# Resin Flow During the Cure of Fiber Reinforced Composites

GEORGE S. SPRINGER

Department of Mechanical Engineering and Applied Mechanics The University of Michigan, Ann Arbor, Michigan 48109

### **ABSTRACT**

Experiments were performed studying resin flow during the cure of fiber reinforced, organic matrix composites using a system in which the resin was simulated by viscous liquids and the fibers either by layers of thin rods or by layers of porous plates. The flow pattern was observed and the flow rate was measured for different applied pressures. The data were compared to the results of the Springer-Loos model and excellent agreement was found between the data and the model.

# INTRODUCTION

Parts made of fiber reinforced, organic matrix composites are fabricated by arranging the uncured fiber-resin mixture into the desired shape and then applying heat and pressure to the material for predetermined lengths of time. The elevated (cure) temperature applied during the cure provides the heat required for initiating and maintaining the chemical reactions inside the resin. The applied pressure provides the force needed to squeeze excess resin out of the material and to consolidate individual plies.

Since the cure temperature and cure pressure have a marked effect on the quality of the finished product they must be selected carefully for each application. Consequently, considerable efforts have been made in the past to determine the appropriate temperatures and pressures which must be applied during cure. As a result of previous studies, the influence of the applied temperature on the curing process is understood reasonably well [1-7]. Relatively little is known, however, of the effects of the applied pressure on the resin flow. Only recently has a model been presented for describing the resin flow during cure [6,7]. The validity of the hypotheses underlying the model and the accuracy of the results could not yet be assessed because of a complete lack of suit-

J. COMPOSITE MATERIALS, Vol. 16 (September 1982), p. 400 0021-9983/82/05 0400—11 \$04.50/0 ©1982 Technomic Publishing Co., Inc. able data. In this paper experimental results are presented which illustrate the mechanism of resin flow and provide means of evaluating the accuracy of analytical models. The data obtained are compared to the results of the Springer and Loos model [6,7], which is the only model presently available for calculating the resin flow. However, the data can also be used to evaluate the accuracy of analytical models which might be proposed in the future.

### THE PROBLEM

This investigation addresses the problem of curing fiber reinforced organic matrix composites made of unidirectional prepreg tape or of woven fabric. Photomicrographs of typical cross-sections of composites made of prepreg tapes are shown in Fig. 1. A porous material (referred to as "bleeder") is placed on one or on both sides of the composite (Figure 2). Restraints are mounted around the composite to prevent lateral motion and to minimize resin flow through the edges.

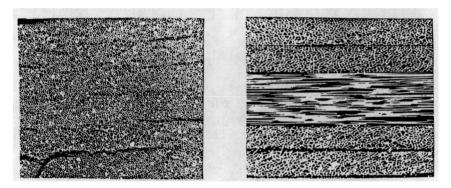


Figure 1. Photomicrographs of unidirectional and crossply composites. Left:  $[O_3J_s]$ Right:  $[O_2/90]_s$ 

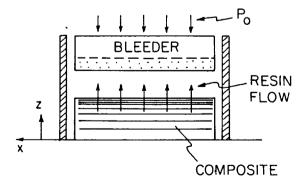


Figure 2. Illustration of the composite-bleeder system.

The cure process involves the application of heat and pressure. The applied heat increases the temperature in the composite, resulting in changes in the molecular structure of the resin and, correspondingly, in resin viscosity. When the resin viscosity has become sufficiently low, a pressure is applied to the system squeezing resin from the composite into the bleeder. This study is concerned with the latter aspect of the cure process, namely with the relationship between the applied pressure and the resin flow. Specifically, the investigation had three major objectives: 1) to observe and examine the mechanism by which resin flows through the composite, 2) to measure the flow rate, and 3) to compare the data obtained with the results of the Springer-Loos model [6,7].

Resin may flow in the directions normal (z direction, Fig. 2) and parallel (x direction) to the plane of the composite. In practice, the flow in the x direction is often negligible because the width and length are large compared to the thickness, and because of the restraints placed around the composite. Some resin may flow through the edges but, in general, the region affected by this lateral flow is small and is confined to the vicinity of the edges. Therefore, only those problems are considered here where the resin flow is restricted to the z direction.

# **EXPERIMENTAL APPARATUS**

During the cure of a composite the viscosity (and hence the resin flow) changes with time due to changes in temperature. In order to eliminate the effects of temperature and to observe the flow pattern, tests were performed with a system simulating composites. The schematic of the apparatus is shown in Fig. 3.

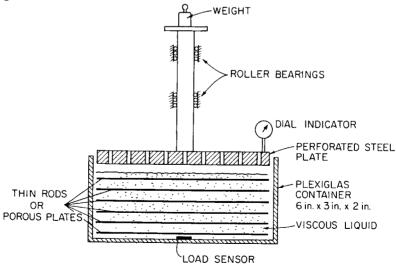


Figure 3. Schematic of the apparatus.

A liquid of known viscosity was placed in a 6 in. long, 3 in. wide, and 2 in. deep Plexiglas container. Two types of liquids were used. One of the liquids was an oil having a viscosity of 16000 cps, the other one was a viscosity standard having a viscosity of 1257600 cps. The characteristics of both liquids were such that they exhibited Newtonian behavior under the shear rates applied in the tests.

Either 3/32 in. diameter plastic rods or 0.068 in. thick perforated fiberboard plates (referred to as "porous plates") were placed in the liquid. The rods were arranged in six parallel layers, the layers being 0.09 in. apart. The horizontal distance between two adjacent rods was approximately 0.1 in. The porous plates (each containing one hundred 0.042 in. diameter holes per square inch) were also arranged in six parallel layers, with a distance of 0.09 in. between each plate. A 1/4 in. steel plate (containing twenty four 1/8 in. diameter holes per square inch) was placed on top of the liquid. The plate was connected by a 1/2 in. diameter steel shaft to a platform on which weights could be placed. The alignment and the motion of the steel plate were controlled by two low friction roller bearings.

During each test a known weight was applied and the vertical displacement of the steel plate as a function of time was monitored using a dial indicator and a stop watch. In addition, the motion of the rods (or plates) was observed. In selected tests the pressure was also measured with a small (0.156 in. diameter and 0.03 in. thick) strain gauge load sensor mounted on the inside bottom surface of the Plexiglas container.

# FLOW PATTERN

The flow pattern was observed in the apparatus illustrated in Fig. 3. As described in the previous section, a force (pressure) was applied to the viscous liquid which contained layers of either thin rods or porous plates. Upon application of the force the first (top) layer (n = 1, Fig. 4) moved toward the second (n = 2), while liquid was squeezed out from the space between the two layers. This liquid seeped through the rods (or through the holes in the porous plate) of the first layer. When the rods (or plates) in the first layer reached the rods (or plates) in the second layer, the two layers moved together toward the third layer, squeezing resin out of the space between the second and third layers. This sequence was repeated for the subsequent layers. Thus the interaction of the layers proceeded in a wavelike manner (Fig. 4).

The wavelike motion described above was observed clearly both with rods and with porous plates in the liquid. A series of photographs illustrating the motion is given in Fig. 5. Initially, the porous plates were separated by an equal distance (Fig. 5a). After the load was applied the first plate near the top moved downward. A plate further down started moving only when the plate above reached it (Figs. 5b-5d).

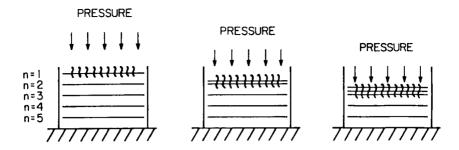


Figure 4. Illustration of the flow during pressure application.

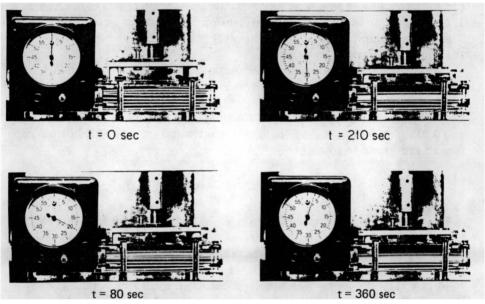


Figure 5. Photographs showing the sequence in which porous plates immersed in a liquid move.

The sequence described above would also occur during the cure of a fiber reinforced composite. The fibers in each ply in the composite will move in unison toward the adjacent ply in the same manner as the layers of rods or porous plates moved in the present tests (Figs. 4,5). Some compacting of the fibers may occur but, in general, this effect will be small.

The foregoing observation shows that during the curing process the resin content in the composite does not vary with position continuously. Instead, the resin is distributed in a nearly discrete manner, with the resin content hav-

ing either a minimum or a maximum value. The resin content is a minimum in those plies across which resin flow takes place. The resin content is maximum (having the same value as in the uncured composite) in those plies across which there is no resin flow. As a consequence, near the center of the composite the plies will be resin rich, unless the appropriate pressure is applied for an appropriate length of time.

The observations described above also imply that there is a pressure drop only across those layers through which resin flow takes place. The pressure is constant (and is equal to the applied pressure  $P_0$ ) across all the other layers. This was confirmed by pressure measurements made on the bottom of the liquid containing layers of either rods or plates. As expected the pressure on the bottom was always the same as the applied pressure. This result is illustrated by the typical set of data given in Fig. 6.

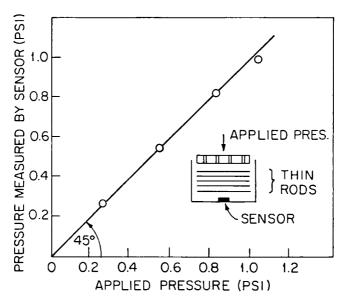


Figure 6. Comparison of the pressure measured by a sensor placed on the bottom of the liquid and the applied pressure.

# FLOW RATE

A model of the resin flow through composites was presented by Springer and Loos [6,7] by taking into account the effects of both the applied temperature and pressure. This model is based on the observation that both the composite and the bleeder resemble porous media. In the case of the

bleeder this resemblance is evident. There is also a strong resemblance between the cross section of the composite and a porous medium, as illustrated in Fig. 1. Accordingly, Springer and Loos applied the expressions developed for quasi-steady, seeping flow through porous media to the composite-bleeder system. Springer and Loos' analysis is not repeated here in detail. In the Appendix the analysis is summarized briefly, and is adapted to the present tests in which the composite consisted of layers of either thin rods or porous plates immersed in a constant viscosity liquid. According to the Springer-Loos model the liquid flow rate out of this composite is (see Appendix)

$$\frac{d(hA)}{dt} = \frac{-1}{n + \frac{S_c}{S_b} \frac{h_b}{h_1}} \left( \frac{S_c F}{\mu h_1} \right)$$
 (1)

where h is the height of the liquid surface at time t, A is the cross sectional area of the composite perpendicular to the direction of the flow,  $S_c$  is the permeability of a single layer of rods or of a porous plate,  $S_b$  and  $h_b$  are the permeability and the thickness of the perforated steel plate above the liquid,  $h_1$  is either the diameter of a rod or the thickness of a porous plate,  $\mu$  is the liquid viscosity, and F is the applied force (Fig. 7). The parameter n is the number of

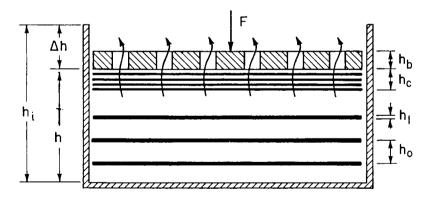


Figure 7. Description of the symbols used in the analysis.

layers through which resin flow takes place. By integrating eq. (1) and by dividing both sides of the resulting expression by  $h_0$  the following expression is obtained for the change in height of the liquid surface

$$\frac{\Delta h}{h_o} = \frac{h_i - h}{h_o} = \frac{1}{n + \frac{S_c}{S_h} \frac{h_b}{h_1}} t^*$$
 (2)

where  $h_i$  and  $h_0$  are, respectively, the height of the surface and the distance between two adjacent layers of rods or porous plates before the load is applied (time t=0).  $t^*$  is a dimensionless time

$$t^* \equiv \frac{S_c F}{\mu h_1 h_0 A} t \tag{3}$$

Equation (2) provides the relationship between the displacement of the liquid surface (which is proportional to the volume of liquid squeezed out of the composite, eq. 1) and the elapsed time. This expression is now in a form which is suitable for comparison with the data obtained in this investigation. Note, that the displacement  $\Delta h$  varies linearly with time. The slope of the line changes, however, as subsequent layers come into contact and, consequently, as the parameter n increases. For the geometry of the present tests n is given by

$$n = I \left( 1 + \frac{\Delta h}{h_o} \right) \tag{4}$$

where I means the integer of the number in the parentheses.

In order to apply eq. (2) the permeabilities  $S_b$  and  $S_c$  must be known. These permeabilities were determined experimentally as follows. First, using a known weight (force) the perforated steel plate was forced through the liquid with neither rods nor porous plates being present in the liquid, and the change in height  $\Delta h$  was measured as a function of time. The results of these measurements were substituted into eq. (2), providing the value of  $S_b = 8.4 \text{ x}$   $10^{-5}$  in<sup>2</sup>. The procedure was repeated with either a single layer of rods or a single porous plate in the liquid resulting in an  $S_c$  value of  $1.6 \times 10^{-4}$  in<sup>2</sup> for each layer of rods and  $5.6 \times 10^{-6}$  in<sup>2</sup> for each porous plate.

The displacement  $\Delta h$  as a function of time was measured both with layers of rods and with layers of porous plates in the liquid. The measurements were repeated with different applied weights (forces). The results are shown in Figs. 8 and 9. In these figures the results of the model (eq. 2) are also included. As can be seen the agreement between the data and the analytical result is excellent. This agreement creates confidence in the model and shows that the model can be used to calculate the flow rate within high accuracy.

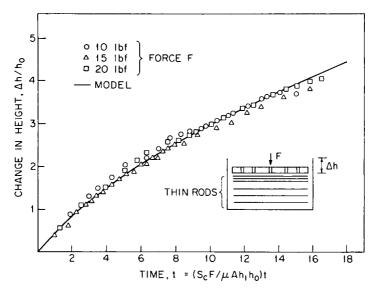


Figure 8. Comparison of the data with the model. Liquid containing layers of thin rods.

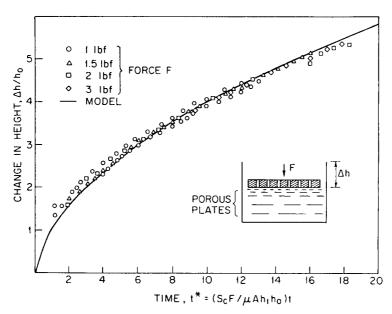


Figure 9. Comparison of the data with the model. Liquid containing layers of porous plates.

### **ACKNOWLEDGEMENTS**

The author is grateful to Messrs. B. Bober and B. Knight for their help in the tests. This work was supported by the U.S. Air Force Systems Command Materials Laboratory, Wright-Patterson Air Force Base, Dayton, Ohio with Dr. S. W. Tsai acting as project engineer.

# REFERENCES

- Progelhof, R. C. and Throne, J. L., "Non-Isothermal Curing of Reactive Plastics," Polymer Engineering and Science, Vol. 15, 1975, pp. 690-695.
- Broyer, E. and Macosko, C. W., "Heat Transfer and Curing in Polymer Reaction Molding," AIChE Journal, Vol. 22, 1976, pp. 268-276.
- 3. Adabbo, H. E., Rojas, A. J., and Williams, R. J. J., "Critical Parameters for Thermoset Curing in Heated Molds," *Polymer Engineering and Science*, Vol. 19, 1979, pp. 835-840.
- 4. Stolin, A. N., Merzhanov, A. G., and Malkin, A. Ya., "Non-Isothermal Phenomena in Polymer Engineering and Science: A Review. Part 1: Non-Isothermal Polymerization," *Polymer Engineering and Science*, Vol. 19, 1979, pp. 1065-1073.
- Barone, M. R. and Caulk, D. A., "The Effect of Deformation and Thermoset Cure on Heat Conduction in a Chopped-Fiber Reinforced Polyester During Compression Molding," International Journal of Heat and Mass Transfer, Vol. 22, 1979, pp. 1021-1032.
- Loos, A. C. and Springer, G. S., "Calculation of Cure Process Variables During Cure of Graphite-Epoxy Composites," Composite Materials: Quality Assurance and Processing. C.E. Browning, Ed. ASTM STP 787, 1983, pp. 110-118.
- Springer, G. S., "A Model of the Curing Process of Epoxy Matrix Composites," Progress in Science and Engineering of Composites, T. Hayashi, K. Kawata, and S. Umekawa Eds. Japan Society of Composite Materials, 1982, pp. 23-35.

# APPENDIX

# CALCULATION OF THE FLOW RATE

In this appendix the Springer-Loos model is adapted to the conditions of the present tests. At any instant of time the liquid velocity in the bleeder  $V_b$  and in the composite  $V_c$  are given by Darcy's law

$$V = -\frac{S}{\mu} \frac{dP}{dz}$$
 (A.1)

For a constant viscosity liquid eq. (A.1) may be integrated to yield

$$V_c = \frac{S_c}{\mu} \frac{P_o - P_u}{h_c}$$
 (A.2)

$$V_{b} = \frac{S_{b}}{\mu} \frac{P_{u} - P_{b}}{h_{b}}$$
 (A.3)

where  $P_u$  and  $P_b$  are the pressures at the composite-bleeder interface and in the bleeder, respectively,  $h_b$  is the instantaneous thickness of the liquid in the bleeder, and h is the thickness of the resin starved layer, i.e., the thickness of the layer through which resin flow takes place (Fig. 7)

$$h_c = nh_1 \tag{A.4}$$

All other symbols were defined in the main text. In the apparatus employed in this investigation  $P_b$  is equal to the atmospheric pressure ( $P_b = P_a$ ),  $h_b$  is the thickness of the steel plate above the liquid, and  $h_1$  is either the diameter of a rod or the thickness of a single porous plate.

The equation of continuity gives the rate of change of volume of the composite

$$\frac{d(hA)}{dt} = -AV_c = -AV_b \tag{A.5}$$

The second equality expresses the fact that, at any instant of time, the flow out of the composite is equal to the flow into the bleeder. The pressure  $P_0$  is related to the applied force by the expression:

$$P_o = \frac{F}{A} + P_a \tag{A.6}$$

By combining eqs. (A.2) to (A.6) the following expression is obtained for the flow rate.

$$\frac{d(hA)}{dt} = \frac{-1}{n + \frac{S_c}{S_b} \frac{h_b}{h_1}} \left( \frac{S_c F}{\mu h_1} \right)$$
 (A.7)