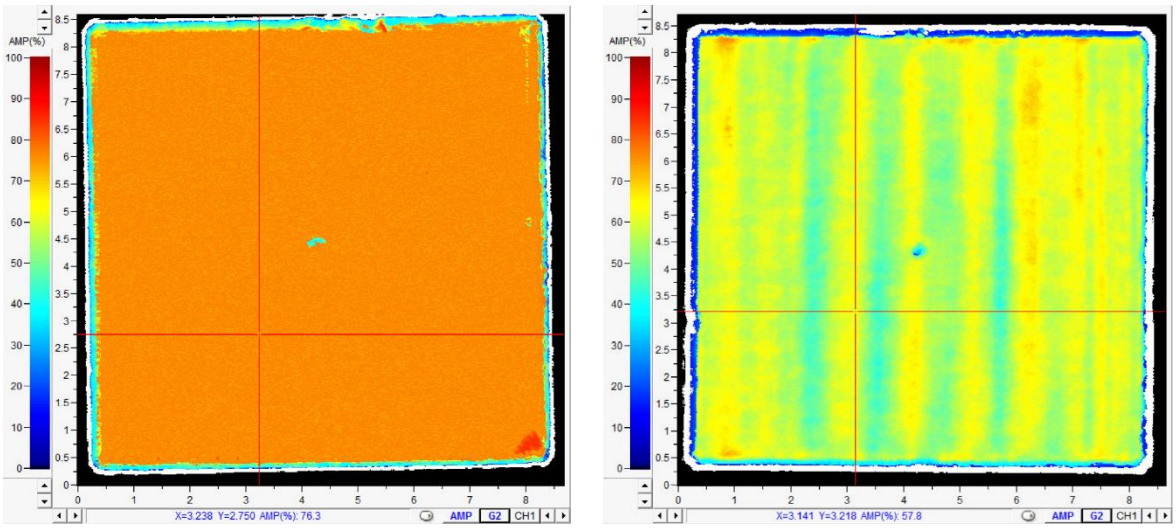

Figure 6. Ultrasonic images of the carbon fiber (left) and fiberglass (right) panels.

The results of the ultrasonic scan suggest that the carbon fiber panel had minimal porosity or other irregularities through its thickness. However, the fiberglass panel seemed to exhibit periodic irregularities in its lower half (tool side).

4.3 Microscopy

In order to evaluate the results of ultrasonic scanning, microscopy was performed on a central cross section of each panel. The sections were cut out using a wet diamond saw, then polished and cleaned to remove machining imperfections. Images obtained using microscopy are shown in Figure 7.

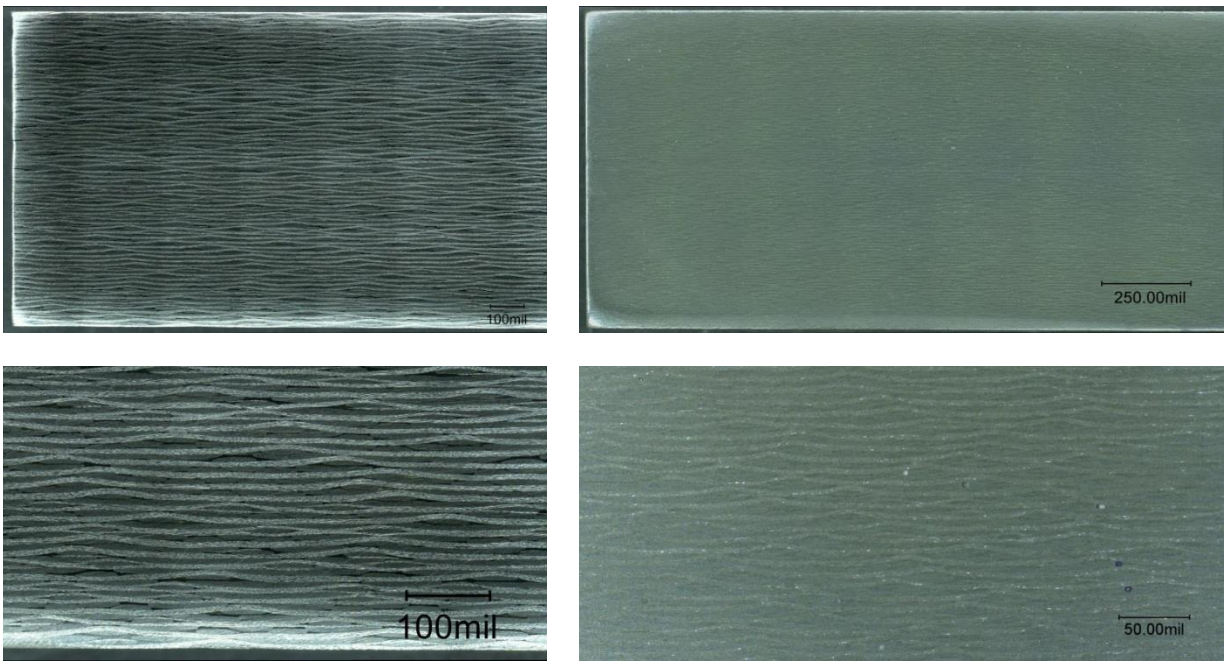

Figure 7. Central cross sections of carbon fiber (left) and fiberglass (right) panels.

The carbon fiber section had no visible voids throughout and exhibited good compaction. The fiberglass panel was also well-compacted, but exhibited porosity on the lower half (tool side). These images confirmed the results of the ultrasonic scanning.

A representative area of the fiberglass cross section was further analyzed to quantify the amount of porosity present in the sample using a digital area ratio approximation. An image of the analyzed area and of the analysis are shown in Figure 8. The analysis revealed an area ratio of 0.12 % porosity in that section.

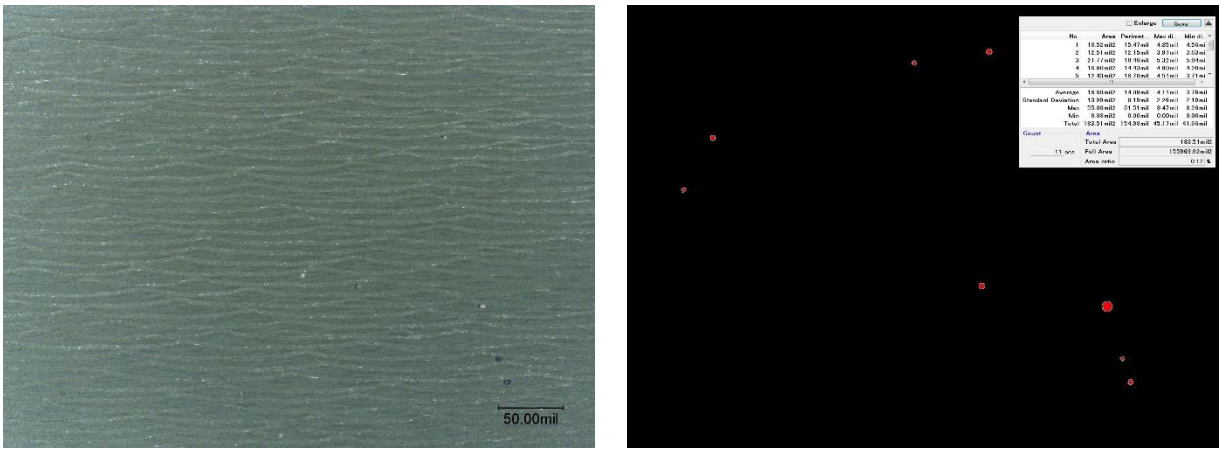

Figure 8. Areal analysis of representative fiberglass cross section with 0.12 % porosity.

4.4 Degree of Cure

To determine the degree of cure afforded by the cure cycle, samples of the finished panels were tested in the DSC. Any exothermic behavior exhibited by the samples would indicate an area of uncured resin in the sample. Samples were cut from both the center and edges of each panel. None of the samples generated heat during a constant 2.71 ºC/min (5 ºF/min) ramp up to and past the cure temperature of 177 ºC, indicating a high degree of cure throughout the panel.

5. SUMMARY

A systematic method of evaluating the reaction of composite prepregs to different cure schedules was employed to develop a cure cycle which was customized to manufacture thick-section composite panels. The cure cycle was designed to mitigate the peak heat generated during the exothermic cross linking of the epoxy resin to prevent excessive process-induced internal stresses in the panels. It was modified to trigger a low minimum viscosity point to allow adequate resin flow and to promote devolatilization.

It was discovered that the epoxy resin in this study was most sensitive to the ramp rates between isothermal holds. A fast ramp kicked off the exothermic reaction and caused high heat generation. However, a fast ramp also triggered a significant decrease in the viscosity of the resin. A slow ramp mitigated heat generation and caused a slow increase in resin viscosity.

A custom vacuum bag configuration was fabricated to preserve the geometry of the laminate and to attempt to preserve the exceptional final thickness of the TSC. The effectiveness of the cure schedule was evaluated by examining the quality of the resultant panels.

The final panels produced by the customized cure schedule exhibited good compaction and low porosity. The vacuum bag and instrumentation used during cure caused over-bleeding in both panels, which resulted in finished panels which were less than their designed thickness. Further research is necessary to determine an improved bagging schedule.

This process may be reasonably applied to future thick-section composite components, which are sensitive to processing parameters. First, the thermal properties of the resin must be determined. The baseline properties of this material were determined by using differential scanning calorimetry to evaluate the material's reaction to the manufacturer's cure cycle. Additional, lower isothermal holds may added before the final cure to cure the composite material more slowly and evenly. The effect of ramp rates on heat generation and viscosity must be determined. In this case, the effects were determined using differential scanning calorimetry and dynamic thermal mechanical analysis. The results of those tests may be used to determine the cure cycle which would best mitigate heat generation while producing a component with low porosity. Finally, the cure cycle must be evaluated by examining the quality of the finished component.

6. REFERENCES

[1] Wichita State University, "Thick-Section Composites," in *Composite Materials Handbook (CMH-17)*, vol. 3, Warrendale, PA: SAE International, 2012, pp. 15-1 - 15-15.

[2] T. A. Bogetti and J. W. Gillespie, "Cure Simulation of Thick Thermosetting Composites," U.S. Army Laboratory Command, Aberdeed Proving Ground, MD, 1990.

[3] A. K. Kulshreshtha and C. Vasile, "Neuroclave: The Intelligent Autoclave," in *Handbook of Polymer Blends and Composites*, vol. 2, Shrewsbury, Rapra Technology, 2002, pp. 27-28.

[4] Z. L. Yang and S. Lee, "Optimized Curing of Thick Section Composite Laminates," *Materials and Manufacturing Processes*, vol. 16, no. 4, pp. 541-560, 2001.

[5] T. A. Bogetti and J. W. Gillespie, Jr., "Process-Induced Stress and Deformation in Thick-Section Thermoset Composite Laminates," *Journal of Composite Materials*, vol. 26, no. 5, pp. 626-660, 1992.

[6] M. R. L. Gower, R. M. Shaw and W. R. Broughton, "Thick Composites: Part 2: Cure Optimisation," National Physical Laboratory, Middlesex, 2008.

[7] T. E. Twardowski, S. E. Lin and P. H. Geil, "Curing in Thick Composite Laminates: Experiment and Simulation," *Journal of Composite Materials,* vol. 27, no. 3, pp. 216-250, 1993.

[8] Mettler-Toledo AG, *Differential Scanning Calorimetry for all Requirements,* Schwerzenbach: Mettler-Toledo, 2011.

[9] T. A. Luckenbach, Ph.D., DMTA: Dynamic Mechanical Thermal Analysis, Piscataway: Rheometrics, Inc..

[10] 3M, "Surface Energy and Wetting," adhesives.org, [Online]. Available: http://www.adhesives.org/adhesives-sealants/adhesives-sealants-overview/structural-design/surface-energy-and-wetting.

[11] P. Oliver, J. P. Cottu and B. Ferret, "Effects of cure cycle pressure and voids on some mechanical properties of carbon/epoxy laminates," *Composites*, vol. 26, no. 7, pp. 509-515, 1995.

[12] J. L. Kardos, M. P. Dudukovic and R. Dave, "Void Growth and Resin Transport During Processing of Thermosetting-Matrix Composites," in *Advances in Polymer Science*, Berlin, 1986.

[13] Wichita State University, "Cured ply thickness," in *Composite Materials Handbook*, Wichita, SAE International, 2012, pp. 40-42.

[14] F. C. Campbell, A. R. Mallow and C. E. Browning, "Porosity in Carbon Fiber Composites: An Overview of Causes," *Journal of Advanced Materials*, vol. 26, no. 4, pp. 18-33, 1995.

[15] "Vacuum Bagging," in *Manufacturing Technology for Aerospace Structural Materials*, Amsterdam, Elsevier, 2006, pp. 307-310.