MOLD FILLING PARAMETERS IN RESIN TRANSFER MOLDING OF COMPOSITES

by

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ABSTRACT

This thesis describes the development of resin transfer molding (RTM) for composite materials, the study of various molding parameters in the process, and their effects on part quality. The resin transfer process involves the flow of catalyzed resin into a closed mold filled with fiber reinforcement to make a composite product. The RTM process is a relatively recent development in composites processing, but is expanding into areas as diverse as aerospace and automotive. Advantages of the process are low volatiles released to the atmosphere, lower tooling costs than some competitive processes, and good part quality.

The main focus of this study was to set up a working RTM process and use it for two purposes: (1) to examine the basic aspects of wetting, flow patterns, pore formation, and the effects of mold deflection, and (2) to manufacture specimens for both educational and research purposes. The fiber and resin materials are representative of those used in industry. The equipment, although smaller in scale, utilizes the same principles as in commercial-scale processes.

The results of this study show the relationship between porosity and flow rate; the importance of capillary action to the wetting process; the significance of mold deflection on part thickness and reinforcement permeability; and the flow pattern as the resin actually fills the mold. It can be concluded that the process works well and produces very good quality parts; however, the mold filling process is quite complex. It is determined that small variations in any of the processing parameters can influence the quality of the finished part.

CHAPTER ONE

INTRODUCTION

Demand for improved part performance has led to efforts to produce products that are lighter, stronger, and more efficient. This is particularly evident in the automotive and aerospace industries where increased fuel costs have forced manufacturers to increase fuel efficiency without increasing product cost. The area of sporting goods has also seen an increase in the demand for improved performance. This has caused an increase in the use of non-traditional materials of construction such as polymer matrix composites.

Polymer matrix composites are made by impregnating very strong fibers with a liquid polymer and allowing it to solidify. The fibers provide strength and stiffness to the structure while the polymer, or matrix, serves to transfer the load between the fibers, protect them, and keep them oriented in the proper direction so as to maximize the composite properties. These components can combine to give a material with a very high strength and stiffness to weight ratio for aerospace applications. In the automotive industry they are used to provide near net shape products, with little machining or waste, that can replace assemblies of metal parts.

Composites are not a new class of materials, but recent advancements have dramatically improved them and given greater range to their properties. Improvements in the matrix chemistry have allowed composites to move into harsher environments. For instance, some polyimides can be used up to temperatures of around 500-600 °F [1]. Changes in reinforcement types and configuration have yielded improved strength and processing characteristics. Most reinforcements are available in woven fabrics, mats, directional fabrics, and braided structures which allow them to be used with different processes. These improvements in the components in conjunction with lower costs and improved processing have allowed them to penetrate a number of different markets. Sporting goods, tanks and pressure vessels, automobiles, airplanes, and consumer goods are all examples of products that make use of polymer matrix composites. The desire to incorporate composites into these various products has led to the development of a number of manufacturing techniques.

CHAPTER TWO

PROCESSING METHODS

The information contained in the following discussion on processing is summarized from information contained in References 2-4. The main purpose of any composites processing method is to bring the resin and the reinforcement together in the correct shape and in such a way so that little porosity remains in the fiber assembly. This is known as wet-out. It is desirable to accomplish wet-out and maintain performance requirements while still achieving the desired rate of production. The degree of wet-out is subject to the processing parameters of the method employed. Such factors as fiber volume fraction, resin viscosity and kinetics, and product geometry all affect the outcome of the finished part, no matter which processing method is used. By varying one of the processing parameters it is possible to affect one or more of the other parameters. It is only by knowing how these factors relate to one another for a given process that it is possible to successfully produce high quality parts.

The strength and stiffness characteristics in a composite come primarily from the fibers, making a high fiber volume fraction (V_f) very desirable. However, as the fiber volume fraction increases, the porosity of the fiber assembly prior to wet-out decreases, and the ability of the resin to infiltrate the fiber bundles and the spaces between them

decreases. This can result in air being trapped and forming pores or in an uneven distribution of resin throughout the part, both of which can affect performance. Proper selection of processing parameters can maximize fiber content for each processing method.

The viscosity and the cure kinetics are critical for thermoset resins which are crosslinked (cured) after wet-out and shaping of the part. The lower the viscosity, the easier it is for the resin to flow and saturate the fiber assembly. The cure kinetics are important in that the viscosity increases as curing occurs. Kinetics also affect the efficiency of the process. If cure takes too long, then it takes longer to produce each part. Many resins have been developed specifically for each particular process, not only for their good processing traits, but for desirable physical properties as well. Heat is often used to lower the viscosity. However, there is a trade-off: increasing the temperature also increases the cure rate, which can increase the viscosity.

The part geometry also influences the permeability of the fiber assembly. Each processing technique has an element of matrix flow involved. As the geometry becomes more complicated, it becomes more difficult to force the resin either into or out of certain domains. Ribs and design features with varying thickness can hinder the movement of resin through the part. The geometry of the part can often dictate the best process. The presence of ribs or other

uneven surfaces, a constant cross-section, or a hollow center all suggest the use of one process over another.

Although there are variations within each, there are six primary methods used to produce thermoset matrix composites: compression molding, filament winding, hand lay-up, autoclave or bag molding, pultrusion, and resin transfer molding (RTM). Each method has carved out a niche based on the above parameters as well as the desired production rate, and the necessary quality. Each process has strengths and weaknesses which make them suitable for particular applications. Injection molding, another composites processing method, is used mainly with thermoplastic matrices and will not be discussed here.

Compression Molding

A material called sheet molding compound (SMC) is often used in compression molding. SMC is made by sandwiching fibers between two layers of catalyzed resin to form a continuous sheet. The flow of resin into the reinforcement is over a short distance and is aided by compaction rollers. The sheet is rolled up between release films after the matrix thickens. This can be cut into sections and stacked to form a charge. A second element of flow occurs when the charge is placed into a two sided, heated mold; as the mold is closed in a press, the charge is forced to fill the mold. The two-sided

mold gives a good finish and allows for varying thickness and the presence of ribs and other variations on both sides.

Increasing the amount of fibers, and thus the fiber volume fraction, decreases the ability to flow. Mold closing speed, temperature, pressure, and the area of the mold base that the charge occupies must be adjusted to insure that the mold fills. It is important to close the mold at a rate that is low enough to allow the material to flow easily. Changing the area that the charge occupies changes the distance that the material must flow. Generally, higher pressures must be used at higher fiber volume fractions and for more complex shapes.

Filament Winding

Filament winding uses a rotating mold called a mandrel to wind up resin impregnated rovings. The process begins by pulling a number of rovings through a resin bath, again utilizing a short wet-out distance. They then are pulled over a roller which helps force the resin into the fiber bundles in the rovings and helps remove the excess resin and porosity. The rovings are then collected together on the carriage, which moves the length of the mandrel. The speed at which the carriage travels, for a given rate of mandrel rotation, determines the angle that the rovings are wound onto the mandrel, giving the desired fiber orientation for a particular layer.

Filament winding uses a resin bath to bring the resin and reinforcement together. After the reinforcement leaves the bath a wiping device is used to control the amount of resin that remains, the amount of resin is also affected by the tension in the strand. The tension can also play a role in the finished piece; if it is too high the resin can be forced out of the first layers on the mandrel as subsequent layers are added, which gives an uneven resin distribution; if it is too low then the fiber content will be low as well.

It is important that the resin not have too high or too low a viscosity. Too low a viscosity will allow the resin to be spun off of the part as it undergoes the winding process. Too high a viscosity will prevent good wetting in the bath, and requires increased residence time so that a slower process results. The resin needs to have a pot life of several hours in order to keep the bath from gelling prior to completion of a large winding.

Filament winding lends itself well to bodies of rotation requiring hollow centers such as tanks and pipes. The structure need not have a circular cross-section, but it is not possible to directly wind shapes with concave surfaces. It is possible to obtain these shapes with an additional molding operation.

Hand Lay-up

Hand lay-up is the least equipment intensive and most labor intensive of the processes. Typically it begins with a one sided mold. The reinforcement is placed in the mold in

the proper orientation. Resin is then applied to the reinforcement and a hand roller or squeegee type device is used to distribute the resin and help force it into the fiber bundles.

The processor can control fiber content in hand lay-up by controlling how much resin is applied to the reinforcement as each layer is added. The amount of resin that remains is then determined by the pressure applied by the spreading device. However, as the layers become thick it becomes difficult to force the resin into them. This can result in an uneven resin distribution.

The fact that there is only one mold face makes it difficult to obtain a high V_f, as the laminate cannot be compressed. The single mold face also limits the possible geometries that can be produced. The viscosity is tied to the shape of the part to some extent. If there are steep sides care must be taken to insure that the resin has a high enough viscosity to keep it from running out of the reinforcement. If the resin is initially applied evenly, the wet-out distances are on the order of the layer thickness. However, if the resin becomes too thick then wetting problems can occur.

Prepreg Molding

In prepreg molding, layers of prepreg tape (unidirectional fibers or woven fabric impregnated with resin which is B-staged or partially cured) are stacked so that they

have the proper orientation. Wet-out has already occurred during the manufacture of the tape. The laminate is then surrounded by bleeder material, and release material is applied to the tool to prevent sticking. This assembly is then placed into a bag. The bag is placed into an autoclave or press which provides pressure and heat, usually a vacuum is used to remove the air from the bag. The combination of pressure and heat, specified by the manufacturer, causes excess resin in the prepreg to flow into the bleeder material. The amount of bleeder material determines how much resin is removed once it begins to flow.

In prepreg molding the fiber volume fraction is controlled by how much resin is in the prepreg, and how much is removed in the autoclave. Prepreg contains more resin than is usually desired. The removal of the excess not only affects the fiber volume fraction, but aids in the removal of air and volatiles from the part. This is accomplished by increasing the processing pressure and the temperature in such a way that pressure is applied at the point when the resin is least viscous. This causes the resin to flow, carrying any entrapped air with it, into the bleeder material. As with SMC, the viscosity decreases with the increase in temperature, then increases as the reaction proceeds. The resin in thicker parts cannot move as readily and care must be taken to ensure that gelation doesn't occur on the surface before the resin in the center of the piece begins to flow. Prepreg materials

usually have high V_f and excellent control of fiber orientation, but a low production rate. It is generally used in the aerospace and sporting goods industries.

Pultrusion

Pultrusion, like filament winding, uses a resin bath to bring the resin and reinforcement together. The reinforcement, often mat or fabric, is pulled through a vat which contains resin. After leaving the bath it is often pulled through a preformer which gives the general shape of the desired part. It is then pulled through a die which finishes forming. Curing is initiated and completed by heaters. As in the other processes the wet-out distance is short.

High fiber content is obtained by first insuring good wet-out in the resin bath. This is controlled by the resin viscosity and the residence time in the bath. After the fibers leave the bath, the preformers distribute and compact the reinforcement, help force the resin into it, and remove the excess resin. It is this final step, along with the pulling force, that determines the amount of resin in the finished part.

Geometries are long strips, generally have a constant cross-section and are usually solid, although it is possible through the use of some tricks with the die to obtain varying

thicknesses and cross-sections. The profiles generally produced are those that are constant along the length.

The viscosity needs to be in a proper range as in the other processes. If it is too low it will drain from the reinforcement prior to entering the die. Too high and the resin won't properly wet-out the fibers unless the residence time in the bath is increased. The pot life of the resin needs to be long, but it must cure quickly in the die at elevated temperatures.

Resin Transfer Molding

Resin transfer molding is not a new process. It has been used in one form or another since the early 1940's [4]. However, its use was limited until the 1970's because of the lack of suitable resins and equipment. In the 1980's fiber preforms and low viscosity resins were developed that allowed the production of more complex geometries and parts for more diverse applications [4]. This, combined with low capital investment and release of volatiles, has dramatically improved the popularity of RTM.

The RTM process begins by placing reinforcement, in the form of properly oriented mats or fabrics, into a two-sided mold cavity. The mold is then closed and the resin is injected until the fibers are saturated and the mold is full. The resin is allowed to cure and the finished part is then removed from the mold and the process repeated. RTM employs four components: the mold, the reinforcement preform, the resin pump, and the resin (Figure 1).

The fact that this process uses a closed mold offers several advantages. First, complex shapes can be produced. Any variations in the geometry, such as ribs and areas of varying thickness, can be molded directly no matter where they are in the part. Second, the closed mold produces a smooth finish on both sides of the part. Third, emission of volatiles, such as styrene in polyester, is greatly reduced during processing. Styrene is a suspected carcinogen [3], exposure is regulated by OSHA and has been reduced to 50 ppm[5]. Finally, production rates can be high enough for automotive parts. These factors make RTM very attractive from both a production and economic standpoint. Disadvantages are

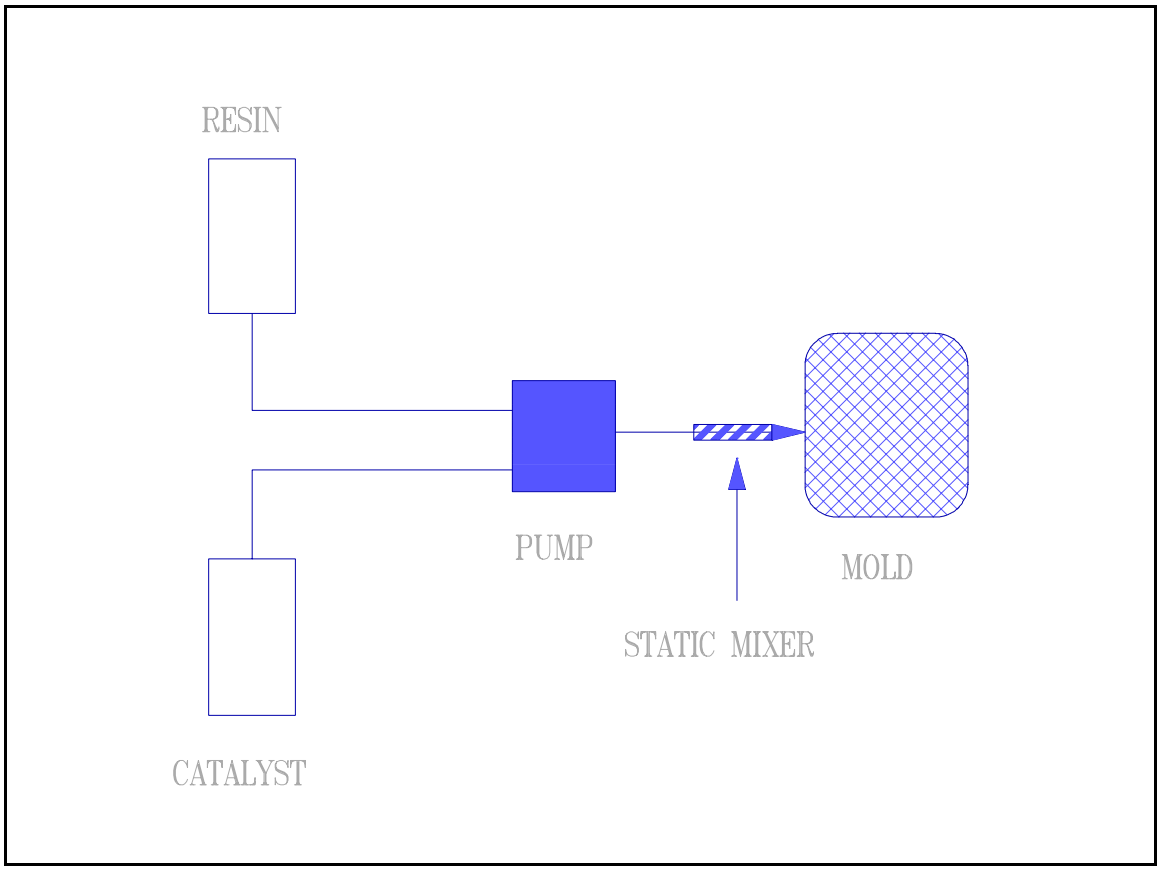

Figure 1. Schematic of the resin transfer molding process.

that wet-out distances are long, requiring lower fiber volume and the use of low viscosity resins which may have less desirable mechanical properties. These limitations are being overcome by continued advancements in equipment and resin chemistry.

The Mold

The process requires a two-sided mold in the shape of the part. The fact that RTM is a low pressure process, typically less than 100 psi, allows molds to be constructed of materials other than tool steel, often composites and aluminum; the molds are often heated to lower the viscosity and increase the cure rate. The use of these alternative mold materials allows lower tooling costs compared to compression and injection molding, and allows manufacturers to have their tooling made in-house.

Molds are the most critical aspect of the process. As the shape of the part becomes more complex the position of the resin inlets and the outlets can determine whether the mold will fill correctly. Experience has shown that injecting the resin into regions with higher fiber content aids the wet-out. Placing vents in areas where air is likely to become trapped can eliminate dry spots.

The Reinforcement

The second component of the RTM process is the reinforcement. There are many types of fibers available, such as E-glass, C-glass, S-glass, carbon, and aramids. These

come in a variety of styles, such as woven roving, chopped strand mats, continuous strand mats, unidirectional rovings, and woven fabrics. These reinforcements can be layered and combined in such a way that the strength properties of the different fibers and configurations are best utilized.

Fiber contents of 5-55 wt% are not uncommon [6]. At the higher values of V_f the location and the number of the inlets and the outlets become very important due to the difficulty of forcing the resin through the preform. It is also beneficial to have a low viscosity resin to help keep the pressures down and to assist wetting.

At a production level, reinforcements are typically made into preforms. A preform is merely reinforcement in which the fibers have been properly oriented, formed, and held in the final shape with a binder. One technique to make a preform is to stitch together layers of fabric or mat. Another technique, used for non-structural parts, blows a combination of chopped fibers and binder onto a screen in the shape of the part. When the binder hardens the fibers are held together in that shape. The use of preforms greatly facilitates the handling of the reinforcement and its placement in the mold, which in turn speeds up production.

The Pump

Most commercial RTM injection equipment centers around a positive displacement pump. There are usually two tanks, one for the resin and one for the catalyst. Metering capabilities are built into the equipment to correctly proportion the two

components. The components are then brought together and mixed in a static mixer located just upstream of the mold inlet. In some cases the holding tanks can be heated in order to lower the viscosity. Solvent tanks are usually included to rinse the catalyzed resin out of the lines between shots.

The Resin

Once the reinforcement is in place the mold is closed and the resin is injected into the mold cavity. RTM requires the use of a low viscosity resin. This assists in wetting out the fiber strands and in flow of the resin through the assembly. RTM relies heavily on capillary forces to get the resin into the fiber bundles. The lower viscosity also permits the use of lower injection pressures and higher injection rates, which in turn allows for the use of smaller pumps and lighter tooling. Resin viscosities range from 100 cP for some polyesters up to 2500 cP for some epoxies.

When the mold is full it is sealed and the resin is allowed to cure. Care must be taken to insure that the resin kinetics match the part being produced. If the cure rate is too fast then the mold will not be full prior to gelling and the part will be ruined. If the cure rate is too slow then the production rate decreases. After the resin is cured the part can be removed from the mold and the process can be repeated.

CHAPTER THREE

LITERATURE REVIEW

An understanding of how all of the processing parameters interact is necessary for accurate predictions of mold filling behavior in RTM. There have been efforts by researchers to model the RTM process and examine some of the factors that affect it. The goal is to ultimately assist in the design of molds and produce better quality parts. Presently, mold making is more of an art than a science and relies heavily on past experience and trial and error [7]. Prediction of flow fronts can lead to faster cycle times, reduce waste, and lead to more efficient placement of inlets and outlets. There is a delicate balance where the pressure drop, the flow pattern, and the resin properties are suitable for good wet-out and a quick cycle time. Too high of a pressure drop in the mold can cause the mold to leak or the reinforcement to be displaced. If the pressure drop is too low the mold may not completely fill [8]. The proper resin processing properties are equally important. If the cure cycle is too slow there is a loss of efficiency. If it is too fast the result can be incomplete mold filling. If the viscosity of the resin is too high then poor wet-out can result.

Much of the work in this area has been done empirically. Many researchers have built molds with which to compare

results of their models [7,9-25] and to observe the actual filling behavior. In some cases the molds are also used to determine the values of processing parameters for use in models, such as the processing pressures and permeability of the reinforcement.

Permeability

The importance of the permeability of the reinforcement has made this parameter the subject of much study [7-10,14,22,27]. The permeability of the reinforcement determines the resistance to resin flow and is a necessary component of all models. Permeability is usually measured in units called darcys, where one darcy is equal to 9.87×10^{-9} cm^2 [28]. This property affects resin wet-out of the fibers as well as the pressure necessary to force the resin through the mold.

The method used by Molnar et al. [8], Fraccia [14], Gauvin [15], Li and Gauvin [20], Martin and Son [21], and Trevino et al. [22] for measuring the permeability of a particular reinforcement was based on Darcy's Law. The reinforcement is placed in a mold and saturated with resin. After saturation, more resin is forced through the mold from one end to the other. Once steady state has been reached, the pressure drop across the length of reinforcement is measured. This value, with the dimensions of the mold cavity, the viscosity of the resin, and the volumetric flow rate can be

substituted into Darcy's Law, and a permeability can be calculated.

Darcy's Law is generally used in the 1-dimensional form of

$$Q = -\frac{K A}{\mu}\frac{\triangle P}{\triangle L} \qquad \textbf{(1)}$$

where Q is the volumetric flow rate, K is the permeability of the porous media, A is the area available for flow, μ is the viscosity of the fluid and △P/△L is the pressure drop per unit length of the medium. This form of the equation can be used in cases where the permeability is isotropic. However, because not all fabrics are isotropic, Darcy's Law is sometimes modified in order to account for anisotropy in the permeability. In the 2-dimensional case a permeability tensor is substituted into the equation and after some manipulation results in

$$\begin{bmatrix} u \\ v \end{bmatrix} = -\frac{1}{\mu}\begin{bmatrix} K_x & 0 \\ 0 & K_y \end{bmatrix}\begin{bmatrix} \frac{\partial P}{\partial x} \\ \frac{\partial P}{\partial y} \end{bmatrix} \qquad \textbf{(2)}$$

Adams et al. [9,10] used a different approach in their study. A square mold with a central injection site, which allowed for radial flow, was constructed. The porous media took the form of various woven fabrics. A hole was cut through the fabric to prevent compression of the fabric over the injection site, which could allow for uneven distribution

of resin. Once the injection was started, the movement of the flow front was timed. Models were developed that allowed the prediction of the permeability in both isotropic and nonisotropic fabrics. Results obtained from these experiments were in accordance with Darcy's Law.

Miller and Clark [27] developed an apparatus to determine the flow resistance of resin normal to the plane of a fabric. This device amounted to a cylinder in which a specimen of the fabric could be mounted. Liquid could be forced through the thickness of the fabric at different rates and the pressure monitored.

Some studies [8,22] have examined the effects of the stacking order of different reinforcement types on the overall permeability of a laminate. The permeability of random mat, bidirectional mat, and unidirectional mat were each determined separately. It was found that the unidirectional mats had a higher permeability in the fiber direction. However, the pressure drop was higher as well for these mats. This was attributed to the unidirectional mats having a lower permeability in the thickness direction because of their packing characteristics. The study also found that a combination of random and unidirectional mats made for a short transition to a stable, steady state flow pattern. This was due to the unidirectional mat allowing the resin to move in the thickness direction and into the random mat which kept the front smooth. Adams and Rebenfeld [9] also found that the addition of a layer with high in-plane permeability aided the

movement of the resin in the thickness direction. This allowed the flow front to remain uniform through the entire thickness.

There has been some disagreement as to whether the fluid behavior is actually described with Darcy's Law in RTM. This has stemmed from the fact that Darcy's Law is based on a saturated, isotropic porous medium. The fluid is assumed to be Newtonian, have a particle Reynolds number less than 1, and not undergo any chemical or physical changes [28]. Because the RTM process has both a saturated and unsaturated region where flow is taking place, involves a chemical reaction, and may use non-Newtonian fluids, some researchers have shown that permeabilities obtained experimentally deviate from Darcy's Law predictions [8,15,21,22]. This has led to the suggestion that there is a transition that takes place where the permeability changes with advancement of the flow front and saturation [29].

On the other hand, several studies have shown that permeabilities based on Darcy's Law in fact are consistent in both saturated and unsaturated porous media [9,10,14].

It should be noted that there are no clear sources of error in these studies. Some of the confusion is due to the lack of detail reported in most of these experiments. Fraccia [14] and Martin and Son [21] both mention that deflection of the mold faces is either a minimal factor or is somehow known not to be a factor. Martin and Son found a deviation from Darcy's Law while Fraccia found there to be agreement.

Furthermore, with the exception of the studies done by Adams et al. [9,10] and Gauvin et al. [15] all of these studies used fluids known to be Newtonian instead of resins. Adams et al. used epoxy with a viscosity of 94.4 poise and stated that the behavior was Newtonian. Good agreement was found between plots of experimental data and predictions from a Darcian-based model. Gauvin used a polyester with unknown viscosity which was assumed Newtonian. Plots of pressure drop *versus* flow rate showed that permeability was a function of flow rate.

In addition to the use of Darcy's Law a number of non-experimental approaches have been taken in order to determine and predict the permeabilities of the reinforcement used in RTM. One type of permeability model is the conduit type. The Bundle of Capillaries Model is one of these. This model attempts to relate the pore structure of the reinforcement to the permeability. It assumes that the reinforcement can be represented by a system of straight, parallel capillaries. This was used by Chan et al. [30] as a basis for a mold filling simulation.

Another conduit type permeability model is the Kozeny-Carmen Equation [28]. This is similar to the capillary model except that it is assumed that there is only one very tortuous conduit of roughly constant cross section through which the fluid flows.

Pore Formation

Pore formation is an important aspect of composites processing. Pores cannot transfer stresses and can serve as stress concentrators. This strongly influences some mechanical properties and the mobility of liquids through the finished part. The interlaminar shear strength and also the compressive strength are adversely affected by the pore content [31]. Pores near the surface can cause flaws in the appearance of the part such as blisters or holes. Pore contents of less than 1% are considered to be acceptable [32].

Broutman and Krock [31] state that pores are commonly caused by the inability of the resin to displace all of the air within the strands. This is affected by the viscosity of the resin, the contact angle, and the rate at which the resin and the reinforcement come together. Pores can also be caused by bubbles, which are entrained in the resin and transported into the mold. It is also possible for volatiles and dissolved air in the resin to form pores, particularly during curing. Broutman and Krock make a distinction between small spherical pores that form in the resin and interstitial pores which form in the strands. The interstitial pores tend to have sharp corners which act as places of stress concentration.

Most of the models examined in this work neglected the contribution of pores in the RTM process; however, there were several exceptions. Chan and Morgan [33] modeled the impregnation of unidirectional reinforcement with pore formation. It was assumed that the contribution of the

capillary pressure was small compared to the injection pressure. The model was based on the assumption that two types of flow were present. One was a macroflow which moved parallel to the strands. The second was a microflow which moved radially into the strands. Furthermore, it was assumed that Darcy's Law described both of these levels of flow. The numerical technique used to solve the equations used in this model was not specified.

Kurematsu and Koishi [17,18] characterized the behavior of epoxy resin impregnating non-woven polyester fabric. In an initial study [17] it was found that the distance that the resin impregnates the fabric increases with an increase in temperature at atmospheric pressure. A modified version of the Carmen-Kozeny equation was used to measure the time dependence of the impregnation. The modification was in the form of a theoretically determined capillary force which was introduced in order to account for the contribution of the fibers. A continuation of this study by Kurematsu and Koishi [18] looked at the kinetics of pore formation. It was found that the interface between the impregnated region and the non-impregnated region was not uniform. Differences in the distribution of the fibers caused variations in the velocity of the resin in very localized areas. Pores were also found to form during this process and a theoretical model was developed to estimate the pore volume. Martin and Son [21] also suggested that the formation of pores are the result of air being trapped by flow fronts recombining.

Parnas and Phelan [29] modeled the flow of resin at both a macro and microscopic level. Pores inside the fiber bundles were also examined as part of this study. Pore size was determined as a function of what was referred to as the sink strength, the ability of the fibers to remove resin from the macroflow. It was also predicted that the pore diameter would be largest at the outlet end and smallest at the inlet end due to the pressures in the mold. As the flow front continued to move through the mold, pressures increase and fiber bundles behind it continue to wet-out, which reduces the size of the pores in the bundles.

Modeling

Most of the research that has been performed in the RTM area has centered around the development of models of mold filling behavior [7,9-26,29,30,33,34]. These models attempt to predict various aspects of the mold filling such as fill times, mold pressures, and flow front positions.

The complexity of solving the partial differential equations that describe the flow of fluids through porous media is greatly eased by the use of numerical techniques. These equations are solved in an effort to predict the position of the flow front [7,9-15,20-22,26,29,30,33,34], mat deformation [16,17], or the pressure distribution in the mold [7,12,13,21,26,29,30]. Crotchet et al. [35] state that for modeling non-Newtonian fluids, finite difference methods are easier to understand and require less processing time than

finite element methods. However, finite element methods have a distinct advantage over finite difference methods when it comes to modeling complex geometries. These and several other techniques have been used to model the RTM process. For instance, the finite element method was used by Ref. [7,11,16,21,23,29]. The finite difference method was used by Refs. 22 and 34. A technique using the numerical generation of a boundary fitted coordinate system was used by Li and Gauvin [20]. Coulter and Guceri [12,13] used a boundary-fitted curvilinear coordinate system. Parnas and Phelan [29] used an explicit Euler algorithim. Um and Lee [25] used a boundary element method. No clear choice seems to have emerged as a superior technique. The accuracy of results obtained from these models has varied from good to bad.

The method for verifying results of models used by most of the previous researchers [9,10,12,13,21,22] has been to construct a square or rectangular mold. One side is usually made of a transparent material, such as glass or Plexiglass® (polymethyl methacrylate), to observe the advancing flow front. These molds use a sandwich design where some sort of gasket material is clamped between a base plate and the clear material plate. A low viscosity, Newtonian fluid is selected to simulate the resin. In many instances the mold is also used to determine the value of the permeability of the reinforcement, which is used in the model. The actual flow front positions are recorded for comparison to the predictions of the model. In several cases [7,11,12,20,22,25] the models

that are subsequently developed are run on different mold geometries. Material properties of the reinforcement and the resin are incorporated into the models. Results of the simulated mold filling such as pressures, flow fronts, mold filling times, and permeabilities are then compared to what has been observed experimentally. In some cases these models have been run for a number of different reinforcement types.

CHAPTER FOUR

EXPERIMENTAL

Materials

The approach taken in this project was to examine the RTM process from an industry standpoint. The mold and the pump, as well as the resin and reinforcement, are representative of those used in an industrial RTM setting.

The reinforcement used in this study was OCF-M8610 from Owens Corning Fiberglas Corporation. This reinforcement is randomly oriented continuous strand mat with a weight of 1.5 oz/ft^2 (Figure 2). The mat can be cut easily and evenly, which is important to insure that there are no regions without fibers near the mold walls.

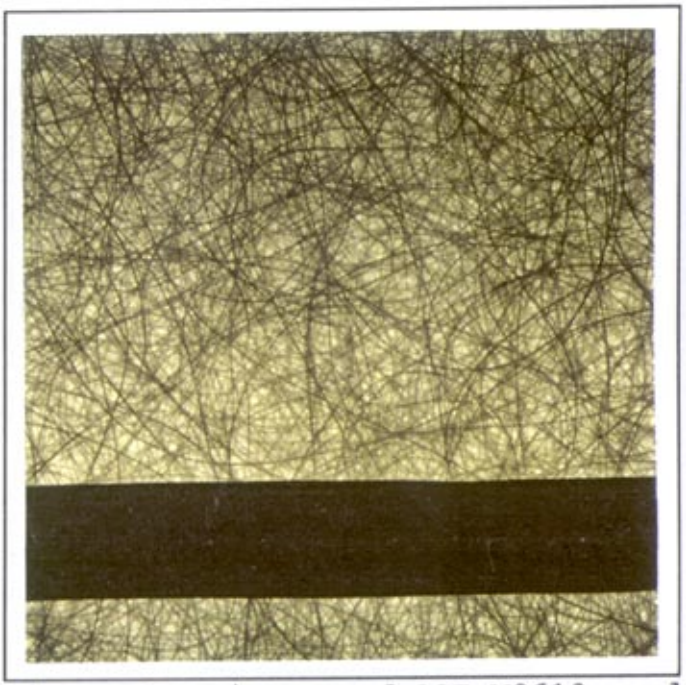

Figure 2. Picture of OCF-M8610 used in these experiments.

mold to fill unevenly, possibly creating pores or dry spots. The second reason for the selection of this reinforcement was the random fiber orientation. The randomness of the fibers permits the assumption that the permeability is the same in all directions. This mat is also commonly used in RTM because the binder and the long fiber length provide resistance to fiber washing when subjected to the flow of the viscous resin during processing. In addition to the binder, the fibers are coated with a sizing that serves to protect the fibers during processing and usually contains a silane. The silane promotes wetting of the fiber by the matrix and protects the interface from moisture [36].

Orthophthalic polyester resin is one of the most commonly used liquid resins in composites processing. Styrene is added to the resin to serve both as a diluent and a crosslinking material when mixed with a curing agent (Figure 3). The resin used in this study was Plast #83, purchased from Fibre Glast Development Co. of Dayton, OH. This resin is an unsaturated polyester resin with a viscosity of approximately 182 cP at room temperature as measured in this program with a capillary rheometer. The viscosity was found to be Newtonian at shear rates spanning the range used in these experiments. The polyesters used here are short chain molecules and as result have a low molecular weight. Polymers below a critical mass exhibit Newtonian or near Newtonian behavior [38]. This resin has a cure time of 25 minutes at 77° F when catalyzed with 1% by volume methyl ethyl ketone peroxide (MEKP). The resin used

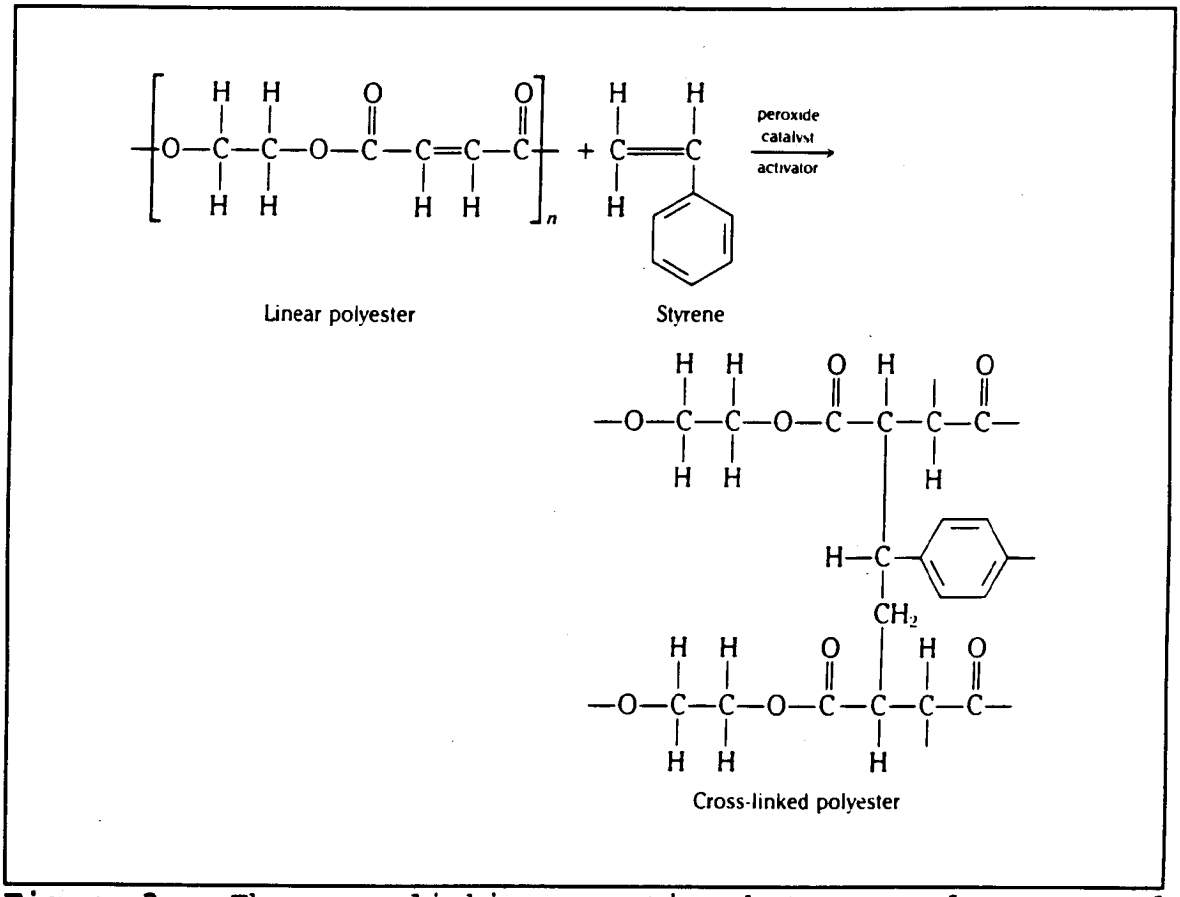

Figure 3. The crosslinking reaction between polyester and styrene [37].

has a cure time of 25 minutes at 77° F when catalyzed with 1% by volume methyl ethyl ketone peroxide (MEKP). The resin used in this experiment was chosen for its low viscosity, fast cure time, and availability in small quantities.

Miscellaneous materials used in this experiment included red, blue, and yellow pigments also purchased from Fiber Glast Developments. These were used to monitor the behavior of the flow behind the main flow front. A proprietary, external release agent (F-57-NC) was purchased from the Axel Corporation of Woodside, NY. A rotary cutter made by Olfa was used to cut the reinforcement.

Equipment

It wasn't feasible to use commercial RTM molds and injection equipment for these experiments due to their large scale and cost. An apparatus was assembled for this study and is procedurally representative of that used commercially. Unlike much of the previous processing research, this study used catalyzed polyester resin as the injection fluid. This imposed some restrictions on the injection device as well as the selection of mold materials. Tempered glass for one mold face allowed visual monitoring of the flow, while the other mold face was aluminum. Figure 4 shows the apparatus.

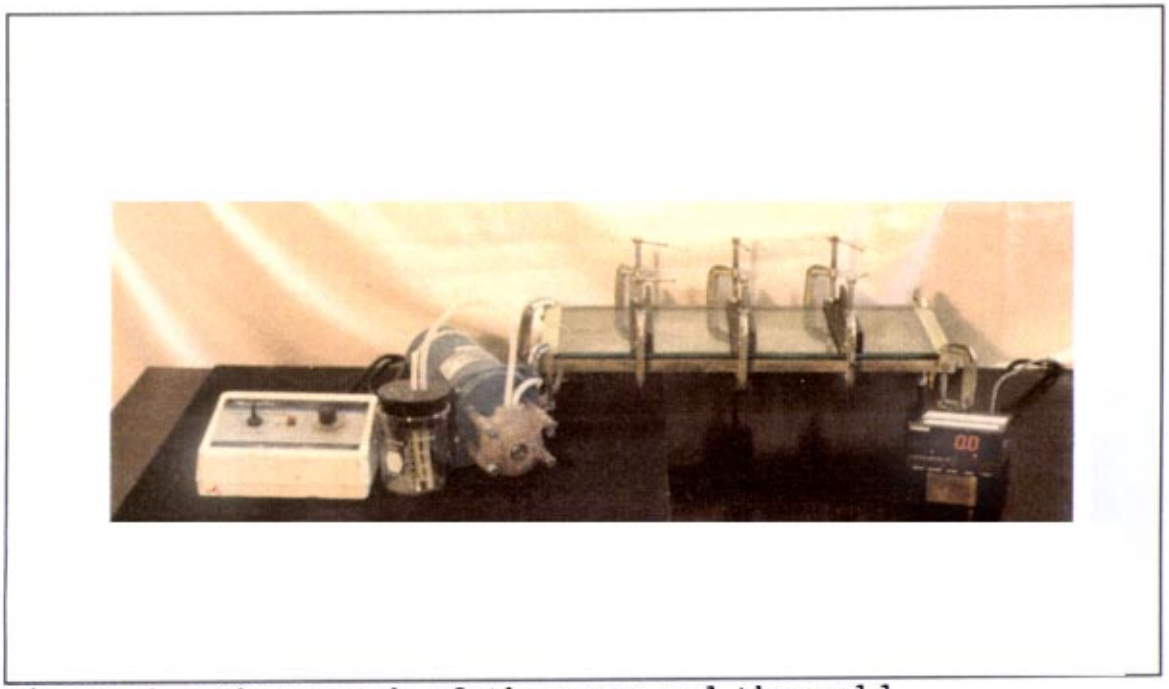

Figure 4. Photograph of the pump and the mold.

The Pump

The use of catalyzed resin requires that contact between the moving parts of the pump and the fluid be minimized. Contacting the resin with moving parts would make it necessary to clean them either with solvents prior to curing or by mechanical removal after curing. It would be difficult to thoroughly accomplish this, and would require that the pump be disassembled to some extent. This is very time consuming and not very representative of industry.

The viscosities of commercial RTM resins vary from about 100-2500 cP. Since there was interest in using more than one resin, the pump had to accommodate a range of different viscosities. The pump also needed to generate enough pressure to fill the mold with a variety of potential reinforcement styles.

A peristaltic pump fit most of these criteria. According to the manufacturer the pump selected could attain pressures of 40 psig, and handle viscosities as high as 10,000 cp. Due to the nature of the pump design, the fluid is contained entirely inside of the tubing, and therefore doesn't touch any moving part of the pump. The peristaltic pump used in this study operates by wrapping a piece of flexible tubing around a set of three rollers which are on a rotor located in the pump head. When the rotor turns, the tubing is squeezed closed at the points contacting the rollers. As the rollers revolve around the rotor, only the pinched off point moves

with them, and the tubing is stationary. The tubing behind this point regains its shape and creates a vacuum. This vacuum draws the fluid into the tubing. Once the fluid gets past the rollers it is also pushed by the fluid being drawn up behind it. Silicone tubing was found to chemically resist the resin used in this experiment and also had the highest burst pressure of those available, and so was used in all of the experiments.

The pump used in these experiments is comprised of four components: the drive, the pump head, the controller, and the tubing. The drive was Cole-Parmer Instrument Co. model 7553-00; the pump head was Cole-Parmer Instrument Co. model number 7016; the controller was a Masterflex from Cole-Parmer Instrument Co; and the tubing used was model number 96400-16, also from the Cole-Parmer Instrument Co. The flow rate of this pump was variable, with outputs between 0.001 and 38.3 ml/sec, based on water [39].

The Mold

The use of catalyzed resin also made the selection of the mold materials important. In order to meet the visibility requirement both sides of the first mold were made out of Plexiglass®. Plexiglass® (polymethyl methacrylate) has been used frequently in RTM studies [7,11-13,20]. However, most of the prior research has not used catalyzed resin as the injected fluid, instead using various oils or other viscous

liquids. It was found that the resin would dissolve the Plexiglass®, so it was unsuitable as a mold material. Tempered glass, 18 in. x 7 in. x 0.25 in., was chosen to form the transparent mold face due to its good strength, chemical inertness, and transparency. Aluminum was chosen as the mold base because of its low cost, availability, and its ability to be machined. Machining was necessary in order to incorporate fittings for the inlets and the outlets. The fittings used as the inlet and the outlets were 1/8-in. NPT x 3/16-in. hose barbs. Three holes 1-in. NPT were drilled down the center line in order to receive an Omega PX 103 (0-100 psi) pressure transducer. The transducer was placed in one of the holes and the other two were plugged. The transducer was connected to an Omega DX 316 digital readout. It was also necessary to machine a groove around the circumference of the mold base to hold the square O-ring that served as the gasket and spacer. The mold cavity with the O-ring in place measured 17.25 in. x 6.1875 in. x 0.1 in.

The Gasket

The choice of gasket material was limited. The selected material had to chemically resist the resin, form a good seal, and have good dimensional stability since it was to be used as a spacer as well. As a first step small pieces of the prospective material were exposed to the resin. Neoprene was tried first. A gasket was cut from a neoprene sheet to fit

the mold periphery. Although it could seal the mold well, it was difficult to obtain consistent molding thicknesses even when a torque wrench was used to tighten the clamps that secured the mold. It was also difficult to obtain straight edges on the molding because of the deformations at the clamping points. Another problem was that when high fiber volume fractions were used, higher injection pressures were required, and the neoprene deformed enough between the clamps that serious leaks developed.

The gasket material that performed the best, and was used for all runs, was a 0.25 in. x 0.25 in. square cross-section O-ring of BUNA N (nitrile rubber) from Parker Seals. A square cross-section forms a square-cornered seal with the glass. A circular cross-section, which tends to have a small gap where the O-ring and the mold face contact, can produce resin channeling. Nitrile rubber offers good chemical and set resistance, but long exposure to uncatalyzed resin produced some deterioration. A groove 0.25-in. wide and 0.15-in. deep was milled in the base plate to receive the O-ring. This left 0.1 in. of the O-ring exposed to act as a spacer. The groove not only constrained the O-ring and prevented it from being forced out from between the glass and the aluminum during injection, but also supported the O-ring evenly when the clamps were tightened. This kept it from deforming unevenly and helped to keep the thickness of the molding uniform. The groove also served to keep the O-ring in

position. This made it imperative that the edges of the reinforcement be cut very precisely in order to prevent resin channeling along the edges. If other thicknesses were desired, shims could be placed in the bottom of the groove which would raise the O-ring and increase the part thickness.

Procedures

Mold Filling

The mold was initially prepared by sanding out any scratches and other roughness on the mold base. The smoother the aluminum base, the easier the finished part released. This step was only necessary prior to beginning the experiments, since most of the roughness occurred during the machining of the groove, the pressure taps, and the holes for the fittings. Acetone was used to remove any residual polyester from previous runs. A pressure transducer was installed into one of three preset positions located down the flow path, the O-ring was inserted into its groove, and all of the surfaces that would come into contact with the resin were coated with the mold release agent according to the manufacturer's instructions.

Reinforcement was cut into rectangles measuring 17.25 in. x 6.1875 in. using a rotary cutter. A straight-edge was used to ensure that the edges were even and straight and would make good contact with the mold walls. Two layers were then placed

into the mold. Two layers should provide a porosity of 0.85, determined from [8]

$$\phi = 1 - \frac{n\xi}{t\rho_f} \qquad (3)$$

where n is the number of layers, ξ is the surface density of the reinforcement, t is the mold thickness, and ρ_f is the density of glass which is taken to be 2.56 g/cm^3. Care was taken to prevent fibers from extending outside the gasket.

When the mold was closed, ten two-inch C-clamps were placed around the perimeter of the mold to provide the necessary clamping pressure. The clamps were tightened with a torque wrench to 30 in-lb to ensure that even pressure was applied.

During the course of these experiments it was noticed that the cross-sections of the finished parts were not even. They tended to increase in thickness near the center. As discussed elsewhere, this was due to bending deflection of the mold. On some runs 1 in. x 1 in. x 0.25 in. steel angle iron stiffeners were placed across the width of the glass (perpendicular to the flow direction) to minimize the deflection of the glass during the injection of the resin. This deflection was not completely eliminated even when the stiffeners were used.

Some runs were made using pigmented resins. Prior to each of the pigmented flow pattern experiments three portions

of resin were measured out. Each of these was then mixed, 3% by volume, with one of the three pigments prior to catalyzation. MEKP was then added and the containers were stirred until the resin was a uniform color, indicating that the catalyst was thoroughly mixed. Each of the three colors were injected into the mold in sequence with the same injection time for each color. The pump controller was set on 2 to give an approximate flow rate of 0.9 ml/s. The flow pattern during filling was recorded with a video camera. This experiment was run three times each with and without the angle irons.

After the mold was full, as indicated by the flow of resin from the outlets, the pump was shut off. The deflected mold sides would force the excess resin out as they returned to a flat condition, after which the outlets were closed. The inlet was also clamped and the tubing was cut loose between the pump and the clamp in order to flush the resin from the tubing with acetone prior to resin hardening. The resin inside the mold was allowed to cure and the finished part removed. After removal the part was placed between two aluminum plates and put in an oven at 140 °F for one hour to post-cure. It was then measured at positions along the edges and in the center to check for uniformity.

Permeability Measurements

Darcy's Law assumes the permeability of a porous medium to be a material property, and therefore to remain constant under different injection rates assuming that the fluid and medium properties are constant [40]. Permeability was determined for two layers of reinforcement, with and without the angle iron stiffeners in place. The reinforcement layers were placed into the mold so that they were just touching the edge of the pressure transducer at the inlet end position. This allowed the pressure drop across the reinforcement to be determined. The resin was injected until the fibers were saturated, then more resin was injected and the pressure measured when a steady state was reached. The volumetric flow rate was determined by measuring the time to catch a certain volume of resin. These data, in conjunction with Darcy's Law (Eq. 1) enabled permeability to be calculated.

Porosity Measurements

Porosity in the cured parts was studied as a function of the flow rate. Specimens made at different flow rates were sectioned to check the porosity level and the degree of wet-out. Microscopy specimens were made from small pieces of the composite, encapsulating them in clear epoxy using standard metallographic techniques. Once the epoxy hardened the specimens were polished on a polishing wheel with very

fine (4000 grit) sandpaper and viewed in a light microscope. The microscope monitor displayed the image of the specimen. A grid was laid over the screen and the number of grid intersections falling within a pore were counted following standard quantitative microscopy techniques. This number was then divided by the total number of intersections on the grid and multiplied by 100 to obtain the percent porosity [41]. Micrographs (hard copies of the monitor screen) were taken of selected areas.

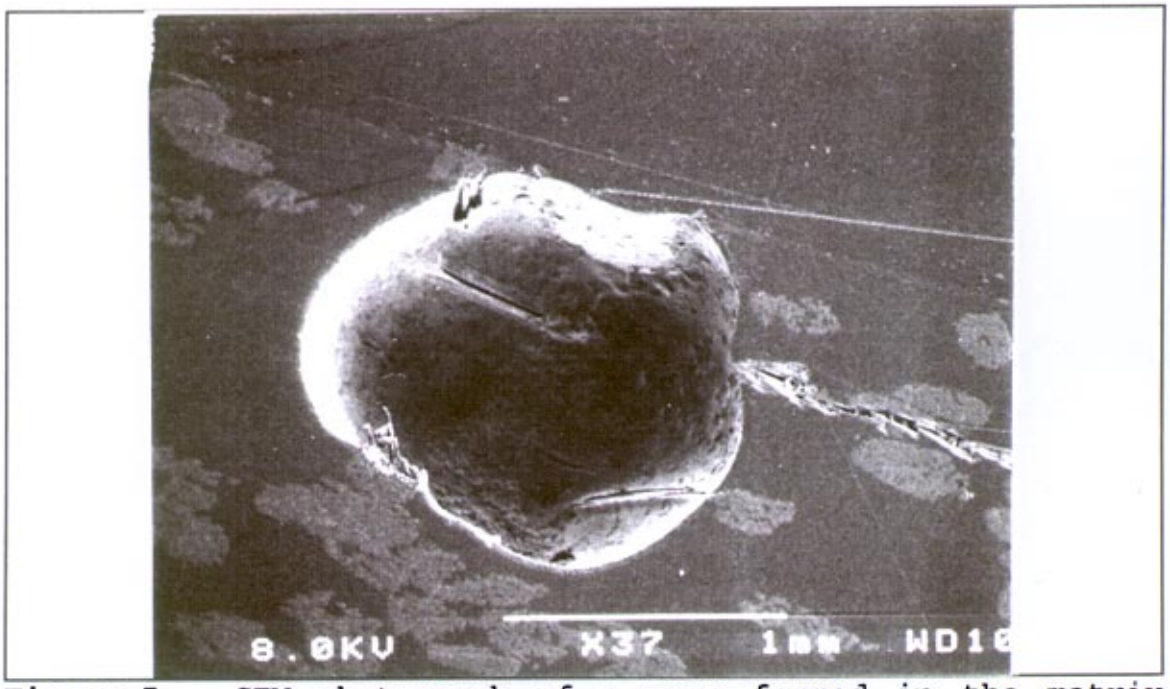

Figure 5. SEM photograph of a pore formed in the matrix region.

At the very slowest injection rate large pores formed between the strands as well as within them. The pores between the strands were assumed to be spherical. This approximation is reasonable based on Figure 5, taken with a scanning

electron microscope. An area was marked off and the diameters of the pores contained within it were measured using a field lens and a set of micrometers. The corresponding pore volumes were calculated. The total volume of pores was then taken as a percentage of the marked off volume. This percentage was then added to the percentage found by the grid method for the intrastrand pores.

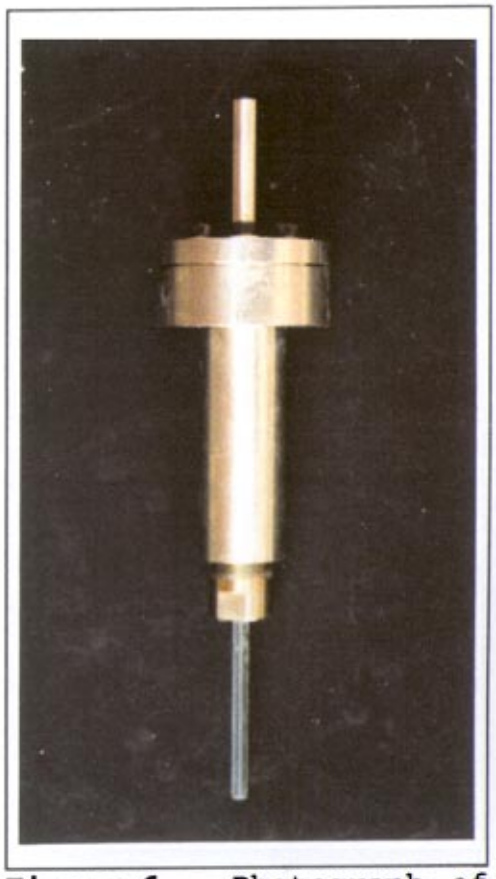

Figure 6. Photograph of the capillary rheometer.

Viscosity Measurements

The viscosity of the resin was measured with a capillary rheometer, designed according to ASTM Standard D3835-79 (Figure 6). Measuring the volumetric flow rate and knowing the applied force, equations supplied with the standard could be used to calculate the shear rate, shear stress, and the viscosity [Appendix C]. The resin used in these experiments was tested at applied forces ranging from 2.7 lb_f to 21.7 lb_f. This produced flow rates comparable to those used in these molding experiments.

CHAPTER FIVE

RESULTS AND DISCUSSION

The central purpose of this study was to develop an understanding of the processing factors in RTM. Very little of the previous work in this area has looked at the mold filling behavior with catalyzed resins. The approach in this study was to use actual resins and reinforcements representative of those in industry to characterize interactions of resin and reinforcement. The sandwich type mold included one glass side to allow visual observations. Pigments were used in selected runs to illustrate the flow pattern behind the main flow front as well as to examine the wetting process. A pressure transducer was incorporated into the mold to examine pressure variations at different positions during the filling process.

Initial Molding Runs

One of the primary goals of this project was to develop a working RTM process representative of that used in industry. Although there were initially some problems determining suitable mold materials and injection equipment, the resulting process does use equipment and principles similar to

commercial ones, but on a smaller scale. This equipment and process have been used to successfully mold parts with different reinforcement types and quantities, and two different resins.

Figure 7. Photograph of moldings made using the RTM equipment developed in this study.

Figure 7 shows some of the typical moldings produced with the equipment and procedure developed in this study. The moldings were generally of very good quality from a porosity standpoint. The porosity was less than 1% under all processing conditions. The reinforcing mat was not distorted appreciably during the mold filling. The molded parts were generally of good quality, with low porosity, little fiber wash, and a fiber volume fraction in the 0.15 range, which is typical for parts made using this reinforcing mat.

The parts were made at room temperature which ranged from around 60° F to 75 °F depending on the season; the resin was at the ambient temperature. The molding pressures were under 15 psig, and vacuum was not used in this study. Variations in this molding technique with other part geometries, and with a pressure bag serving as one mold face have also been successful. Molding geometries have included channels, circular tubes, airfoil shapes, and large plates.

In the initial runs the dimensional quality suffered. Prior to the use of the square gasket, the edges of the parts were very irregular. More importantly, as will be discussed in detail later, the thickness was not uniform in the initial moldings, with a greater thickness in the center of the molded plates.

Wetting Process

The strands in the reinforcement are made up of a collection of approximately fifty to one hundred individual fibers which are continuous over the length of the strand. Long channels are formed in the gaps between these fibers which behave like capillaries (Figure 8). This Figure also shows that the channels have a non-circular cross-section which, due to variations in fiber packing and diameter, change with the length and the width of the strand. The strands are randomly oriented so that these channels are oriented from

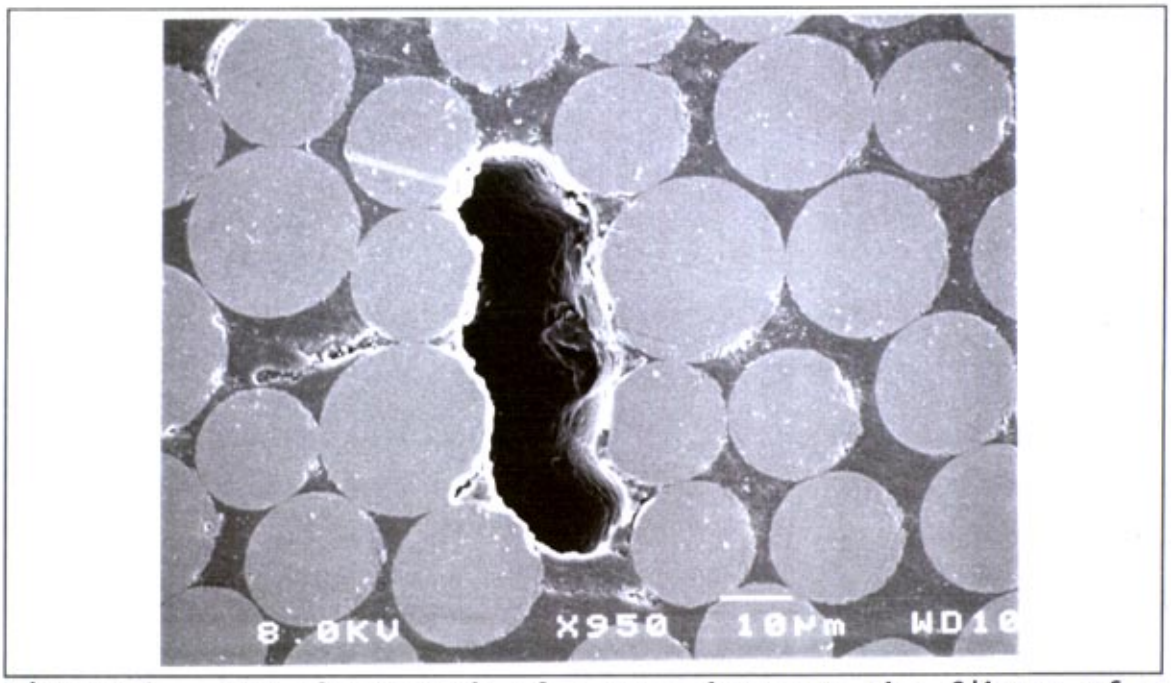

Figure 8. SEM photograph of a pore between the fibers of a strand, also showing fiber spacing (polished cross-section).

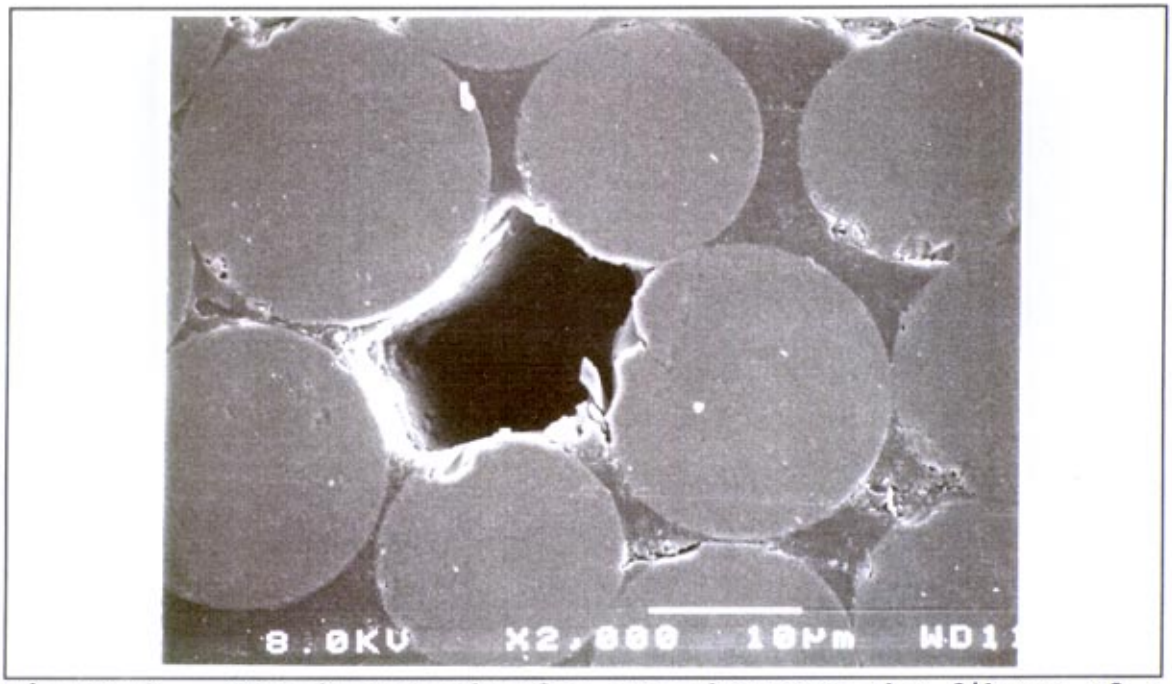

Figure 9. SEM photograph of a pore between the fibers of a strand.

parallel to perpendicular to the flow.

As can be seen in Figures 8 and 9, small gaps exist between the fibers; when the resin approaches the strand from the side, they will appear as thin slits. These slits can act as capillaries, and resin is drawn into them. Therefore, it is not necessary for the resin to enter the strand from an end in order to wet it out. The resin has been observed entering through the sides of strands oriented perpendicular to the principal flow direction, then flowing in both directions away from the point of entry. The fibers tend to pack together so that they are touching the surrounding fibers in some cases (Figures 8 and 9). These Figures also show that the resin is able to get into the tight corners formed where fibers come into contact. The pores present in these photographs suggest that the resin is penetrating from all sides at once, thus trapping air.

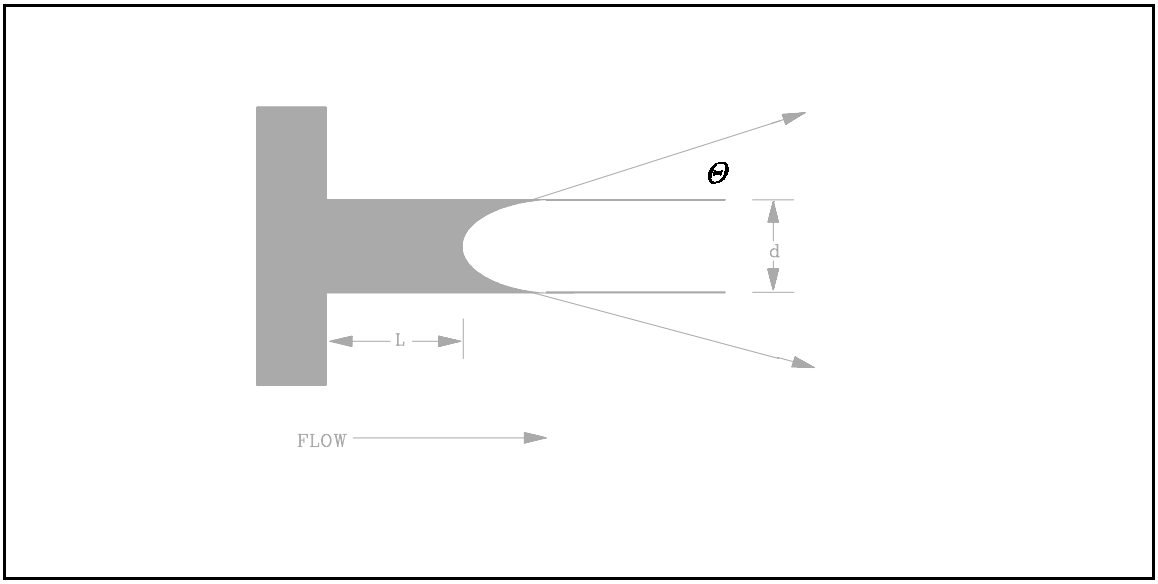

Figure 10. Diagram of capillary flow.

The following equation describes the flow of a fluid in a horizontal circular capillary [42]:

$$v = \frac{d\,\gamma_{lf}\cos(\theta)}{8\,\mu\,L} \qquad \textbf{(4)}$$

In this equation d is the diameter of the capillary, γ_{lf} is the interfacial tension of the liquid-fluid interface, θ is the contact angle, μ is the viscosity, and L is the length of the fluid in the capillary (Figure 10). The driving force pressure for penetration is given by [42]

$$\Delta P = \frac{4\,\gamma_{lf}\cos(\theta)}{d} \qquad \textbf{(5)}$$

Although the cross-sections of these capillaries are not circular, resin can move into the strands without an induced pressure drop. This indicates that the channels in the strand are behaving in a fashion similar to a circular cross-section capillary. It is reasonable to assume that the same factors which affect the movement of fluid in a capillary are important in the strands, even though the geometry is different. The fibers are treated with a proprietary surface agent (probably a silane) to enhance the bonding and the wetting of the fibers with the matrix. This affects the contact angle which (shown in Eq. 5) determines the driving force of capillary penetration [42]. When the gaps between the strands become very small the flow transversely into the

strand will become very limited. Similarly, the length of gap, L, that can fill from one entry point is limited (Eq. 4).

Moldings were made using a sequence of three colors of pigmented resin, so that each color made up about one third of the total resin. It was then possible to see the filling pattern in the plane of the sheet and through the thickness at different stages of filling. It was of particular interest to section the molding to examine the resin inside the strands as compared to that in the pure matrix regions between them. Examination of cured plates that were made using pigmented resin showed that the matrix inside the strands appeared to be the same color as the resin that was injected into the mold first. Specimens were prepared, polished, and examined under a light microscope. Figure 11 shows the cross sections of fiber bundles after wet-out. The blue- colored resin between the fibers (inside the strands) went into the mold first. The red- colored resin surrounding the strands was the last to be injected. Yellow- colored resin was injected between the blue and the red, none of which is seen at this position. This shows that the first resin to contact the bundles is drawn into the space between the individual fibers within a strand primarily by capillary action [8]. Once the resin is inside the strands it is not displaced by the subsequent flow of the resin through the spaces between the strands as the mold fills. There is some threshold distance between the individual fibers around the perimeter of the bundle which

allows the resin to be displaced. Gaps as small as 3.5 μm were large enough to allow subsequent resin to move in and displace the original resin at the outside of the strand.

There was no apparent difference in this characteristic for strands that were perpendicular or parallel to the flow direction. This observation lends strength to the argument that it is capillary forces that most strongly influence the wetting phenomenon within the strands, so that strand wet-out is not primarily induced by the pressure drop of the pump. Once the strands are wet-out, further flow of the resin occurs almost entirely in the pure resin spaces between the strands, which is a tortuous route in random mat. Once filled by resin, the capillary forces driven by γ_{lf} no longer exist, so new resin does not enter.

Figure 11. Microphotograph of interface of a matrix region and a fiber bundle.

The Effect of Flow Rate on Porosity

The preceding discussion is not intended to imply that the overall wet-out of a composite is not affected by the pressure drop of the pump. Molnar et al. [8] demonstrated how the position of the gross flow front in relation to the wet-out region can vary depending on the rate of injection. If the rate of injection is relatively fast, then the gross flow front stays physically ahead of the position where the strands are completely wet-out. This is readily observable through the glass mold face, as the strands appear white until they are completely wet-out with resin.

In industrial applications of RTM the resin is injected into the mold as fast as possible in order to maximize the number of pieces that can be made in a given amount of time. There has been some general recognition in the literature [8,12] that flow takes place at a micro level within the strands and a macro level between the them. This section describes the effect of injection rate on both the micro and the macro level of flow, and how this ultimately affects wet-out and the retained porosity in the finished part.

To study the effects of flow rate on porosity, different flow rates were selected to produce different relative positions of the macroflow front and strand wet-out position. A specimen was sectioned from each of the cured plates,

polished, and examined under the microscope to establish the size, quantity and the location of the resulting pores.

Flow Rate #1

The first molding was made at a flow rate of 0.05 ml/sec. This was the slowest that the pump was capable of turning while still producing a consistent flow rate. At this condition resin could be seen (through the glass mold face) moving into the individual strands. The resin could move through the strand either from the end or through the side depending on the orientation of the strand where it contacted with the resin. The distance that the resin moved into the strand ahead of the macroflow front was approximately 0.02-0.03 in., as measured with a calliper through the glass mold face. However, it was difficult to measure accurately due to the orientation of the strands and the variations in the flow front across the width. The region directly behind the flow front appeared to be mostly translucent, indicating that the reinforcement was saturated.

It was thought at the outset of the experiment that because the resin was moving so slowly, the strands would have plenty of time to wet-out and the resulting part would be nearly perfect. In fact, pores formed within the strands amounted to 0.17% of the total plate volume in these specimens, which is very low. However, in addition to the pores within the strands, larger pores (0.02-2 mm) formed

unexpectedly in the regions between the strands. These pores could be easily seen with the unaided eye when the specimen was held up to the light. The large pores were unique to the slowest flow rate case, and because of their size could cause significant deterioration of some mechanical properties of the part.

The large pores between the strands form in certain instances when the spaces between the strands are oriented and spaced in such a way so as to behave as capillaries. These capillaries have a much larger diameter than those formed between the individual fibers within the strands. This would allow the resin to suddenly move ahead and overwhelm the flow within the strands in localized areas as shown in Figure 12. Equation (4) shows that the velocity of resin flow in a capillary increases as the diameter increases. When the resin moved ahead in two adjacent locations it was possible for the two capillaries between strands to join together ahead of the main flow front. Upon recombining, air would become trapped, forming a large bubble behind the flow front. In most instances the bubbles would escape through some path to the flow front, and could be seen moving. However, because of the slow flow of the resin, there was little driving force for the resin to dislodge and carry the bubbles along. If the trapped air could not find a path, it would remain in the composite as a pore. The pores residing between the strands, although individually large in size, only made up .049% of the total

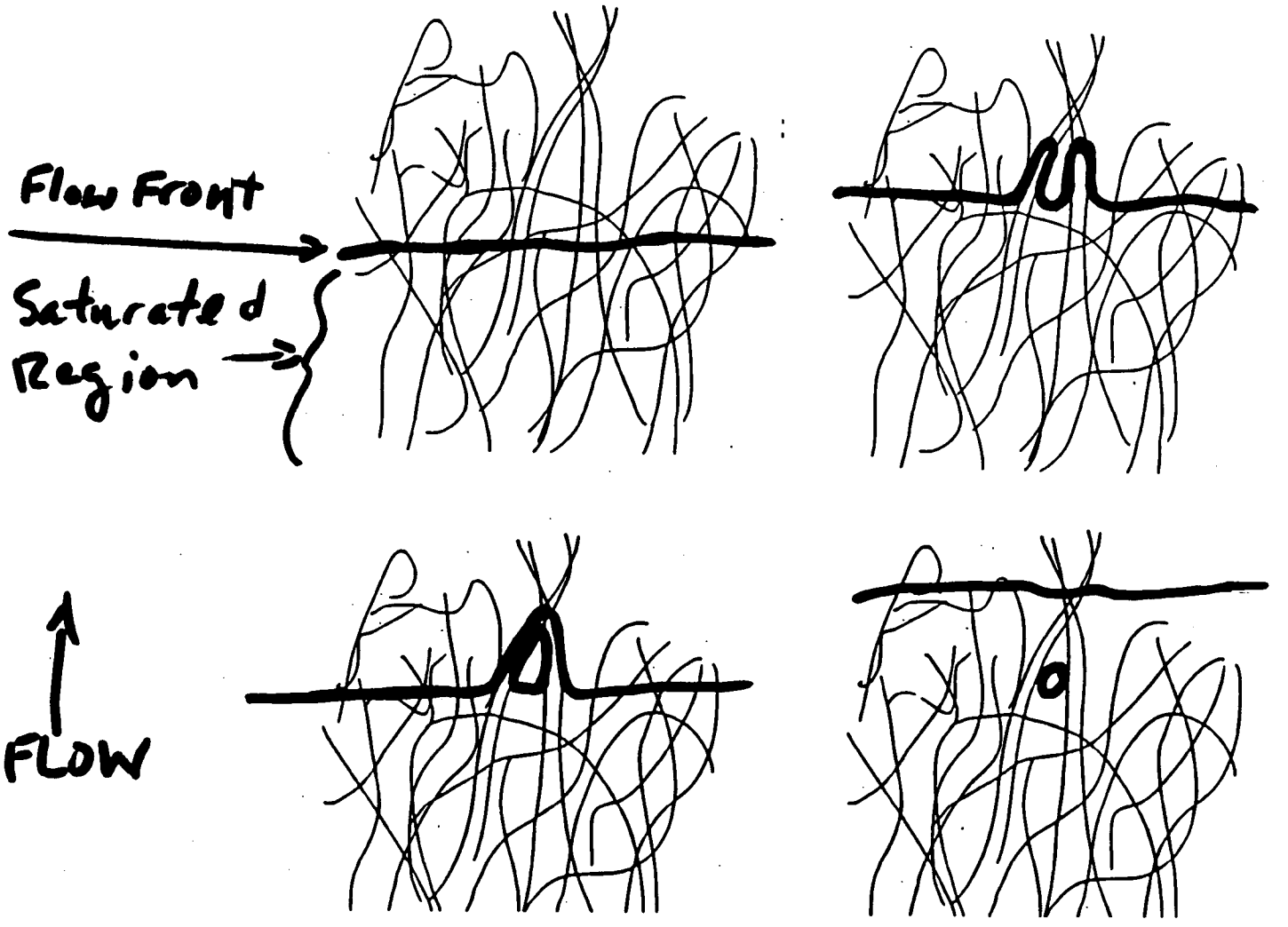

Figure 12. Sketch of the encapsulation of air by the recombining flow front at slow speeds.

volume of the molding.

Flow Rate #2

The second molding was made at a rate of 0.1 ml/s. At this rate the flow front position between the strands was approximately equal to the wet-out position within the strands. The resin could no longer be seen moving ahead of the macroflow front within the strands. However, the resin could be seen being deflected by the strands and moving in directions other than that of the main flow front movement. This indicated that the flow in the capillaries formed by the randomly oriented strands was still slightly ahead of the macroflow front. The macroflow front became much smoother and the large bubbles that were seen forming at the slower rate were no longer present.

Table 1. % Porosity at Different Flow Rates.

Flow Rate Number	Volumetric Flow Rate (ml/s)	% Porosity	Average Pore Diameter mm*	
1	0.05 ml/s	0.22 %	Between strands	0.474 mm
			Within strands	0.015 mm
2	0.1 ml/s	0.27 %	0.022 mm	
3	0.4 ml/s	0.39 %	0.026 mm	
4	0.9 ml/s	0.53 %	0.028 mm	

* pores within strands are elongated, so that their length is much greater than the pore diameter.

Examination of the specimens from this flow rate experiment showed that the porosity was entirely within the strands, with no pores in the pure resin regions between the strands. As shown in Table 1 the porosity of these specimens was approximately 0.27%. Referring back to the method of measuring porosity (see Porosity Measurements in CHAPTER FOUR), the average pore had a diameter of 0.022 mm. No pores were found outside of the strands.

Flow Rate #3

A third flow rate of 0.4 ml/sec was chosen in which the macroflow front was coincident to the flow in the large capillaries formed between the strands. At this rate the flow could no longer be seen changing directions around the reinforcement at the flow front. The flow front was very smooth and linear and moved down the mold in a uniform manner.

As Table 1 shows, the level of porosity was 0.39% and again this was located entirely within the strands. The average diameter of the pores was 0.026 mm.

Flow Rate #4

The highest flow rate used was 0.9 ml/s. At this rate the macroflow overwhelmed all aspects of the microflow. The front was very linear, smooth, and progressed uniformly down

the entire length of the mold. Strand wet-out lagged far behind the macroflow front.

The porosity at this flow rate was approximately 0.53%. Pores were located within the strands and the average diameter was 0.028 mm.

The results from this study indicate a distinct dependence of the size and location of pores on the resin injection rate, for this particular reinforcement type and fiber content. The slower the flow rate the lower the porosity, although it doesn't appear possible to eliminate porosity completely with these materials and under these molding conditions.

Microflow Lag Distance

The microflow lag distance is the distance between the macroflow front and the position where the strands are completely wet-out. An unexpected aspect of the study was the linearity of the relationship between the flow rate and the position of saturation in the strands. In the slowest case the degree of wetting was indicated by the resin moving into the strands ahead of the macroflow, a distance of approximately 0.02-0.03 in. (a negative distance since it preceded the macroflow front). For the three higher flow rates this was manifested as a whitish, hazy region immediately behind the macroflow front, and is referred to as

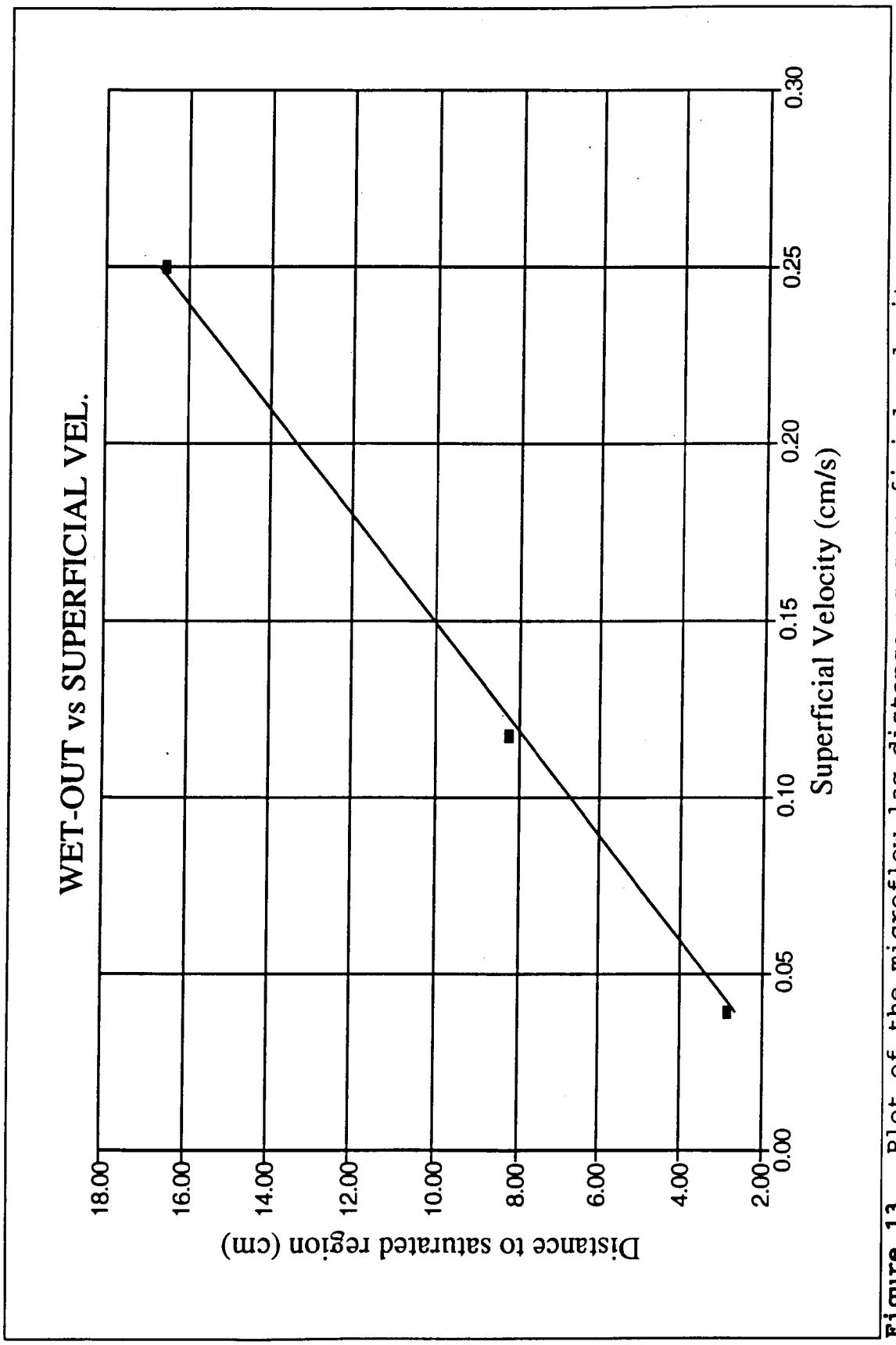

Figure 13. Plot of the microflow lag distance vs superficial velocity.

the microflow lag distance. The whitish color indicated that the strands had not completely wetted out. The lag distance ranged from approximately 1 in. for flow rate (2) to approximately 6.5 in. for flow rate (4).

The length of the microflow lag distance combined with the overall flow front velocity can be used to determine the time it takes to thoroughly wet-out the strands after the passing of the macroflow front. The superficial velocity is calculated from the volumetric flow rate divided by the total cross-sectional area, ignoring the reinforcement areas. When plotted with the microflow lag distance, this yields a straight line (Figure 13). The slope of this line is the time it takes to saturate the strands, in this case 66 seconds.

The microflow lag distances used in Figure 13 were the maximums measured in each case. There was some variation in this distance at each flow rate indicating that some regions wet-out more quickly, apparently due to factors such as local strand integrity and fiber packing. Also, the measurement of this distance was difficult due to the absence of a sharp line dividing the saturated region from the slightly-less-than-saturated region. However, the measurements were done consistently at each flow rate and the saturation time predicted by this plot is close to the measured saturation time of 79 seconds. The measured saturation time was determined by using a stopwatch to measure the time necessary to wet-out the reinforcing mat after the passing of macroflow

front. Again, the point of saturation is subjective and difficult to define consistently. Improved measurements of the microflow lag distance would allow improvement in the prediction of saturation time.

It should be noted that the linearity of Figure 13 shows that the wetting of the strands has little dependence on the induced pressure drop of the pump. This further supports the contention that capillary action is primarily responsible for the wetting of the strands.

Mold Deflection

The mold in most of this study includes a top face of 0.25-in. thick tempered glass. As noted earlier, parts made conventionally in this mold did not have a uniform cross-section. The plate was as much as 0.01-in. (10%) thicker in the very center than at the edges, tapering down to the thickness of the gasket at the edges.

Initially, it was thought that this problem was due to the glass being bent over the reinforcement during clamping. Three attempts were made to combat this problem. First, a harder gasket material was used. It was thought that if there were less deformation of the gasket there would be less bending of the glass. Second, care was taken to insure that the reinforcement did not extend over the top of the gasket. Third, stiffeners made of angle iron (1 in. x 1 in., 0.25 in.

thick) were clamped across the mold face to resist bending. None of these procedures completely eliminated the problem.

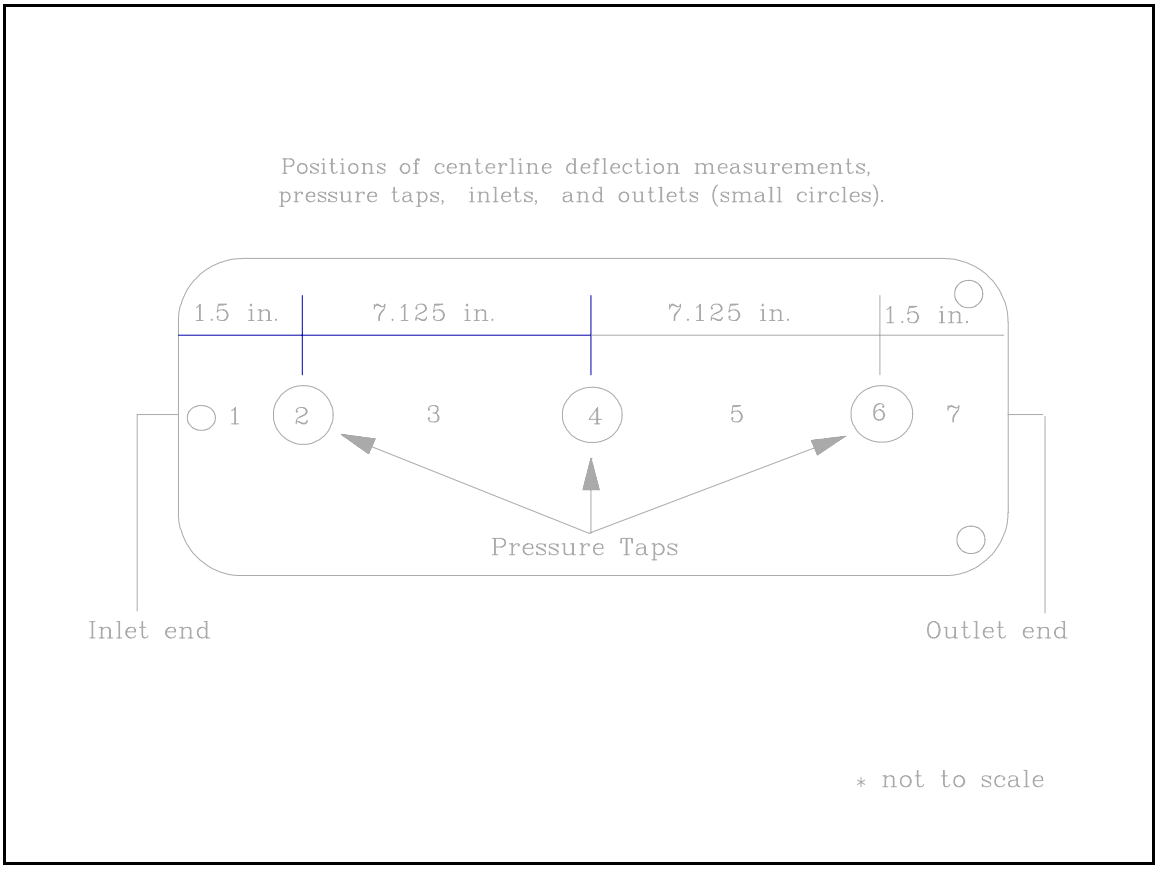

Figure 14. Positions of deflection measurements, pressure taps and inlets and outlets.

Through the use of the digital displacement indictor (Mitutoyo 543-531A), the deflection of the glass under both flow and static pressurized conditions was measured at the points shown in Figure 14. With the pressure transducer in place and the vents closed, the mold was filled to a constant pressure with uncatalyzed resin. Deflection readings were taken prior to pressurization, after the desired pressure was reached, and after the pressure was relieved. The difference

between the pressurized reading and the initial reading was the deflection at that pressure. Figure 15 shows that the glass deflects in a linear fashion with increasing pressure, with the mold deflection increasing from the edge to the center of the mold lengthwise, as shown by the positions in Figure 14. Figure 16 shows that, at a constant mold pressure, the response is the same on both ends of the mold, indicating that this is not a clamping phenomenon. Figure 17 shows that during resin flow (variable pressure down the mold) there is more centerline deflection at the inlet end than at either the center or the outlet end, with or without the stiffeners. This reflects the pressure drop down the length of the mold. Measurements of the pressure at the pressure tap locations down the length of the mold for both the constrained (stiffeners) case and the unconstrained case are shown in Table 2. It should be noted that maximum deflections occurred between the stiffeners.

Table 2. Deflections and Pressures at each Pressure Tap in both the Unconstrained and Constrained Cases During Flow.

Tap Number	Unconstrained		Constrained	
	Pressure	Deflection	Pressure	Deflection
1	10.1 psig	0.0123 in.	11.8 psig	0.0022 in.
2	5.1	0.0075	6.5	0.0015
3	0.5	0.0034	0.9	0.00063

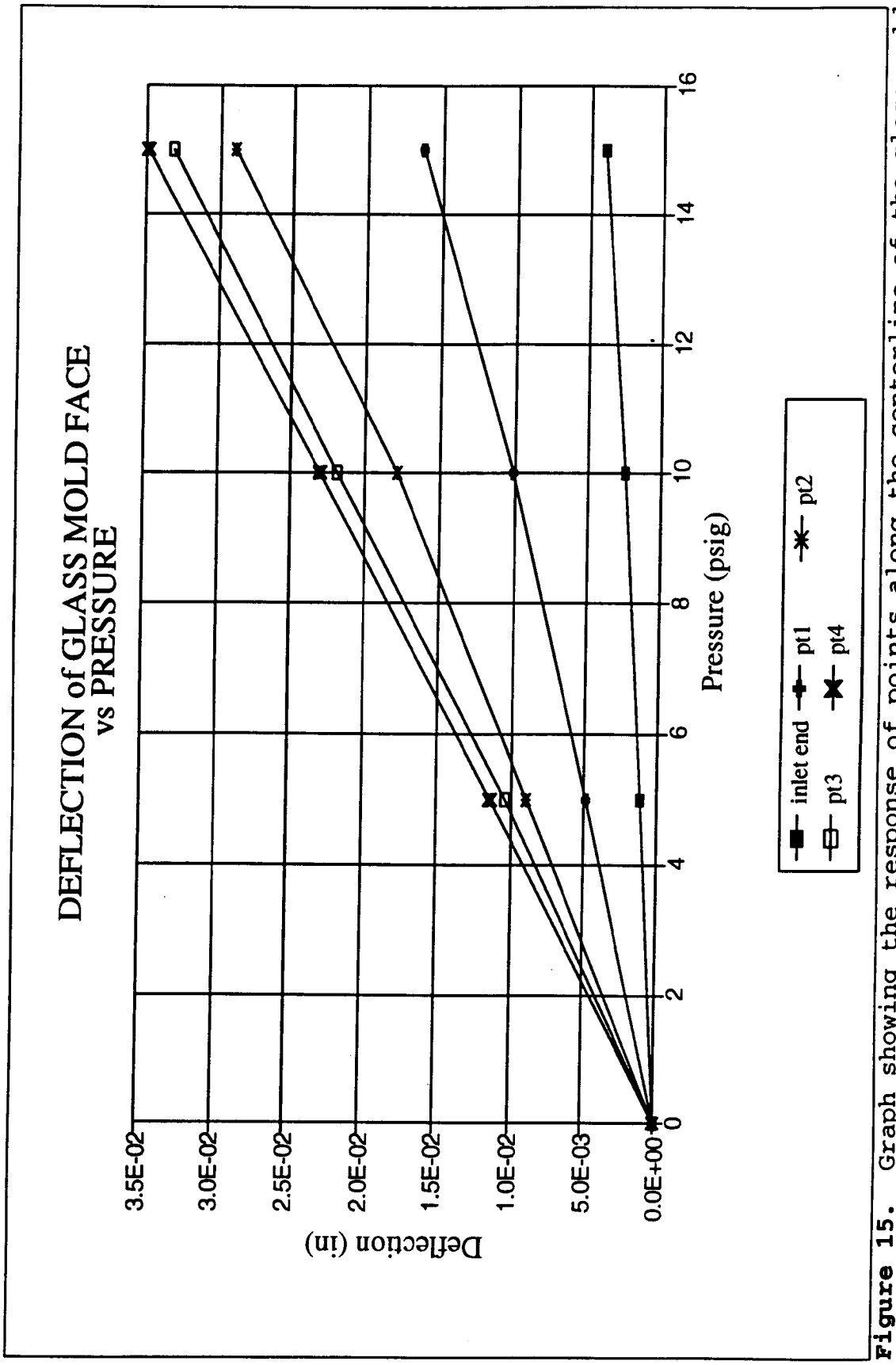

Figure 15. Graph showing the response of points along the centerline of the glass mold face (at constant pressure) from the inlet to the center of the mold.

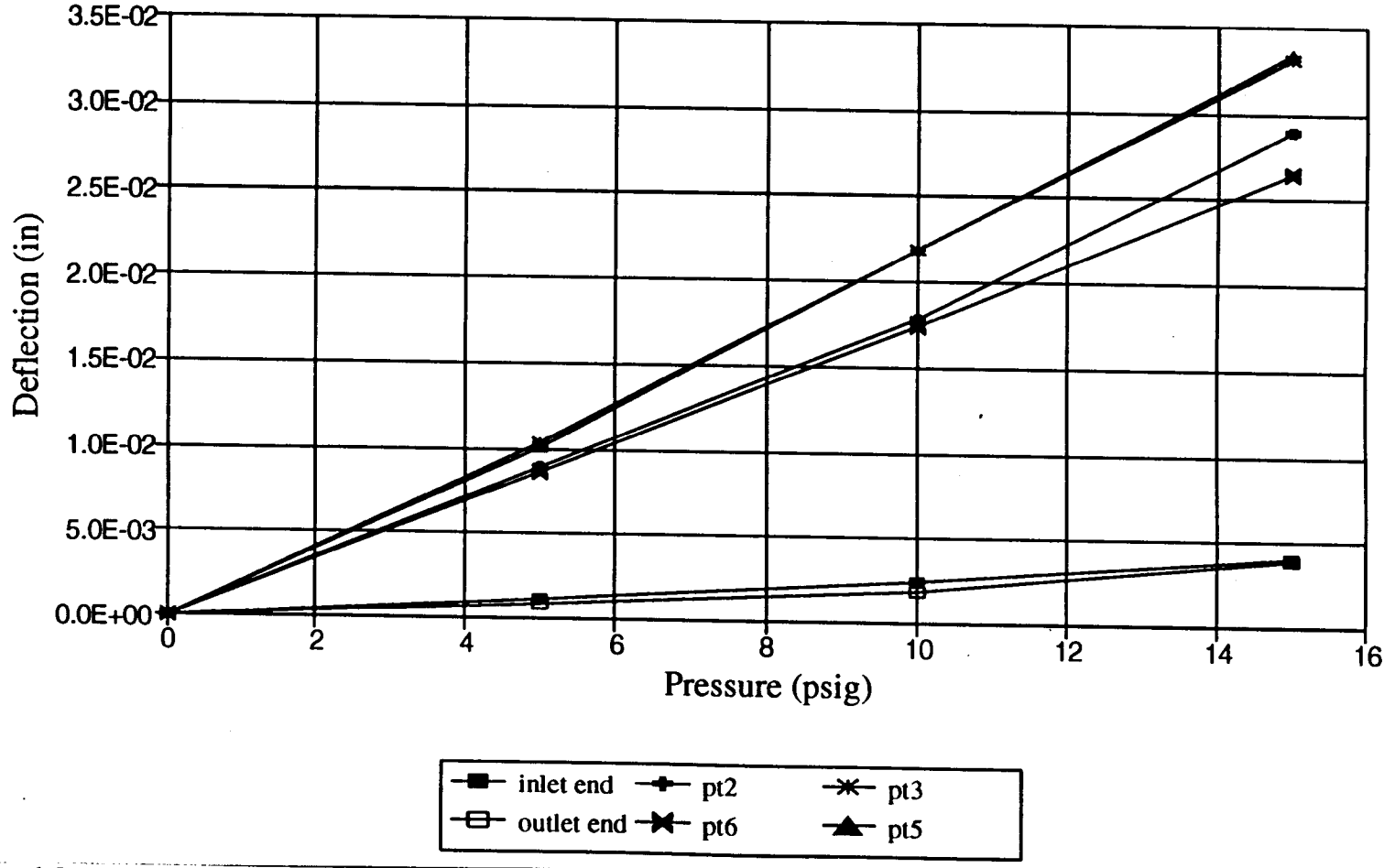

Figure 16. Graph showing the response of points along the centerline of the glass mold face (at constant pressure) at both ends of the mold.

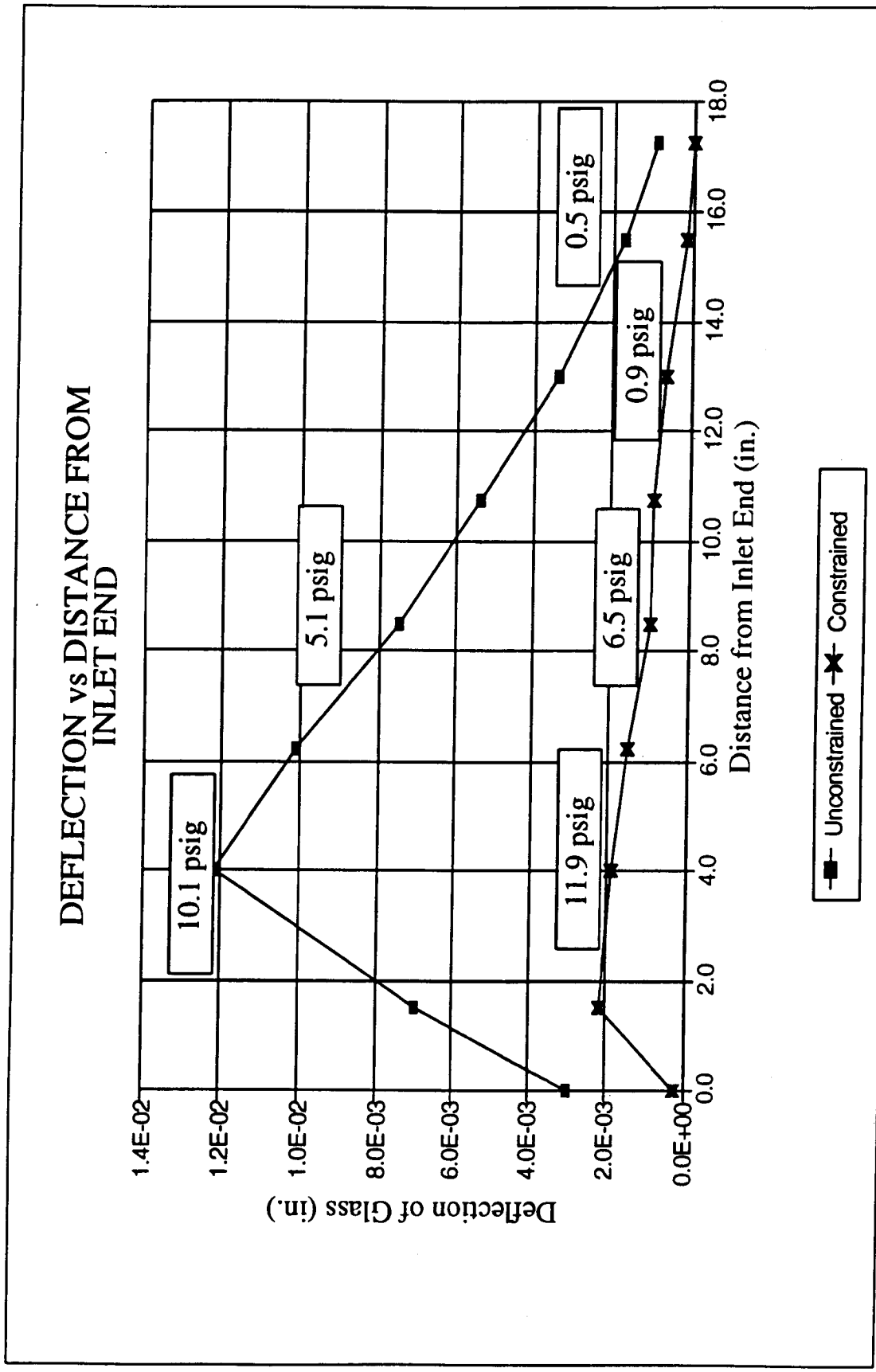

Figure 17. Deflection profiles for both the constrained and unconstrained cases during flow.

The mold deflection translates into added volume at the one end, along the central area of the mold, and thus excess resin is present in this area. The reinforcement doesn't appear to shift during the molding process. In the initial molding procedure the pump was shut off after the resin reached the outlets, and the vents were closed immediately. This had the effect of trapping the excess resin in the mold; resin would then flow to evenly distribute along the length, but mostly near the center of the width, at equilibrium. This is further supported by Figure 18, which shows that a uniform pressure of 5 psig causes a maximum deflection in the center (lengthwise) of the mold. Pressures of this magnitude were measured inside the mold if the vents were closed immediately after the pump was shut off. Allowing for a slight contraction during cure, this amount of deflection matches the amount of variation measured in the center of the cured molding.

Thus, mold deflections due to the pressure and mold dimension caused the variations in thickness observed in the early moldings.

Other evidence of excess resin becoming trapped in the mold was the behavior of the resin if the vents were left open after the pump was shut off. In this case the resin continued to flow out of the vents for some time. This subsequent flow was caused by the unbending of the glass mold face as the pressure declined, forcing the excess resin out of the mold.

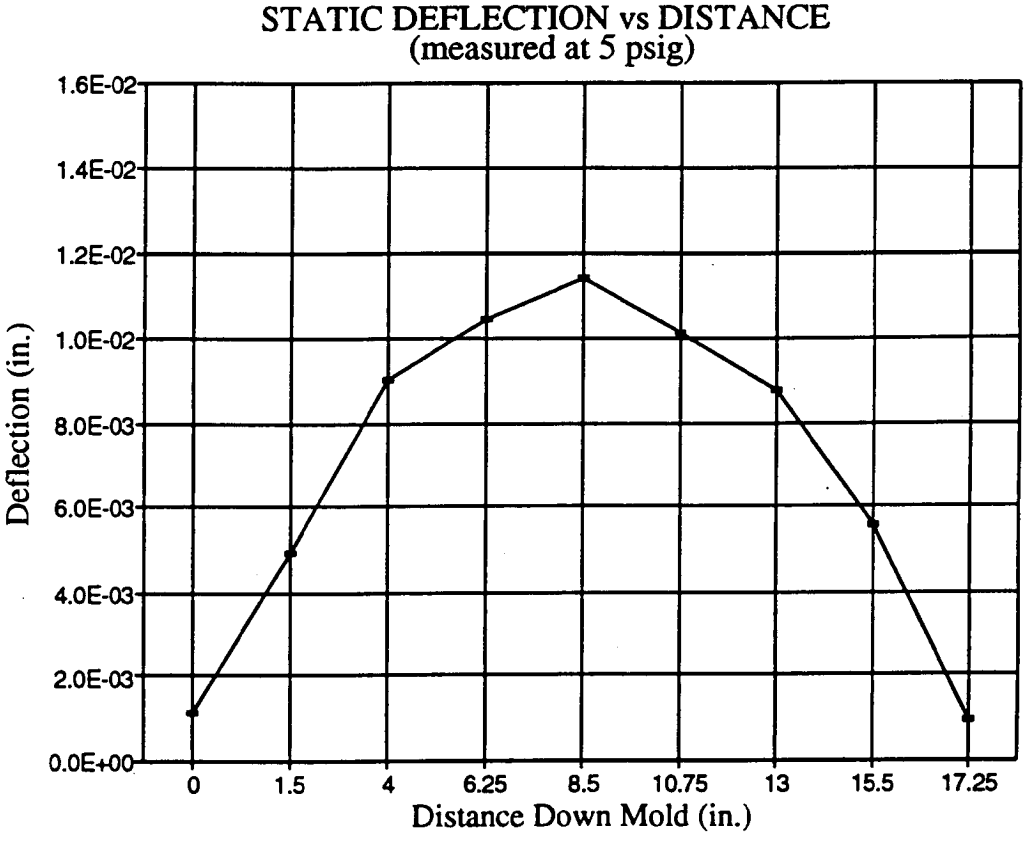

Figure 18. Static deflection from a uniform 5 psig in the unconstrained case.

The same behavior was seen if the stiffeners were used, but to a lower degree due to the much smaller deflection. Allowing the resin to flow after pump was shut off, regardless of whether the stiffeners were used, resulted in a molding of uniform thickness.

Along with the change in volume caused by deflection of the mold came a change in the cross-sectional area available for flow. As can be seen in Figure 17, this deflection, and thus the cross-sectional area down the length of the mold, was dependent on whether the stiffeners were used.

This change in cross-section complicates the use of Darcy's Law. To determine a value for the area available for flow, an average of the deflections down the length of the mold was taken for both the constrained and unconstrained cases. Adding this deflection to the non-pressurized mold cavity height and assuming that the deflection was a circular arc across the width, equations for the area of a circular segment were used to find the change in cross-sectional area of the mold cavity due to pressure. In the case where stiffeners were used the average overall mold deflection was 0.00094 inches. This resulted in a change of 0.0039 in^2 in the mold cavity which is a 0.63% increase in cross-sectional area. In the case where stiffeners were not used the average deflection was 0.0057 inches for a cross-sectional change of 0.024 in^2 or a 3.9% increase in cross-sectional area. The effects of this were most strongly felt in injection pressures and permeabilities for the different cases, as described later.

The theoretical deflection of the unconstrained case was calculated with plate equations. Equations for both a simply supported (hinged) plate and a plate with fixed (clamped)

Table 3. Predictions of Maximum Deflections Using Plate Equations [43].

	Static Pressure		Uniformly Decreasing Pressure	
	Measured	Predicted	Measured	Predicted
Simply Supported	0.023 in	0.017 in	0.0122 in	0.0104 in
Fixed Edges		0.0036 in		0.0019 in

edges were used [43]. As Table 3 indicates, the simply supported model gives a reasonable prediction of the deflection actually measured in both the uniform pressure and decreasing pressure (along the length) cases. Differences between the measured and the predicted values are probably due to deflections incurred in the compression of the reinforcement prior to molding, which are not accounted for in the predicted values.

Permeability

The permeability was determined according to the procedure described previously. Results show that small changes in the processing conditions can have a pronounced effect on the calculated values. The permeability was found to have different values depending on the flow rate and whether stiffeners were used.

Applicability of Darcy's Law

This section explores the suitability of using Darcy's Law to determine the relationship between the resin flow rate and pressure drop. The stiffeners were used throughout to minimize the deflection problem; results for the unconstrained case are given at the end of this section. Calculations for the permeability follow the method described in Chapter Four. The reinforcement in all cases began at the downstream edge of the pressure transducer as also described in Chapter Four.

Figure 19 shows a typical plot for flow rate *versus* pressure at the first pressure measuring position. The slope of the experimental line reflects the permeability and should be straight if Darcy's Law (Eq. 1) applies. The deviation from linearity indicates that the permeability is not a constant, but varies by a factor of two over the range of pressures and flow rates examined (Table 4). Although the details are uncertain, Gauvin et al. [15] found a similar

Table 4. Permeability at Different Flow Rates and Pressures.

Flow Rate Number	Permeability case #1 (darcys)	Permeability case #2 (darcys)
1	1800	1703
2	----	2297
3	2938	2999
4	3232	3300

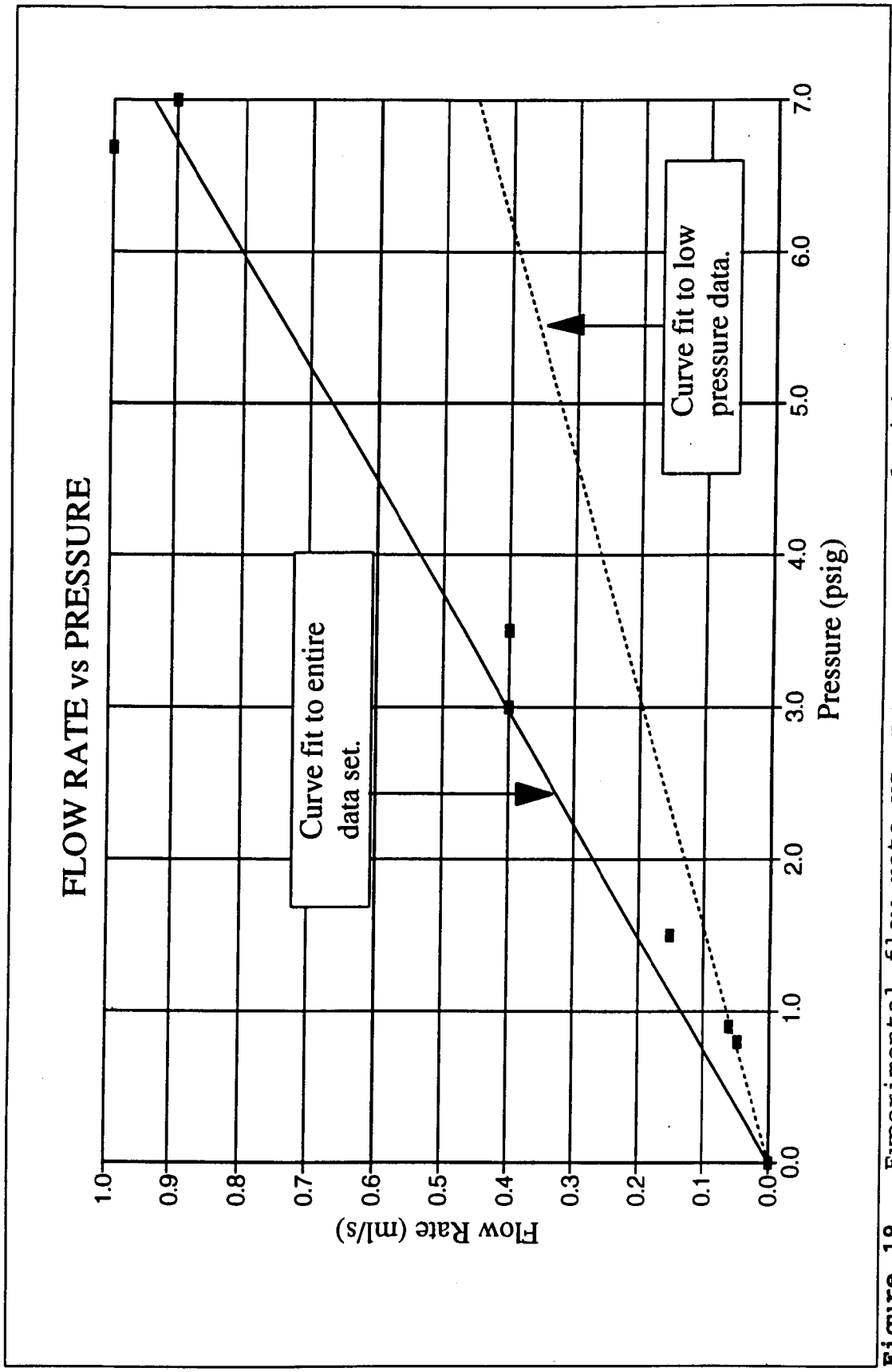

Figure 19. Experimental flow rate vs. pressure compared with Darcy's Law predictions based on curve fits to low pressure data and to the entire data set.

trend in experiments using uncatalyzed polyester, and Trevino et al. [22] showed the same behavior with a low viscosity oil. The slope of the theoretical (constant permeability) line was obtained by performing a least squares fit to the pressure-flow rate data. The permeability predicted from this line falls between the upper and the lower measured values. Table 4 shows how the permeability typically increases with flow rate for two different trails. This increase at the higher rates brought into question whether permeability is in fact a constant and whether Darcy's Law can be used to predict the resin flow in RTM with geometries of this type. Possible causes of this phenomenon were investigated, as described in the following sections.

Channeling

One possible cause of the deviation from Darcy's Law was considered to be the compression of the reinforcement, which could provide a large channel between the mat and the mold face. Trevino et al. [22] cite work done on channeling caused by mat deformation. Han et al. [16] report a study which found that mat deformation caused channeling and they were able to model this behavior. The mat compression was found to lead to channeling between the mold face and the fiber assembly. Due to the rather spongy nature of the mat used in our study, it was thought that mat compression may be

occurring in these experiments. To explore this question, a small amount of pigmented resin was added to a steady state flow of resin through saturated reinforcement. The pump was shut off just after the colored resin entered the mold for a short distance, and the part was allowed to cure. It was thought that a gradient of color would exist through the thickness if channeling along the mold faces was present. The result of this experiment did not show any color gradient. The flow seems to not only be a characteristic parabolic in-plane flow front, but uniformly distributed in the thickness direction as well. This indicates that the resin is moving throughout the thickness, and that the reinforcement is uniformly distributed through the thickness, possibly due to the low fiber volume fraction used in these experiments. Thus, channeling was effectively ruled out as a cause of the variable permeabilities.

An interesting aside is that although the general flow front shape was parabolic in the mold plane, the front was not smooth. Figure 20 shows that small branches of the colored resin (following the clear resin) seem to extend slightly ahead in certain areas, giving the front a rough appearance. This suggests that there may be local variations in the permeability from point to point, despite of the random orientation of the fibers on the average. As noted above, this is for flow of colored resin into saturated mat, as opposed to the macroflow front in unsaturated mat.

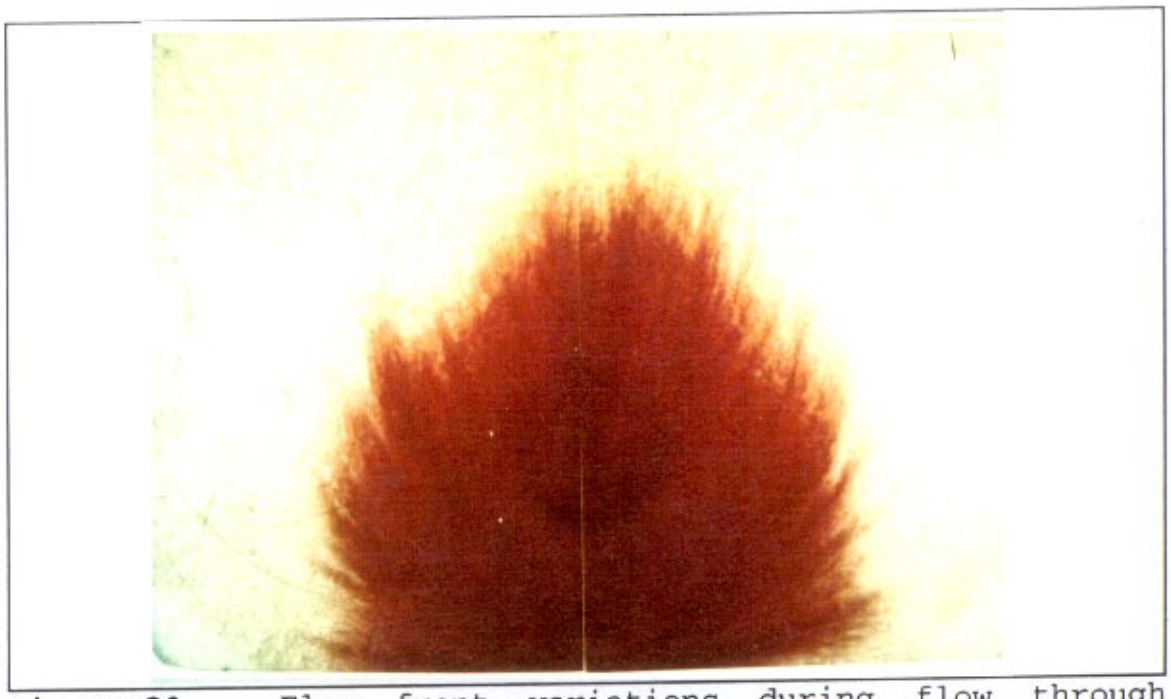

Figure 20. Flow front variations during flow through saturated reinforcement, in the plane of the mold.

Resin Characteristics

The second possible cause of the deviation from Darcy's Law was the potential non-Newtonian nature of the resin, which could change the viscosity at different shear rates. There were no data available from the literature to show how the resin behaved at different shear rates. It was originally assumed that, over the small shear rate range used here, it was Newtonian or nearly Newtonian. However, in light of the variations seen in the permeability, it was thought that the resin might be shear thinning at the higher flow rates. A

capillary rheometer was constructed and employed to determine resin behavior.

Figure 21 shows that the relationship between shear stress *versus* shear rate for uncatalyzed resin; the linearity of the relationship indicates a Newtonian behavior over the range examined. These rheometer rates produced volumetric flow rates reaching values in excess of those used in the molding experiments. The value obtained with the rheometer at 71° F was an average of 182 cP compared with the range supplied by the distributer of 400-500 cP at 77° F. In order to check the accuracy of the rheometer, a viscous, Newtonian liquid with a known viscosity (Glycerin with 96.0 % minimum glycerol) was tested. The value obtained was 628 cP at 70 °F. According to Ref.44 the viscosity of this solution should be 610 cP at 68 °F and 635 cP at 77 °F. The experimental value falls between the two standard values which verifies that the measurements made with this instrument were correct.

A related possibility is that the ongoing reaction in catalyzed resin may have an effect on the viscosity. Polymers with a molecular mass below a critical value behave in a Newtonian manner [34]. However, the addition of the MEKP starts some crosslinking of the polyester molecules immediately. This causes an increase in the molecular mass and was thought to possibly cause a non-Newtonian behavior after a certain amount of time. Resin catalyzed with 0.5%

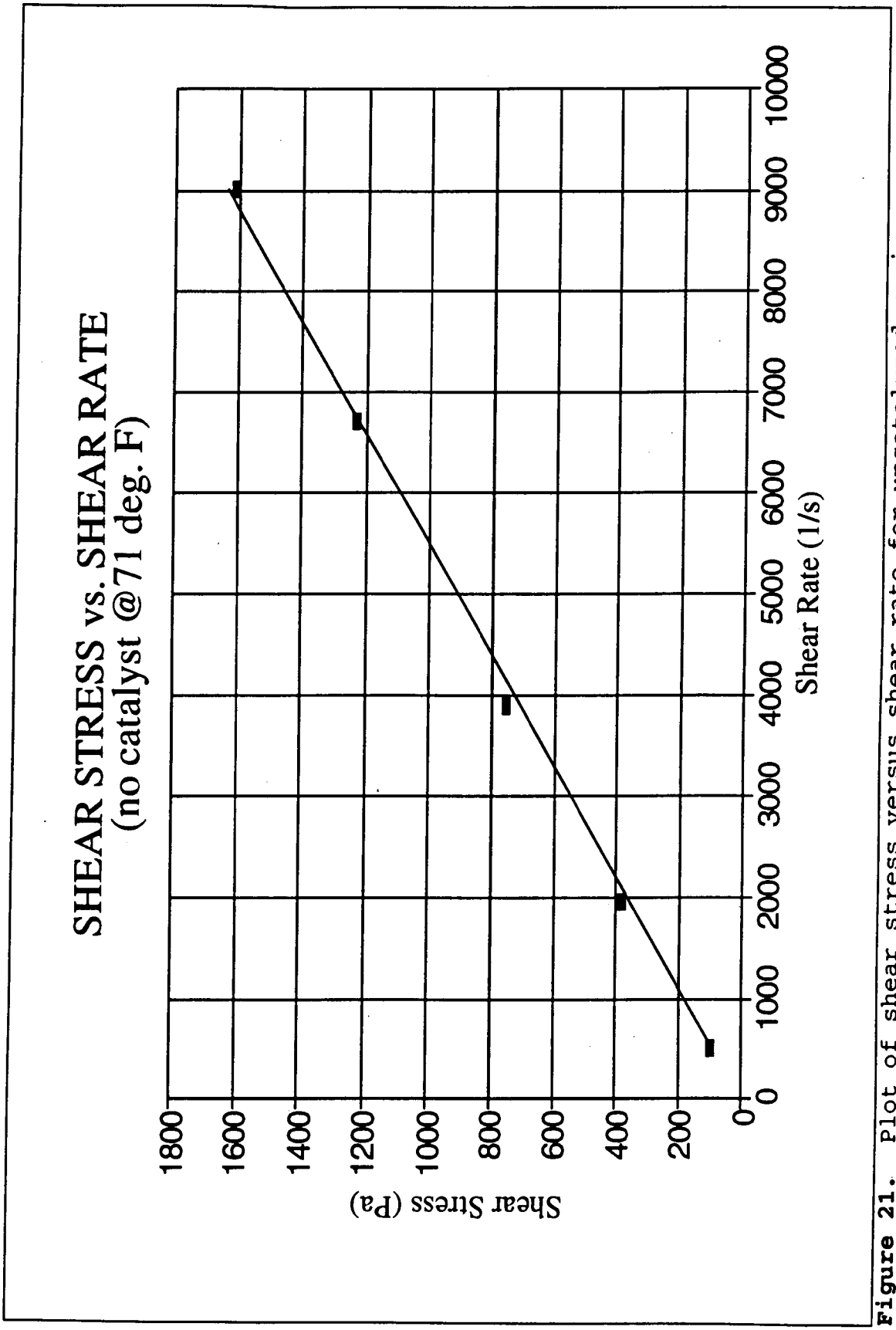

Figure 21. Plot of shear stress versus shear rate for uncatalyzed resin.

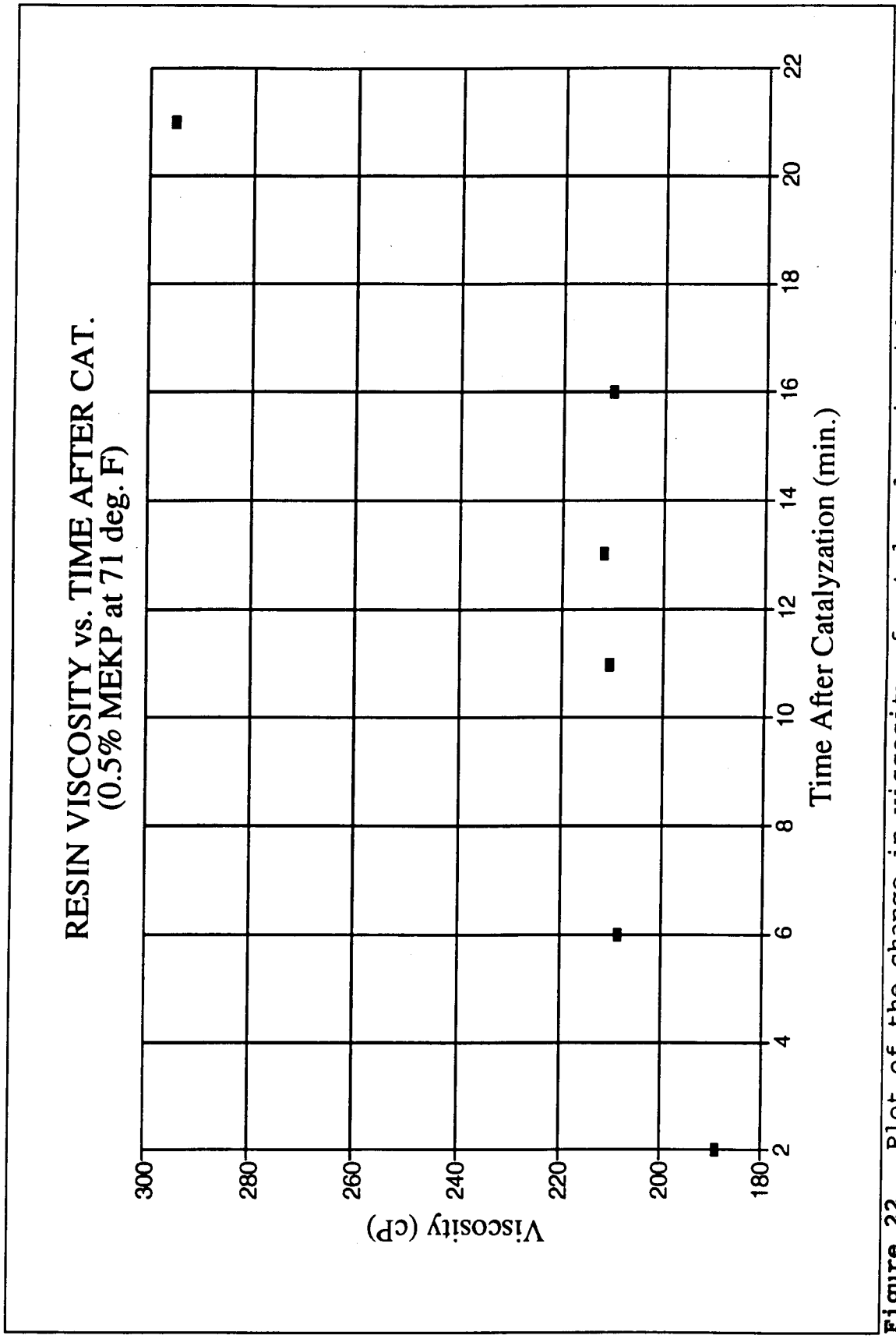

Figure 22. Plot of the change in viscosity of catalyzed resin with time.

MEKP was run through the rheometer using a constant force. The results of this show that the viscosity is approximately constant over the first sixteen minutes after the addition of the MEKP (Figure 22). This period of time is longer than the typical experiment. Thus, non-Newtonian behavior was ruled out as the cause of variation in the measured permeabilities *versus* flow rate.

Reinforcement or Mold Effects

The final possibility explored was that the permeability effect might be due to mold geometry rather than to resin/reinforcement interactions. Young et al. [7] report that other experiments have found the mold walls to have an effect on flow resistance when mold cavities are thin and reinforcement porosities are high (low fiber content). To explore this, a molding was made without any reinforcement present. Due to the low pressures needed to flow the resin, the mold was positioned vertically with the outlet at the top, so that the flow would be better controlled. The pressure exerted by the column of resin in the mold was subtracted from the transducer reading.

Figure 23 is a plot of the flow rate *versus* pressure for the neat resin case. Comparing these data to those in Figure 19, the trend of the experimental line appears similar, although the magnitude of the deviation from the theoretical line is much smaller. However, the pressures measured in this

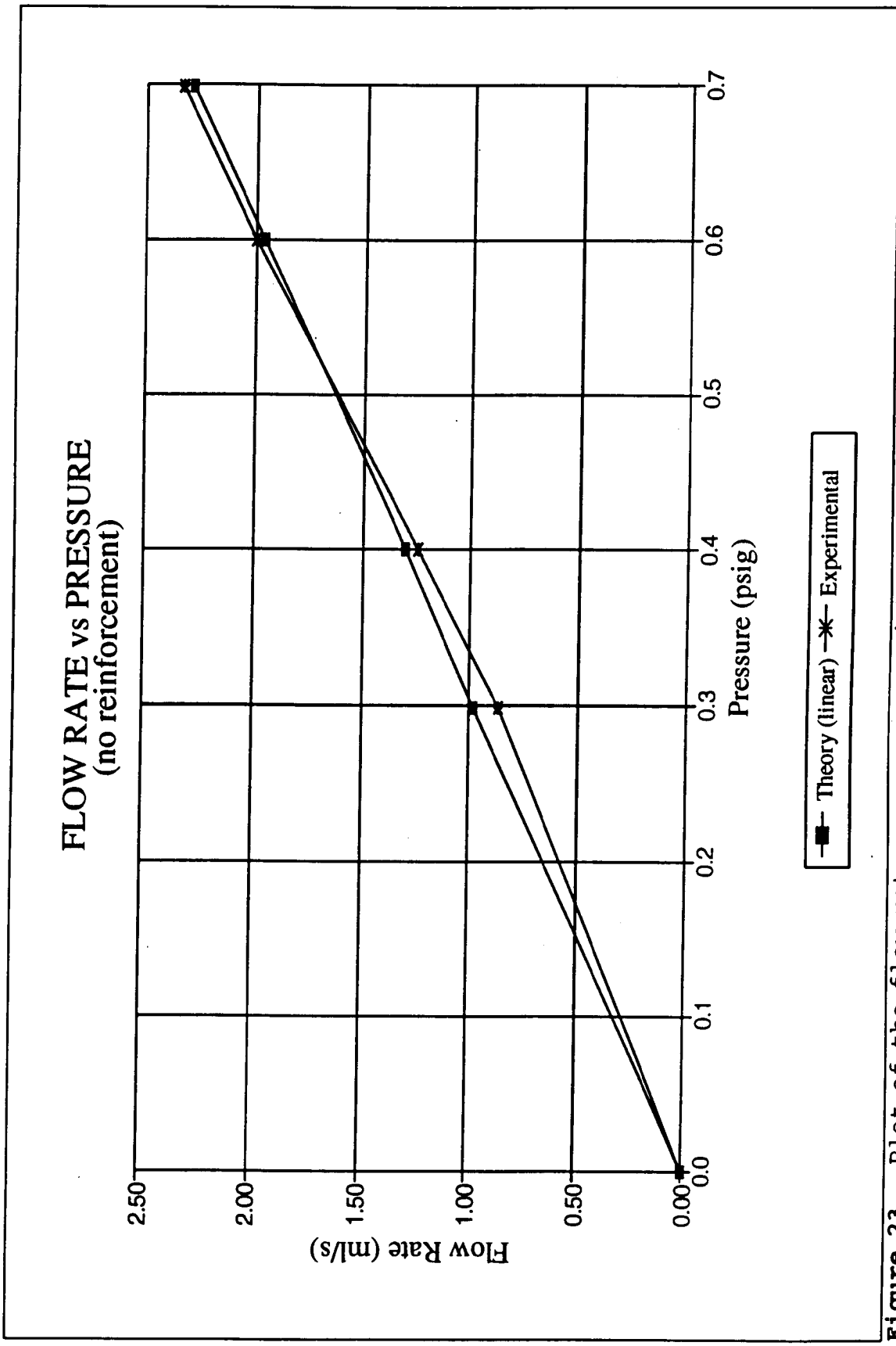

Figure 23. Plot of the flow rate vs pressure with no reinforcement present (neat resin).

case are questionable because their magnitude is less than the reported accuracy of the transducer (± 0.25 psi), which makes the amount of deviation uncertain.

The results of the neat resin case are not conclusive with regards to the contribution of the mold walls to the non-Darcian behavior observed. It can be concluded that the presence of the reinforcement significantly changes the

Table 5. Permeability at Different Flow Rates and Pressures without Reinforcement (neat resin).

Flow Rate Number	Volumetric Flow Rate (ml/sec)	Pressure (psig)	Permeability (darcys)
1	0.9 ml/sec	0.3	85414
2	1.3 ml/sec	0.4	92041
3	2.0 ml/sec	0.6	98178
4	2.33 ml/sec	0.7	98037

magnitude of the permeability (compare Tables 4 and 5), and that the non-Darcian behavior is probably not entirely due to the effect of the mold boundaries. It is apparent that when reinforcement is present, a correction factor is necessary to bring about better agreement with the experimental data. Nevertheless, error is small enough that Darcy's Law could be used to obtain an estimate of the pressure drop or the flow rate at other points within the range of collected data. However, because of the divergence of the lines outside the

measured range, it cannot be used to extrapolate these values without potential significant error (Figure 19).

The real question then becomes not whether Darcy's Law is valid, but whether it is useful. If experiments must be performed with each mold and fiber content in order even to obtain an average permeability over a given range using Darcy's Law, one may as well make the measurements at the desired level, which would give an exact value. Another aspect, is that because the mold may influence the permeability as well as the reinforcement, characterizing the permeability is only meaningful for the particular mold used. Any changes in the geometry may require remeasurement of the permeability. Thus, Darcy's Law is of no use unless it is modified to account for mold geometry.

The data reported in this section do not clearly indicate the cause of the deviation from Darcy's Law shown in Table 4, although several possibilities have been eliminated. Darcy's Law was developed large volumes and may not be applicable for thin molds.

Effect of Mold Stiffeners

Using Darcy's Law, there was a distinct difference in the values of permeability obtained with and without the stiffeners at the flow rate of 0.9 ml/sec. The addition of the angle irons has the effect of lowering the area available for flow by approximately 3% at the center of the mold. The

highest pressures measured inside the mold were 10.1 psig in the unconstrained case, and 11.7 psig in the constrained case. This is a 15.8% increase in the mold cavity pressure due to the constraints. Using the previously calculated cross-sectional areas the value of the permeability in the case of the constrained glass was 1991 darcys *versus* 2234 darcys without the stiffeners. This is an increase of 12.2% in the calculated permeability in the mold when the constraints are not used, despite accounting for the increased cross-sectional area in the calculation.

This decrease in permeability is the result of maintaining a more constant mold volume. When the glass mold face is constrained the volume remains nearly constant. If the glass is unconstrained, then it can deflect, resulting in an increase in the volume of the mold and a non-uniform distribution of resin, with a higher resin content at the inlet end and down the centerline. The increase in the volume of the mold causes the reinforcement porosity to increase which in turn causes the permeability to increase as well. Figure 17 shows the amount of centerline deflection with and without the stiffeners.

Another aspect of the study done with pigments was to look at the effect of the stiffeners on the flow of resin within the mold. The photographs of the cured parts in Figure 24 show that the red and the blue appear to extend farther down the center of the mold when the constraints were not

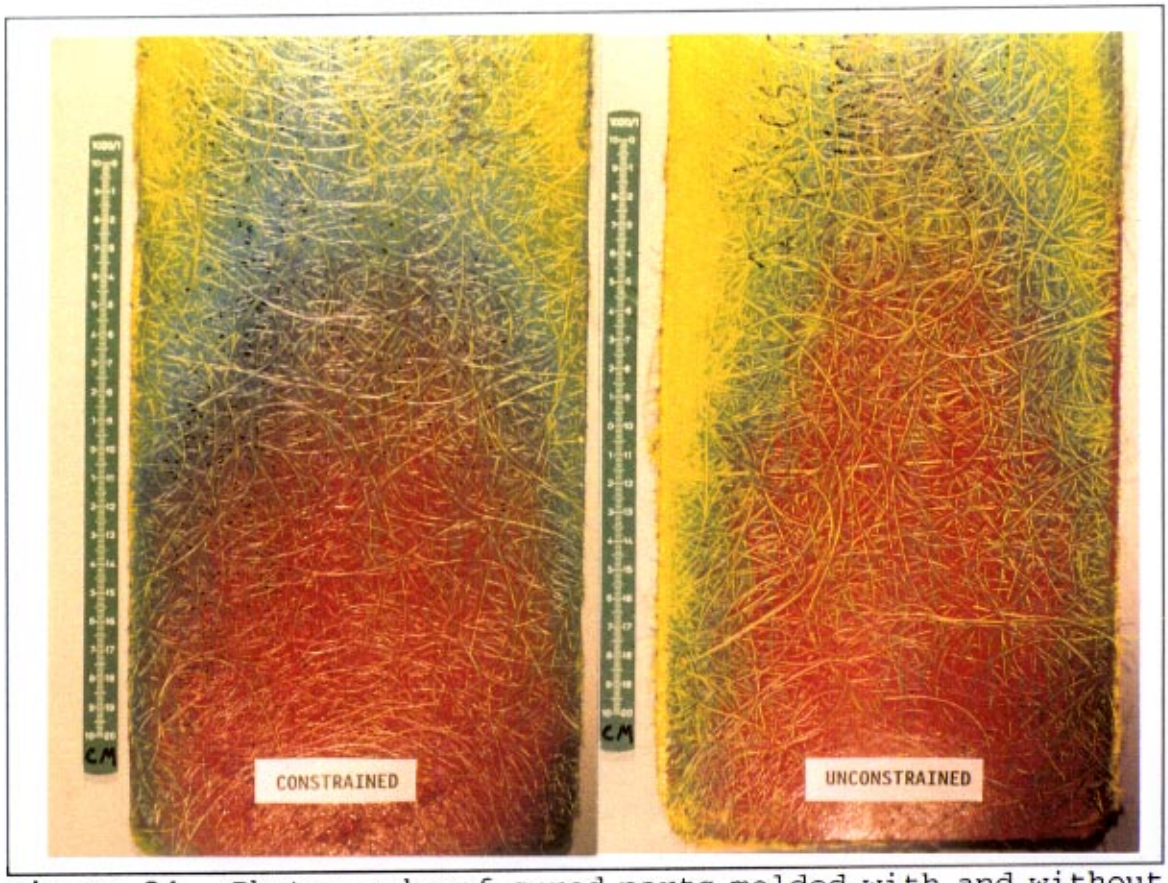

Figure 24. Photographs of cured parts molded with and without stiffeners.

used. This shows that even small deflections in the mold face can alter the resin flow pattern.

Modeling

Originally, this research was to include a model of the mold filling behavior. The model development was to be done with commercial computational fluid dynamics software, using a finite element method. The finite element method, as stated earlier, is one of the techniques used to model the RTM process. The package selected was FIDAP, written by Fluid

Dynamics International (FDI). This choice was made because of initial discussions with the staff at FDI, and because of its availability on the supercomputer at the National Center for Supercomputing Applications (NCSA) at the University of Illinois Urbana-Champaign. Several problems arose that hindered and finally prevented this model from being developed (see Appendix B).

CHAPTER SIX

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

RTM should continue to rise in popularity as a method of processing composites because of its low cost, versatility, and the high quality of molded parts. However, in order for it to move into advanced composites there will need to be a better understanding of the fundamental principles involved. This will not only improve the quality, but will also enable better models to be developed. In turn, this will help lower the costs as well as improve the overall efficiency of the process.

The findings of this study show that the permeability may be affected by mold geometry as well as the reinforcement, and Darcy's Law is of limited use in predicting the flow conditions in thin molds. Although Darcy's Law can provide a good approximation to the permeability, it can only be used for the range of pressures and flow rates measured in a given mold. Attempts to use the permeability value outside of the measured range could result in a large error.

Capillary action is by far the strongest influence on the wetting of the strands. Results of the experiments using pigments show that the first resin to flow past the strands is drawn into them and is not removed by the subsequent flow.

This is further supported by the fact that the time to saturation is independent of the pressure drop and flow rate through the mold as seen with the microflow study.

Mold deflections play a large part in the permeability of the reinforcement. Even small deflections can affect the permeability and the resin flow through the mold. When mold deflections are present, care must be taken to ensure that the excess resin is removed in order to obtain the desired dimensions. It was also found that deflections predicted using plate equations were in approximate agreement with measured deflections.

The study of porosity showed two effects. First, the location of the pores and the mechanism of their formation underwent a change between the slowest rate and the next highest. Second, the amount of porosity increased as the flow rate increased. The mechanism of large pore formation in the slowest case may be unique to this type of reinforcement. It was also found that even at very slow speeds it is not possible to totally eliminate porosity. However, porosity was low at all rates, fiber wash was very low, and the overall quality was excellent. These characteristics have also been found with other reinforcement types and mold geometries used in related studies.

Recommendations

In the past there have been significant efforts at modeling the RTM process. These models have met with reasonable success as far as predicting the mold filling behavior. There are even a number of commercial programs available which claim to aid the design of molds. The majority of this work has looked at mold filling from a macroscopic level. Therefore, it is suggested that some of the future work in RTM modeling should include the microflow.

A study of the effect of the injection rate on the porosity needs to be continued. A logical step would be to examine this effect on lay-ups made of well-aligned fibers which tend to be used more in structural composites. It is also important to explore the effect on mechanical properties of the location (i.e., within the strands or between them) and size of pores.

Another area that deserves some attention is the deformation and relaxation of the mold. An intriguing aspect of this behavior is its possible exploitation in order to lower the molding pressures, increase permeability and still maintain dimensional tolerances. Research into techniques and materials that would allow deformation that would then either regain the original shape on its own or by external means could help keep tooling and equipment costs down. It may also be possible to use the deformation to obtain certain geometries without having a matched (outer and inner) mold.

Further investigation is needed into the effects of the mold walls and the reinforcement on the accuracy of Darcy's Law. It is apparent that a correction factor is necessary to effectively determine the permeability of a given reinforcement independent of mold geometry.

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APPENDICES

APPENDIX A

Molding

The molds used at Montana State University were for the most part simple geometries. However, in the course of their development certain problems arose during this study which had to be overcome. An offshoot of this work was the development of some general guidelines which can be useful for future designs.

The first consideration is the channeling of resin in the mold. It is imperative that there not be any regions in the cavity that do not contain reinforcement. Channeling can cause uneven mold filling which can result in the waste of materials. Channeling can occur where the reinforcement is unevenly cut, and where the mold faces meet the seal.

The second consideration is the reinforcement type. Certain configurations can cause the flow to be anisotropic. This can cause the resin to flow unevenly in different directions and result in improper filling. For instance, when using unidirectional mats it is helpful to use a manifold to distribute resin uniformly along the leading edge before it begins moving downstream. Some reinforcements are not suitable for use with RTM. One type of chopped strand mat used contained a binder that was soluble in the resin. Under

flow conditions it was possible to wash the fibers down the mold.

A third consideration is the placement of the inlets and the outlets. It is important that they be positioned so that air does not become trapped in any part of the mold. Placement of the vents in corners can alleviate this problem. Some fabrics can lead to higher pressure drops than expected which may make it advantageous to inject the resin into the center of the mold instead of the end.

Care should be used when selecting mold materials. Significant mold deformations were found to occur even when seemingly stiff materials were used. This can lead to a change in the dimensions of the finished part. In some cases, if the deflection is extreme, the filling pattern can change as well. Thick mold walls or the use of stiffeners can help minimize the deflections. Dimensional changes can be lessened by allowing the vents to remain open after the pump is shut off. This will allow any excess resin to be forced out of the mold as the deflected surfaces regain their original shape. Efforts should be made to be sure that the resin does not react with any of the materials it will come in contact with, particularly the seals.

APPENDIX B

Modeling

The first problem was finding a suitable graphics interface between the computers at MSU and NCSA. This interface was necessary to review the results of the model, and had to be an X-terminal. This was solved by changing the operating system of an existing pc to UNIX, which allowed it to become an X-terminal.

The second problem was that the manuals for the version (version 6.0) on the supercomputer were not available. Manuals for the latest version (version 7.0) were purchased. There were enough differences between these two versions so as to make writing the input file very difficult.

The third problem developed when version 7.0 was finally loaded onto the supercomputer. For some reason, which neither the staff at FDI nor at NCSA understood, the graphics interface that was previously established would not work with this version. This meant that the model had to be developed using version 6.0.

The input file was sent to FDI in an effort to find why the model would not run. It was originally thought that the difficulties that came up were the result of having a poorly stated boundary condition, or other improper statement in the

input file. These problems could have come about trying to translate the version 7.0 manuals into a version 6.0 input file. Subsequent conversations with the staff at FDI however, brought to light, belatedly, the fact that neither FIDAP version 6.0 nor version 7.0, would be capable of modeling an RTM mold filling in spite of earlier conversations to the contrary. Efforts in this area were then abandoned.

APPENDIX C

Capillary Rheometer

The following equations were used to calculate the shear rate, shear stress, and the viscosity of the resin used in this experiment.

$$Shear\ Rate = \frac{4V}{\pi r^3 t} \quad \textbf{(1)}$$

$$Shear\ Stress = \frac{Fr}{2\pi R^2 L} \quad \textbf{(2)}$$

By dividing equation 6 by equation 5 the following equation is obtained which can be used to calculate the viscosity directly.

$$Viscosity = \frac{Fr^4 t}{8R^2 LV} \quad \textbf{(3)}$$

r= capillary radius (0.5 mm)
R= barrel radius (0.25 in.)
F= force
V= volume
t= time
L= capillary length (4.6 in.)
The mass of the piston is 152.2 grams.

This rheometer was designed according to ASTM Standard D3835-79. Figure 25 is a sketch of the rheometer used in this study. This device was originally intended to have an Instron 8562 apply the force to the rheometer piston. However, because this model is not hydraulic and the load cell was not sensitive enough it was unable to keep up with the movement of the rheometer piston movement during the test. Instead, a set of calibrated weights were used.

The first step is to make sure that the barrel of the rheometer is clean. The barrel is then filled to approximately 0.5 in. of the top with resin. The piston can then be inserted. Trapped air is removed by inverting the rheometer, waiting a brief period for the air to travel to the other end, and pushing the piston, thus purging the air. The rheometer can then be placed into the stand and the initial height of the piston above the base measured. It is necessary to hold the piston in place before starting the test in order to prevent it from displacing any resin. The application of the weight requires two people; one to lower the weight onto the piston and one to run the stopwatch. It is recommended that the test be allowed to run as long as possible. At the end of the test the stopwatch is stopped at the same time that the weight is removed from the piston. The height of the piston above the base is then measured and a volume of displaced resin calculated. It was found that better results were obtained between each run if the piston was cleaned with acetone and allowed to air dry.

The values of volume, time, and the applied force, which must include the weight of the piston, can be put into the above equations and the shear rate, shear stress, and the viscosity calculated. This procedure should be repeated several times at each force level in order to ensure that the results are consistent. Plotting the shear stress *versus* the shear rate will indicate whether the resin is Newtonian.

This rheometer was designed to test a variety of resins. However, if other resins to be tested have very low viscosities it will be necessary to use a smaller diameter capillary. The capillary is held in the end cap with epoxy. To remove it first remove the O-ring from the top and heat the end cap and capillary assembly in an oven to burn off the epoxy. It will be necessary to use a bushing to make the new capillary fit if the outside diameter is smaller than the original. There are a very small number of different capillary diameters available locally. The Thomas Register® is a good source of names of companies that supply glass capillaries in small quantities. If the fluid to be tested has a very low viscosity, requiring a small diameter capillary, hypodermic tubing can be used, however a new end cap would have to be manufactured in order to install it.

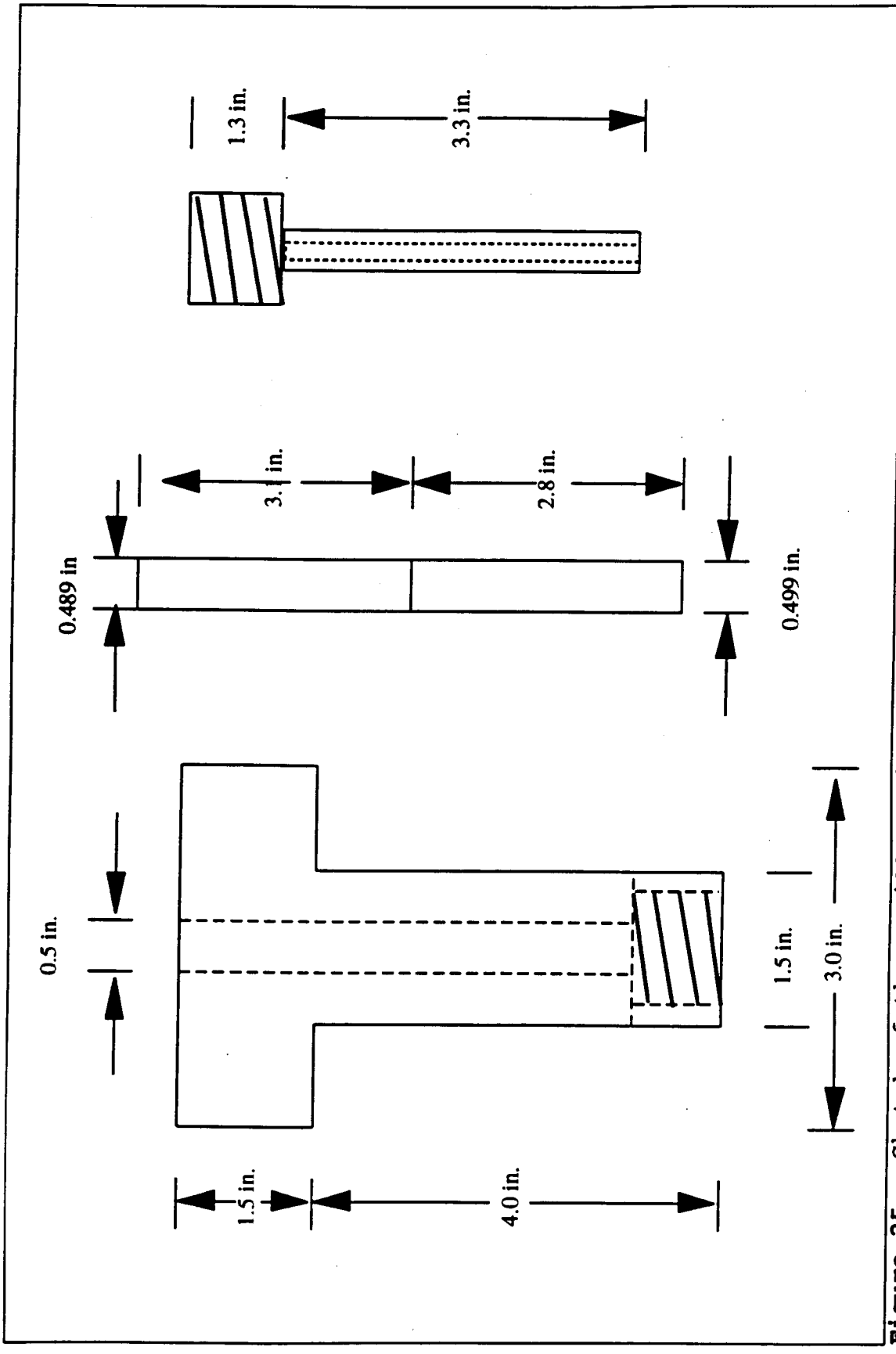

Figure 25. Sketch of the capillary rheometer.