1995122614

N95-29035

ADVANCED RESIN SYSTEMS AND 3-D TEXTILE PREFORMS FOR LOW COST COMPOSITE STRUCTURES

J. G. Shukla* and T. D. Bayha Lockheed Aeronautical Systems Company Marietta, GA 30063

51: 34

ABSTRACT

Advanced resin systems and 3-D textile preforms are being evaluated at Lockheed Aeronautical Systems Company (LASC) under NASA's Advanced Composites Technology (ACT) Program. This work is aimed towards the development of low-cost, damage-tolerant composite fuselage structures. Resin systems for resin transfer molding and powder epoxy towpreg materials are being evaluated for processability, performance and cost. Three developmental epoxy resin systems for resin transfer molding (RTM) and three resin systems for powder towpregging are being investigated. Various 3-D textile preform architectures using advanced weaving and braiding processes are also being evaluated. Trials are being conducted with powdered towpreg, in 2-D weaving and 3-D braiding processes for their textile processability and their potential for fabrication in "net shape" fuselage structures The progress in advanced resin screening and textile preform development is reviewed here.

INTRODUCTION

The NASA ACT program underway at LASC is evaluating advanced toughened epoxy resin systems and 3-D textile preforms to develop low-cost and damage tolerant fuselage structures such as frames, window belts, keel beams/frames and skin-stiffened fuselage panels. Phase II, entitled "Development and Verification of Technology", is focused on the evaluation of advanced epoxy resin systems which have been developed both for resin transfer molding (RTM) and preimpregnation of carbon fiber tow with powdered resin systems in Task 1. Mechanical tests of flat panels using 2-D woven preforms will be performed to make a resin selection for both composite fabrication processes.

The selected resin systems will be used in Task 2 to evaluate the advantages of advanced textile preforms, made by processes such as 2-D braiding, 3-D through-the-thickness braiding, 3-D interlock braiding, 3-D weaving and multi-axial knitting/stitching. Flat preforms using the above processes were made for the Task 2 work, and fabricated into panels by RTM; the powder coated preforms were only fabricated by 2-D braiding and 3-D through-the-thickness braiding for autoclave processing.



TASK 1: ADVANCED RESIN SYSTEMS FOR TEXTILE PREFORMS

Resin Transfer Molding

The resin transfer molding (RTM) manufacturing process offers major cost advantages in the fabrication of "net shape" composite structures from textile preforms. However, high performance resin systems are needed to meet aircraft structural requirements. Three emerging epoxy resin systems (RSL-1895 (Shell), PR-500 (3M) and E-905L (BP)) were evaluated for RTM. Flat panels were fabricated at Brunswick Defense Corporation using AS-4 (6k) carbon fiber tow woven into 8-harness satin fabric; a schematic of the RTM process is shown in Figure 1.



Figure 1. Schematic of the Resin Transfer Molding (RTM) process.

Two part systems such as RSL-1895 and E-905L were mixed and degassed at the required temperature before transfer to the injection cylinder. The process parameters for the resins under study are presented in Table 1. The one-part resin, PR-500, was also heated and degassed before processing. The resin was vented from the center and back of the mold to ensure wet-out of the preform and removal of entrapped air.

The resin systems are being compared using the screening test matrix in Table 2. Of particular interest are the room temperature, hot-wet strengths, notch sensitivity and damage tolerance of the resin systems, since conventional RTM resin systems generally do not have optimal hot-wet and damage tolerance properties.

The Shell resin (RSL-1895) showed the best processability among the three systems evaluated. The shelf life of RSL-1895 is 48 hours at room temperature, and at the injection

Table	1.	Processing	Parameters	for	RTM

Resin System	Temperature at	Injection, °F	Injection	Cure Temp., Time	Post Cure
	Resin Temp.	Mold Temp.	Pressure, psi	(°F, minutes)	Conditions
RSL-1895	175°	250°	20-80	350°, 30 min.	350°F, 2 hrs.
PR-500	225°	325°	20-80	350°, 60 min.	350°F, 2 hrs.
E905-L	<u>180°</u>	225°	40-80	350°, 30 min.	350°F, 2 hrs.

temperature of 140°F, this system also has a low viscosity (< 100 centipoise at 140°F). The RSL-1895 system is easier to vent and process the part compared to either the 3M PR-500 or BP E-905L resin systems. Temperature control is critical in liquid molding with PR-500; drops in temperature at the injection port, the injection line or in the tool during injection can result in dry spots, voids in the part, and cooling of the resin in the venting lines. E-905L behaves in a similar fashion to RSL-1895 in its flow characteristics, however, temperature control during mixing is required to control viscosity.

Microscopic analysis of the laminates made with all three resin systems indicates good fiber wetout, however, surface pitting was observed in some laminates made with 3M PR-500. Surface pitting was eliminated in subsequent panel fabrication by adequately degassing and controlling temperature. The Shell RSL-1895 laminates showed virtually no porosity, while those panels molded using BP E905-L and PR-500 showed some level of porosity. Photomicrographs of RTM panels are presented in Figure 2. The ultrasonic C-scanning met Lockheed's acceptance criteria.

The bulk cost of the Shell 1895 is \$12.00/lb., that of BP E905L is \$27/lb., and that of PR-500 is \$40.00 per pound.

The testing of RTM panels is being conducted at Delsen Laboratories. Open hole tensile strength (OHT), open hole compression strength (OHC), compression modulus, and Poisson's ratio at room temperature have been completed, and the results are shown in Table 3.

Processing Science for RTM

Resin transfer molding has been used in automotive and other industrial applications where up to 50 volume percent (v/o) fiber is adequate. Aircraft structures require a minimum of 55-60 v/o as well as selected fiber architectures. 2- and 3-D tightly woven or braided preforms being evaluated under this program will require a better understanding of permeability and resin flow through the preform.

Currently, a process model of the RTM process is being developed at the University of Delaware. This modeling effort is to help tool design by providing the location of gates, ports, and vents for optimum mold filling. The flow simulation has been developed based on finite element/control

TABLE 2 : TEST MATRIX

TEST TYPE	LOADING	TEST ENVIRONMENT	INSTRUMENTATION	TEST METHOD (COUPON DWG. NO)
0° Tension	Tension	RTA	T-gage	SACMA SRM4-88 (C1AB)
0° Tension	Tension	-65°F	T-gage	SACMA SRM4-88 (C-1AB)
0° Compression	Compression	RTA	BBTG	SACMA SRM1-88
0° Compression	Compression	180°F, Wet	BBTG	SACMA SRM1-88
Moisture Absorption Travellers				
± 45° Tension	Tension	RTA	T-Gage	SACMA SRM7-88 (C-3)
Unnotched	Tension	RTA	ASG	SACMA SRM4-88 (C-23)
Open Hole	Tension	RTA	_	SACMA SRM5-88 (C-4NB)
Open Hole	Tension	-65°F	_	SACMA SRM5-88 (C-4NB)
Unnoiched	Compression	RTA	B/B	TPS 86-2256 (C-23)
Unnotched	Compression	180°F, Wet	B/B	TPS 86-2256 (C-23)
Moisture Absorption Travellers				—
Open Hole	Compression	RTA		SACMA SRM3-88 (C-4NB)
Open Hole	Compression	180°F, Wet		SACMA SRM3-88 (C-4NB)
Comp. after Impact	Compression	RTA	B/B	SACMA SRM2-88
Comp. after Impact	Compression	RTA	B/B	NASA 1142 (C-8)



Shell RSL-1895





Figure 2. Photomicrographs of RTM Panels.



BP E-905L

Figure 2. Photomicrographs of RTM Panels, cont.

Table 3. Test Data* for RTM Laminates (8-Harness Fabric).

<u>Test Type</u>	<u>Condition</u>	3501-6**	<u>Shell-1985</u>	<u>E-905</u>	<u>PR-500</u>
Open Hole Tension	RT	48.0	54.1	48.1	48.7
Strength, ksi	-65°F	47.3	57.0	45.7	47.9
Open Hole Compression Strength, ksi	RT	48.6	43.8	45.7	44.8
Compression Mod, msi		7.5	8.9	8.7	9.3
Poisson's Ratio		0.027	0.024	0.034	0.028
* not normalized					

** prepreg

volume method to calculate the flow pattern for mold filling of generalized fluids in anisotropic media. The simulation takes into account the heat transfer between the heated mold, the resin and the fiber perform. Also, resin cure during and after the mold-filling stage is taken into account. The dependence of the resin viscosity on the local temperature and the degree of cure is incorporated in the simulation. Presently, the simulation can model thin planar parts of otherwise arbitrary shape. Since actual resins exhibit a shear-thinning behavior, the simulation takes into account the dependence of the fluid viscosity on the shear-rate. Also along with the flow pattern, the simulation allows one to obtain the progression of the resin cure during the filling stage, and monitor the final cure after the part is filled. Preform permeability studies for 3-D textile architextures and flow simulation for the RTM tool design are underway for the location of gates, vents, and ports for optimum mold filling.

Powder Coating Processes

Two methods of fiber tow impregnation have been studied under the contract: a slurry process developed at BASF Structural Materials, and an electrostatic process devised by Custom Composites. A general schematic for all powder coating processes is given in Figure 3.



Figure 3. Schematic of Powder Coating Processes.

The BASF process suspends the powder particles in a water-based slurry, and the tow is passed through a bath containing the powder. In line with the slurry bath is an oven to sinter the particles to the carbon fibers before bobbin winding for textile operations. Towpreg was produced with very little variation in resin content by this method.

The electrostatic process uses ionization of the powder particles to induce attraction to the tow. The tow passes through a cloud of powder in the impregnation chamber before sintering in the oven. This process, once stabilized, produces a fairly uniform towpreg; however, some

clumping of powder particles occurs occasionally for some types of powdered resins, especially when a high level of moisture is present.

It is evident from early results that some powder resin variables affect each process differently. For example, in some processes, powder particle size and particle size distribution are critical issues for a consistent, homogeneous coating of powder on the fiber tow. Spreading of the fiber tow is also very important; fibers in the tow need to be individualized in order to guarantee that each interacts with the powder in the impregnation chamber. The degree of fusion of the powder to the fibers is important; the flexibility of the fibers for weaving or braiding into a textile preform is a function of the furnace temperature and speed of the moving tow. The chemical and thermal stability at room temperature to accommodate weaving, braiding and storage are equally important for towpreg materials to be accepted for use in textile preforms.

Advanced Resin Systems for Powder Epoxy Technology

Three emerging epoxy powder systems, RSS-1952 (Shell), Dow CET-3 and PR-500 (3M), are being evaluated. All three resin systems have been screened using a slurry process at BASF. RSS-1952 was chosen to conduct feasibility studies for various textile processes because of its ample supply and processability. Recently, processing criteria for PR-500 and CET-3 have been determined. PR-500 powder was successfully coated by the Georgia Institute of Technology and later by Custom Composites using an electrostatic coating process. Both powder systems were applied to AS4 carbon fiber tow. The processing conditions used to fabricate panels from the two powder systems are listed in Table 4.

Table 4	Processing	Parameters	for	Powder	Towpreg	Laminates
---------	------------	------------	-----	--------	---------	-----------

Powder Resin	Coating Method	Autoclave	Conditions	Curing	Post Cure
System	-	Heating Rate	Pressure, psi	Conditions	Conditions
RSS-1952	slurry	5°F/min	85	350°F, 1 hr.	400°F, 4 hrs.
PR-500	electrostatic	5°F/min	85	350°F, 1 hr.	350°F, 2 hrs.

Powder towpreg was woven into 8-harness satin fabric at Fabric Development from towpreg produced at BASF and at Textile Technologies, Inc. (TTI) for the Custom Composites material; the resin content for this fabric construction was 35 ± 2 weight percent (w/o). Flat panels having fiber volume fraction (v/o) of 58%-60% were fabricated and evaluated by Lockheed to compare the two powder coating processes. Photomicrographs indicate good fiber wetout in the Shell RSS-1952 panels and in the 3M PR-500 panels. The photomicrographs of these materials are shown in Figure 4.

Mechanical testing of these panels is currently being performed. The screening test matrix is the same as shown in Table 2 for the panels fabricated by RTM.

Shell RSS-1952



3M PR-500

Figure 4. Photomicrographs of Powdered Epoxy Panels.

TASK 2: PREFORM DEVELOPMENT AND PROCESSING

Task 2 is focused on the evaluation of preforms produced by advanced textile processes. The preform architectures are designed so as to provide improved damage tolerance due to 3dimensional fiber reinforcement, and have potential cost reductions in the fabrication of composite structures. 3-D textile processes also provide "net" or "near-net" shape preforms, and subsequent fabrication of composites using low cost processes such as resin transfer molding, pultrusion and resin infusion methods reduces part count and therefore assembly steps.

The development of textile processes with fiber continuity between the skin and stiffeners eliminates the problems associated with stiffener separation and significantly improves the structural efficiency of many fuselage structures. Similarly, composite fabrication with textile preforms using low cost powder towpreg offers the same benefits.

The following textile processes are currently being evaluated under Task 2:

- 3-D through-the-thickness weaving with biased yarn;
- 2-D braiding;
- 3-D multi-layer weaving/stitching;
- 3-D interlock braiding;
- 3-D through-the-thickness braiding;
- Multi-axial knitting/stitching; and
- Near-net Fiber Placement (N²FP) stitching.

Quasi-isotropic flat panel preforms have been produced using most of the above processes with dry Hercules AS-4 carbon fiber tow. These preforms are being fabricated into flat panels using RTM, and will be mechanically tested. The test matrix is identical to the screening test matrix used in Task 1, and presented in Table 2, with the exception that 0° tension and compression, or $\pm 45^{\circ}$ tension tests will not be performed. The potential applications, benefits and limitations for preforms made by the respective textile processes are reviewed below. The preform architectures resulting from the above textile processes are shown in Figures 5 and 6.

3-D Weaving

3-D multi-layer [0°/90°] weaving (Figure 5) of preforms was carried out at Textile Technologies, Inc. (TTI). Multi-layer fabrics were stacked to achieve a quasi-isotropic orientation before RTM. This process offers advantages over two dimensional processes due to its relatively low cost, improved damage tolerance and its ability to produce near-net shape preforms. However, the process as it is currently applied has some limitations in introducing biased yarns during weaving, and fiber volume is limited to about 55%.

A 3-D through-the-thickness process has been developed by Techniweave, Inc. The process is capable of weaving multi-axial preforms that include biased plies integral with the skin. The through the thickness yarn is introduced by a chain lock stitch. Window belt panels with window cutouts, stiffened panels and fuselage frame applications all have the potential to be fabricated by this process. Currently, window belt panels with cutouts and stiffeners in both longitudinal and



3-D Multi-layer Weaving



Multi-axial Knitting/Stitching

Figure 5. Textile Architecture for Weaving and Knitting Processes.



2-D Braiding



3-D Interlock Braiding



3-D Through the Thickness Braiding

Figure 6. Textile Architecture for Braiding Processes.

transverse directions are being produced, however, the stiffeners will not have biased plies and will have to be stitched. Quasi-isotropic flat preforms are also being produced for testing and evaluation.

2-D and 3-D Braiding

The textile architecture is shown in Figure 6 for various braiding processes. Generally, braiding operations are very versatile, offering reduced cost preforms that can be made in any desired length.

2-D braiding is a low cost textile process to produce preforms to be used in subsequent fabrication of stiffeners and frames by RTM or pultrusion, which offers advantages over conventional autoclave hand layup processes in cost and handling reductions. Low cost, near-net shape preforms can be made by 2-D braiding processes that have improved delamination resistance over hand layups. Preforms manufactured in this manner have only 2-D reinforcement, as well as limitations on the size, thickness and shapes that can be fabricated. Quasi-isotropic ($\pm 60^\circ$, 0°) flat panels have been made by tri-axial (braid-over-braid) braiding.

An advancement over 2-D braiding is a 3-D interlock braiding process. In this textile process, multi-layer fabric is braided with a layer-to-layer angle interlock. The interlocking fibers provide for z-directional reinforcement, which improves damage tolerance. The cost of these preforms is relatively low, and they can be made in near-net shapes. Potential applications for the 3-D interlock braided preforms include stiffeners and curved frames.

Quasi-isotropic flat panel preforms were made at Albany International on their circular braider. This 3-D braider is currently limited in preform thickness; only five layers can be braided for a given preform with their R & D machine. However, a flat braider is available to make higher thickness braids, as well as braided "net" shapes. Thick panel preforms will be made on this machine in the near future.

Atlantic Research Corporation has an automated 3-D through-the-thickness braiding process that allows production of 72-inch wide preforms. Circular braiding with their bifurcation technique allows integral braiding of stiffened panels and frames in "net" shape. As with the other braiding processes, near-net shapes can be fabricated; however, this process has a greater flexibility in the range of fiber orientations available. At this time, the process is slow and costly, and efforts are underway to increase the speed and reduce preform costs. Quasi-isotropic flat panel preforms have been made by this process at Atlantic Research.

Multi-Axial Knitting/Stitching

Multi-axial stitched panels are currently being fabricated at Hexcel-Hitech. Multi-axial knitting, as shown in Figure 5, provides multi-layer (4-7 layers) fabric with 0°, 90°,45°,135° fiber orientation, and is probably the least expensive method available to produce multi-layer stitched

preforms for stiffened panels and stiffeners. Multi-layer fabrics will be stacked and stitched for quasi-isotropic laminate and resin transfer mold.

Near-Net Fiber Placement

Quasi-isotropic, multi-layer flat preforms are currently being made at Cooper Composites with their near-net fiber placement technique. The N^2FP process is a fully automated computer-interfaced system which is capable of placing fibers in any predetermined orientation. Volume fractions up to 60% are obtained and near-net shape preforms can be fabricated. While some z-direction reinforcement is available using this method, currently there are limitations in the sizes and thicknesses which can be made. Window frames and window belts are potential applications for composites made from this type of preform.

Powder Coated Advanced Textile Structures

The 2- and 3-D braiding feasibility studies are being conducted with Shell RSS-1952 towpreg produced by BASF. Similarly, feasibility studies are being carried out on 3M PR-500 towpreg. Both materials are braidable, however, both 2- and 3-D braiding processes require an initial resin content on the order of 39 ± 2 w/o resin due to the abrasive nature of the processes. The textile operations reduced the resin/fiber content between 2% and 3% by weight during processing. These preforms are currently being made and tested at Lockheed. BASF/Textile Technologies, Inc. has already woven 3-D multi-layer fabrics from the towpreg and processed them. The 3-D fabrics required higher temperature and pressure to fully consolidate, compared to the 2-D fabrics.

The Custom Composites PR-500 towpreg was woven into 8-harness satin fabric at TTI. The towpreg has been evaluated for its braidability at both Fiber Innovations and Atlantic Research Corp. The initial abrasion tests indicated that the towpreg is braidable. Flat preforms will be braided early in the summer to evaluate any advantages in braiding of the electrostatically towpregged materials over towpreg made by the slurry process.

SUMMARY AND CONCLUSIONS

Resin transfer molding evaluation of all three advanced resin systems was accomplished. The processability of the Shell RSL-1895 resin system was better than the 3M PR-500 and BP E-905L systems. Photomicrographs showed excellent fiber wet-out with all three systems. The current cost of the Shell system is lower than either of the other two. Mechanical testing of all RTM panels is underway. A selection of an optimal resin system will be made based on processability, cost and performance.

Both powder epoxy resin systems, the Shell RSS-1952 and the 3M PR-500, show promise. Studies of the 2-D and 3-D weaving and braiding feasibility for both powder towpreg systems have been completed, and flat panel evaluation has begun.

The mechanical testing of the autoclave processed panels is expected to be completed by mid-summer of 1992.

.