Experiments Related to the Fabrication of a Graphite/Epoxy Tube by the Resin Transfer Molding (RTM) Process

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ABSTRACT

Graphite/epoxy composite tubes can be fabricated by several manufacturing processes. Probably the least expensive method for producing a composite tube would be by pultrusion followed by prepreg mandrel wrapping, centrifugal casting and filament winding. This paper however describes the application of Resin Transfer Molding (RTM) to composite tube fabrication. Early RTM experiments with the fabrication of flat panels by the RTM method clearly indicated the value of vacuum assisted RTM when the level of microvoid content is considered. A brief summary of this work is included in this paper. The tubes fabricated were two inches (5.08 cm) in outside diameter and thirty six inches (91 cm) long with a 0.125 inch (0.32 cm) wall thickness. Braided graphite fiber preforms were used as the reinforcement and an epoxy resin was used as the matrix. The braid was packed into the mold to achieve a 50% fiber volume. The matrix was mixed, degassed, and pumped into the evacuated mold containing the preform with a peristaltic pump with a pressurized reservoir. The tooling consisted of a one-piece steel tube for the tube's outside diameter and a Teflon rod to form the tube's inside diameter. This paper covers the details of the RTM fabrication method for composite tubes as well as a description of the tooling required.

Keywords: RTM, resin transfer molding, graphite-epoxy composites, composite tubes, composite tube fabrication, void content

INTRODUCTION

Although composite tubes can be fabricated by several established processes, this work was felt to be important for those applications where the braided preform fiber architecture is of significant value. Resin Transfer Molding (RTM) is felt to be an excellent fabrication method for tubes fabricated from a braided preform. However, it must be acknowledged that composite tubes of excellent quality and a reasonable cost can be fabricated by established processes such as filament winding, pultrusion, centrifugal casting and prepreg mandrel wrapping. Each of these fabrication methods have characteristics that make them suitable for various applications based on production quantities required, cost and/or mechanical properties.

Braided preforms can be produced relatively inexpensively, have good uniformity and provide composites with a reasonable set of mechanical properties. Of particular interest is the torsional strength of composites fabricated with braided preforms. Researchers have found many applications for torque tubes and other similar applications. They have also found that composites based on these preforms tend to retain a greater percentage of their mechanical strength after impact than regular laminated constructions. Composite fabricated from braids have also been found to have an enhanced appearance which is important for some composite applications. Although braids are primarily produced in tube form, they can be formed on a shaped mandrel. In addition, they can be further shaped into many configurations by the mold. Some fabricators use preforms made from slitted braids, however this type would probably not be appropriate for net shape molding.

RTM is still an emerging composite fabrication process and is being addressed by extensive research at many manufacturing, research and university facilities throughout the world. Much of the research interest is directed toward obtaining high fiber volume, controlled fiber architecture encapsulated in a strong, tough matrix with a low void content such as those composites obtained by the autoclave processing of prepreg.

The fiber architecture provided by braids is attractive for many structural applications. Commercial braids can be readily obtained in a variety of forms. Simple biaxial tubular braids can be obtained in a variety of fiber orientations. Biaxial braids with ±30 and ±45 degrees are popular and often are off-the-shelf items at braid suppliers but many others are also available. Triaxial braids are also commercially available but usually must be specially prepared. Triaxial braids are attractive because axial tows are included in the biaxial. The addition of the axial tows improves the tensile and compressive strength along the axis of the tube with little loss in the torque strength. Both the biaxial and the triaxial braids are usually plied to obtain the required preform thickness.

A relatively new braid form for preforms is the 3-D braid. In this type, each tow will connect both surfaces and form a thick preform which may eliminate the need for plies. With all the plies tied together, failure by delamination is impossible. Like the biaxial braids, axial tows may be added to provide a 3-D braid with axials with a similar increase in tensile and compression strength. The commercial availability of 3-D braids is limited and they are very expensive.

PRIOR WORK

One of the primary areas of research being conducted at this facility in RTM is the formation (and minimization) of microvoids. These voids are contained within the matrix and no dry fibers are involved. Ghiorse (Ref. 1) studied these voids and found they had a significant effect on the mechanical properties of laminated composites. The properties most affected are those influenced by interlaminar shear. This property is reduced by the presence of the small (microvoids) between the plies of the composite.

A study (Ref. 2) was conducted utilizing a 10" X 11" X 0.100" (25.4 cm X 27.9 cm X 0.254 cm) panel mold with a transparent (methyl methacrylate) cover plate. A preform was devised which provided a controlled variable permeability. The preform was constructed of plies of 8-harness satin weave graphite cloth and provided for a controlled fiber volume variation from 40 to 54%. This preform is illustrated in figure 1. To obtain the controlled fiber volume the preform was compressed to a thickness of 0.100 inches. Various matrix fill techniques were utilized to explore some of the processing variables that affected the microvoid content. The most significant finding involved the use of vacuum assisted RTM. Figure 2 illustrates the experimental setup for vacuum assisted RTM used for that study. Within the range of the research, when the vacuum assisted RTM was used, the matrix fill rate did not affect the microvoid content, whereas, without the vacuum the void content increased as the fill rate increased. Another conclusion drawn from this research was that a panel fabricated with a vacuum fill without a vacuum tight mold contained more microvoids than the panels fabricated without vacuum assistance.

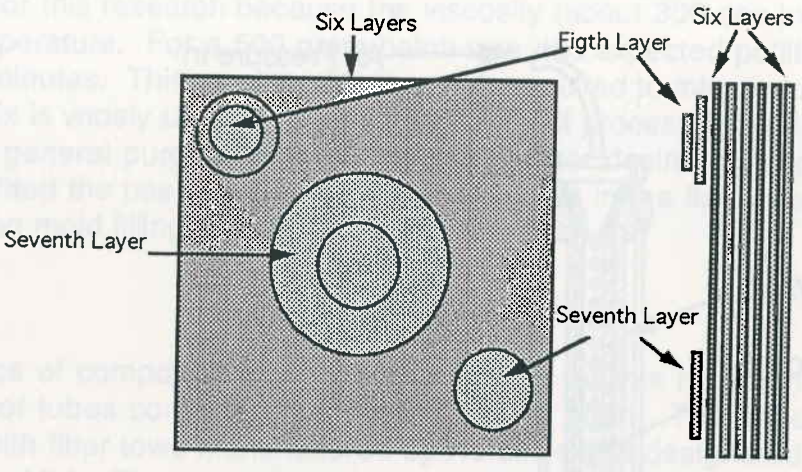

Figure 1, Stacking Sequence to Obtain a Preform with Controlled Variable Fiber Volume

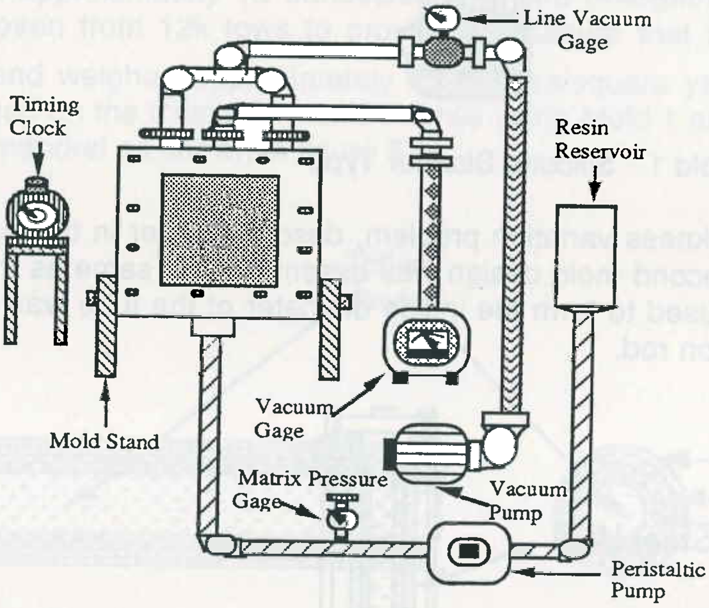

Figure 2, RTM Setup for Fabricating Panels Using Vacuum Assisted Process

EXPERIMENTAL PROCEDURE

RTM Mold Design

Based on the results of the panel experiments, a mold was designed with a 36 inch (91 cm) X 2 inch (5.08 cm) ID steel tube (no taper) to form the outside diameter of the composite tube with an end cap on each end. The inside diameter of the composite tube was formed with a silicone elastomer bladder. The bottom end cap had one hole for the introduction on the liquid matrix and the other end cap had two holes. One of the two holes was for excess matrix to escape and the other hole was to introduce air to inflate the silicone bladder. The matrix vent hole also provided an opening to pull a vacuum on the preform. Silicone elastomer gaskets were used between the ends of the steel tube and the two end caps to: (1)

prevent the liquid matrix from leaking out of the mold and (2) to prevent the vacuum from leaking.

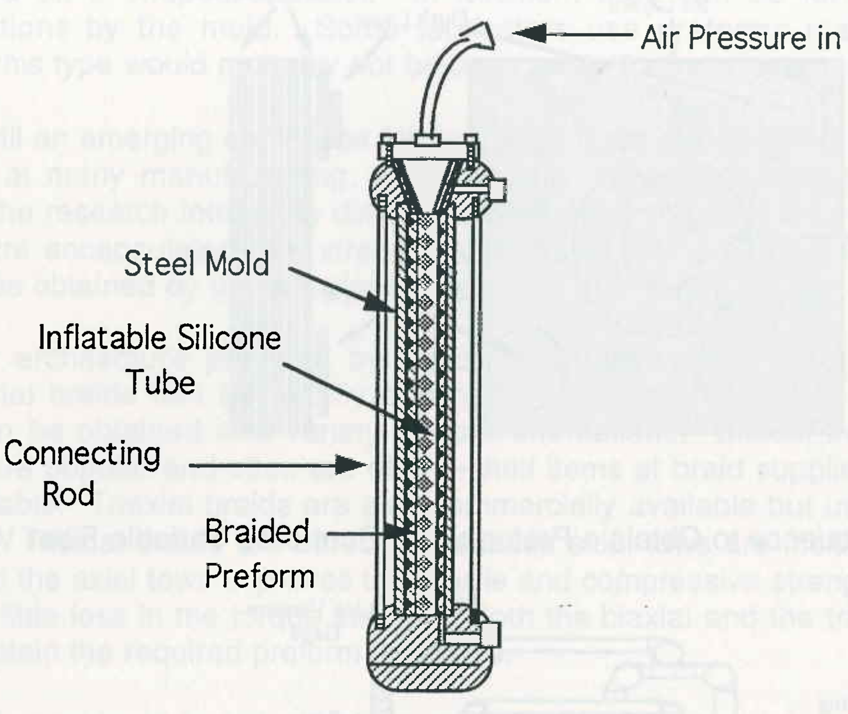

Figure 3, Sketch of Mold 1 - Silicone Bladder Type

Because of a wall thickness variation problem, described later in this paper, a second mold was designed. The second mold design was essentially the same as the initial mold except the inflatable bladder used to form the inside diameter of the tube was replaced with a 1.75 inch (4.45 cm) OD Teflon rod.

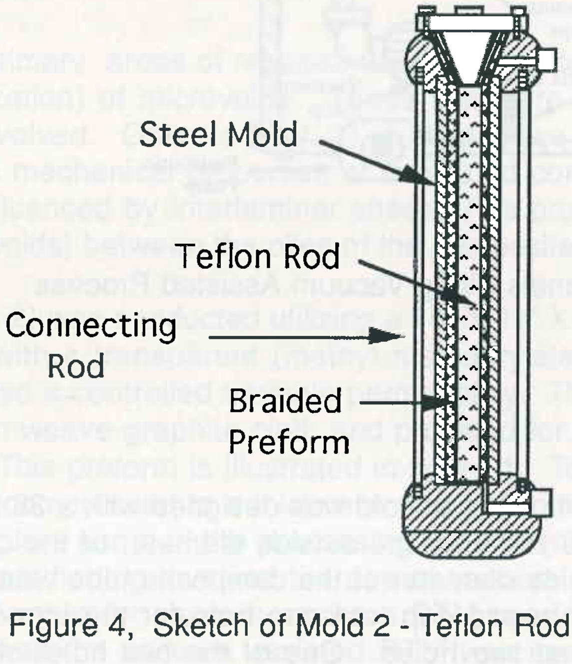

Figure 4, Sketch of Mold 2 - Teflon Rod Type

Matrix

The matrix system was Dow Chemical's Tactix 123 epoxy resin and the catalyst was Pacific

Anchor's Ancamine 1770 Curing Agent. The ratio of of these two ingredients was 100 parts by weight resin to 17 parts by weight catalyst. This particular matrix system was primarily selected for this research because the viscosity (about 300 cps.) was suitable for injection at room temperature. For a 500 gram batch size, the expected potlife at room temperature was 70 to 80 minutes. This much potlife time was required to mix, evacuate, transfer and cleanup. This matrix is widely used in the industry for RTM processing and has mechanical properties similar to general purpose prepreg matrix. Another desirable property included a cure cycle that permitted the use of a transparent mold plate in the flat plate mold which was essential for studying mold filling.

Preform

Two groups of composite tubes were fabricated for this research. The initial group (called Group 1) of tubes contained five plies of biaxial braid. The braid plies were woven at ±45 degrees with fiber tows manufactured by Hercules and designated as MAGNAMITE Graphite fiber, Type AS4. The particular braids used for the Group 1 tubes were Atkins & Pearce's Gammasox GM2.00 and GH2.00. GM2.00 braid (M=medium and 2.00=2.00 inch (5.08 cm) OD) was woven from 6k tows to provide a structure that was about 0.046 inches (0.117 cm) thick and weighed approximately 13 ounces/square yard (440gm/m^2). The GH2.00 braid (H=heavy) was woven from 12k tows to provide a structure that was about 0.074 inches (0.188 cm) thick and weighed approximately 20 ounces/square yard (677 gm/m^2). These braids were arranged on the inflatable mandrel while using Mold 1 and later on Mold 2 which utilized the Teflon mandrel as shown in figure 5.

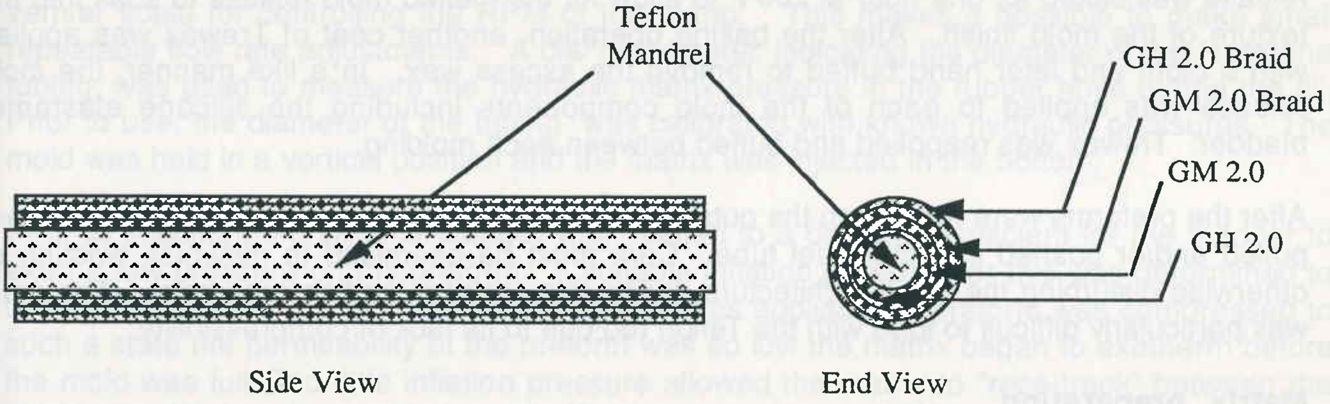

Figure 5, Illustration of Preform for Group 1 Tubes with Mold 2 Mandrel

The next group (called Group 2) of composite tubes contained four plies of braid. The inner and outer plies were triaxial braids, also supplied by Atkins & Pearce and the two inside was the same braid, GM 2.00, about 0.074 inches (0.188 cm) thick and weighed about 24 ounces/square yard (972 gm/ m^2). The triaxial braid was also woven at ± 45, as before with the biaxial braids, but had axial tows incorporated within the ± 45 architecture. The preform ply construction for Group 2 tubes is illustrated in figure 6. As a result of adding the triaxial braids to the preform, the molded composite tube contained 14% axial fibers.

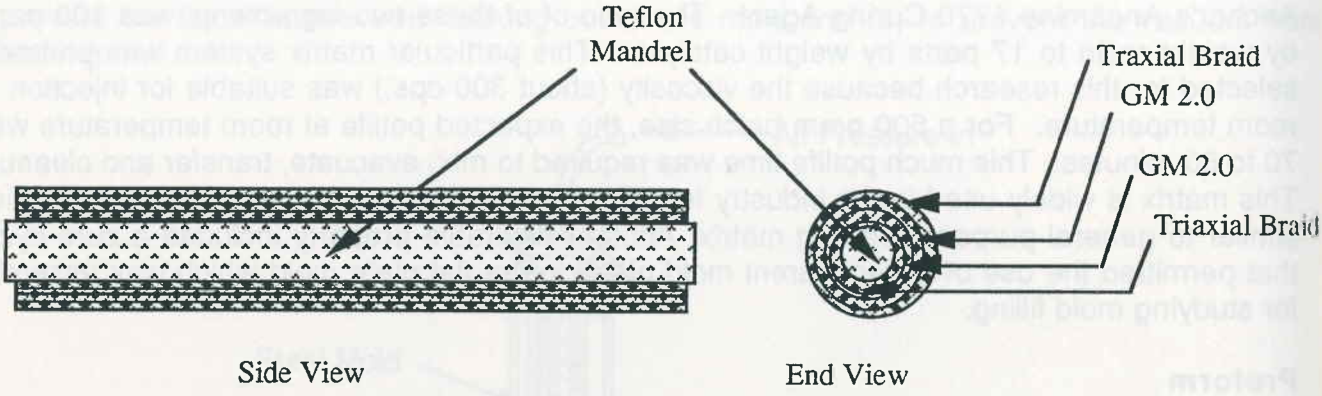

Figure 6, Illustration of Preform Ply Arrangement for Group 2 Tubes on Mold 2 Mandrel

To obtain the proper preform thickness, one ply was slipped over the mold mandrel (silicone bladder for Mold 1 and the Teflon mandrel for Mold 2) and then the additional plies were added on top of the first ply. The diameter of biaxial braids expanded under compression which made it easy to add plies on top of each other which was required to obtain a preform of suitable thickness.

Mold Preparation

The inside surface of the steel tube was sanded and polished to improve the mold finish. A good finish was felt to be essential for easy removal of the composite tubes from the mold. After polishing the steel tube, mold release (Trewax) was applied to the tube surface. Trewax is a commercial floor wax compound containing carnauba wax. After application, the mold release was baked for one hour at 200 F to allow for the melted mold release to soak into the texture of the mold finish. After the baking operation, another coat of Trewax was applied with a cloth and later hand buffed to remove the excess wax. In a like manner, the mold release was applied to each of the mold components including the silicone elastomer bladder. Trewax was reapplied and buffed between each molding.

After the preforms were nested on the outside of the mandrel, the mandrel and preform were pulled and/or pushed into the steel tube. Care must be exercised to minimize twisting or otherwise disturbing the braid architecture during this preform loading procedure. The mold was particularly difficult to load with the Teflon rod due to its lack of compressibility.

Matrix preparation

After weighing out the two ingredients and mixing, the liquid matrix was degassed in a vacuum chamber for 5 minutes at 700 millitorr to eliminate the air incorporated by the pouring and mixing operation. Injecting void free matrix is essential for producing composites fabricated by the RTM process with low microvoid content. About 500 grams of matrix was required for molding each tube.

Mold Filling

Mold 1 Filling Process

A sketch of the laboratory set-up to fill the tube Mold 1 is shown below

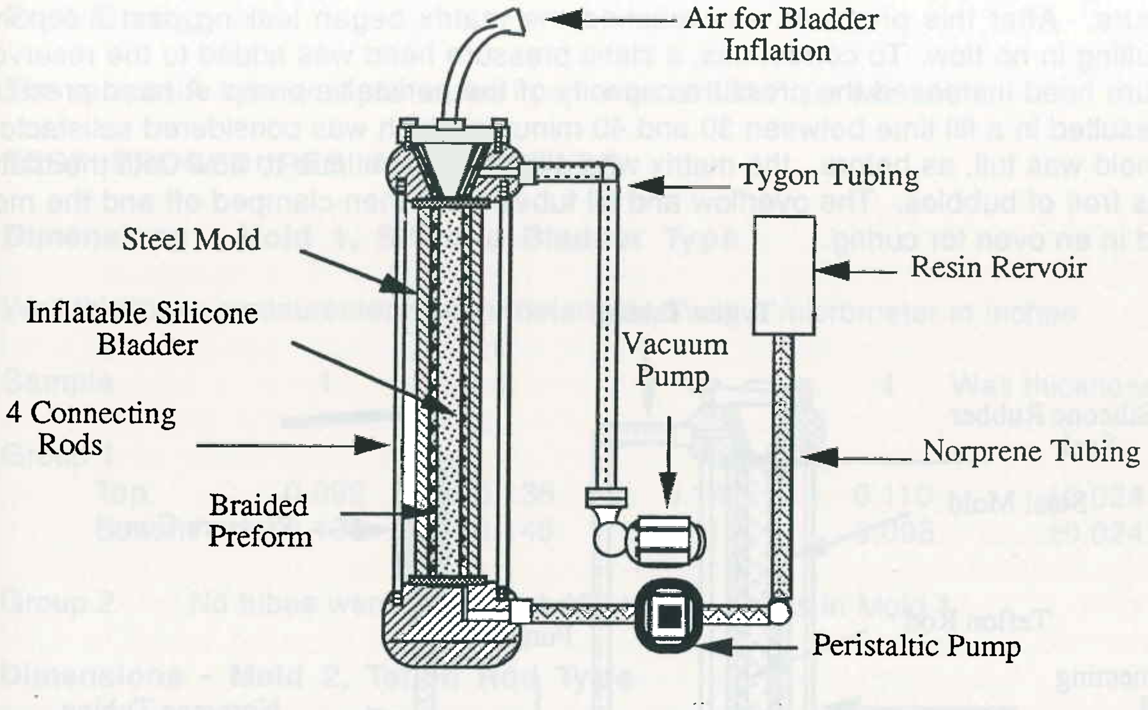

Figure 7, Laboratory Setup for Filling Mold 1 Containing the Silicone Elastomer Bladder.

The evacuated matrix was carefully poured into the resin supply tube. Norprene tubing with an outside diameter of 7/16 inches and 3/32 inches wall thickness was used to connect the bottom of the supply tube and the bottom of the mold. This tubing also passes through a peristaltic pump, model 7520-35, purchased from Cole Palmer. This particular pump has a vernier scale for controlling the RPM of the pump. This makes it possible to make small repeatable flow rate adjustments. A dial micrometer, indicating the diameter of the Norprene tubing, was used to measure the hydraulic matrix pressure in the rubber hose during the fill. Prior to use, the diameter of the tubing was calibrated with known hydraulic pressures. The mold was held in a vertical position and the matrix was injected in the bottom.

Prior to injection, the silicone bladder was inflated to a diameter sufficient for the bladder to exert a low pressure on the preform. A suitable inflation pressure for this was determined to be about 25 psi. If too much bladder pressure was applied, the preform was compressed to such a state the permeability of the preform was so low the matrix began to exotherm before the mold was full. Too little inflation pressure allowed the matrix to "race-track" between the bladder and the preform which resulted in a composite tube with excessive porosity. When the inflation pressure was about right, the mold filled in 30 to 40 minutes.

After the mold was full, the matrix was allowed to continue to flow until the matrix stream was relatively free of bubbles. Generally, the matrix flow was clear after a few minutes. The mold set-up was placed in a Blue M oven, the injection and the overflow hoses were clamped off and the bladder pressure was increased to 80 psi and maintained until the matrix had solidified..

Mold 2 Filling Process

The filling procedure for Mold 2 was essentially the same as for Mold 1 except the mandrel did not require inflation. The first filling attempt resulted in a partial mold fill before the exotherm of the matrix stopped the flow. The peristaltic pump was limited to about 25 psi fluid pressure. After this pressure was reached the matrix began leaking past the pump rollers resulting in no flow. To correct this, a static pressure head was added to the reservoir. The pressure head increased the pressure capacity of the peristaltic pump. A head pressure of 40 psi resulted in a fill time between 30 and 40 minutes which was considered satisfactory. After the mold was full, as before, the matrix was allowed to continue to flow until the matrix stream was free of bubbles. The overflow and fill tubes were then clamped off and the mold was placed in an oven for curing.

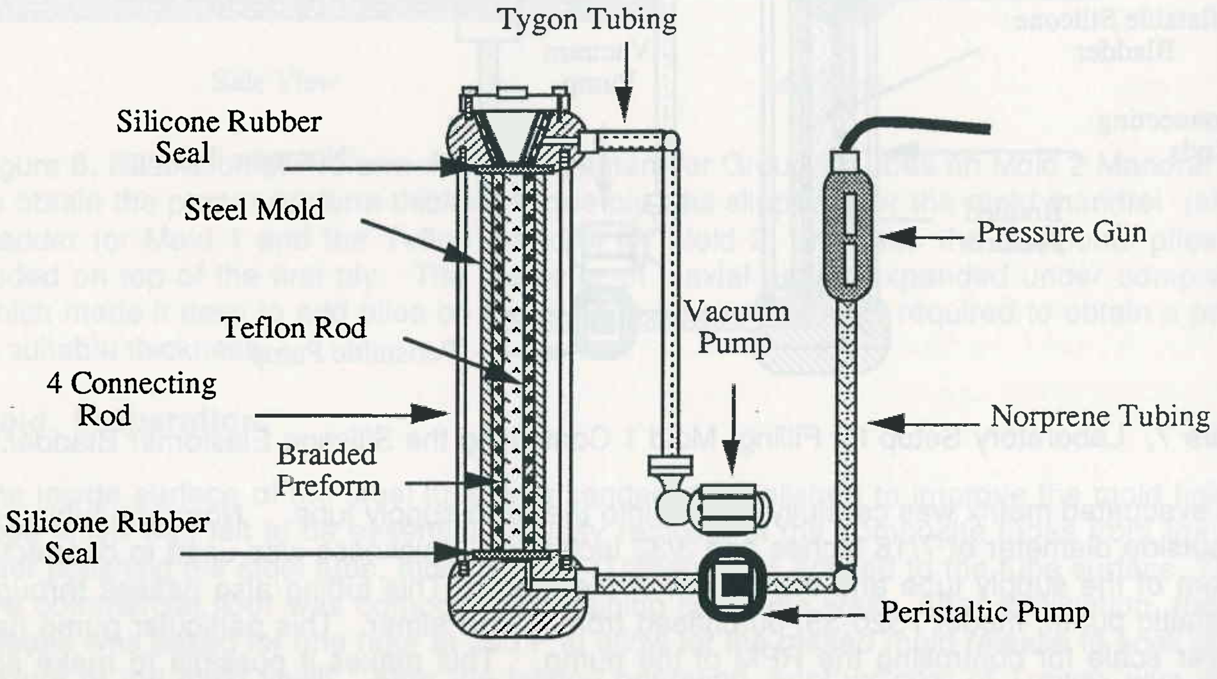

Figure 8, Setup for Filling Mold 2 (Teflon Rod)

Initial Curing

The initial cure temperature was at 175 F and held for two hours.

Tube Extraction From Mold

The initial efforts to remove the composite tube from the inside of the steel mold tube were a failure. The first composite tube was finally removed from the mold with an application of acetone and dry ice to the inside of the composite tube. The extreme cold of this mixture provided enough thermal coefficient shrinkage to provide sufficient clearance for the composite tube to be pushed out of the steel tube. An inspection of the “stuck” composite tube revealed it contained gross surface porosity. It was theorized that the surface skin of the bubbles had broken off and acted as micro wedges to lock the tube into the mold. Future tubes came out relatively easily if they were free of surface bubbles.

The final composite tube removal process consisted of placing an aluminum plug in the end of the mold and tapping the plug with a plastic faced hammer. The secret of easy removal was a very close fitting aluminum plug which minimized “cocking” caused by misalignment. Even with this procedure, every molding with porosity was difficult to remove.

Post Curing

The composite tube was post cured in an oven at 350 F for two hours.

TEST PROCEDURES AND RESULTS

Dimensions - Mold 1, Silicone Bladder Type

Wall thickness measurements were determined with a micrometer in inches.

Sample	1	2	3	4	Wall thickness variation
Group 1					
Top	0.092	0.135	0.140	0.110	±0.024
Bottom	0.130	0.146	0.117	0.098	±0.024
Group 2	No tubes were fabricated with triaxial braids in Mold 1				

Dimensions - Mold 2, Teflon Rod Type

Wall thickness measurements were made with a micrometer in inches.

Sample	1	2	3	4	Wall thickness variation
Group 1					
Top	0.124	0.126	0.124	0.125	±0.001
Bottom	0.125	0.124	0.124	0.123	±0.001
Group 2					
Top	0.126	0.124	0.126	0.123	±0.0015
Bottom	0.124	0.125	0.124	0.126	±0.001

Specific Gravity - Mold 2, Teflon Rod Type

The specific gravity of the composite tubes was determined by the "water displacement" method as described in ASTM D792.

Sample	1	2	3	4	Average
Group 1	1.38	1.38	1.37	1.39	1.38
Group 2	1.38	1.38	1.37	1.37	1.375

Fiber Volume - Mold 2, Teflon Rod Type

The fiber volume percentage was determined by the nitric acid digestion method described in ASTM D3171.

Sample	1	2	3	4	Average
Group 1	39.6	38.8	39.5	38.1	39.0

Group 2	40.1	40.5	39.2	40.1	40.0

Ultrasonic Scanning of Tubes for Defects

The tubes were ultrasonically scanned using an Ultrasonic C-scan. A fixture was designed and fabricated for use in a water tank to enable the step rotation of the tube during the scanning process. The fixture, illustrated in figure 9, was designed for indexing the x-axis of the tube with a stepper motor. The fixture consist of a plastic table with four legs with leveling screws, two plastic bearing blocks secured to the table with plastic screws. The tubular specimen is placed on a stainless steel rod and is centered and secured at both ends by a set of tapered plastic bushings. These bushings make a snug fit with the steel rod and are secured on the rod by means of Allen screws, with their tapered ends sliding into the tube. The tapered ends of the bushings as as a self-centering device and allow a concentric rotation of the tubular specimen about the axis of the steel rod. The steel rod with the specimen was fixed into the plastic bearing blocks of the plastic table. One end of the rod is fixed with a set of stainless steel collars to restrict the lengthwise movement during the scanning process, while the opposite end is connected to a sprocket which is in turn connected to the X-axis stepper motor by means of a plastic chain. The whole unit with the exception of the stepper motor is submerged in the water tank. Settings and gating method was kept the same as described in reference 2. Figure 9 below shows the set-up used.

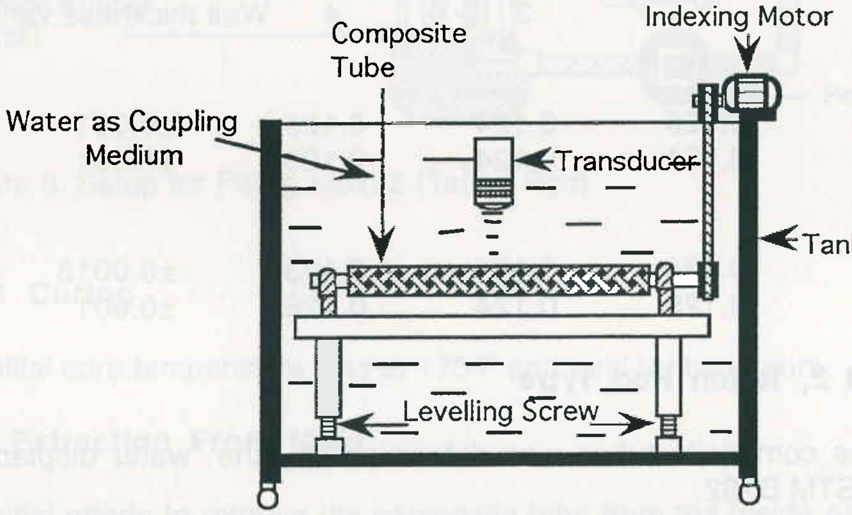

Figure 9, Setup for C-Scanning Composite Tubes

Table 1, Void Content of Tubes Fabricated from Braided Biaxial Preforms (Group 1) Tubes by C-Scan

Tube Number	Location of Specimen	Void Content %
1	Top	0.00
	Bottom	0.01
2	Top	0.09
	Bottom	0.01
3	Top	0.01
	Bottom	0.00
4	Top	0.01
	Bottom	0.00

5	Top	0.01
	Bottom	0.00

Table 2, Void Content of Tubes Fabricated from Braided Triaxial Preforms (Group 2) by C-Scan

Tube Number	Location of Specimen	Void Content %
1	Top	0.00
	Bottom	0.00
2	Top	0.02
	Bottom	0.01
3	Top	0.00
	Bottom	0.00
4	Top	0.01
	Bottom	0.01
5	Top	0.02
	Bottom	0.01

DISCUSSION

Processing

Many experiments were performed with Mold 1(silicone bladder type). The mold filled satisfactorily but the wall thickness variation problem was never solved. If the bladder inflation pressure was increased to a pressure sufficient to thoroughly consolidate the preform, the mold couldn't be filled within the time limit established by the matrix potlife. When the inflation pressure was reduced the composite wall thickness varied to an unacceptable degree. When the mold was vented at the ends during cure to allow the matrix to bleed, the wall thickness was thinner at the ends than at the middle.

In the interest of development time, the bladder type mold was abandoned in favor of Mold 2 which utilized a Teflon rod as the center core rather than the inflatable bladder. The first efforts to fabricate a composite tube with this mold were unsatisfactory because the mold failed to fill within the allotted potlife because of the high fiber volume (low permeability) of the compressed preform The matrix injection system was then modified to add a compressed air static head to the reservoir. This was accomplished by using a Semco Model 550 (20 ounce) Sealant Gun. It was found that 40 psi air pressure provided a reasonable fill time. No attempt was made to try this static pressure arrangement on the inflatable bladder type mold. The tubes obtained from Mold 2 with the added pressure arrangement fully met our expectations.

Tube Quality

The tube wall thickness variation obtained with Mold 1 (inflatable bladder type) were so large (±0.024 inches) no other quality features were determined. However, except for the wall thickness defect, the tubes did meet our visual expectations.

The tube wall thickness variations obtained with Mold 2 were small (±0.001 inches) and considered to be acceptable. The Teflon rod was allowed to "float" in the mold because of the interference fit of the compressible tubular preform. The fiber volume determinations performed on Group 1 parts were lower than expected. The target fiber volume for the Group 1 tubes was 45%. The thickness of preform used seemed to be the most that could be slipped into the steel tube mold. If preform plies of lesser thickness had been available, perhaps an additional ply could have been squeezed into the mold. This will be attempted in future work.

Group 2 tubes still did not contain the desired 45% fiber volume. However, a slightly slower fill time was an indication the fiber volume was higher than the Group 1 tubes.

The Group 1 and 2 tubes were both scanned for uniformity. Very little nonuniformity was found. There seemed to be a relative absence of voids, dry areas or delaminations as illustrated in tables 1 and 2. Where there was a difference between the top and bottom of a tube, the bottom (where the matrix enters the mold) has a lower nonuniformity.

CONCLUSIONS

This research indicates the following conclusions can be made:

- Composite tubes 36 inches long, 2 inches in diameter with a 1/8 inch wall thickness can be fabricated in a tubular steel mold with no draft utilizing braided preforms.
- RTM is a viable process for fabricating composite tubes.
- A Teflon rod makes a good core for a tubular mold.
- Silicone elastomer inflatable bladder presents dimensional problems.
- A static pressure head assist extends the pumping pressure range for a peristaltic pump.
- A fixture can be built to provide a technique for C-scanning tubular composites.

Acknowledgements

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