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RESIN TRANSFER MOLDING OF TEXTILE PREFORMS FOR AIRCRAFT STRUCTURAL APPLICATIONS

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INTRODUCTION

The NASA Langley Research Center is conducting and supporting research to develop cost-effective fabrication methods that are applicable to primary composite aircraft structures. One of the most promising fabrication methods that has evolved is resin transfer molding (RTM) of dry textile material forms. RTM has been used for many years for secondary structures, but has received increased emphasis because it is an excellent method for applying resin to damage-tolerant textile preforms at low cost. Textile preforms based on processes such as weaving, braiding, knitting, stitching, and combinations of these have been shown to offer significant improvements in damage tolerance compared to laminated tape composites. The use of low-cost resins combined with textile preforms could provide a major breakthrough in achieving cost-effective composite aircraft structures. RTM uses resin in its lowest cost form, and storage and spoilage costs are minimal. Near net shape textile preforms are expected to be cost-effective because automated machines can be used to produce the preforms, post-cure operations such as machining and fastening are minimized, and material scrap rate may be reduced in comparison with traditional prepreg molding.

Successful RTM is dependent upon many factors, including tooling approaches, resin characteristics, and textile preform architecture. Location of resin injection ports in the mold, resin viscosity variation with time and temperature, and compaction and permeability characteristics of preforms are all important factors that must be understood. Many of the RTM processes used in the past were developed using trial and error methods. Low fiber volume fraction (50 percent or less) secondary structures using fiberglass mats are readily molded by RTM. However, aircraft-quality primary structures have stringent structural requirements and processing conditions, necessitating a science-based approach to RTM process development. The purpose of this paper is to discuss experimental and analytical techniques that are under development at NASA Langley to aid the engineer in developing RTM processes for airframe structural elements. Included are experimental techniques to characterize preform and resin behavior and analytical methods that have been developed to predict resin flow and cure kinetics.

The NASA Langley RTM development team includes in-house staff devoted to process development, element fabrication, and composite material characterization; Virginia Polytechnic Institute and State University performing preform characterization and analytical modeling; the College of William and Mary developing resin characterization data and sensor systems; and industrial contractors (Douglas, Boeing, Lockheed, and Grumman) involved in process development, tooling studies, and subcomponent fabrication.

RESIN TRANSFER MOLDING

The term RTM is often used rather loosely to include several processes such as vacuum/pressure infiltration, resin film infusion, and liquid pressure injection. In all cases, liquid resin infiltrates a dry preform, air is evacuated, and heat is applied to cure the composite. Several different types of tooling concepts have been developed to accommodate RTM variations. The preform may be placed in a matched cavity mold or on a single-sided mold and covered with a flexible membrane. Based on the characteristics of the resin (liquid or solid at room temperature, short or long gel time), infiltration may occur isothermally or during heat-up. Pressure used to compact the preform may also drive the flow of resin, or the compaction and injection pressures may be controlled separately.

Figure 1 illustrates the process (vacuum/pressure infiltration) used at NASA Langley to fabricate panels from preforms having through-the-thickness reinforcement using hot melt epoxy resins. Flow occurs through the thickness of the preform as the resin melts and pressure is applied in a heated press. A tight fit is required between the preform and mold cavity to avoid a resin leak path around the preform. The pressure performs two functions: debulking the preform, and forcing the resin into the preform.

Figure 2 shows a typical matched cavity process employing pressure injection of resin. Flow occurs in the plane of the preform. Resin is contained in an external tank, which may be heated to lower the viscosity. Compaction pressure is used to close the mold and debulk the preform and is independent of resin injection pressure.

Some versions of RTM employ a combination of features of the two processes shown, leading to many variations of mold construction and process parameters. Analytical methods are needed to aid the mold designer and process engineer to minimize the waste of time and material. Successful modeling requires data on preform and resin processing properties; however, most of these data are not supplied by the preform or resin manufacturers. NASA Langley has sponsored the development of characterization methods for constituent materials and analytical models describing the RTM process. Recent results of this research are discussed herein.

Preform Behavior

Textile preforms are deformable and porous in their dry state. The amount of rigidity and permeability is related to the fiber architecture. Several preform architectures are illustrated in figure 3. Another factor affecting preform rigidity and permeability is the size and type of the constituent fibers. High modulus, brittle fibers impose limits on the amount of crimp and tightness of the preform as it is produced. When compressed in the thickness direction, preforms exhibit nonlinear behavior as shown in figure 4. The reasons for the nonlinearity are that fibers deform by bending, behaving as constrained beams, by buckling, behaving as constrained columns, and also slide past each other with variable friction effects.

Compaction characteristics of preforms are quantified by mounting a sample between rigid plates, applying a compaction load and measuring the resulting thickness as shown in figure 4. Data are plotted in terms of fiber volume fraction, which is directly related to thickness, against pressure in figure 5. As indicated in the figure, none of the preforms meets the typical goal of 60 percent fiber volume in the uncompacted condition. Stitching helps to compact the Hexcel[1] preform, but it still falls short of the goal. In all cases shown, pressure in excess of vacuum (14.7 psi) must be applied to achieve the desired compaction.

Permeability is also a nonlinear function of preform compaction pressure. Pores exist between fiber bundles (tows) and at the tow intersections in the preform. If a preform is visualized as a collection of planar pores, the pressure drop at a fixed volumetric flow rate is proportional to the third power of pore height (ref. 1), which is proportional to fiber volume fraction. In addition, pores may change shape or close off entirely as compaction pressure and fiber volume fraction increase. Pores formed by aligned tows form a fairly smooth flow path, whereas pores formed by crossed tows create a much more tortuous path for resin flow as shown in figure 6.

[1]The use of trademarks of names of manufacturers in this paper does not constitute an official endorsement, either expressed or implied, of such products or manufacturers by the National Aeronautics and Space Administration.

Permeability is measured in a specially designed fixture as shown in figure 7. The sample is placed in a sealed chamber of variable height. The chamber is configured to allow flow in only one of the three primary directions at a time. Fluid of a known viscosity is pumped through the preform, and the resulting pressure drop is recorded. Permeability is calculated by multiplying flow rate, viscosity, and flow length and dividing by the product of flow pressure and area. This test is repeated at several preform thicknesses. Results are shown in figure 8 for a stitched and unstitched quasi-isotropic uniweave preform. Two effects are significant: permeability is much higher in-plane than through-thickness, and the presence of stitches affects flow differently in the two directions. The stitches create channels through the thickness of the preform which increase permeability in that direction. However, they also form a partial barrier to flow in the plane of the preform, thereby reducing in-plane permeability.

Permeability information is useful in process development at a number of levels. Molds are sometimes designed so that infiltration occurs below the target fiber volume fraction of 60 percent so that permeability is high; then full compaction is applied mechanically prior to gelation to squeeze out excess resin. However, this procedure is difficult for complex shapes. Another technique is to take advantage of thermal expansion differences between mold components to achieve the same effect. For example, silicone blocks placed inside an aluminum mold will expand and compact the preform as temperature increases. Injection occurs at moderate temperature and low fiber volume fraction. The final fiber volume fraction is attained at the higher cure temperature.

Mold design criteria can be partly established by consideration of permeability variation over a range of fiber volume fractions. Mold cavity machining tolerances and rigidity determine the uniformity of permeability of the preform. Figure 9 shows the permeability variation of 16 plies of 8 harness satin, IM7 graphite fabric. The nominal cavity thickness required for a 60 percent fiber volume is 0.264 inches. If this dimension is allowed to vary by ± 0.005 inches, the fiber volume ranges from 59 to 61 percent. If the dimension varies by $\pm .020$ inches, the fiber volume ranges from 55 to 65 percent. The corresponding permeabilities are affected to a far greater degree than the fiber volume fractions. The nominal permeability, in units of 10^{-10} in^2, is 6.0. Permeability variation with the small tolerance ranges from 5.0 to 7.4. However, the variation with the large tolerance is 2.0 to 15.6, a range of more than twice the nominal value, which will most likely cause problems during resin infiltration.

<u>Resin Characterization</u>

Resins are primarily characterized by viscosity measurements under both isothermal and increasing temperature conditions. Since viscosity behavior is affected by the existing degree of resin cure, this parameter must also be quantified. The degree of cure α of a resin varies with time and temperature and is measured by differential scanning calorimetry (DSC), which records heat of reaction during time. This test is run under both isothermal and rising temperature conditions.

Resin viscosity can range from 10 cp to 10^{15} cp before molecular forces restrict fluidity and elastic behavior sets in (ref. 2). Mold design features and process parameters must be selected to account for this behavior so that a fully impregnated void-free structure is produced. Examples of viscosity behavior are shown in figure 10, which shows viscosity as a function of time for two epoxies, each at two selected temperatures. An important point is that these data are for freshly formulated resins with no impurities; viscosity may be drastically affected by improper storage or formulation, and its dependence on time at temperature can vary substantially.

For RTM, it is desirable to tailor a mold and process window for minimum viscosity for the longest time. A relatively viscous resin such as 3501-6 (see figure 10) is recommended for a through-thickness RTM process only. A resin with low viscosity and short gel time such as

E905L can be used for in-plane injection, but would require multiple inlet ports to rapidly fill large molds. The E905L has acceptable pot life as long as the temperature is carefully controlled. Other resins that have been evaluated by NASA Langley are listed in figure 11. Difficulties arise when a process is optimized for a specific resin, but a decision is made to alter the resin formulation or switch to an entirely new resin. Such changes may not require mold redesign in the case of a prepreg material, but may require an existing RTM mold to be discarded in favor of a new design. Adjusting the process parameters may, however, in some cases allow continued use of the existing mold. These examples highlight the need for a predictive model of resin behavior if high-quality, defect-free composites are to be fabricated.

Tackifiers

In some cases, textile processes yield a nearly net-shaped preform, such as braiding directly over a mandrel which is part of a RTM mold. However, in most cases, the textile product is supplied on a roll. The composite fabricator unrolls the material and cuts patterns that are assembled into the final three-dimensional preform. For complex shapes, the assembly process must be aided either mechanically, such as by stitching, or by bonding with tackifying agents as described below. Figure 12 illustrates a preform for a sine spar. Several plies of a multiaxial warp knit fabric were assembled using a tackifier and a shaping mold.

Another reason for using these tackifier preforming aids is to debulk the preform. As shown previously, even high density stitching does not fully debulk preforms. Tackifiers applied only to surfaces of plies do not fully debulk the entire thickness, as shown schematically in figure 13. The only ways to fully debulk a preform are by distributing a tackifier through the entire thickness (as by using powder-coated tows for the weaving process), by maintaining in-plane tension (as when braiding over a mandrel), or upon closing the mold. Considerable effort sometimes is needed to devise methods of closing molds without pinching or wrinkling preforms. The proper use of a tackifier would simplify the mold design.

Several types of tackifiers are listed in figure 13. They are available in a wide range of application temperatures and physical forms, from room temperature sprays to higher temperature scrims. Some are thermoplastics that remain as a discrete phase in the composite after cure, while others are formulated to dissolve in the matrix resin during infiltration. In any case, two criteria must be met: they must have minimal impact on resin infiltration, and they must have minimal impact on mechanical properties.

The effects of one type of tackifier (a polyamide scrim interleaved between each ply) on processing and mechanical properties of an eight harness satin fabric are shown in figure 14. Both the compaction pressure and the permeability at 60 percent fiber volume are very similar both with and without the tackifier. There is also little difference in room temperature static compression strength.

SCIENCE-BASED RTM PROCESSING

RTM Process Modeling

The approach in developing a process model for RTM has been to derive expressions for one-dimensional flow including all relevant physical effects, verify the model experimentally, then expand to complex geometries.

The flow of fluid through porous media obeys Darcy's law:

$$Q = \left(\frac{K}{\mu}\right) \frac{\Delta P}{X} A \tag{1}$$

where Q is the volumetric flow rate, K is the preform permeability, μ is the resin viscosity, ΔP is the pressure difference between the resin source and the flow front over a distance X, and A is the area normal to the direction of flow. This is a simple equation, but complexity arises because K varies with fiber volume fraction V_F and fiber orientation; μ varies with temperature T, time t and degree of cure α; and complex geometries include the Y and Z dimensions in which A may also vary.

For a simple isothermal one-dimensional case with a nonreacting resin and homogeneous incompressible preform, infiltration pressure and mold filling time can be calculated by hand using equation (1). However, the vacuum/pressure through-thickness process involves the simultaneous interaction of all of the parameters in both time and space. A computer is required to expedite the solution.

A computer model of the RTM process has been under development at Virginia Polytechnic Institute and State University. Basic features of this model are shown schematically in figure 15. The user specifies the process conditions and mold configuration. The main program calls up subroutines that contain data for the specific preform and resin. The program outputs values of temperature, viscosity, position of flow front, degree of cure, fiber volume fraction, and thickness.

Details concerning the calculations performed in the main program are described in reference 3. Compaction behavior is modeled with a logarithmic expansion which requires four constants to be determined experimentally. Permeability is related to fiber volume fraction using the Kozeny-Carman or Gebart equations, each of which requires an experimentally-determined constant. Resin viscosity is modeled using either an exponential or a William-Landel-Ferry expression, which relate viscosity to temperature and degree of cure. Material properties for these expressions are determined from viscosity and DSC tests. The degree of cure of a given resin is related to heat of reaction and can be modeled with exponential equations relating time and temperature history. The form of these equations can be very different for various resins, and considerable judgement and analysis of DSC information are required for accurate modeling. Once the forms of the degree of cure equations are selected, several material constants must be determined from DSC data.

Transient conductive heat flow is a major factor in the through-the-thickness process with hot melt resins. The heat flow model includes the change in thermal conductivity as resin infiltrates the preform and accounts for heat generated by the resin cure reaction. Convective heat transfer between the preform and slowly moving resin is neglected. Material thermal properties are taken from published sources. Heat generation from the resin reaction is determined from the degree of cure calculation.

The flow front position is calculated using a finite element procedure in discrete time increments. Darcy's Law is applied to each element between the resin source and the flow front, using the instantaneous values of permeability and viscosity for each element.

Computing requirements are related to the dimensions of the problem as indicated in figure 15. The one-dimensional model will run on a personal computer in 1/2 hour or less. However, the two- and three-dimensional models require larger computers and more time. The added complexity of higher order models arises because of the orthotropic permeability of preforms and the need to calculate flow front position in multiple directions.

RTM Process Monitoring and Control

The College of William and Mary has been developing a sensing system for the cure state of resins based on dielectric measurements. This system, termed Frequency-Dependent Electromagnetic Sensing (FDEMS), figure 16, utilizes a thin (0.003 inch) sensor consisting of interdigitated electrodes mounted on a substrate. The sensor is excited by an alternating electric current at various frequencies, which in turn causes molecular vibration in the resin. The molecular action changes as the reaction proceeds, which alters the electrical conductivity and capacitance of the resin. FDEMS correlates electrical conductivity and capacitance to resin viscosity and degree of cure. This system can be applied to any of the RTM process variations to detect the presence or absence of resin, local viscosity, and degree of cure.

Another use of FDEMS is to enable process control based on sensing the direct process variables (viscosity, degree of cure) in real time, as opposed to the traditional method of controlling indirect variables (mold temperature, time) per a predefined cycle. This procedure is illustrated in figure 17. Panels have been made with the through-the-thickness infusion process under direct control of temperature by the FDEMS system. The system was programmed to maintain a preheat temperature until both bottom and top sensors indicated wet-out, then to proceed with the cure cycle.

The use of the FDEMS system in a control mode is being expanded to include larger parts with more complex flow paths, emerging resins, and control of pressure cycles.

Model Verification and Utilization

The one-dimensional model has been verified using a through-the-thickness infusion process with three different preforms, 3501-6 resin, and several cure cycles (ref. 3). The mold was instrumented with thermocouples for determining temperature distribution, a dial gage to measure platen deflection for correlation with flow front position, and the FDEMS system to monitor viscosity and degree of cure, and to verify complete infiltration. Results for one case are shown in figure 18. Agreement is very good for temperature and flow front calculations; predictions of viscosity and degree of cure, although showing some disagreement with experimental observations, are still reasonable.

The model was used to derive alternate cure cycles for panels of 0.25 inch nominal thickness as shown in figure 19. The resin manufacturer recommended a moderate temperature hold during infiltration, cycle A. The model was run with the manufacturer's and two other heating cycles: ramping to the cure temperature, cycle B, and preheating the platens, cycle C. Predicted infiltration times agree well with experimentally determined values, with cycle C reducing infiltration time by 50 percent.

A further example of the utility of the model is in deciding whether to use or discard an aged sample of resin. The degree of cure advancement during storage, α_o, is determined from a DSC test. For example, hypothetical values of 0.02 and 0.30 were entered in the model, which was run with one of the above cure cycles. The case with aged resin predicts infiltration into only about 1/3 of the full thickness before gelation occurs, whereas full infiltration was predicted for fresh resin. The reason for the difference can be explained by plotting the reciprocal of viscosity against time for the resin flow front, as in figure 20. The areas under the curves are related to the process window. For fresh resin, the area is .00900 min/cp, whereas for the aged resin, the area is only

.00045 min/cp. Further model runs with aged resin and fewer plies indicate more complete infiltration, with saturation occurring at 0.063 inch thickness. If this is not thick enough for the user, the resin should be discarded.

SUMMARY

Resin transfer molding (RTM) is a promising method for cost-effective fabrication of composite structures having a wide range of preform architectures and resin processing requirements. The large range of variations of the process and of material behavior requires that a science-based understanding be applied to the design of molds and the development of cure cycles.

Characterization tests on textile preforms have shown that compaction pressure in excess of ambient vacuum is required to achieve a 60 percent fiber volume. Permeability of preforms can differ by at least two orders of magnitude between the in-plane and through-thickness directions. The effects of secondary materials such as stitches and tackifiers on compaction and permeability should also be quantified.

Resin flow and cure kinetics have been successfully modeled mathematically. Flow simulations can be performed on a computer in order to save labor and materials during process development. Frequency-dependent dielectric sensors have been shown to be beneficial in both monitoring and controlling the RTM process.

Research supported by NASA Langley in developing the required process models, databases, and sensing systems is beginning to yield solutions to practical problems. The work is currently being expanded to encompass a wider range of geometries and materials.

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2. Van Vlack, L. H.: Materials Science for Engineers, Addison-Wesley Publishing Co., Inc., 1970, p. 232.

3. Weideman, M. H.: An Infiltration/Cure Model for Manufacture of Fabric Composites by the Resin Infusion Process, Masters Thesis, Virginia Polytechnic Institute and State University, Blacksburg, Virginia, 1991.

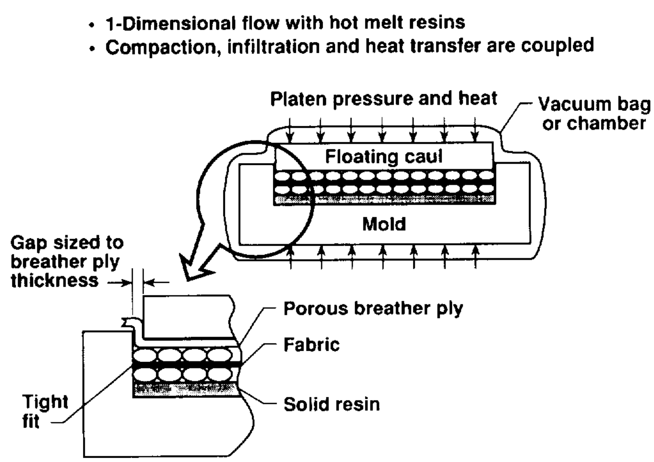


Figure 1. Through-Thickness Vacuum/Pressure Process

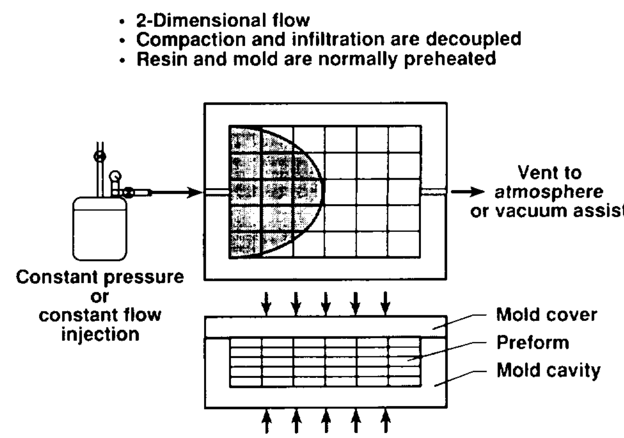


Figure 2. Pressure Injection

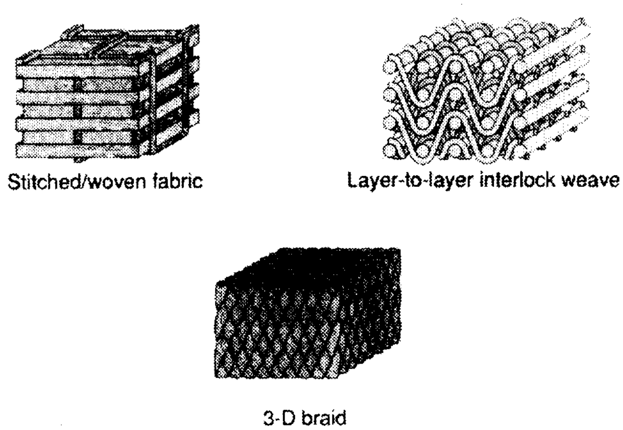


Figure 3. Through the Thickness Reinforced Textile Forms

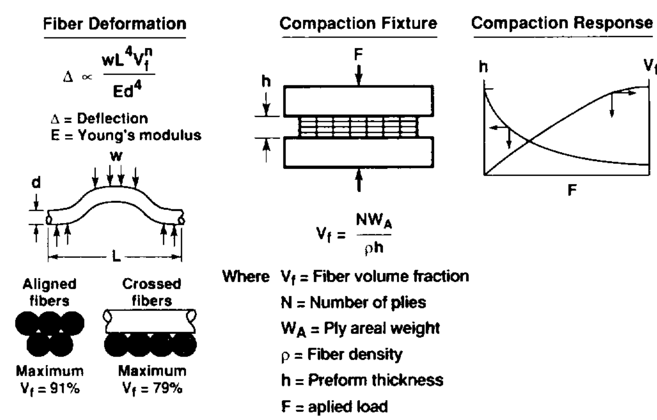


Figure 4. Preform Compaction Behavior

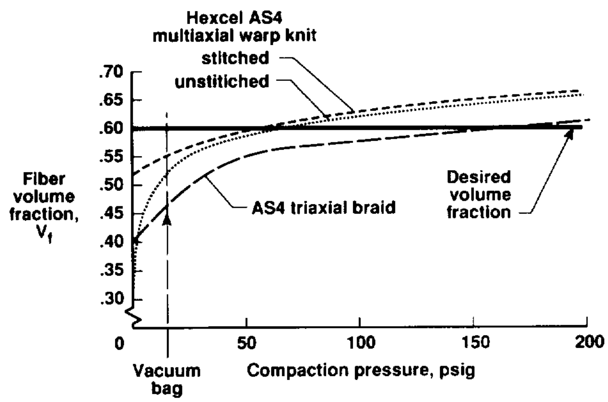


Figure 5. Preform Compaction Data

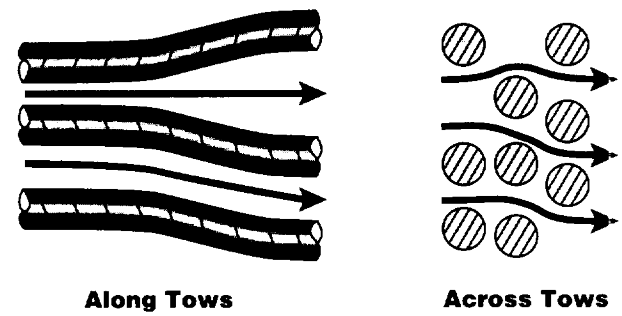


Figure 6. Flow Through Pores in Fibrous Preforms

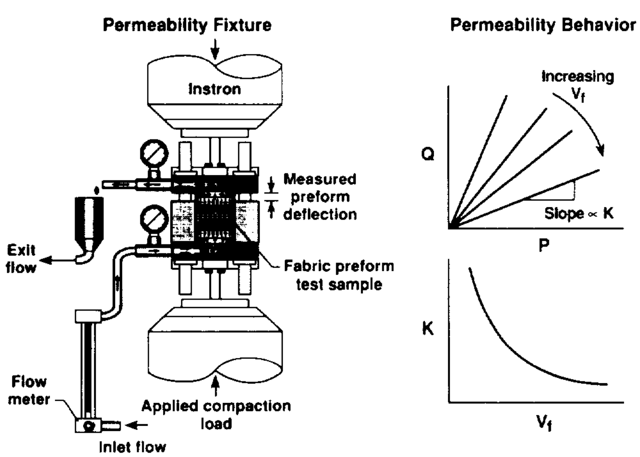


Figure 7. Preform Permeability Measurement

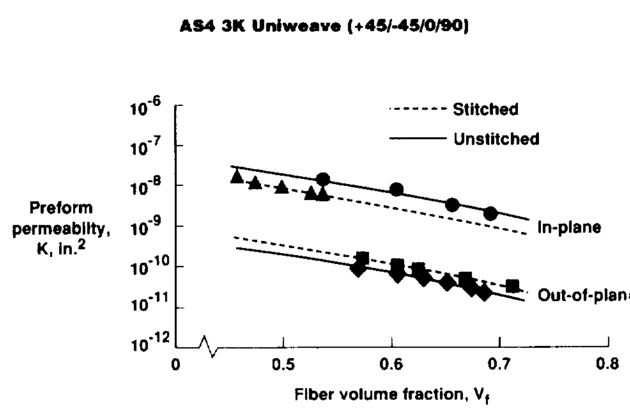


Figure 8. Preform Permeability Data

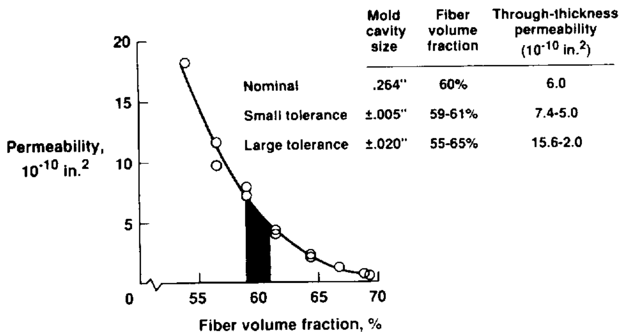


Figure 9. Permeability Variation of Satin Fabric

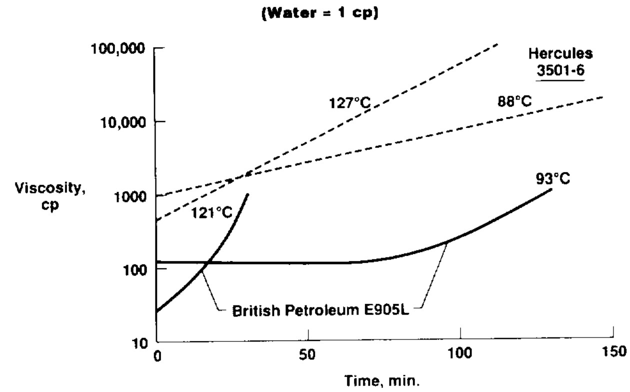


Figure 10 Effect of Time and Temperature on Viscosity of Epoxy Resins

Hercules	3501-6	Hot melt
Dow	CET 2	Hot melt
3M	PR500	Paste
Dow	Tactix 138/H41	Liquid
BP	E905L	Liquid
Shell	1895/W	Liquid
Shell	862/763	Liquid

Figure 11. Resins Evaluated

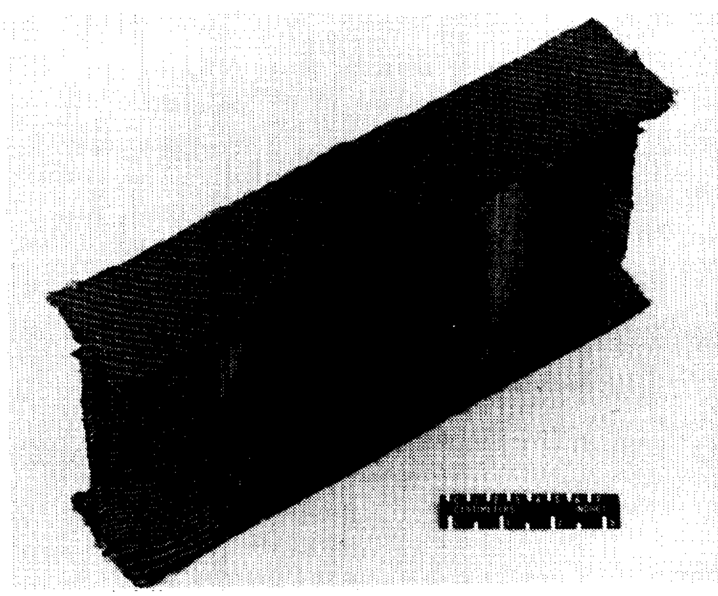
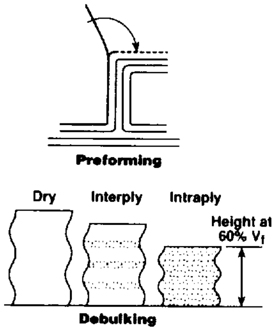


Figure 12. Preform for Sine Wave Spar



Type	Method of application
Spray adhesive	• By hand, from aerosol can to broadgoods
Film adhesive	• By hand, transfer from backing paper to broadgoods
Powdered polymer	• By hand, from shaker to broadgoods, heat activated • Automated, from applicator to broadgoods, heat activated • Automated, powder coated tows prior to fabricating broadgoods, heat activated
Polymer scrim	• By hand or automated, interleaved between plies, heat activated

Figure 13. Tackifier Materials

(16 plies, 8 harness satin, 0/90, IM7
15 plies polyamide scrim, interleaved)

	Compaction pressure @ 60% V_f (psi)	Through-thickness permeability @60% V_f (10^{-10} in.^{2})
Without tackifier	32	7.0
With tackifier	34	8.5

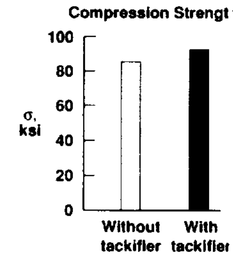
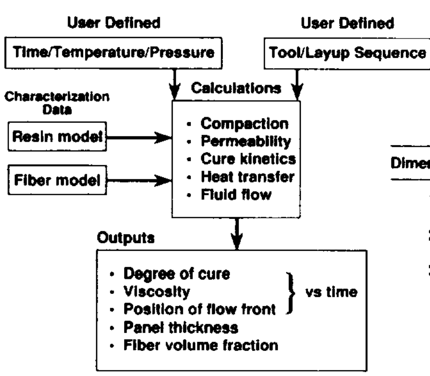


Figure 14. Effect of Tackifier on Processing and Properties



Computing Requirements

Dimensions	Computer	Run time
1	PC	30 min.
2	Mainframe	3 hr.
3	Cray	?

Figure 15. Computer Simulation of RTM Processes

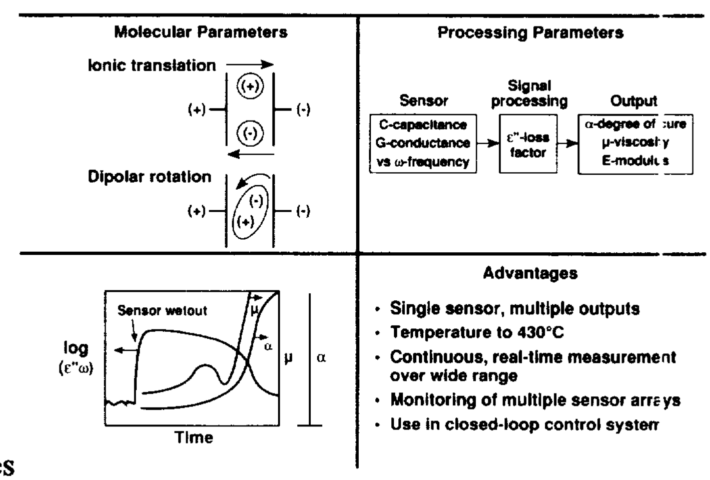


Figure 16. Frequency-Dependent Electromagnetic Sensors (FDEMS)

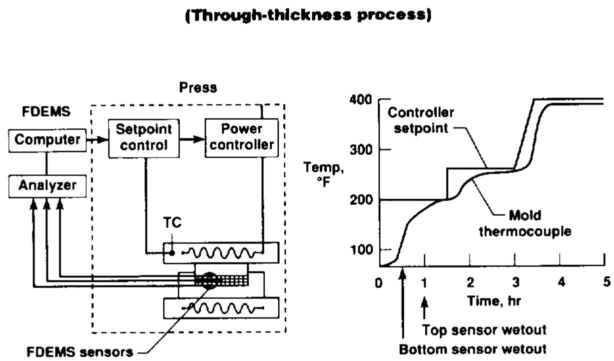


Figure 17. Prototype FDEMS Expert Cure System

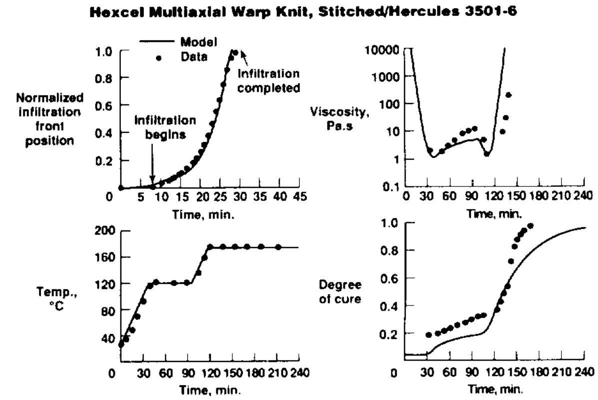


Figure 18. 1-D Model Verification

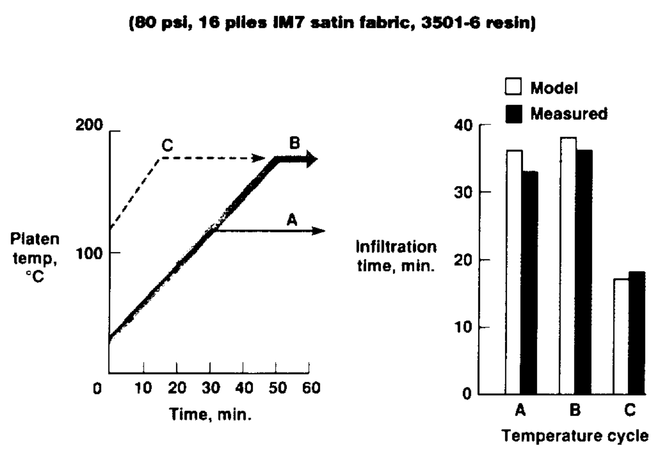


Figure 19. Utility of 1-D Model to Alter Infiltration Time

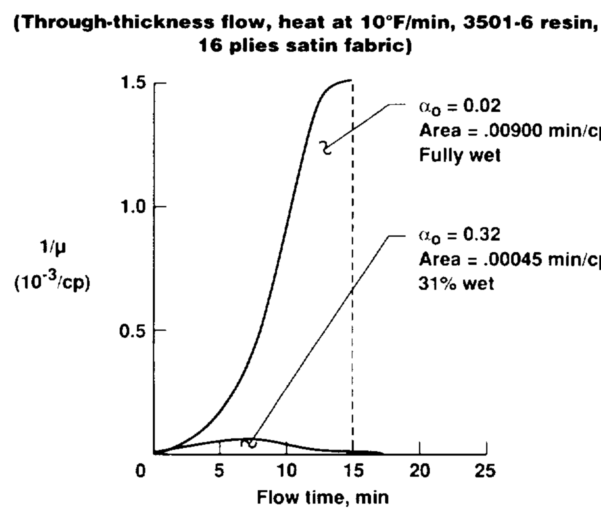


Figure 20. Use of RTM Flow Model to Define Process Window

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SESSION VIII

DESIGN APPLICATIONS (B)

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