Fall 2014

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Recommended Citation

Taylor, Gregory A., "Fabrication of glass fiber-reinforced transparent composites using vacuum assisted resin transfer molding process" (2014). *Masters Theses*. 7862.
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FABRICATION OF GLASS FIBER-REINFORCED TRANSPARENT COMPOSITES USING VACUUM ASSISTED RESIN TRANSFER MOLDING PROCESS

by

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A THESIS

Presented to the Faculty of the Graduate School of

MISSOURI UNIVERSITY OF SCIENCE AND TECHNOLOGY

In Partial Fulfillment of the Requirements for the Degree

MASTER OF SCIENCE IN AEROSPACE ENGINEERING

2014

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ABSTRACT

The most commonly used transparent material is glass. Traditionally, glass is not desired for applications involving a low weight material while preserving high strength such as aerospace and military applications where armor must also be transparent. Some applications may include aircraft canopies and other vehicle windows. Development of a reliable transparent composite would fill a need for many of these applications where a transparent structure must both be strong and lightweight. A transparent polymer reinforced with a glass fiber fabric is a viable solution. To ensure transparency, both fiber and matrix must match in refractive index.

In the present work, transparent composites are manufactured using two vacuum assisted resin transfer molding (VARTM) processes. An E-glass fiber fabric is used as reinforcements and an epoxy-based resin developed at the Missouri University of Science & Technology is used as the matrix. In the VARTM process, the glass fiber preform is placed between two molds for part quality and the mold is sealed with a vacuum bag. Resin is drawn into the mold by a combination of a vacuum and atmospheric pressure to infuse the preform and the part is cured. A second, yet similar VARTM process has been developed to decrease void content and increase transparency of the composite panels through the elimination of air bubbles concentrated at the initial stages of infusion.

ACKNOWLEDGMENTS

I wish to express my deepest gratitude and appreciation to my academic advisor Dr. K. Chandrashekhara whose guidance has been so vital throughout the pursuit of my Master's degree. Both his support and knowledge have been driving influences throughout my graduate research.

I would like to thank the members of my advisory committee, Dr. A. C. Okafor and Dr. T. Schuman for their support and cooperation. I would also like to specially thank Dr. T. Schuman for his knowledge and insight into the chemistry and manufacture of the transparent composites.

I would also like to thank all the members of the Composite Research group, especially Dr. R. R. Vuppalapati, who worked extensively with me in the transparent composites.

Finally, I would like to give special thanks to my father, mother, and sister for their continued support and love through my graduate research. Without their support, I would not have been able to achieve what I have today.

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1. INTRODUCTION

1.1. POLYMER-MATRIX COMPOSITES

Polymer-matrix composites are utilized in a variety of structural applications due to their low density, high strength-to-weight ratio, high strength-to-modulus ratio [1]. A composite consists of a reinforcing material (fibers or particles) surrounded by a matrix, often a polymer resin [2]. While particles are often uniformly distributed, fibers (with the exception of short chopped fibers) are generally placed in a cross-ply or unidirectional pattern to aid in structural rigidity.

In structural applications, composites offer significant advantages over other materials because of their low density and high specific strength. The military and aerospace industries are interested in composites for uses in ballistic and explosive armor due to a weight reduction without decreasing structural performance [3]. This reduction in weight while maintaining or improving structural performance can have substantial benefits including more efficient vehicle operation and greater protection of human life.

1.2. TRANSPARENT COMPOSITES AND APPLICATIONS

Currently, a need exists for the improvement of ballistic armor in military applications. Modern transparent armor consists of laminates fabricated from ceramics or glass and a transparent polymer such as polycarbonate (PC) as shown in Figure 1.1 [4, 5]. The thickness of the modern transparent armor consists of 25 mm thick glass plates stacked with at least three layers and a thin transparent polymer layer. Glass is used due to its hardness, strength, chemical and abrasion resistance, and low cost, but suffers in performance due to its brittle and heavy nature [6]. Transparent composites fill the need to replace the heavier glass or sections of glass due to their lightweight but durable characteristics [7, 8]. A proposed solution to the conventional transparent armor is shown in Figure 1.2 where a thin layer of transparent composite material is inserted between a glass and transparent polymer. The thickness of the proposed transparent armor consists of a 25 mm thick glass plate , a 6 mm thick transparent panel, and a thin transparent polymer layer. An additional advantage of the transparent composite layer is the structural rigidity given by the composite material.

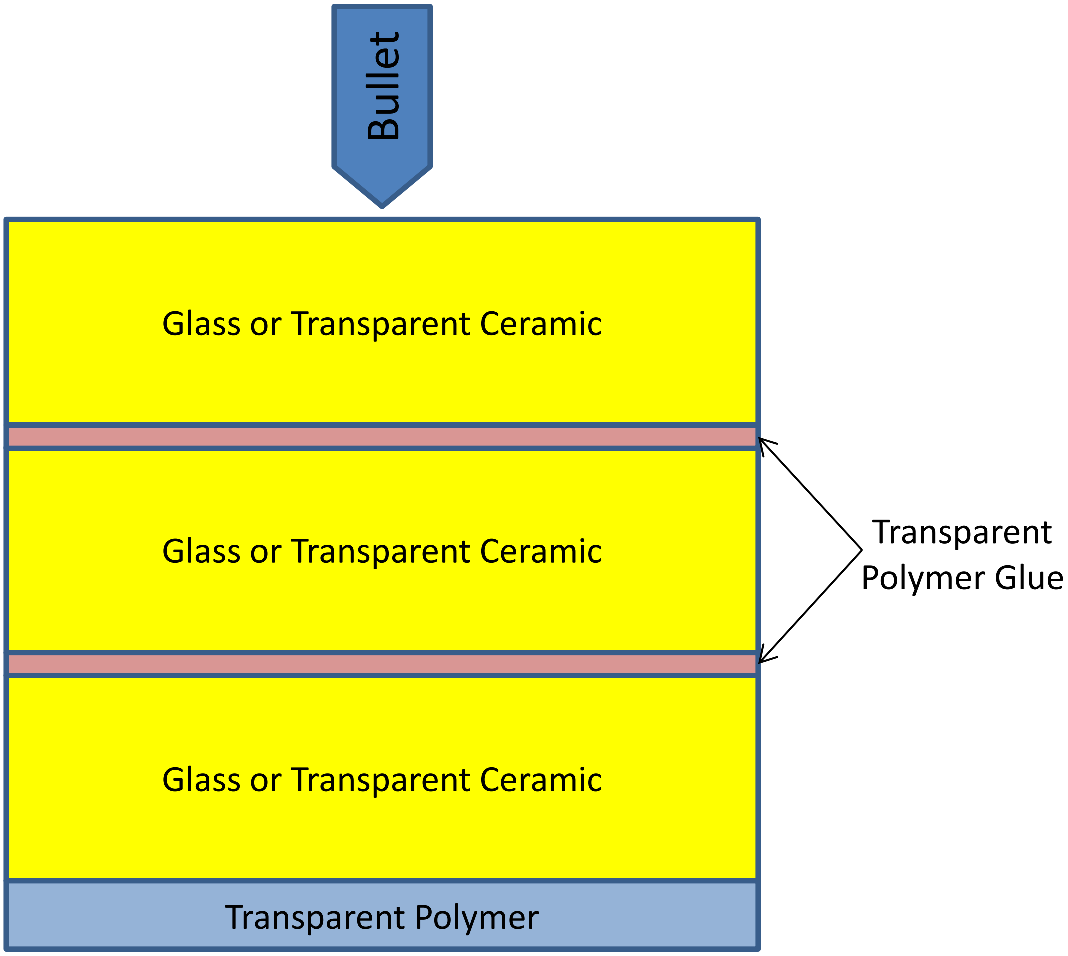

Figure 1.1. Conventional transparent armor

For both the conventional and proposed transparent armor, materials can vary depending on requirements of the transparent armor. The glass or ceramic layer can be manufactured from soda-lime silica, other strengthened glasses, or a ceramic such as aluminum oxynitride (ALON). The transparent polymer layer can be manufactured from polycarbonate, poly(methyl methacrylate) (PMMA), or polyurethane (PU) [9]. The additional transparent composite layer in the proposed solution is made from a glass fiber-reinforced composite with a thermoset resin matrix such as epoxy.

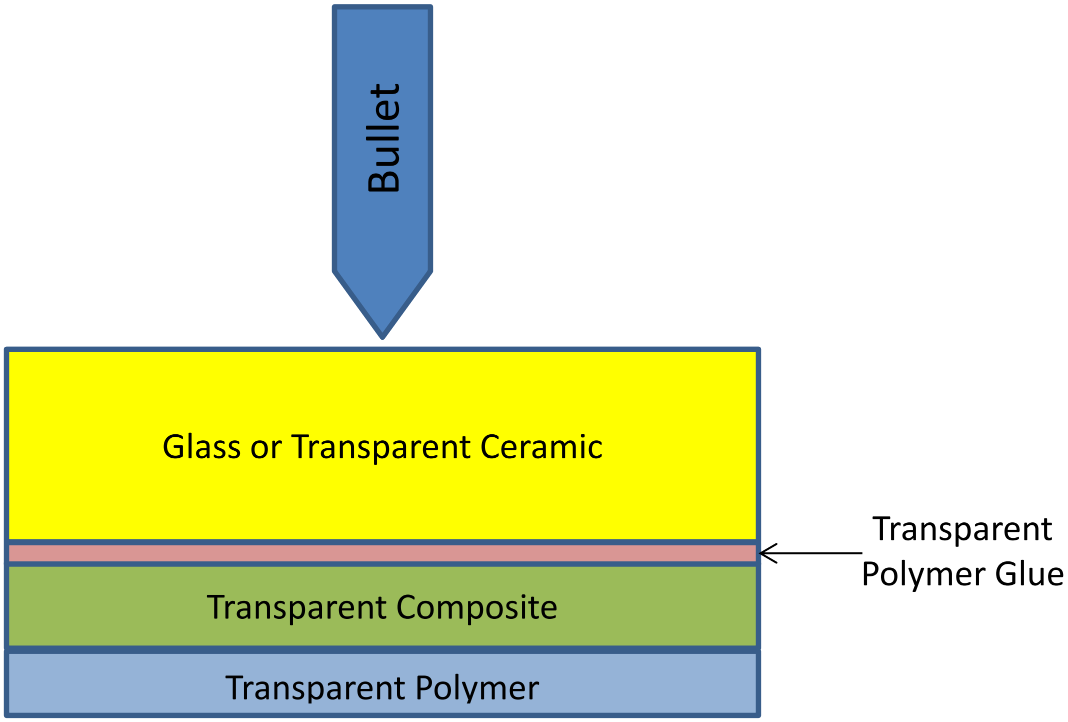

Figure 1.2. Proposed transparent armor with addition of transparent composite layer

The idea of manufacturing a transparent composite relies heavily on matching and maintaining the refractive index match between both the fiber and the matrix (Figure 1.3). If the refractive index of the fiber and matrix does not match, the light passing through the composites will refract or scatter, resulting in a distorted or opaque composite. The bending of light through different media is theorized in Snell's law (1):

$$n_1 \sin\theta_1 = n_2 \sin\theta_2 \tag{1}$$

In Snell's law (Figure 1.4), n represents the refractive index of the respective material while θ represents the angle of incident of the light rays. In order for light to pass through and create a transparent material, n_1 and n_2 must be equal.

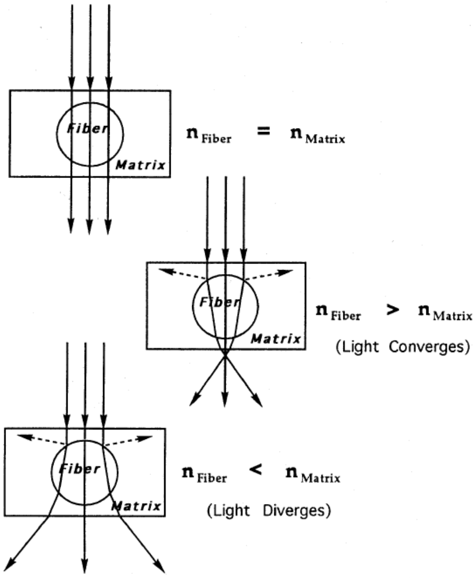

Figure 1.3. Refraction and reflection of light rays passing through a composite where "n" equals the refractive index of the material

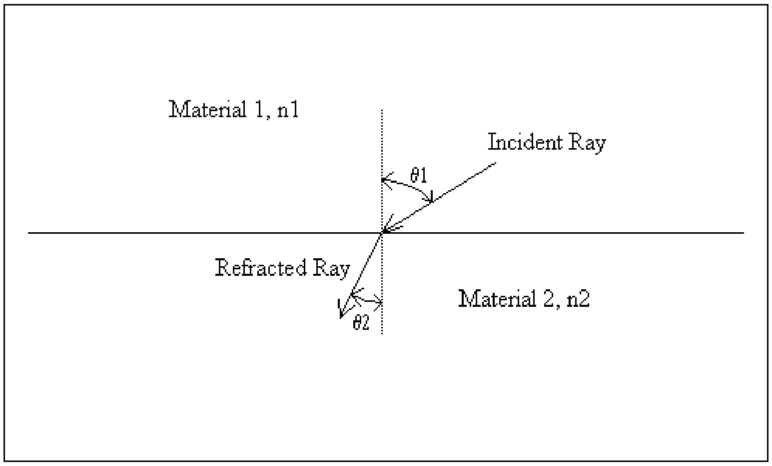

Figure 1.4. Snell's Law

In order to achieve transparency for the composite, one material's refractive index must be changed to match the other's refractive index. The glass fibers' refractive index can be adjusted by altering the chemical composition, controlling the thermal history during manufacture, or changing the sizing of the fiber. The sizing of the fiber is a chemical added to the glass surface during manufacture that forms bonds to specific resin matrices. The glass's chemical composition controls the density of the glass. This density relates to the refractive index in that, as density increases, so does the refractive index.

The matrix's refractive index can be modified by altering the (polymer) chemical structure. If the matrix is a polymer, the refractive index depends on the chemical initiator for chain growth, the amount of cross-linking between polymer strands, and the temperature and time for which the polymer is cured. Excessive or prolonged exposure to the elevated temperatures can thermally degrade and discolor the matrix.

1.3. COMPOSITE MANUFACTURING PROCESSES

Composites for the military and aerospace industry are commonly cured inside and autoclave at both elevated temperature and pressure. The added pressure, of typically 60 to 100 psi, reduces the size of entrapped air in the fiber-matrix layup to greatly reduce void content and improve the overall part quality [10]. The primary problem with the autoclave process is the cost of operation of maintaining an elevated temperature and pressure on an inert environment to prevent chemical reactions with the manufacture part. The environment can typically be nitrogen or argon gas.

To reduce the overall costs of the composite fabrication, composite manufacturers want to switch to low cost out-of-autoclave (OOA) processes. Similar to an autoclave process, the composites are manufactured under a sealed vacuum bag and at an elevated cure temperature for the resin. The primary differences for the OOA processes are the lack of elevated pressure and/or lack of inert atmosphere required to achieve a complete cure of the resin. The OOA processes also enable the manufacture of much more complex structure due the absence of the pressure vessel required for an autoclave. The downside of OOA processes are the potentially higher void content in comparison to autoclave [11]. The overall advantages include a greatly reduced operating cost, initial investment in manufacturing, and energy cost.

One of the OOA processes in use today is the vacuum assisted resin transfer molding (VARTM) technique as seen in Figure 1.5 [12, 13]. VARTM provides several advantages over the autoclave process such as low cost of mold or tool, increased variability and complexity in the composite parts, and reduction of volatile chemicals commonly dispersed during the cure cycle [14]. The quality of composite parts manufactured through VARTM is controlled by numerous parameters including resin viscosity during infusion, ease of penetration of resin into the fibers during infusion, locations of inlets and outlets, and resin cure cycle [15]. The resin viscosity depends on chemical structure of the resin, temperature during infusion, and fiber wetting. The ability for the resin to penetrate into the fiber layup greatly relies on the fiber density, resin viscosity, and vacuum pressure at the outlet [16]. Locations of the inlets and outlets can enhance or reduce the resin flow characteristics during infusion. The cure cycle controls volatility of the chemicals as well as rate at which a resin cures.

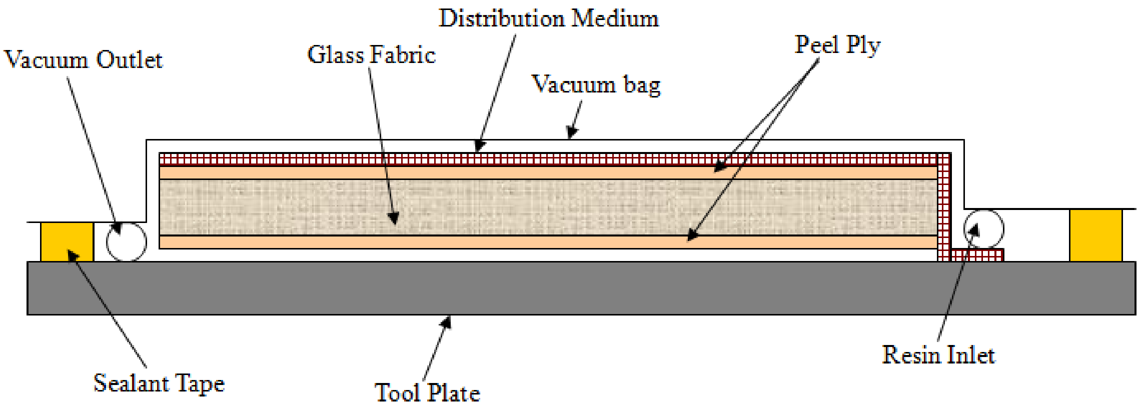

Figure 1.5. Typical VARTM process for manufacture of composites

1.4. CONTRIBUTION OF THIS WORK

In the present work, a vacuum assisted resin transfer molding process was used to manufacture transparent composites. The work presented within this thesis specifically involves the synthesis of a refractive index matched epoxy-based resin, production of transparent composite panels, and performance of the transparent panels. The resin composition was varied to refractive index match the glass fibers. The fabrication of the transparent composite panels utilizes the VARTM process while altering the traditional process to improve the quality of the composite parts. The performance evaluation was conducted using a clarity test and two mechanical tests, tensile and flexure. Through each step of the resin synthesis and production and evaluation of the transparent panels, a better understanding of the entire transparent composite production process was developed.

2. MATERIALS

2.1. GLASS FIBER REINFORCEMENT

An E-glass woven fabric manufactured by Owens Corning Composite Materials was used as the fiber reinforcement in the transparent panels (Figure 2.1). The fabric weight is 18 oz/yd^2. The reinforcement consisted of either a bi-directional (0°/90°) woven fabric or a unidirectional layup formed by the removal of one direction of fibers from the woven fabric. Removing one direction of fibers from the bidirectional fabric ensured all fibers had matching refractive indices as the fibers are from the same fabric roll or batch. Having each layup from the same fabric roll or batch is important due to E-glass fibers having varying refractive indices from batch to batch even from the same manufacturer.

Figure 2.1. Conventional E-glass fibers manufactured by Owens Corning

2.2. EPOXY-BASED POLYMER MATRIX

A one-part transparent resin system was developed at the Missouri University of Science and Technology. The epoxy resin system was created to match the refractive index of the fibers by the blending of two epoxy resins and a cure hardener (Table 2.1).

The two epoxies include the aromatic Epon 826 resin produced by Momentive and aliphatic Epalloy 5200 resin produced by Emerald Performance Materials. Hexahydrophthalic anhydride (HHPA) produced by Dixie Chemical was used as the cure hardener. In Table 2.1, the refractive index of the E-glass fibers is a calculated value from the synthesis of the resin while Owens Corning only provides a range of possible refractive indices. The refractive index of 1.54 was determined from the liquid resin system after degassing

Table 2.1. Resin system

RESIN SYSTEM	Manufacturer	R.I.	Mass (g)
Epon 826	Momentive	1.573	99.5
Epalloy 5200	Emerald	1.486	2.6
HHPA	Dixie	1.47	45
E-glass	Owens Corning	1.54	

The process for making the resin system first involves measuring out a desired amount of Epon 826. Then, Epalloy 5200 was added to the Epon 826 until the total mass of both epoxies added to a fixed amount, e.g., ~100g. While holding the total epoxy at a fixed amount, the ratio of Epon 826 to Epalloy 5200 could be altered to tune the refractive index.

After combining the epoxies, hexahydrophthalic anhydride was heated (~80°C) and added to the mixture as a weight ratio of total epoxy to HHPA. The purpose of heat is to melt the HHPA to a liquid state as the HHPA is normally crystalline at room temperature. A tiny amount (negligible mass) of transparent catalyst was added to the mixture to initiate chain formation of the resin.

The mixture was thoroughly stirred until the blend was visibly clear. Finally, the new resin system was degassed at approximately 50°C for a short period of time to evacuate any excess air contained with the resin from the mechanical stirring. Multiple samples of the resin system with varying chemical ratios were synthesized and cured until a resin system was found to match the E-glass fibers upon curing.

The cure cycle for the fiber and resin samples was set at 80°C for 12 hours after a steady ramp at 1-2°C/min (Figure 2.2). A sample of one of the resins with chopped fibers can be seen in Figure 2.3. This specific sample had slightly different refractive indices for the fiber and resin as the fibers are just visible over the colored background. After finding a suitable matching resin for the fibers, a larger quantity of the resin was synthesized to use in the VARTM process.

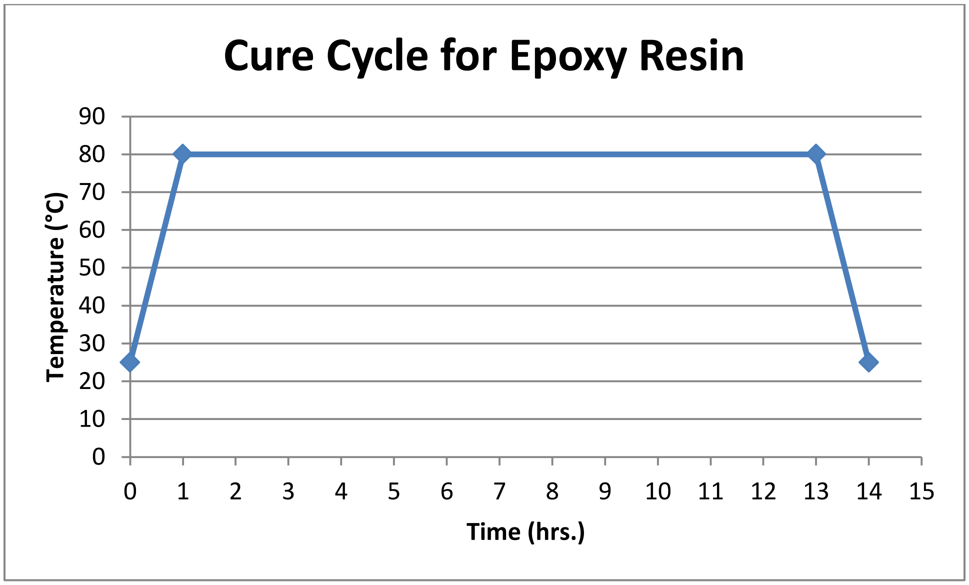

Figure 2.2. Resin system cure cycle

Figure 2.3. Fiber and resin sample for refractive index matching

The new resin system has low viscosity and a polymer glass transition temperature (~60°C) to allow use in the VARTM process. The low viscosity gives the resin a sufficient flow to infuse into the glass fibers during VARTM, and the glass transition temperature is high enough to allow the formation of a glassy polymer matrix.

Several other resins were manufactured using similar techniques in attempts to raise the glass transition temperature while maintaining a strong transparent composite sample. The idea of raising the glass transition temperature was to raise the maximum possible cure temperature. Raising the cure temperature allows for an increased infusion temperature. The increased infusion temperature provides a lower viscosity during infusion, resulting in better fiber-to-matrix attachment in the composite samples. A side goal of the raised glass transition temperature was to hopefully reduce air bubbles entering the fiber layup during infusion. These other attempted resins involved increasing the anhydride-to-epoxy weight ratio and/or altering the type of anhydride in the blend while maintaining the anhydride-to-epoxy ratio. However, no resin composition was found that raised the glass transition temperature while maintaining a transparent composite upon curing.

3. FABRICATION USING VARTM

3.1. MOLD AND VARTM PREPARATION

The two part mold consists of a large glass mirror and a small glass square. The mirror and glass materials are used for their high-quality surface in order to improve the surface quality of the manufactured transparent panel. The bottom mold consists of a glass mirror measuring 60 cm by 60 cm, and the upper mold consists of a glass square measuring approximately 15 cm by 15 cm. The mold was cleaned using solvents (Acetone) and cotton cloths to remove all excess residues. Upon cleaning, Frekote was applied as a release agent to ease separation between the manufactured panel and the mold. The release agent also aids the infusion process by allowing quicker infusion due the compatibility with epoxy resin. Six layers of E-glass woven fabric or twelve unidirectional fibers were then laid on to the mirror mold and the glass square mold was placed on top of the fibers.

Resin inlet and vacuum outlet lines were positioned on opposites sides of the fiber layup as seen in Figure 3.1. No peel ply or distribution medium was present in the preform as these materials can damage surface quality and transparency of the composite panel. The preform was then sealed with a vacuum bag and high tack sealant tapes. A two-stage vacuum with an ultimate vacuum of 0.013 Pa ($1x10^{-4}$ torr) was connected to the preform and all excess air is removed. Upon the removal of most of the air, any air leaks found were sealed to prevent any air from entering the part during curing.

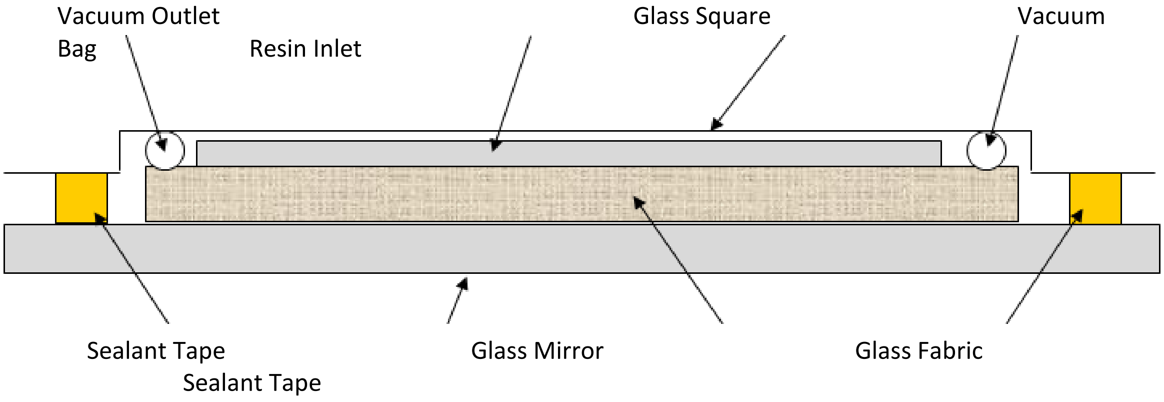

Figure 3.1. VARTM process schematic for transparent composites

On sealing of all air leaks, the mold and resin was degassed and heated to between 50˚C and 80˚C, usually 60 ˚C, to remove any remaining air and lower the resin's viscosity. The effect of different degassing temperatures on part quality was checked throughout the experiment, but little or no difference was observed. Lowering the viscosity improved the rate and quality of infusion.

3.2. INFUSION PROCESS

To begin the infusion process, the inlet line was opened and the epoxy resin was free to flow into the preform. Throughout infusion, the mold and resin were kept at the elevated temperature to lower the resin's viscosity. The infusion is aided by atmospheric pressure pushing the resin through the preform into the space evacuated by the vacuum pump. Any excess resin that flows through the outlet line was captured in a resin trap. The vacuum pump remained running throughout the experiment to ease the infusion as the process which took anywhere from fifteen to sixty minutes. Upon the panel being fully infused with resin (Figure 3.2), both inlet and outlet were closed. The vacuum was left on to aid in a possible sealant or valve failure. Occasionally, the infusion process fails and the part will have insufficient resin or high quantities of voids. These failures are most often due to improper sealing of the preform or insufficient resin.

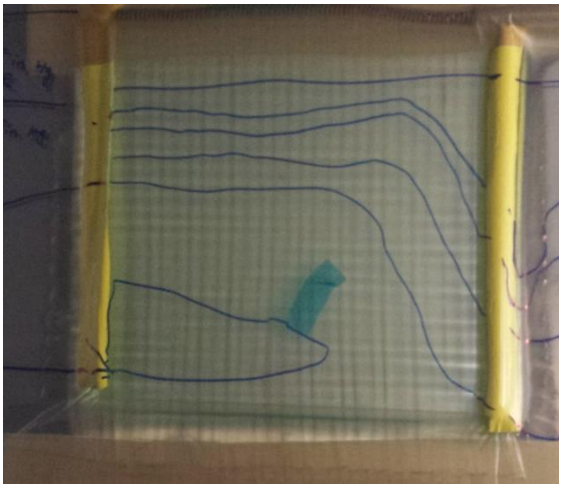

Figure 3.2. Fully infused panel before curing (not transparent at this temperature)

3.3. CURE CYCLE

Transparent panels were manufactured with a 12 hour cycle at 80˚C (Figure 2.2). Resulting transparent panels were shown to be promising in terms of void quantity, and all parts in this study were manufactured using the 80˚C cure cycle. Ramp rates of the cure cycle was set at ~1-2˚C/min. On completion of the cure cycle, the resin has hardened, and the part was cooled for any post fabrication.

Initially, transparent panels were intended to be manufactured with a two-stage cure to reduce residual stress in the composite parts. This cure utilized an initial soak at 80˚C before raising the temperature. This two-stage cure was abandoned due to discoloring from the elevated temperatures.

3.4. TEST SAMPLES

Once the part was cured and mold has cooled to room temperature, the part was separated from the mold. The transparent panel was then examined for visible voids, microscopic voids, surface finish, and refractive indices matching.

4. IMPROVED FABRICATION USING VARTM

4.1. REASONING FOR MODIFICATION

Initial attempts of the VARTM manufacture of transparent panels yielded large size (macroscopic) and quantity of voids. Voids damage the transparency of the panel. Since VARTM traditionally yields higher void contents, a modification to the process was created to reduce void content and improve transparency of the composite panels.

4.2. MOLD AND VARTM PREPARATION

Mold preparation was unaltered from the previous VARTM setup. The mold was cleaned and release agent was applied. The details to the preparation of the mold are found in Section 3.1.

Resin inlet and vacuum outlet lines were positioned on opposites sides of the fiber layup as before, but additional vacuum lines and resin reservoirs were added to trap air. The additional lines aid the infusion process in removing resin containing air from entering the fiber layup. The top-down view of the modified VARTM process is shown in Figure 4.1. The goal of the modified VARTM process was entirely focused on aiding the infusion of the resin into the panel.

Similar to the older VARTM process for transparent panels, no peel ply or distribution medium was present in the preform. The preform was then sealed with a vacuum bag and high tack sealant tapes. A two-stage vacuum pump was connected to the preform and all excess air was removed. Upon the removal of most of the air, any air leaks found were sealed to prevent any air from entering the part during curing. On sealing of all air leaks, the mold and resin were degassed and heated to between 50˚C and 80˚C (usually 60˚C) to remove any remaining air and lower the resin's viscosity.

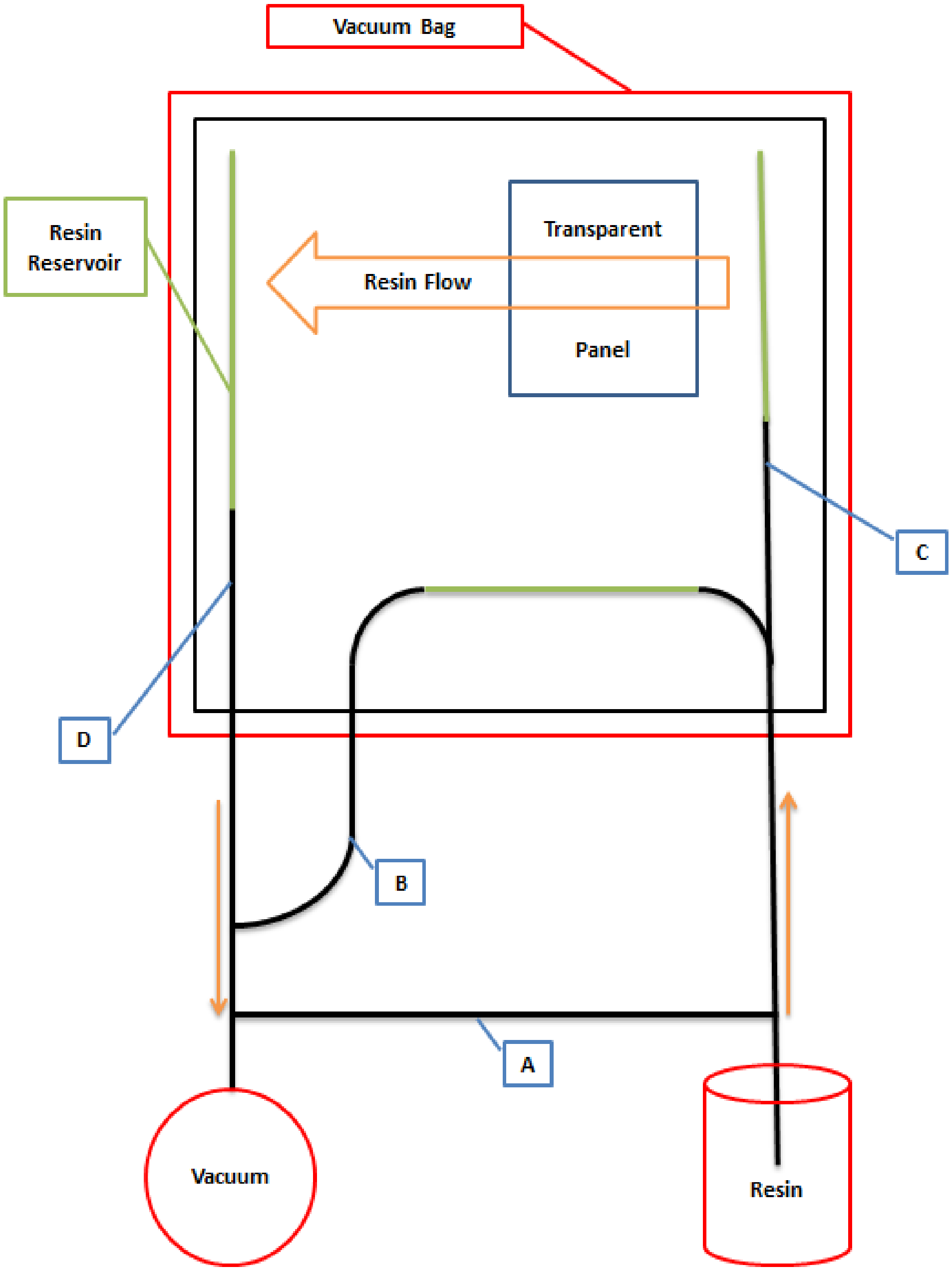

Figure 4.1. Top-down view of schematic of the modified VARTM process

4.3. INFUSION PROCESS

For the infusion process, please refer to Figure 4.1 for the schematic of the modified VARTM process. Initially, the primary inlet line was closed, and the preform and resin were then thoroughly degassed at an elevated temperature (~60˚C).

Previously, the older VARTM process showed large quantities of air bubbles contained with the initial resin entering the preform and fiber layup. This air proceeded to get trapped in the fibers and produce the voids. To counter these pockets of air, the initial resin was evacuated into two reservoirs.

To begin the infusion process, the first reservoir (designated A in Figure 4.1) line was opened and the epoxy resin was free to flow into the reservoir A. Reservoir A consisted of a standard vacuum/infusion tube that could be closed or clamped at both ends. The outlet of reservoir A was held at an increased height relative to the inlet to ease the flow of the air bubbles into the reservoir through a buoyancy effect. Air bubbles noticeably tend to float on top of the resin even in the vacuum lines. Upon filling of the first reservoir, the reservoir was closed at both its inlet and outlet.

The primary infusion line into the preform was opened at the inlet of the preform. This line feeds directly to both the second reservoir (B) and panel inlet (C). Since the desired effect was for the air to become trapped in the reservoir, the outlet of the preform was closed while the outlet of reservoir B was left open. Reservoir B consisted of a standard vacuum/infusion tube that could be closed or clamped at both ends as well as a steel spring inside the preform to help trap air. Any remaining of the initial resin containing air was evacuated into reservoir B until filled. The entire preform was tilted with the outlet above the inlet during this process to ease the flow of air bubbles into the reservoir. Upon filling of reservoir B, the outlet of B was closed to hinder flow of the resin both forward and backward in the reservoir. The inlet of B is part of the preform and cannot be closed.

The primary outlet line (D) was opened to allow the resin to infuse around the fibers. Throughout the entire infusion process, the mold and resin were kept at the elevated temperature to lower the resin’s viscosity. Due to reservoirs A and B, a larger quantity of resin was initially degassed in order for the fibers to fully infuse. Again, the infusion was aided by atmospheric pressure pushing the resin through the preform into the space evacuated by the vacuum pump. During the manufacturing with this process, a weaker atmospheric pressure occasionally struggled to fully infuse the fibers, so reservoirs A and B were limited to a length of no more than 1 meter.

The vacuum pump was left on to ease the infusion. The addition of the reservoirs added about 15 minutes to the total infusion process (30-75 minutes). Once fully infused, both inlet and outlet of the preform were closed, and the cure cycle was initiated. The vacuum was left on to aid in a possible sealant or valve failure.

4.4. CURE CYCLE

Only one cure cycle was utilized for the manufacture of the transparent panels as seen in the previous VARTM process with a 80˚C cure cycle. The cure cycle of 12 hours above the glass transition temperature of the resin (80˚C) was used along with the ramp rate set at ~1-2˚C/min. On completion of the cure cycle, the resin has hardened, and the part was then cooled to room temperature for any post fabrication.

4.5. TEST SAMPLES

Once the part was cured and mold has cooled to room temperature, the part was separated from the mold. The transparent panel was then examined for visible voids, microscopic voids, surface finish, and refractive indices matching. If the sample contained few or no visible (non-microscopic) voids, the sample was cut and prepared for clarity or mechanical testing.

5. PERFORMANCE EVALUATION AND RESULTS

5.1. CLARITY EVALUATION OF SAMPLES

Photographs testing the clarity of a transparent panel were taken. Figure 5.1 shows a printed paper background without transparent composite, and Figure 5.2 shows the background with a 15 cm by 15 cm transparent composite panel manufactured at Missouri S&T resting on top. Figures 5.3-5.5 show the clarity of the composite panel when the panel was placed at distances (d) of 7.5 cm, 15 cm, and 30 cm from the background, respectively. While the transparency matched at zero distance, the transparency decreased when the distance between the background image and the composite increased. The most likely cause of the decrease in clarity was microscopic voids between groups of fibers. These voids can be attributed to either volatile gases developing during the infusion or air passing through the inlet during infusion. The voids concentrated along some fiber bundles producing vertical streaks where the volatiles and/or air could not find a flow path to escape.

Figure 5.1. Background image with no transparent panel

Figure 5.2. Transparent panel with background (d = 0 cm)

Figure 5.3. Transparent panel with background (d = 7.5 cm)

Figure 5.4. Transparent panel with background (d = 15 cm)

Figure 5.5. Transparent panel with background (d = 30 cm)

5.2. TENSILE TESTING

The transparent composite panels were cut and along with neat resin samples using an Instron-5985 testing machine based on their tensile modulus, tensile strength, and strain to failure in accordance with the ASTM D-3039 standard test. An extensometer measuring strain was attached to five specimens measuring 152 mm x 13 mm x 1 mm. and tested with a crosshead speed of 1.27 mm/min. The tension test setup is shown in Figure 5.6.

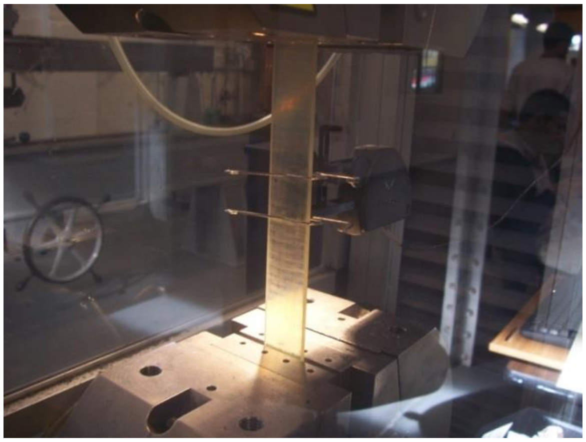

Figure 5.6. Tension test setup

Table 5.1 lists the average results from the tensile tests. The tests indicated the transparent composites had a tensile modulus of 31.74 GPa, tensile strength of 374.90 MPa, and strain to failure of 1.84% while the neat resin samples only had a tensile modulus of 3.17 GPa, tensile strength of 46.75 MPa, and strain to failure of 2.14%.

Table 5.1. Tensile test results

Samples	Tensile Strength (MPa)	Tensile Modulus (GPa)	Strain to Failure (%)
Neat Resin	46.8 ± 2.5	3.17 ± 0.13	2.14 ± 0.11
Composite	375 ± 24	31.7 ± 1.9	1.84 ± 0.09

5.3. FLEXURE TESTING

Composite panels were also cut, tested, and compared to neat resin samples for their flexural modulus and strength on an Instron-5985 testing machine in accordance with the ASTM D-790 standard test with a crosshead speed of 0.25 mm/min. Five specimens measuring 152 mm by 13 mm by 1 mm supported over a span of 25 mm were tested as shown in Figure 5.7.

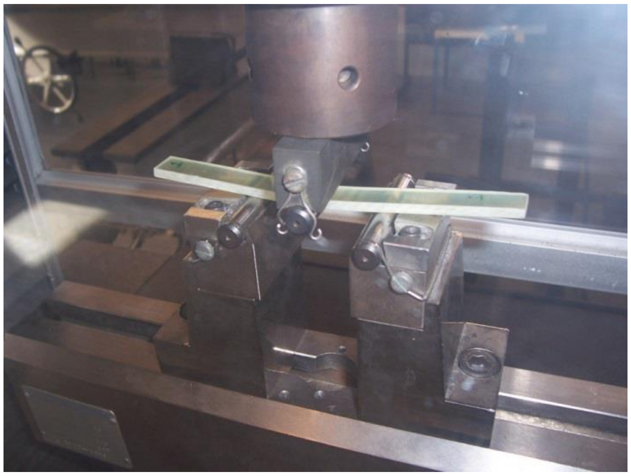

Figure 5.7. Flexure test setup

Table 5.2 lists the average results from the flexural tests. The tests indicated the transparent composites had a flexural modulus of 19.01 GPa, flexural strength of 629.70 MPa, and strain to failure of 3.50% while the neat resin samples only had a flexural strength of 125.30 MPa and a flexural modulus of 4.28 GPa, and strain to failure of 4.90%.

Table 5.2. Flexural test results

Samples	Flexure Strength (MPa)	Flexural Modulus (GPa)	Strain to Failure (%)
Neat Resin	125 ± 6	4.3 ± 0.3	4.9 ± 0.4
Composite	630 ± 35	19.0 ± 1.1	3.5 ± 0.15

6. CONCLUSION

An glass fiber and resin system with matching refractive index was developed for use in transparent composite panels. A modified VARTM process has been developed to eliminate as many voids as possible from manufactured transparent panels. Optically transparent composites were manufactured using the vacuum infusion process. Results show the feasibility of manufacturing mechanically strong and light weight transparent composites using low cost continuous E-glass fibers. Clarity, tensile, and flexure tests have been conducted to evaluate the performance of new materials. The manufactured panels show good quality and mechanical properties. These new transparent composites will find possible applications in windows, windshields, and other components where a strong, lightweight transparent material is desirable.

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