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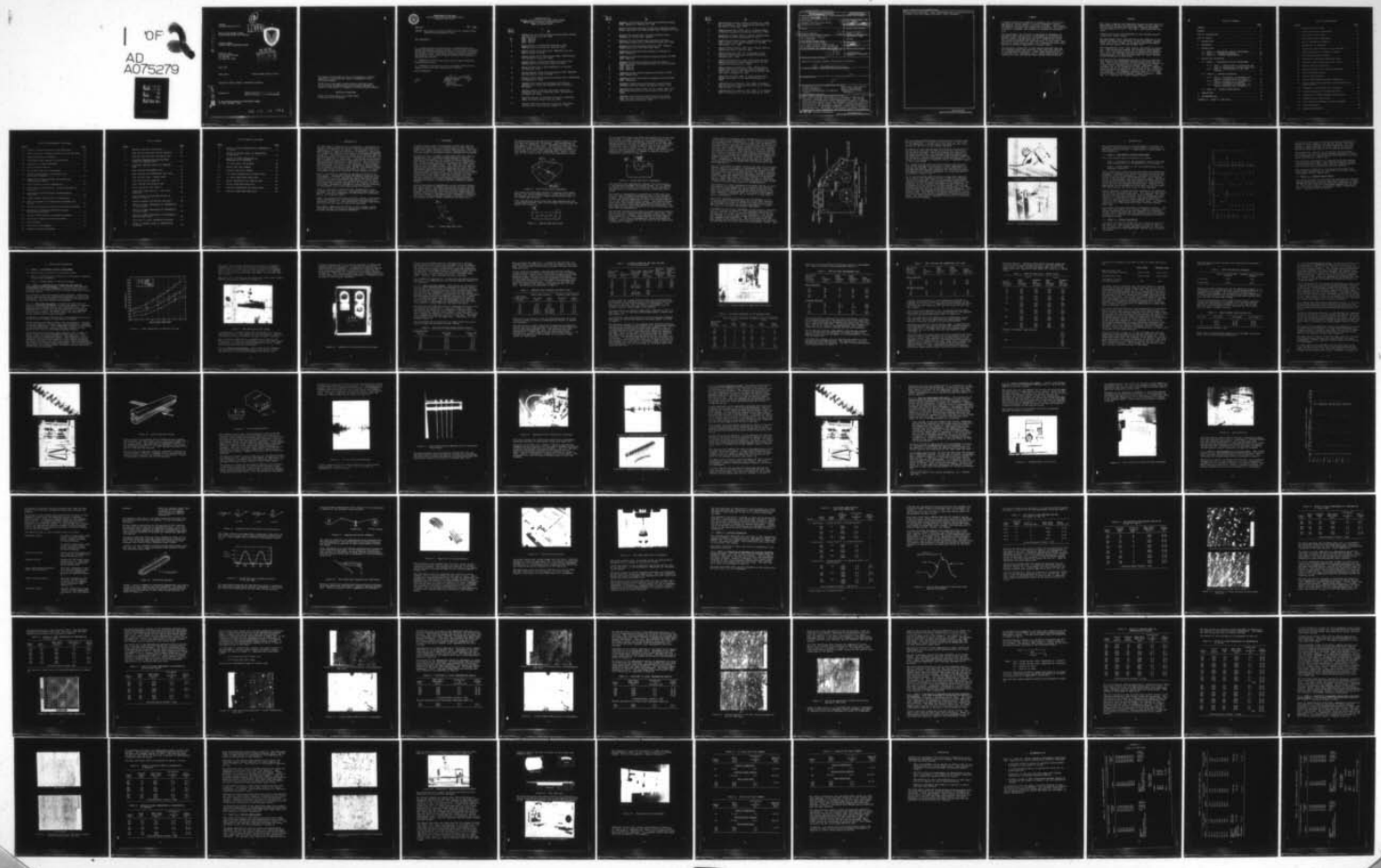

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Manufacturing Methods Program
Manufacturing Methods and Technology

FILAMENT WINDING
PRECISION RESIN IMPREGNATION SYSTEM

Richard R. Zeits
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Final Report

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2. AVRADCOM report TR 79-15 has been provided to supply information concerning this program.

3. If additional information or reports are needed, contact Mr. Dan Haugan at Autovon 693-1625, Commercial (314) 263-1625.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report presents the results of a program to develop and evaluate a precision resin impregnation system. The program objective was to develop a resin impregnator capable of consistently wetting S-glass, Kevlar and graphite roving with resin controlled within ±2% by weight. The tests conducted demonstrate the ability of the developed equipment to meet resin content control objectives. The effect of the process variables of fiber temperature, resin temperature and winding speed on impregnation

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quality were evaluated. Short beam shear tests and microstructure analysis were the primary evaluation tests conducted.

SUMMARY

A 3-phase program was conducted to develop a wet resin direct impregnation system capable of wetting fiberglass, Kevlar, and graphite roving within ±2 percent by weight. These phases consisted of equipment development and evaluation, an evaluation of process variables, and a mechanical properties determination.

This development has resulted in impregnation equipment improvements which enable resin and hardener to be separately metered, mixed, and applied at elevated temperatures within the ±2 percent program objectives for a given roving yield. The process evaluation has indicated that, in most cases, best wetting of fibers occurs using a low or medium viscosity resin applied at elevated temperature to heated fibers.

The mechanical properties of test samples produced with the wet winding process were generally lower than published data for preimpregnated materials. These lower properties are attributed to air entrapment due to unresolved problems associated with fiber wetting and resin system characteristics.

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PREFACE

This report compiles and summarizes the work accomplished as a Manufacturing Methods and Technology (MM&T) Project under U.S. Army Aviation Research and Development Command (AVRADCOM), Contract DAAJ01-77-C-0777. This was a firm fixed-price contract.

Technical direction from AVRADCOM for this program was provided by Mr. Daniel Haugan.

The development effort reported herein was conducted in the Methods and Materials Laboratory at Bell Helicopter Textron (BHT), Fort Worth, Texas, by the Manufacturing Development Group between August 1977 and October 1978.

The contributions of Mr. Larry Ashton, who served as consultant to BHT during the program, are gratefully acknowledged. Also, the investigators would like to extend their appreciation to Dr. Jan Cernosek, Methods and Materials Laboratory Group Engineer, and laboratory personnel for their interest and participation in this study.

This project was accomplished as part of the U.S. Army Aviation Research and Development Manufacturing Technology program. The primary objective of this program is to develop, on a timely basis, manufacturing processes, techniques, and equipment for use in production of Army materiel. Comments are solicited on the potential utilization of the information contained herein as applied to present and/or future production programs. Such comments should be sent to: U.S. Army Aviation Research and Development Command, Attention: DRDAV-EXT, P. O. Box 209, St. Louis, Missouri 63166.

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1. INTRODUCTION

The development of manufacturing technology is recognized as a necessary prerequisite for the cost-effective application of composites to aircraft structures. The process of wet filament winding of composite structures is generally acknowledged as a potentially cost-effective method for automated component production. The material cost savings to be realized from the direct procurement of fiber and resin materials from their respective suppliers is significant. Limitations to the process have included an inability to thoroughly wet the fibers while maintaining accurate resin-to-fiber ratios. This project has addressed these limitations. The objective of this program was to develop a wet resin direct impregnation system capable of consistently wetting S-2 glass, Kevlar, and graphite roving with resin within ±2 percent by weight or better. A 3-phase development program was conducted to meet these objectives.

Phase I consisted of impregnation equipment evaluation and development. Impregnation system development centered on the improvement of a roller impregnator employing metering pumps to meter the resin and hardener applied to the fibers and a series of rollers to work the resin/hardener into the fibers. This system was selected subsequent to an industry survey of resin/fiber impregnation concepts as having the most potential for development into controllable production hardware.

Phase II consisted of evaluating the impregnation process parameters of resin viscosity, impregnation speed, fiber temperature, and resin temperature as applicable to fiberglass, Kevlar, and graphite fibers.

Phase III consisted of testing S-2 glass, Kevlar, and graphite fibers impregnated under the optimum process parameters established in Phase II. Short-beam shear, tubular compression, and unidirectional tension tests were conducted.

This report summarizes the results of this 3-phase program conducted by BHT to develop an impregnation system and to evaluate impregnation process parameters.

2. BACKGROUND

Filament winding came into prominence around 1955 when the process was being used in the fabrication of high pressure rocket motor cases and other similar applications. The origination of filament winding occurred at approximately the same time that epoxy resins became readily available on the market.

Through the years, a number of approaches have been used in impregnation, all meeting different degrees of success. A commonly held opinion in the filament winding industry was that control of the fiber-to-resin ratio is not important as long as high winding tension is maintained and rather thick parts are to be filament wound. The reason for the lack of concern about the fiber-to-resin ratio was the fact that the high tension provides a natural squeezing-out action which removes the excess resin and provides the structure with a very high fiber-to-resin ratio. Since the use of the filament winding process was aimed primarily at pressure bottles or rocket motor cases where a high fiber-to-resin ratio was desired, there was little concern for absolute accuracy. Precision winding of aircraft components requires precise impregnation control to achieve optimum strength-to-weight ratios.

Initial approaches to impregnation control involved winding dry fibers around the mandrel and coating the wound surface with epoxy resins. The theory behind this process was that the fibers would become saturated, would be impregnated thoroughly, and would cure into a structurally sound component. This early method of impregnation was unsuccessful and led to the use of a funnel-type bath (Figure 1) wherein the fibers were passed through a funnel containing the epoxy resin.

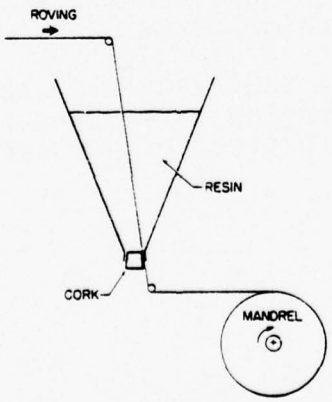

Figure 1. Funnel-Type Resin Bath

The fibers exited at the bottom of the funnel around a "cork" and then were wound onto the mandrel. The disadvantage of this type of impregnator is poor impregnation caused by the entrapment of air in the fiber. Air is pulled into the resin bath by the fibers resulting in the formation of bubbles within the resin which makes thorough impregnation difficult. This system is still used in modified form. Another variation of this system is the orifice-type impregnator (Figure 2).

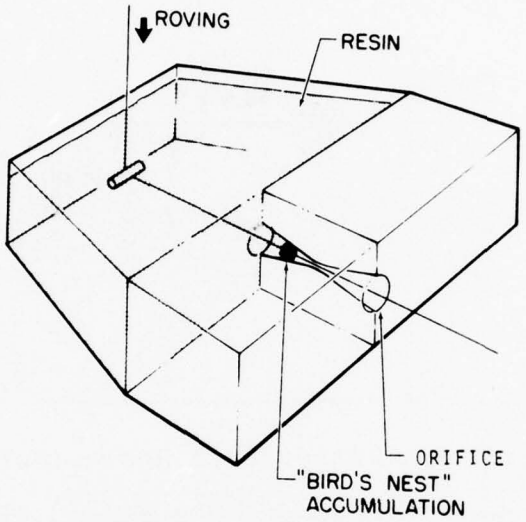

Figure 2. Orifice-Type Resin Impregnator

This system provides resin control at constant fiber speed; however, the rapid accumulation of a fiber "bird's nest" at the entrance to the orifice renders the method difficult in wet winding applications.

Other impregnating devices that have been experimented with include dipping baths, both with stationary and revolving submerged bars (Figure 3).

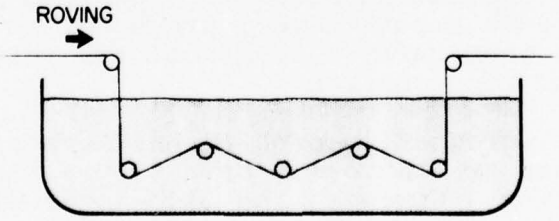

Figure 3. Dipping-Type Resin Bath

Roller-type baths where the fibers are passed over rollers that are running in a bath of resin, as well as many other variations of the bath-type impregnator, have been attempted. An improved degree of control has been achieved in the bath-type impregnator where the fiber is passed over a roller running in a resin bath with a "doctor blade" to control the amount of resin on the roller (Figure 4). This type of impregnator requires constant operator attention to vary the "doctor blade" pressure that compensates for changes in ambient and resin temperature, and resin viscosity.

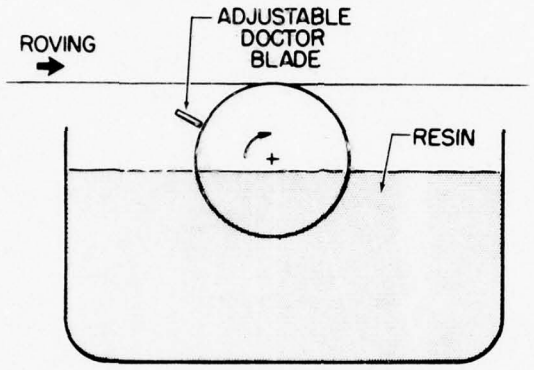

Figure 4. Roller/Bath Resin Impregnator

The existing resin impregnation systems, with the exception of the operator-attended roller/bath system, are not capable of meeting the ±2 percent resin control that is required for the fabrication of helicopter structures, especially rotor blades.

The degree of impregnation or penetration of the fiber with resin is another problem plaguing the users of wet filament winding. This penetration is referred to as the "wetting out" of the fiber during impregnation, which is a problem exclusive of the fiber-to-resin ratio control. This problem becomes severe when high winding speeds are desired and the rovings must pass through a bath-type impregnator at high speed. The high speed rovings carry a sheath of air which effectively prevents thorough impregnation.

The thorough impregnation problem has heretofore been resolved best by the use of stationary bars within a resin bath. The bars aid wetting. However, the high-speed winding entrains a large amount of air, which causes foaming in a bath-type im pregnator. This type of impregnation, using the saturation concept, always provides far too much resin on the fiber which is difficult to remove.

A major factor in achieving good impregnation is low viscosity of the resin during impregnation. A low viscosity resin generally produces better "wetting" and is much less susceptible to the entrainment of air and other problems that are usually associated with the impregnation of resins having high viscosity. For this reason, many filament winders utilize resin systems that have the viscosity reduced by heat or by either reactive or nonreactive diluents. The reduction of viscosity with the use of heat is very successful and produces good wetting; however, few resin systems can tolerate the heat and temperature required to reduce viscosity sufficiently without causing a drastic reduction in the pot life or gel time of the resin bath. Consequently, the heated bath concepts for reducing viscosity are limited to resins with slow reactivity at the bath temperature.

The use of diluents to reduce viscosity has been very prevalent. However, the use of diluents is considered to be a poor tradeoff in filament winding because the diluents detract from the physical properties of the resin system. The highest performing resins, in terms of interlaminar shear strength, structural toughness, and high temperature performance are those epoxies that are based upon either very high viscosity or semisolid resins. These types of resin systems are used in the prepreg industry where a hot-melt or a solvent-dissolve process is used to reduce viscosity sufficiently for preimpregnation.

Consideration of these impregnation variables led BHT to select the nonbath roller impregnator as the process with the most potential for development into controllable production hardware. The nonbath roller impregnator does not require a concentrated mass of catalyzed resin to be held at high temperature. Therefore, premature gel or set-up of a large mass of resin is minimized.

The roller impregnator dispenses resin and hardener to the rollers by means of metering pumps set at the rates required to impregnate the fibers at the desired fiber-to-resin ratio. Heat to reduce the resin viscosity can be applied to either the fibers or the resin and hardener separately.

A schematic of the roller impregnator in its original configuration is presented in Figure 5. The impregnator operates as follows. The dry fiber is passed as multiple rovings through the spreader bar at the rear of the impregnator. The rovings pass through a quartz-element hooded oven and can be heated to a predetermined temperature. The roving then passes over a dual chamber manifold where the resin and hardener are delivered through separate orifices directly onto the passing roving. The

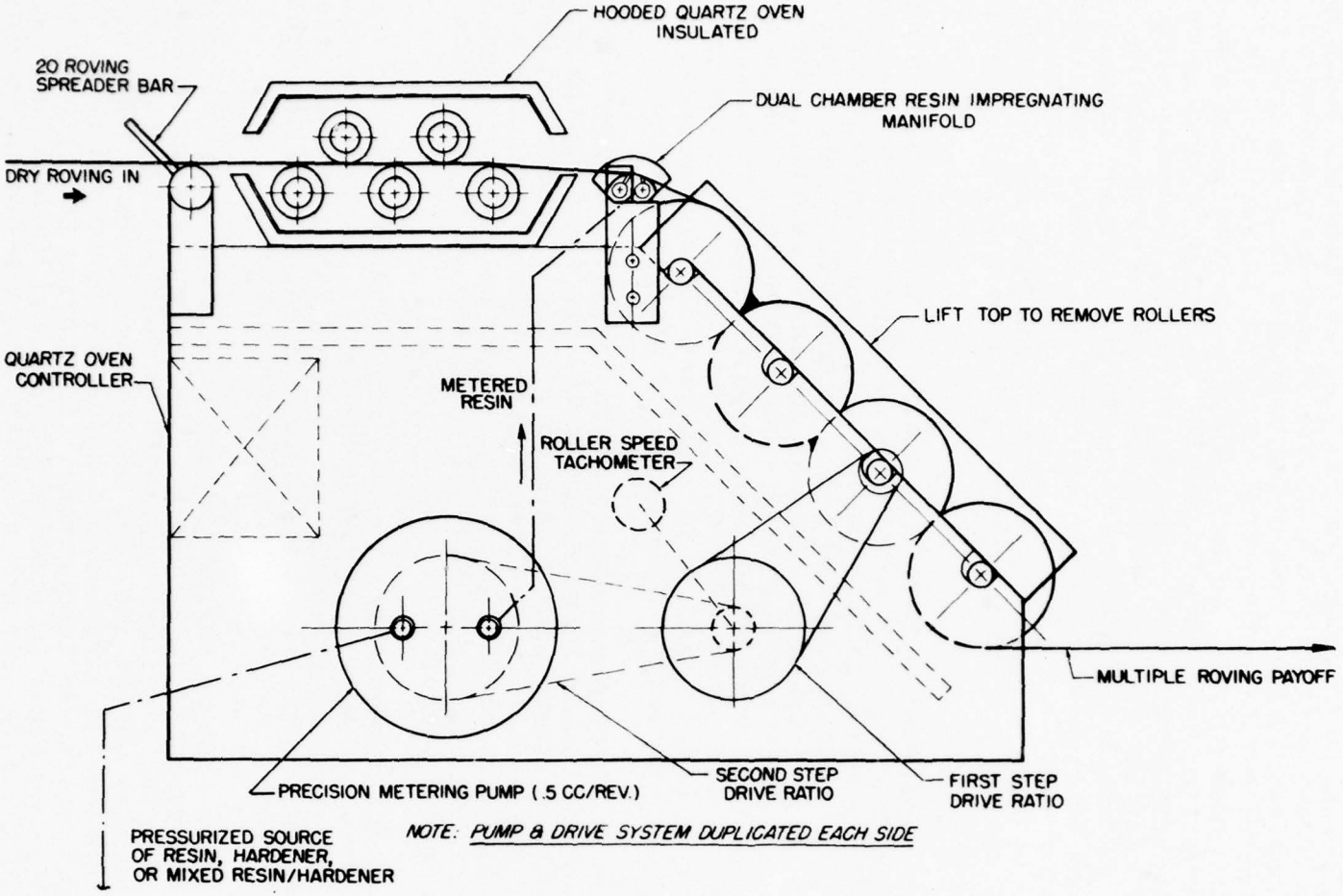

Figure 5. Roller Impregnator Schematic

wetted roving proceeds through a series of four rollers where the resin/hardener is worked into the roving. At the output of the impregnator, a second spacer bar maintains the roving spacing for delivery to the winding equipment.

The amount of resin and hardener delivered to the manifold is controlled by adjusting the speed ratio between the rollers and the metering pumps. Resin and hardener are supplied to the pumps through lines from separately heated pressure pots. The preheating oven is heated by five quartz heating elements controlled by a variable percentage on/off SCR controller. The current to the heaters is varied by use of a tachometer coupled to the roller system.

BHT, recognizing the potential cost savings to be realized from direct impregnation, purchased a roller impregnator for in-house development. The timing of this purchase was such that BHT had this equipment available for the subject program. Photographs of the roller impregnator as it was received are presented as Figures 6 and 7.

Due to the allowable yield variation of some fiber materials (±8.5 percent for fiberglass roving), a need for in-process control was recognized. Beta ray transmission measurements conducted on both the input and output fibers appeared to have potential for the in-process adjustment of resin delivery in accordance with the variation in fiber weight. Beta ray measurement basically consists of passing the material to be measured between the beta ray source and pick-up transducers. The amount of beta ray absorption realized is proportional to the material mass. This method is currently being used to measure the resin content during the impregnation of fiberglass broadgoods. The feasibility of applying this process to roving was studied in this program.

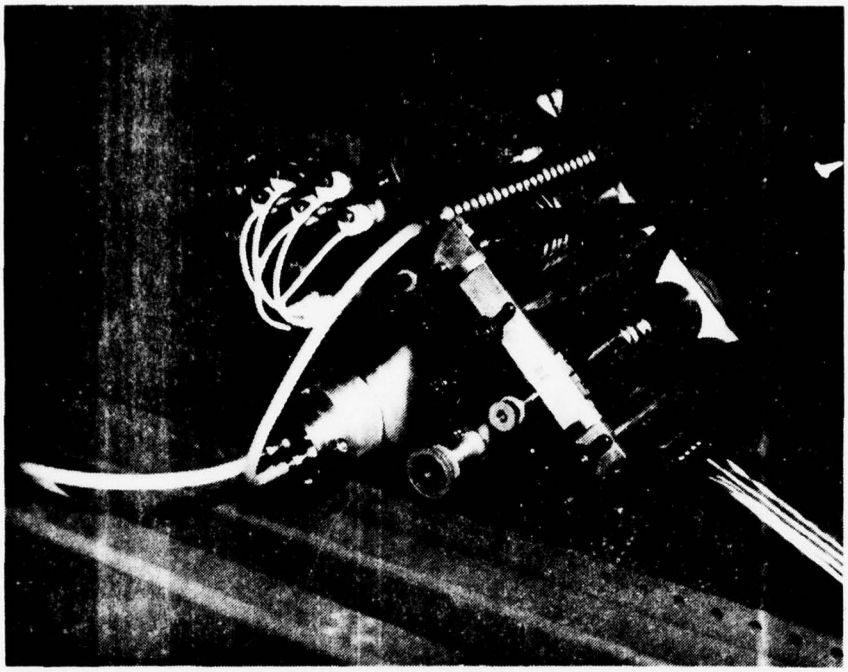

Figure 6. Impregnator as Received

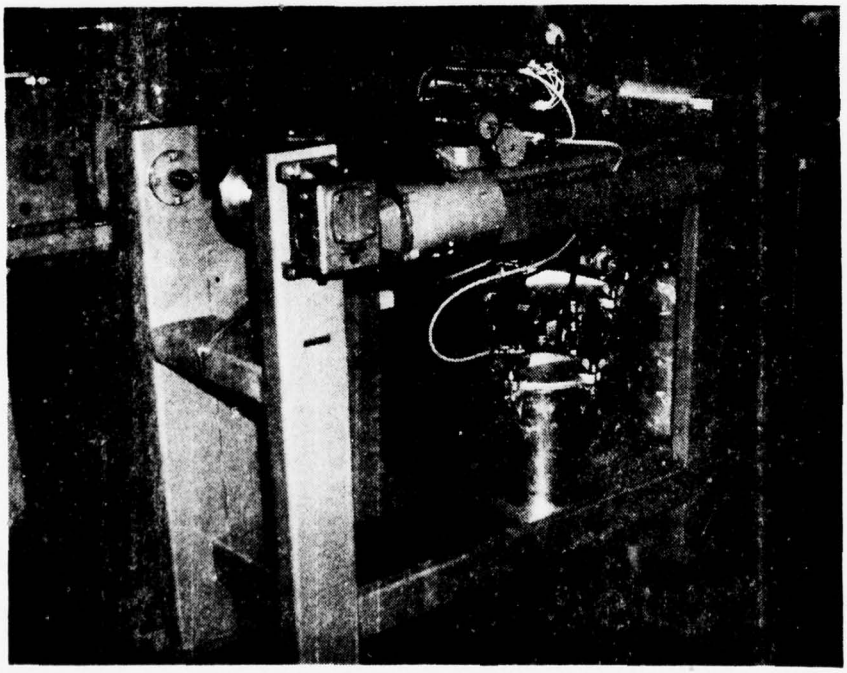

Figure 7. Impregnator and Pressure Pots as Received

3. PROGRAM PLAN

The program approach used in the development of a direct impregnation system consisted of three phases, as described in BHT's proposal 299-199-278. A description of these phases is presented below.

3.1 PHASE I - ENGINEERING PROCESS DEVELOPMENT

This first program phase consisted of two tasks:

- Task 1 - Evaluation of the impregnator and the beta gage measurement process for determination of roving mass.
- Task 2 - Establishment of the test program based on the equipment evaluation.

Task 1 consisted of operating and evaluating the impregnation equipment to determine its operational nature and the process parameters affecting impregnation. Equipment evaluation consisted of evaluating the fiber heating unit, resin heating system, metering pumps, and resin delivery system. The feasibility of applying beta gage measurement for determination of roving mass was explored through discussions with preimpregnators and beta ray equipment manufacturers. Samples of non-impregnated and preimpregnated roving were sent to two equipment manufacturers for trial tests on their equipment.

Task 2 consisted of formalizing the variables to be evaluated and the detailed program test plan. This task included procuring the fiber and resin materials to be evaluated. The fiber materials selected for evaluation were S-2 fiberglass roving, "Kevlar" 49, and graphite roving. The resins selected consisted of high and low viscosity bisphenol-A resins and a solid (at room temperature) epoxy-novalac resin. The hardener used with all of the resins was a high viscosity aromatic amine type.

The program test matrix proposed is presented in Table 1. The basic test philosophy was to conduct a general evaluation of process parameters using the less expensive S-2 fiberglass roving, and to determine the most suitable resin type and temperature to be used for the succeeding evaluations of Kevlar and graphite fibers.

3.2 PHASE II - PROCESS EVALUATION

This phase consisted of conducting the evaluative portion of the test plan, test series AA through JC (refer to Table 1). The evaluation test methods employed consisted of resin content determination, short-beam shear testing, and microscopic evaluation.

TABLE 1. PROPOSED PRINCIPAL TEST MATRIX

MATERIALS/VARIABLES	SAMPLE CODE	PROCESS CONDITIONS: RESIN			PROCESS CONDITIONS: RESIN TEMPERATURE				PROCESS CONDITIONS: FIBER TEMPERATURE				PROCESS CONDITIONS: ROVING VELOCITY			TEST TYPE				
		LOW VISCOSITY	HIGH VISCOSITY	HOT MELT	ROOM TEMP	100°F	150°F	200°F	ROOM TEMP	100°F	150°F	200°F	1 FPS	2 FPS	4 FPS	SEM	RESIN CONTENT	TUBULAR COMP.	UNI-DIRECTIONAL TENSION	SHORT-BEAM SHEAR
PHASE II																				
20-End S-Glass Winding Speed	AA	■			■				■				■			A/Req	■			■
	AB	■			■				■					■		A/Req	■			■
	AC	■			■				■						■	A/Req	■			■
Resin Temp.	BA		■			■			■				■			A/Req	■			■
	BB		■				■		■				■			A/Req	■			■
	BC		■					■	■				■			A/Req	■			■
Fiber Temp.	CA		■							■			■			A/Req	■			■
	CB		■		Optimum						■		■			A/Req	■			■
	CC		■									■	■			A/Req	■			■
Fiber Temp. Hot-Melt Resin	DA			■	Manufacturer's Recommendation					■			■			A/Req	■			■
	DB			■							■		■			A/Req	■			■
	DC			■								■	■			A/Req	■			■
60-End S-Glass	EA	Optimum			Optimum				Optimum				Optimum			A/Req	■			■
Kevlar	FA								■				■			A/Req	■			■
	FB	Optimum			Optimum				■					■		A/Req	■			■
Winding Speed	FC								■						■	A/Req	■			■
Fiber Temp.	GA									■			■			A/Req	■			■
	GB	Optimum			Optimum						■		■			A/Req	■			■
	GC											■	■			A/Req	■			■
Graphite	HA								■				■			A/Req	■			■
	HB	Optimum			Optimum				■					■		A/Req	■			■
	HC								■						■	A/Req	■			■
	JA									■			■			A/Req	■			■
	JB	Optimum			Optimum						■		■			A/Req	■			■
	JC											■	■			A/Req	■			■
PHASE III																				
S-Glass	KA															A/Req	■	■		
	KB	Optimum			Optimum				Optimum				Optimum			A/Req			■	
	KC															A/Req				■
Kevlar	LA															A/Req	■	■		
	LB	Optimum			Optimum				Optimum				Optimum			A/Req			■	
	LC															A/Req				■
Graphite	MA															A/Req	■	■		
	MB	Optimum			Optimum				Optimum				Optimum			A/Req			■	
	MC															A/Req				■

The resin-to-fiber ratio of uncured roving was determined by weighing 30-foot lengths of dry and wet roving. The resin content of cured specimens was determined by the "burn-off" method per ASTM D 2584 for S-2 glass, and the nitric acid extraction method per ASTM D 3171 for Kevlar and graphite.

Short-beam shear testing, in conjunction with microscopic examination, was used to measure wetness consistency or quality of impregnation. The short-beam shear specimens were prepared and tested per ASTM D 2344.

The microscopic evaluation was conducted using the scanning electron microscope (SEM). This evaluation method provides visualization of the resin/fiber structure enabling observation of resin/fiber distribution and void location.

The evaluations conducted in this phase were divided into three tasks representing the evaluation of S-2 glass, Kevlar, and graphite fibers, respectively.

3.3 PHASE III - PROCESS VERIFICATION

This phase consisted of evaluating the unidirectional tension, tubular compression, and short-beam shear properties of fibers impregnated using the process conditions established in Phase II. Three task areas were established representing the testing of S-2 glass, Kevlar, and graphite fibers, respectively. Table 1 represents the test matrix.

4. RESULTS AND DISCUSSION

4.1 PHASE I - ENGINEERING PROCESS DEVELOPMENT

This program phase consisted of two primary tasks:

- Task 1 - Identification, definition, and study of impregnation process parameters.
- Task 2 - Establishment of the test program.

4.1.1 Task 1 - Identification, Definition and Study of Impregnation Process Parameters. This initial task consisted of evaluating the roller impregnator and the feasibility of applying beta ray transmission for providing in-process control of resin content.

This evaluation entailed operating the equipment, determining general improvements and conducting evaluations of each of the subsystems. The fiber heating, resin heating, resin delivery, and delivery manifold systems were evaluated.

4.1.1.1 Fiber Heating System. A series of tests were conducted to determine the heater voltage requirements to maintain specific roving temperatures at given delivery rates. The determination of heater voltage was required for setting the high and low limits on the heater control for the applicable roving velocity range. For these tests, the roving was in direct contact with the quartz heaters.

The general relationship between the roving temperature and the relative heater voltage is illustrated in Figure 8.

Roving tension was found to affect this relationship. Although it was not measured, increasing the tension generally increased the roving temperature. This temperature increase is attributed to tension keeping the roving in intimate contact with the quartz heater surface, thus improving heat transfer.

The quartz heater protective tubes required 15-20 minutes to reach a stable operating temperature after start-up. This delay caused a corresponding delay in the temperature rise of roving processed through the heaters. The electronic control system for the heaters was found to "drift" upward increasing the heater voltage as the controller became warm with operation, thus requiring continual manual adjustment. Due to the above considerations, an alternate heater was used to provide fiber heat for the evaluation program.

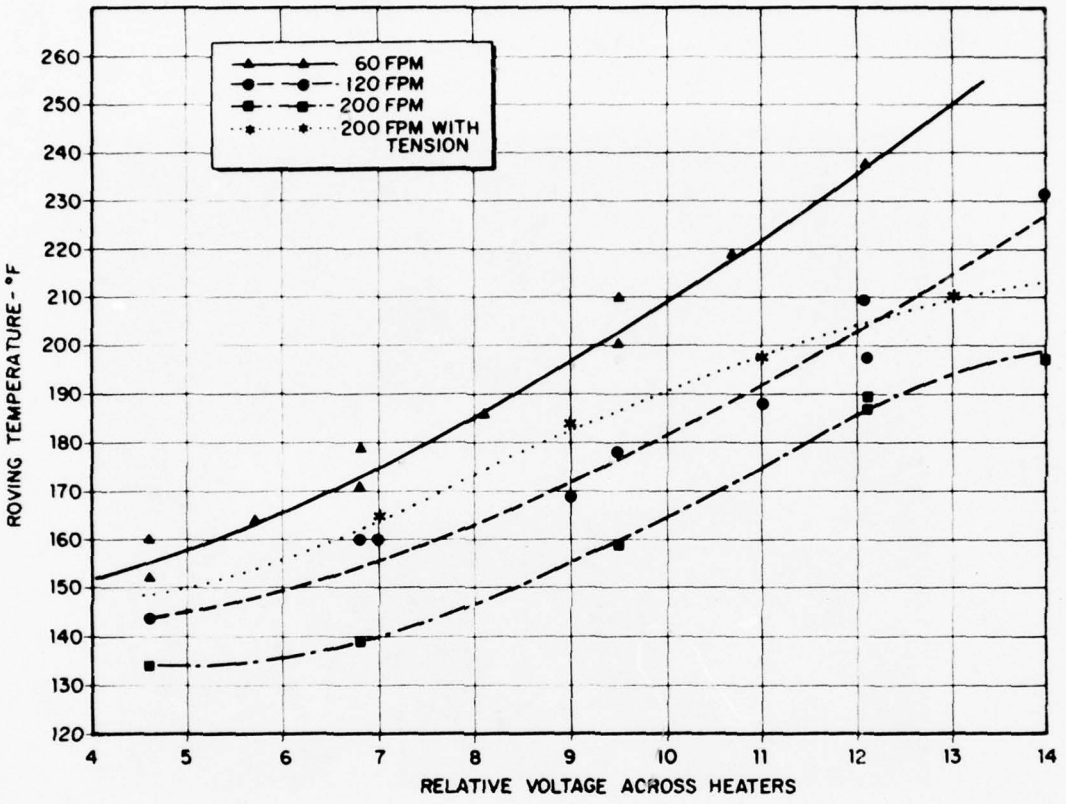

Figure 8. Fiber Temperature vs Relative Voltage

The quartz tube heating system could be used in a production winding situation (with correction of the control problem) where fibers are wound over a period of time and the response time is less critical. However, for the short runs required for winding the short-beam shear specimens, the response time was too slow.

The heater unit employed was one previously fabricated in BHT's Methods and Material Laboratory (Figure 9).

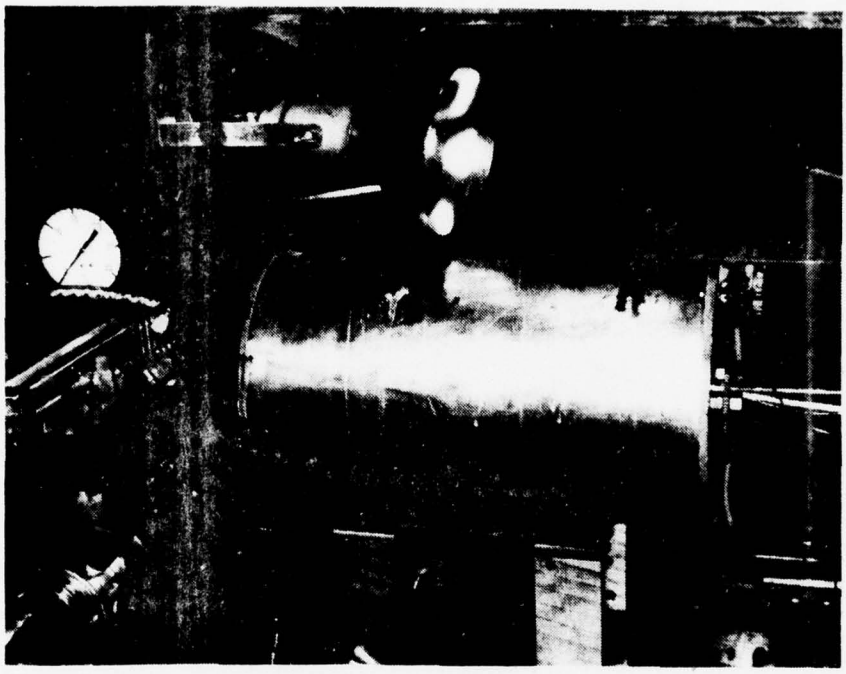

Figure 9. BHT Fabricated Fiber Heater

It consisted of a central glass tube surrounded with a resistance heating unit. For purpose of the test program, this unit was manually controlled using a variable voltage controller.

For a production application, a chamber which preheats the entire roll of roving or a preheating oven of sufficient length to heat the roving to the desired temperature without a high oven temperature should be considered.

4.1.1.2 Resin Heating System. This system was evaluated on its capability to provide heated resin and hardener to the delivery manifold at specified temperatures up to 200°F.

Initial modifications to this system consisted of adding temperature indicator-controllers to replace the controllers supplied with the equipment. A temperature indicator coupled to a 6-position thermocouple switch was also installed for monitoring temperature at a multiplicity of system locations (Figure 10). The indicator-controller units enabled monitoring of the actual resin and hardener temperature, a capability not provided on the original equipment.

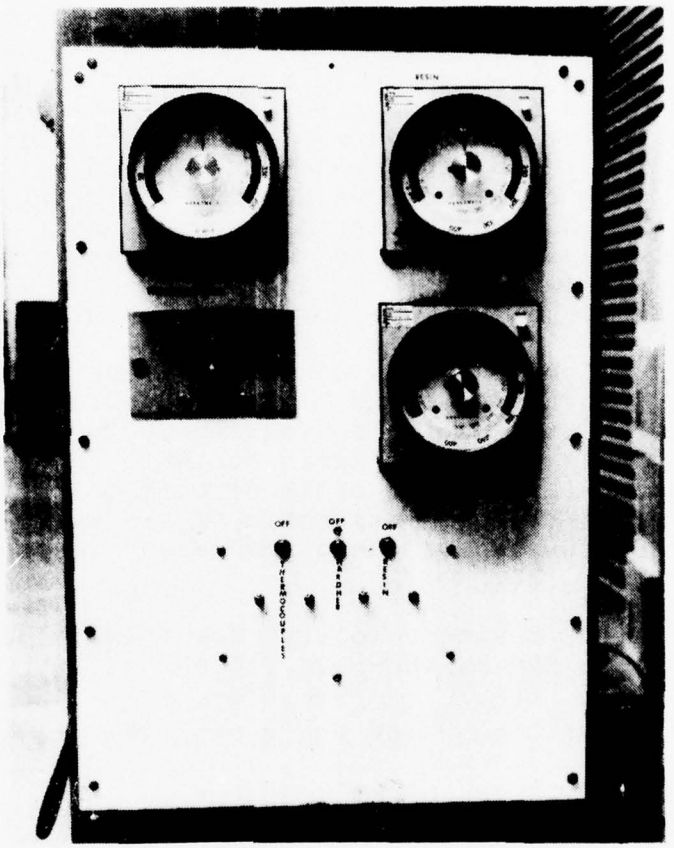

Figure 10. Temperature Indicator-Controller Panel

The resin and hardener pots were equipped with a heating system consisting of electrical resistance coils attached to the bottom of a 1/4-inch aluminum plate. It was found that the latent heat of the coil and plate caused the temperature of the resin and hardener baths to "overshoot" after controller shut-off. This condition was alleviated somewhat by replacing the 1/4-inch thick plate with a .090-inch thick plate. However, some overshoot was still realized.

For a production application, an insulated and heated pressure container is recommended with the heating elements external to the container.

4.1.1.3 Resin Delivery System. This system was evaluated on its ability to deliver resin and hardener of varying viscosities at the required delivery rate within ±2 percent accuracy. This evaluation centered primarily on the metering pumps which are the principal components of the resin delivery system. Evaluation of the delivery characteristics of the resin/hardener metering pumps entailed considerable searching for a common set of operating conditions that would enable attainment of reliable pump delivery. The metering pumps are a gear type that (typical of gear pumps) are affected by fluid viscosity, inlet pressure, outlet pressure, and operating speed.

The data from the pump evaluation is presented relative to the "equivalent roving velocity." This is a relative figure considering the amount of roving employed, the mix ratio of the resin system, and the resin content. For these tests, a resin content (by weight) of 28 percent on four 20-end rovings and a hardener-to-resin-mix ratio of 1:5 was used. The pump/roller speed ratios for the pumps were: resin pump 1:4; and hardener pump 1:20.

The "equivalent roving velocity" for these conditions corresponds to pump RPM as shown in Table 2 below.

TABLE 2. PUMP RPM FOR EQUIVALENT ROVING VELOCITY

Equivalent Roving Velocity (ft/min)	Resin Pump (rpm)	Hardener Pump (rpm)
30	14.3	2.9
60	28.6	5.7
90	43.0	8.6
120	57.3	11.5
150	71.6	14.3
180	86.0	17.2
200	95.6	19.4

When reviewing the pump data, it should be realized that the hardener pump is operating at one-fifth the speed of the resin pump, thus affecting the discharge pressures realized and other characteristics.

Initial testing consisted of determining the back pressure (output pressure) produced by the manifold at the anticipated delivery rates. These initial tests were conducted using room temperature Epon 828 resin in both the resin and hardener delivery systems. These tests were conducted with and without an adjustable needle valve in the output line downstream from the pump outlet pressure gage. Initially a 30 psi gage was used. No pressure was applied to the pressure pots for this test. The results are presented in Table 3.

TABLE 3. METERING PUMP PRESSURE EVALUATION DATA

Equivalent Roving Velocity (ft/min)	Resin Pump Pressure		Hardener Pump Pressure	
	No Valve (psi)	Valve (psi)	No Valve (psi)	Valve (psi)
30	8.4	9.5	1.8	1.7
60	15.6	20.0	3.2	3.5
90	23.2	29.0	4.7	5.0
120	25-26	off scale	6.0	7.0
150	25-25.5	off scale	8.0	8.3
180	25-25.5	off scale	9.7	10.5

These tests and several of the following tests were of an exploratory nature in order to understand the effects of operating parameters.

The next series of tests consisted of measuring the pump discharge rates with some pressure on the resin pot and back pressure on the pump discharge line. The delivery rates were determined by weighing the amount of resin and hardener delivered for a given number of pump revolutions (Table 4). No pressure was applied to the hardener pot or hardener pump outlet for these tests.

TABLE 4. DISCHARGE PRESSURE AND PUMP DELIVERY VS POT PRESSURE DATA

Equivalent Roving Velocity (ft/min)	Pot Pressure (psi)	Resin Pump Discharge Pressure (psi)	Resin Pump Delivery Rate (cc/rev)	Hardener Pump Discharge Pressure (psi)	Hardener Pump Delivery Rate (cc/rev)
60	10	20	.563	3.5	.514
120	10	20	.402	7.0	.495
120	10	20	.430	7.0	.508
180	10	off scale	.279	10.5	.508
180	10	off scale	.279	10.5	.499
60	20	20	.563		
180	20	off scale	.399		
180	20	off scale	.395		

A severe drop off in pump delivery rate with increasing speed was observed. The delivery rate at 180 feet/minute was 50 percent of the delivery rate obtained at 60 feet/minute. This variation in delivery rate was totally unacceptable and indicated a need to control pump inlet pressure.

Sixty psi and 30 psi capacity gages were installed on the inlet and outlet lines of the resin and hardener pumps, respectively (see Figure 11).

Following this gage installation a series of pressure readings were taken at various pot pressures and pump speeds, as shown in Table 5.

The discharge pressure of the hardener pump remained relatively constant with increasing pot pressure for a given speed, whereas the discharge pressure of the resin pump increased. The increased pressure realized from the resin pump is indicative of a greater delivery rate since the output conditions remained the same. Thus, in the case of the resin pump, the discharge was affected by the amount of pot pressure over the range evaluated. The hardener pump discharge did not indicate such an increase. It was concluded that, at the pot pressures used, the pressures were sufficient to maintain a supply of resin at the hardener pump inlet due to its slower speed, whereas they were insufficient for the resin pump at its higher speed thus affecting delivery.

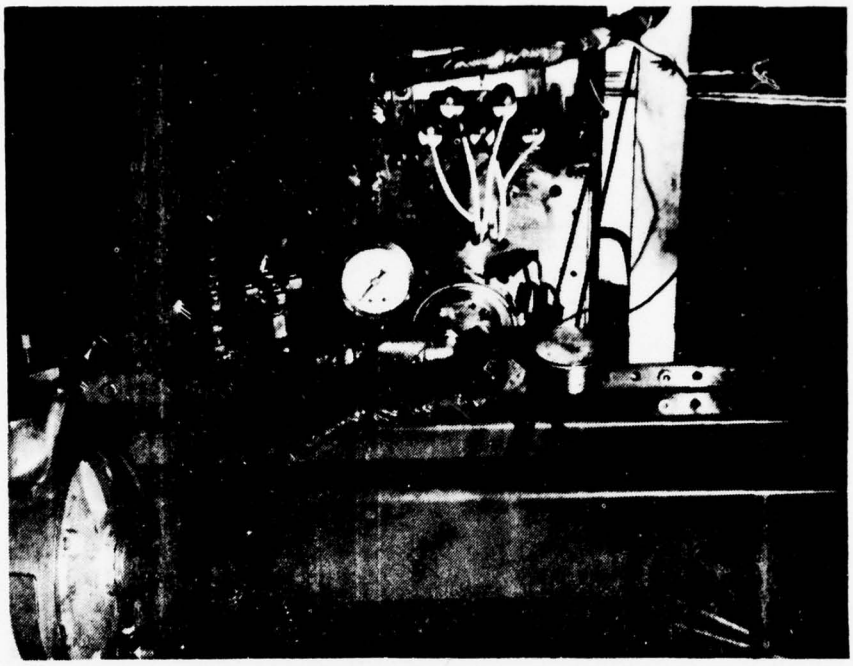

Figure 11. Pressure Gages on Pump Inlet and Outlet

TABLE 5. DISCHARGE PRESSURE VS POT PRESSURE DATA

Equivalent Roving Velocity	Resin Delivery Pressures (psi)			Hardener Delivery Pressures (psi)		
	Pot	Pump Inlet	Pump Outlet	Pot	Pump Inlet	Pump Outlet
60	20	2	17	10	6	5
120	20	0	34	10	3.5	10
180	20	0	38	10	0	16
60	30	13	19	20	17	5
120	30	0	36	20	14.5	10.2
180	30	0	36	20	11	17
60	40	25	21	30	26.5	6
120	40	10	40	30	24	11
180	40	0	62	30	21	16.5

Based on the above pressure and delivery results, measurements were taken at slightly higher pressures (Table 6).

TABLE 6. PUMP DELIVERY MEASUREMENT DATA

Equivalent Roving Velocity (ft/min)	Pot Pressure (psi)	Pump Inlet Pressure (psi)	Pump Outlet Pressure (psi)	Pump Delivery (cc/rev)
Resin Delivery				
60	40	22	21	.571
120	40	6	40	.564
180	40	0	62	.557
120	50	20	40	.588
180	50	0	62	.569
Hardener Delivery				
60	30	26	6	.528
120	30	23.5	11	.508
180	30	20.5	16.5	.508
120	40	40	40	.519
180	40	30	16.5	.499

These measurements indicated very little difference in either the discharge pressure or the delivery rate at the higher pressures, thus indicating the pressures were sufficient for this set of operating conditions. The high pressures required, however, were at the limit of the pressure pot, thus a need to reduce the resin viscosity through heating was recognized.

Due to the high pressure requirements indicated and intended use of heated resin, the delivery lines were changed from plastic on the original equipment to a medium-pressure, teflon, steel-braided line.

Following this change, resin in the pots was heated to 160°F and pressure measurements made. The temperature of the discharged resin was also measured. Results of these measurements are presented below in Table 7.

TABLE 7. PUMP PRESSURE AND TEMPERATURE LOSS DATA

Equivalent Roving Velocity (ft/min)	Pot Pressure (psi)	Pump Inlet Pressure (psi)	Pump Outlet Pressure (psi)	Outlet Temperature (°F)
Resin Delivery				
60	20	12	18	100
60	20	12	17	100
120	20	10	30	100
Hardener Delivery				
60	10	8	7	95
60	10	9	7	95
120	10	55	13	95

A higher inlet pressure at a given speed was realized (as compared to results in Table 4) indicating the effect of the reduced resin viscosity. A temperature loss through the delivery lines was also realized; 60°F for the resin and 65°F for the hardener.

Due to the above temperature loss, resistance heating tapes were added to the delivery lines and band heaters were mounted around the metering pump bases (refer to Figure 11).

The line temperature was monitored by a thermocouple on the line surface. The line temperature and band heaters were manually controlled through a variable potentiometer.

Following the addition of the heating tapes, a pump delivery rate determination was made using Epon 828 resin delivered at 150°F and 200°F. The pump inlet and outlet pressures were "balanced" at 6 psi on both the resin and hardener pumps (see Table 8).

The balancing of the pump inlet and outlet pressures was significant in reducing the variables to be controlled using this delivery system. This approach was selected because it was adaptable to automated control, and effectively eliminated the effects of two significant variables, pump inlet pressure and pump discharge pressure. With balanced inlet and outlet pressure there is a zero (negligible) pressure differential across the pumps enabling them to perform solely a metering

metering function. Although sufficiently high pot pressures could be used to maintain pump delivery, the high pressures would cause "leakage" through the pumps under static or low-speed conditions, especially when using low viscosity or heated resins.

TABLE 8. PUMP DELIVERY DATA - HEATED RESIN

	150°F		200°F	
Roving Velocity (ft/min)	Resin Pump Delivery (cc/rev)	Hardener Pump Delivery (cc/rev)	Resin Pump Delivery (cc/rev)	Hardener Pump Delivery (cc/rev)
50	.543	.531	.540	.718
50	.557	.539	.553	.786
50	.548	.511	.550	.728
71	.551	.529	.550	.707
71	.551	.517	.548	.691
71	.548	.502	.538	.624
100	.552	.499	.545	.660
	.548	.505	.543	.684
	.550	.505	.545	.671
142	.557	.511	.546	.662
	.548	.531	.550	.669
	.550	.522	.543	.661
200	.558	.529	.549	.652
	.552	.514	.546	.612
	.567	.514	.550	.580
280	.559	.537	.545	.523
	.547	.534	.552	.538
	.555	.517	.548	.546
Recheck of Hardener Pump Delivery				
50				.517
				.514
				.512
				.517
280				.506
				.523
				.511
				.519

Statistical treatment of the data in Table 8 yields the following:

	Resin Pump	Hardener Pump
Mean delivery rate (150°F and 200°F results)	.5495 cc/rev	.5179 cc/rev
Standard deviation	.0056 cc/rev	.0109 cc/rev
Confidence interval for ±2% variation	95%	65%

The above analysis indicates that the resin pump will operate within the ±2 percent target range 95 percent of the time and the hardener pump 65 percent of the time. The scatter associated with the hardener pump results is considered to be due to the slow operating range of the hardener pump which magnifies the percent of delivery change due to leakage caused by minute pressure imbalances. Due to the reliability exhibited by the resin pump operating in a higher range, it is safe to conclude that the hardener pump would exhibit similar accuracy when operated in a production situation employing more moving and thus a higher rate of delivery, or operating at a higher hardener-to-resin-mix ratio.

As can be noted from the data, both pumps had a very consistent delivery rate using 150°F resin over a wide speed range. At 200°F, however, the hardener pump delivery rate was higher than anticipated and did not agree with the results obtained at 150°F. An evaluation of the test method revealed that the amount of discharge measured at low rates was affected by the orientation of the discharge line due to resin running out of the line rather than being pumped out. To eliminate this factor, an eye dropper-shaped piece of glass tubing was installed in the discharge line and a recheck conducted of the hardener pump delivery rate at the high and low speeds (refer to Table 8). As can be noted from the data, the delivery rate was similar and consistent at both speed extremes.

Based on the data in Table 8, it was concluded that the pump discharge was predictable and relatively constant over a wide speed range using balanced inlet and outlet pressures. While conducting these tests, the tendency of the pumps to leak resin while stationary with positive pressure on the inlet was noted. This characteristic was especially severe with the resin at 200°F (lower viscosity). This leakage was attributed to gear-gear and gear-housing clearance in the pumps.

The pump gear-housing clearances were measured and recorded in Table 9 below.

TABLE 9. PUMP GEAR-HOUSING CLEARANCE

	Resin Pump Clearance (inches)	Hardener Pump Clearance (inches)
Drive Gear	.0013	.0011
Driven Gear	.0008	.0019

Based on the pump construction and the experience gained, it is recommended that the pump delivery rate be measured whenever the pump gears are changed. Care also should be exercised in assembly and disassembly of the pumps to preclude forming burrs on the housing details which would increase gear-housing clearance and thus increase pump leakage.

Following determination of the pump delivery rates, the resin content was determined on 4 strands of S-2 roving processed through the impregnation equipment at 50, 100, and 200 fpm. Epon 828 resin was heated to 150°F and used for these tests. Data are shown in Table 10 below.

TABLE 10. RESIN CONTENT VERIFICATION DATA

Trial No.	50 Ft/Min	100 Ft/Min	200 Ft/Min
1	29.63	29.07	28.88
2	28.7	29.25	27.14
3	29.82	28.31	29.04

Predicted Resin Content = 28%

These tests reconfirmed the capability of the pumps to deliver within the ±2 percent program objective.

4.1.1.4 Delivery Manifold System. The delivery manifold was evaluated on its capability to effectively distribute the resin uniformly onto the roving, and also on its capability to mix the resin and hardener. The original manifold consisted of a dual chambered unit where the resin and hardener were pumped through their respective chambers and applied through separate orifices to the bottom of the roving as it passed over the orifices. This unit applied the resin/hardener to the bottom of the roving and depended on the mechanical working of the resin and roving passing through the rollers to accomplish mixing.

Initial operation of the impregnator produced very uneven distribution of the resin to the 4 rovings processed. The distribution was uneven between strands and nonuniform within a given strand. It was found that the roving, under slight tension, passing over the orifices tended to seal them causing back pressure to build in the manifold. A relaxing of tension on one roving (due to nature of winding pattern on the roving roll) caused a spurt of resin to be applied to that particular roving. Removing the roving tension minimized this condition, but uneven resin application was still realized.

This problem was alleviated by machining a relief in the manifold surface creating a small reservoir for the resin and hardener discharge that could not be sealed off by the roving. This alteration eliminated the pressure build-up/relief condition.

During the course of operation, it was observed that improved wetting of the fibers could be realized by applying the resin to the top as well as to the bottom of the roving. To accomplish this using the original manifold, two holes were drilled (above the roving location) through the manifold divider fins at an angle to intersect the roving chamber (refer to Figure 12).

This modification enabled the resin to be pumped above as well as under the roving strands. A crude brush arrangement was rigged in order to create a resin dam, thus creating a puddle through which the roving was drawn and in turn applied the resin to all external surfaces (see Figure 13).

The application of resin to both the upper and lower surfaces significantly improved wetting of the roving. The brush arrangement, however, required constant attention and continual adjustment.

A roller manifold was designed and fabricated whereby the resin was applied through rollers contacting the upper and lower roving surfaces. The resin was applied to the rollers through orifices behind the rollers, as shown in Figure 14.

Figure 12. Modified Delivery Manifold

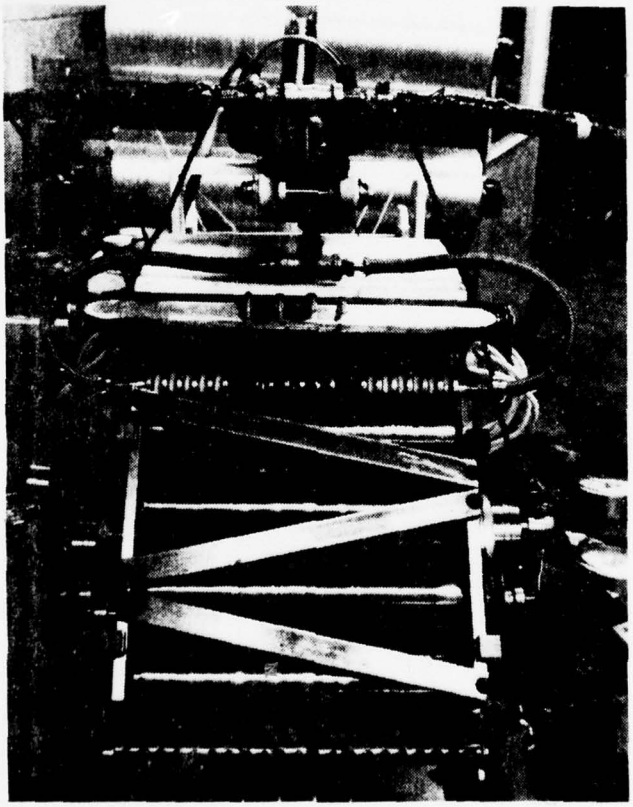

Figure 13. Modified Manifold with Brush Arrangement

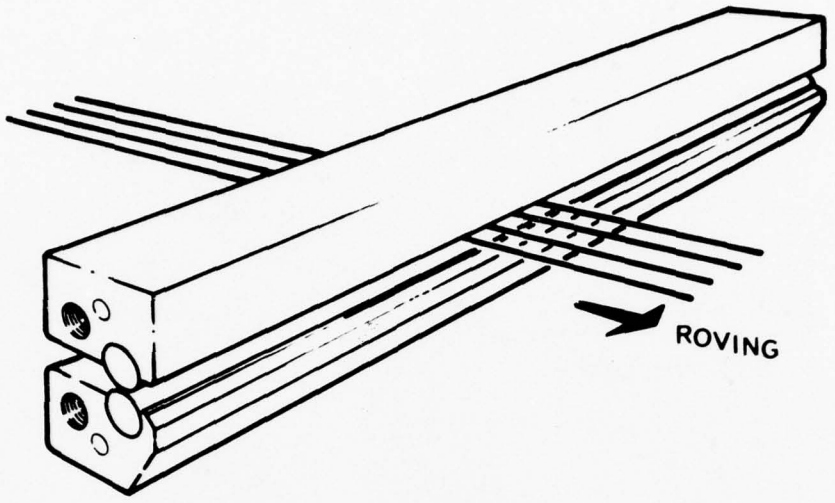

Figure 14. Roller Manifold Concept

This unit was not successful due to nonapplied resin accumulating between the fiber paths, and in turn being scraped off as the roller turned. This approach was abandoned; however, it was recognized this approach would be feasible if the roving was uniformly spread (i.e., in a tape impregnating application) prior to entering the manifold.

The next manifold approach attempted consisted of passing the roving through a .090-inch diameter orifice and applying the resin to the roving near the entrance to the orifice. Figure 15 represents a section view of this manifold.

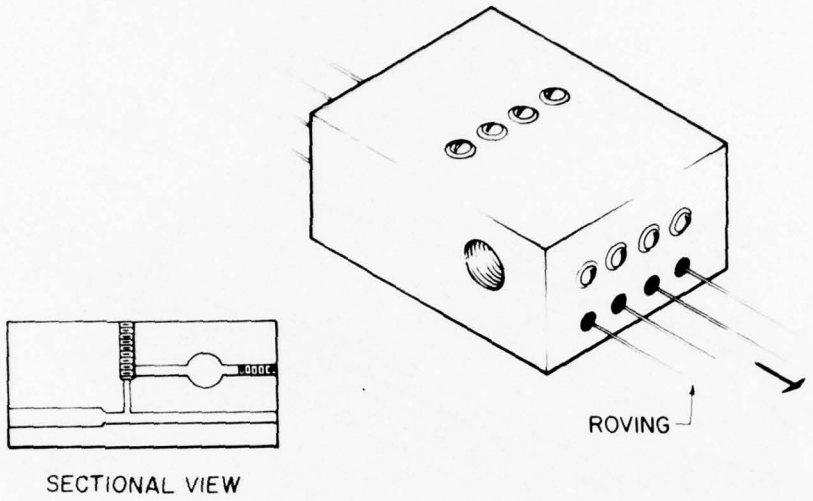

Figure 15. Orifice-Type Manifold

This method relied on the close proximity of the roving and resin passing through the orifice to apply the resin to all surfaces. This concept distributed the resin as effectively as the brush manifold without the adjustment problems. Later in the program, during the impregnation of graphite fibers, this approach was found to have a definite draw back. A considerable amount of "fraying" of the fibers was experienced during the evaluation, and the fibers were found to be susceptible to breakage. The loose or frayed fibers tended to accumulate at the orifice entrance causing a constriction, which in turn produced more fraying. Frequent clearing of the manifold was required.

The manifold concepts tried to date are not considered suitable for production use. Additional development is required in this area to produce a manifold that will uniformly apply resin to the upper and lower roving surfaces without fiber damage.

The quality of mixing was evaluated by adding a blue colorant to the resin in the resin pot and a yellow colorant to the resin in the hardener pot. The quality of mixing was determined visually by the uniformity of the "green" resin produced by mixing the colored resins. The colored resins were pumped

through their respective delivery systems and applied to roving being processed through the impregnator. Poor distribution and mixing of the resin components was observed. A predominance of hardener was distributed onto the roving nearest the hardener inlet, with little hardener reaching the fourth roving from the inlet. A similar condition was observed with the resin distribution. These conditions are shown in Figure 16.

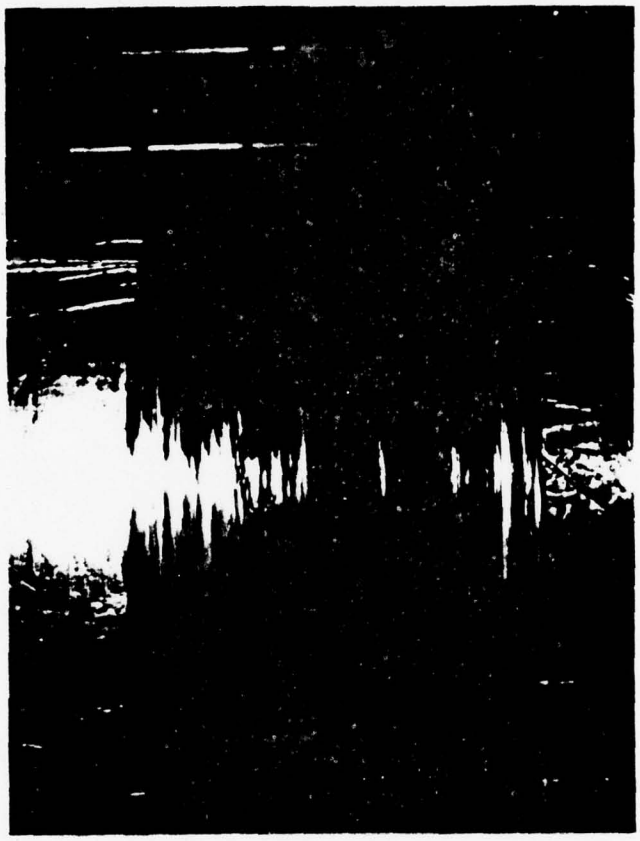

Figure 16. Poorly Mixed Pigmented Resin

A color variation was also experienced along a given roving strand (refer to Figure 17). This color variation is indicative of a lack of mixing.

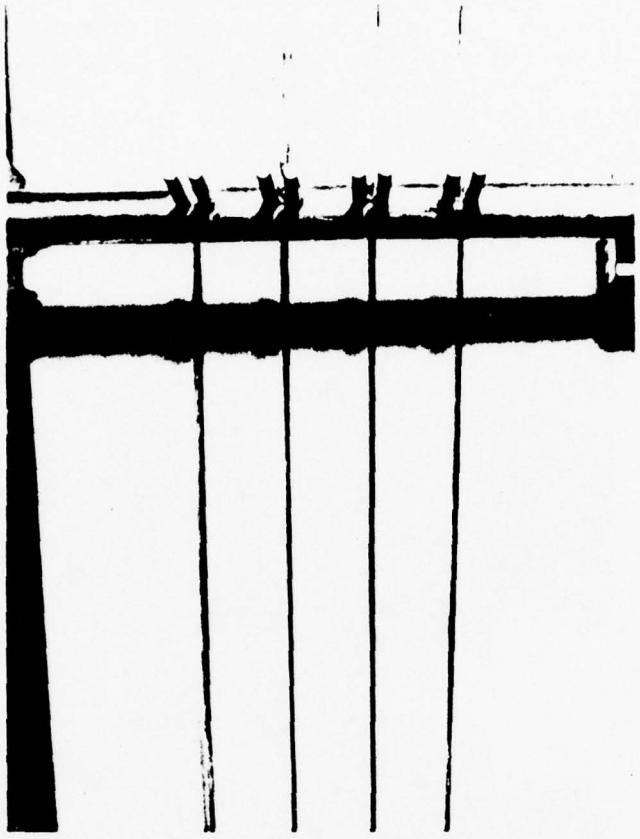

Figure 17. Poorly Mixed Resin Component Color Distribution Along Roving

The mixing problem was eliminated by flowing the resin and hardener through a static mixing unit (Figure 18) and porting the mixed resin through both ends of the distribution manifold. Heaters were added to the mixer to eliminate heat loss.

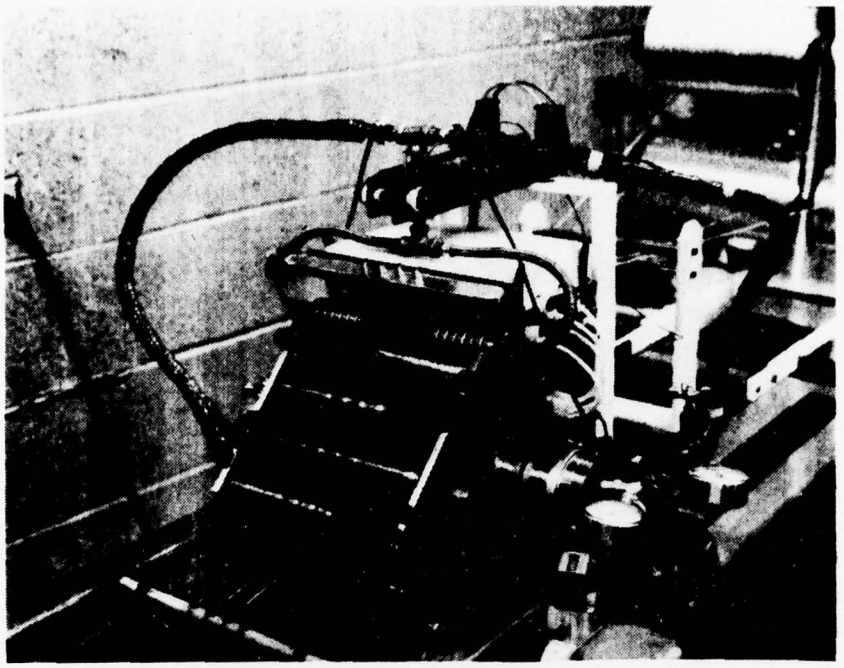

Figure 18. Impregnator With Mixing Unit Installed

The static mixing unit effectively mixed the two components producing uniformly colored green roving (see Figure 19).

Cleaning of the above unit, however, required complete disassembly and the cleaning of a multitude of internal deflectors, which was inconvenient and time consuming. This unit was subsequently replaced by a disposable unit consisting of 2 plastic spiral-shaped deflector units inserted in a plastic tube. Figure 20 represents a comparison of mixing units. The disposable unit also provided thorough mixing of resin and hardener.

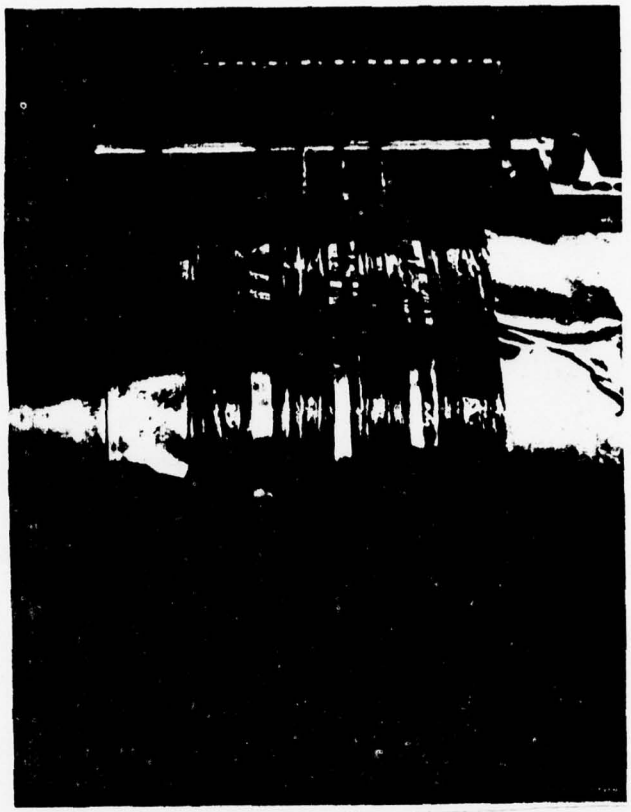

Figure 19. Uniformly Mixed and Distributed Resin

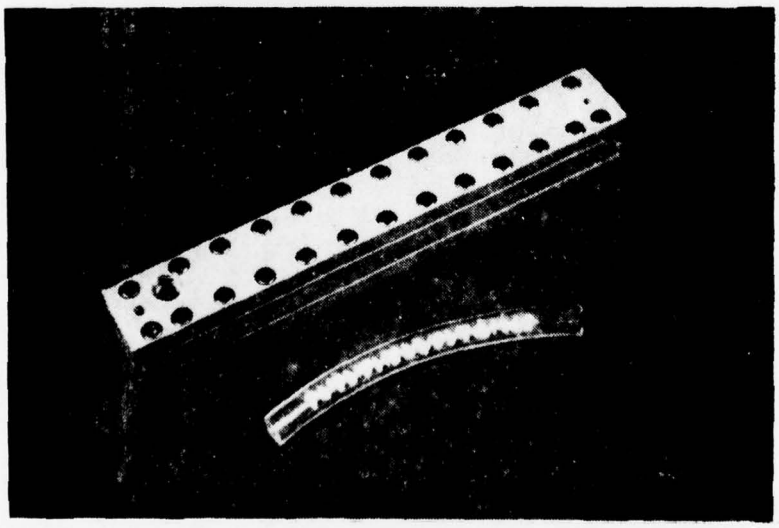

Figure 20. Comparison of Resin/Hardener Mixing Units

4.1.1.4 Delivery Manifold System. The delivery manifold was evaluated on its capability to effectively distribute the resin uniformly onto the roving, and also on its capability to mix the resin and hardener. The original manifold consisted of a dual chambered unit where the resin and hardener were pumped through their respective chambers and applied through separate orifices to the bottom of the roving as it passed over the orifices. This unit applied the resin/hardener to the bottom of the roving and depended on the mechanical working of the resin and roving passing through the rollers to accomplish mixing.

Initial operation of the impregnator produced very uneven distribution of the resin to the 4 rovings processed. The distribution was uneven between strands and nonuniform within a given strand. It was found that the roving, under slight tension, passing over the orifices tended to seal them causing back pressure to build in the manifold. A relaxing of tension on one roving (due to nature of winding pattern on the roving roll) caused a spurt of resin to be applied to that particular roving. Removing the roving tension minimized this condition, but uneven resin application was still realized.

This problem was alleviated by machining a relief in the manifold surface creating a small reservoir for the resin and hardener discharge that could not be sealed off by the roving. This alteration eliminated the pressure build-up/relief condition.

During the course of operation, it was observed that improved wetting of the fibers could be realized by applying the resin to the top as well as to the bottom of the roving. To accomplish this using the original manifold, two holes were drilled (above the roving location) through the manifold divider fins at an angle to intersect the roving chamber (refer to Figure 12).

This modification enabled the resin to be pumped above as well as under the roving strands. A crude brush arrangement was rigged in order to create a resin dam, thus creating a puddle through which the roving was drawn and in turn applied the resin to all external surfaces (see Figure 13).

The application of resin to both the upper and lower surfaces significantly improved wetting of the roving. The brush arrangement, however, required constant attention and continual adjustment.

A roller manifold was designed and fabricated whereby the resin was applied through rollers contacting the upper and lower roving surfaces. The resin was applied to the rollers through orifices behind the rollers, as shown in Figure 14.

Figure 12. Modified Delivery Manifold

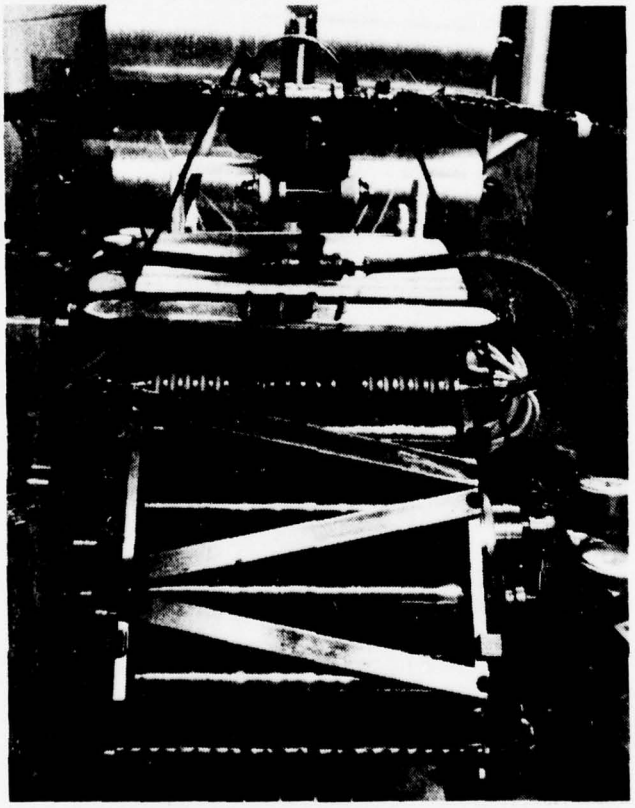

Figure 13. Modified Manifold with Brush Arrangement

Premixing the resin and hardener prior to introduction to the manifold solved the mixing problem. Flowing the resin through both sides of the manifold and applying it around the fiber improved distribution. It is recognized, however, that additional improvements could be realized through development of a better manifold.

4.1.1.5 Beta Ray Measurement Evaluation. The feasibility of applying beta ray measurement for in-process control was explored through discussions with beta ray equipment manufacturers and the submission of samples for evaluation. Samples of dry and preimpregnated roving were sent to 2 equipment manufacturers for trial on their equipment. The general conclusion of both manufacturers was that additional development would be required to apply this measuring technique to roving (even though it is currently being used in prepreg tape and broadgoods manufacturing). The beta-gage readings obtained were erratic and greatly dependent on the orientation of the strands under the beta ray beam. A quotation from the laboratory report of one of the manufacturers is as follows:

> "The readings on the bare samples were erratic, depending greatly on the geometry of the strands when being measured. The grouping of the strands was found to have a significant effect upon the readings. The coated samples gave readings that also depended upon the geometry of the strands while being measured. Since the changing geometry of the sample makes it impossible to zero the instrument and get acceptable repeatability, it is not possible to monitor the impregnation of the fiberglass by beta transmission. Geometry refers to the vertical-horizontal pattern of the cross sections of the glass fibers as they are being measured."[1]

The basic problem of applying beta ray measurement to roving, as contrasted to preimpregnated tape or broadgoods, is: (1) the inability to cover beta ray emmision source window with roving; and (2) the orientation of the fiber under the beam.

On broadgood applications, the beta ray beam scans the material and is completely covered. In the case of roving, an extremely small, emission window is required which creates a problem of back scatter. In addition, a uniform roving cross section would be required for measurement. It is doubtful that the roving geometry could be controlled with sufficient accuracy to enable ±2 percent resin variation measurement. Based on the above, it is concluded that substantial development would be required to apply beta ray measurement for roving mass measurement.

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[1]Laboratory Report, Unit Process Assemblies, Inc., Syosset, New York.

4.1.1.6 General Equipment Development. Several other general equipment and test arrangement modifications or additions were made to facilitate testing.

The pulley and belt arrangement for driving the metering pumps was replaced by a stepping-motor drive system. Since, on the original equipment, the pumps were driven from the third roller, the resin and hardener delivery rates were obtained by selecting the pulley diameter and belt size combinations that provided the desired speed ratio between the roller and pumps. This process was time consuming and in some cases the desired ratio could only be approximated. The roller torque required to drive the pumps through the belts also had the effect of increasing tension on the applied roving.

The stepping motors were mounted inside the impregnator housing, as shown in Figure 21.

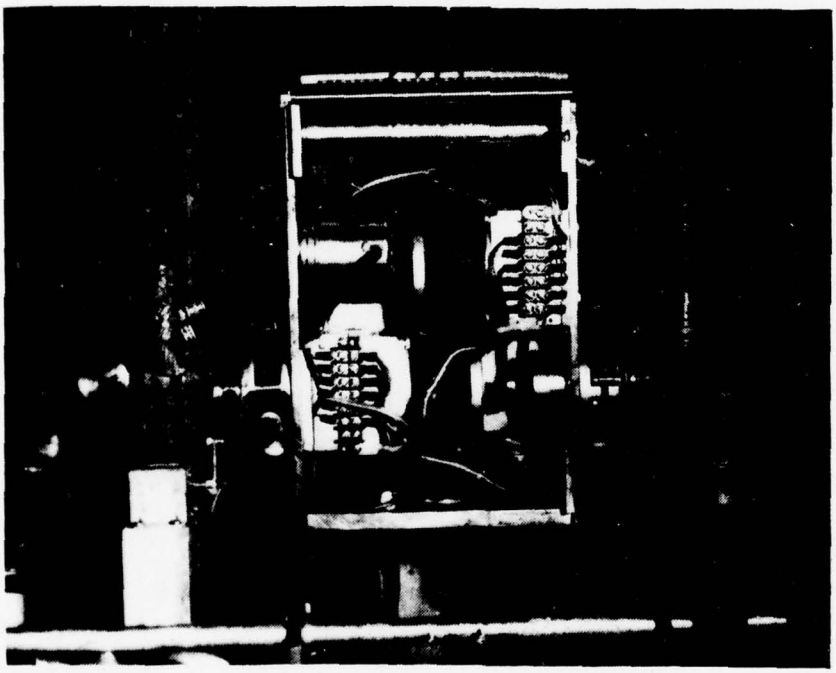


Figure 21. Stepping Motor Installation

The stepping motors are controlled through a signal generator/ translator control. The resin and hardener delivery ratio are selected at the control panel using a digital divider network employing integrated circuit chips.

Additions to the equipment arrangement to facilitate testing included roving creels with tensioners, a leveling device to distribute the applied roving, an enclosure to house the fibers at an elevated temperature, and a dust collector to contain loose graphite fibers. These items are presented in Figures 22 and 23.

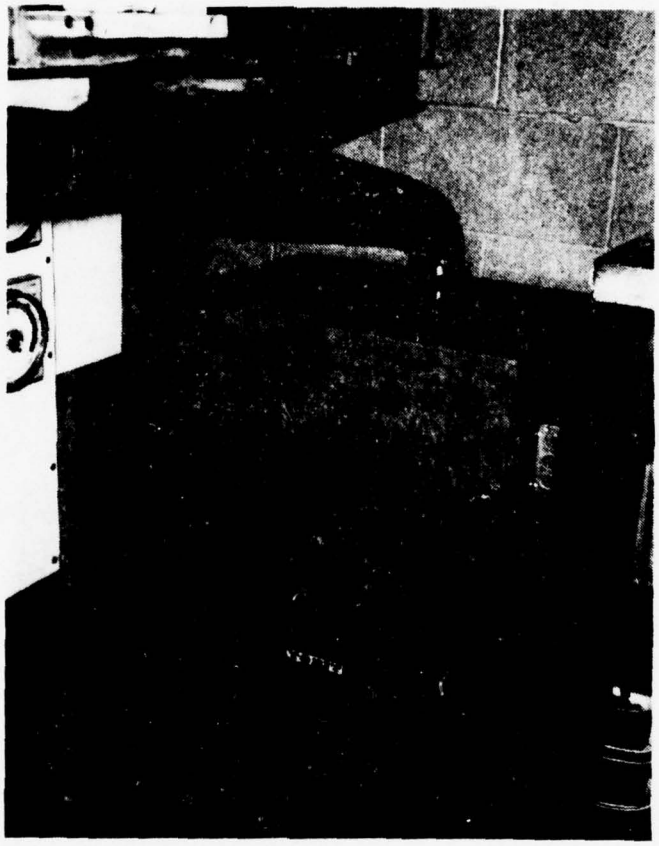


Figure 22. Dust Collector Attached To Fiber Enclosure

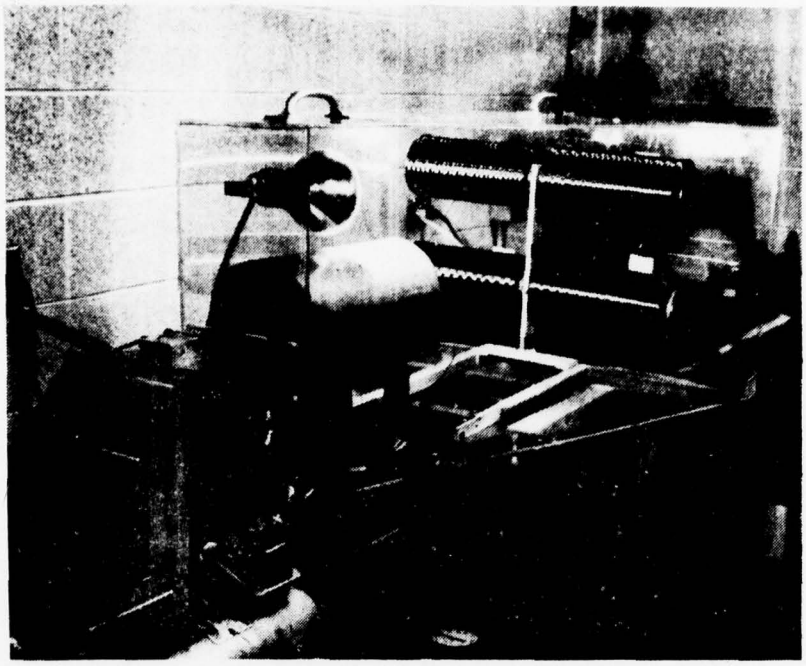


Figure 23. Fiber Enclosure

The modifications made at BHT to the basic impregnation equipment have been made with the full cognizance of Ashton Engineering, the equipment manufacturer, and in some cases were provided by the manufacturer. The stepping motor drive, static mixing, pressure balancing, and heated resin delivery modifications have been incorporated into new equipment designs by the manufacturer and are currently available to industry.

4.1.2 Task 2 - Establishment of the Test Program. The primary purpose of the test program was to evaluate the process parameters affecting impregnation quality and consistency. The finalized test program established is presented in Table 11.

The principle process variables evaluated were resin viscosity, impregnation speed, fiber temperature and resin temperature as applicable to fiberglass, Kevlar, and graphite fibers. The basic test philosophy was to conduct a general evaluation of process parameters using the less expensive S-2 fiberglass

TABLE 11. ACTUAL PRINCIPAL TEST MATRIX

MATERIALS/VARIABLES	SAMPLE CODE	RESIN: LOW VISCOSITY APCO 2434	RESIN: HIGH VISCOSITY EPON #828	RESIN: HOT MELT DEN 438	PROCESS CONDITIONS: RESIN TEMPERATURE: ROOM TEMP	RESIN TEMPERATURE: 100°F	RESIN TEMPERATURE: 150°F	RESIN TEMPERATURE: 200°F	FIBER TEMPERATURE: ROOM TEMP	FIBER TEMPERATURE: 110°F	FIBER TEMPERATURE: 150°F	FIBER TEMPERATURE: 200°F	FIBER TEMPERATURE: 250°F	ROVING VELOCITY: 50 FPM	ROVING VELOCITY: 100 FPM	ROVING VELOCITY: 200 FPM	TEST TYPE: SEM	TEST TYPE: RESIN CONTENT	TEST TYPE: TUBULAR COMP	TEST TYPE: UNI-DIRECT. TENSION	TEST TYPE: SHORT-BEAM SHEAR
PHASE II																					
20-End S-Glass	AA	0			0				0					0			A/Req	0			0
Winding Speed	AB	0			0				0						0		A/Req	0			0
	AC	0			0				0							0	A/Req	0			0
Resin	BA		0			0			0					0			A/Req	0			0
Temperature	BB		0				0		0					0				0			0
	BC		0					0	0					0			A/Req	0			0
Fiber Temp.	CA		0					0		0				0			A/Req	0			0
	CB		0					0			0			0			A/Req	0			0
	CC		0					0				0		0			A/Req	0			0
	CD		0					0					0	0			A/Req	0			0
Fiber Temp.	DA			0				0		0				0			A/Req	0			0
Hot-Melt Resin	DB			-				-			-			-			A/Req	-			-
	DC			0				0				0		0			A/Req	0			0
	DD			0				0					0	0			A/Req	0			0
60-End S-Glass	EA		0					0					0	0			A/Req	0			0
Kevlar	FA		0					0		0				0			A/Req	0			0
Winding Speed	FB		0					0		0					0		A/Req	0			0
	FC		0					0		0						0	A/Req	0			0
	GA		0					0		0				0			A/Req	0			0
Fiber Temp.	GB		0					0			0			0			A/Req	0			0
	GC		0					0				0		0			A/Req	0			0
	GD		0					0					0	0			A/Req	0			0
Graphite	HA		0					0		0				0			A/Req	0			0
Winding Speed	HB		0					0		0					0		A/Req	0			0
	HC		0					0		0						0	A/Req	0			0
Fiber Temp.	JA		0					0		0				0			A/Req	0			0
	JB		0					0			0			0			A/Req	0			0
	JC		0					0				0		0			A/Req	0			0
PHASE III																					
20-End S-Glass	KA		0					0					0	0			A/Req		0		
	KB		0					0					0	0			A/Req	0		0	
	KC		0					0					0	0			A/Req				0
Kevlar	LA		0					0					0	0			A/Req		0		
	LB		0					0					0	0			A/Req	0		0	
	LC		0					0					0	0			A/Req				0
Graphite	MA		0					0				0		0			A/Req		0		
	MB		0					0				0		0			A/Req	0		0	
	MC		0					0				0		0			A/Req				0

roving and to determine the most suitable resin type and temperature to be used for succeeding evaluations of Kevlar and graphite.

The resins for this program were selected on the basis of their respective viscosities and compatibility with a diamine hardener system. Basic resin systems common to the industry were selected. The diamine hardener system was selected primarily due to its reduced hygroscopic characteristics (as compared to an anhydride system). It was decided that a better test of the impregnation process variables could be realized without introducing the additional effects of moisture absorption on the resin system.

The materials used in this evaluation were as follows.

Fiberglass Roving	S-2 Glass, 20-end count, 449A finish, 750 yd/lb nominal yield, produced by Owens/ Corning Fiberglass.
	S-2 Glass, 60-end count, 283A finish, 250 yd/lb nominal yield, produced by Owens/ Corning Fiberglass.
Kevlar (R) Roving	Kevlar (R)-49 4560 Denier roving, Type 969, produced by E.I. duPont de Nemours & Co.
Graphite Roving	Thornel (R) 300 carbon fiber, grade WYP 30 1/0, 2600 yd/lb nominal yield, produced by Union Carbide Corp.
Resins and Hardener Materials "Low" Viscosity Resin	APCO 2434, Bisphenol-A type, 1250-1500 CPS nominal viscosity at room temperature, produced by Applied Plastics Co.
"High" Viscosity Resin	Epon 828, Bisphenol-A type, 10,000-15,000 CPS nominal viscosity at room temperature, produced by Shell Chemical Co.
"Hot Melt" Resin	DEN 438, Epoxy-Novolac Type, "Solid" at room temperature, produced by the Dow Chemical Co.

Hardener	APCO 2330, Aromatic Amine Type, 30,000-40,000 CPS nominal viscosity at room temperature, produced by Applied Plastics Co.

The hardener used with all the above resins was APCO 2330 that was mixed with the resin at 90 percent of the stoichiometric equivalent.

Neither wetness consistency nor the quality of fiber wetting are characteristics that can be measured directly. Since the interlaminar shear strength in composites is generally a function of the resin system and processing, short-beam shear tests were therefore selected as the means for determining impregnation quality.

As long as identical fiber and resin systems are used, the interlaminar shear test provides a good indication of fiber wetness. Poorly wetted fibers exhibited low interlaminar shear strength; well wetted fibers exhibited higher values.

Initially, it was intended to obtain the short-beam shear specimens for test from a race-track-shaped wound specimen (Figure 24) previously used at BHT.

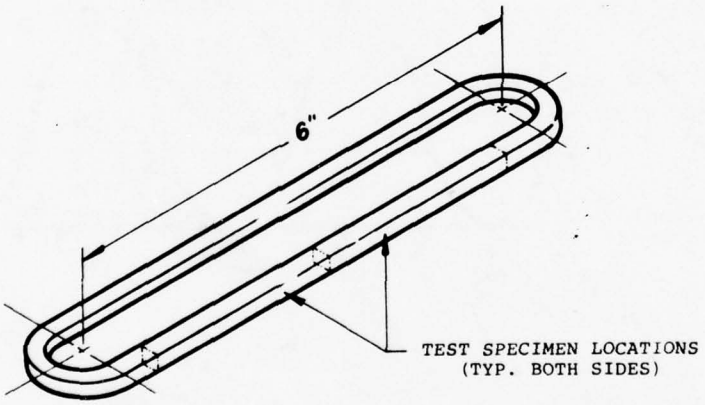


Figure 24. Race-Track Specimen

However, initial attempts at high-speed impregnation and winding of the race-track specimen were not successful. The abruptly changing winding radius resulting from this configuration produced severe fluctuations in the linear roving velocity, as shown in Figure 25.

1/4 Rev 1/2 Rev 3/4 Rev

Figure 25. Winding Fixture Positions in One Revolution

The linear velocity plot approaches a sinusoidal shape with the wave varying from near zero to maximum delivery velocity (Figure 26). This feature occurs twice with each mandrel revolution.

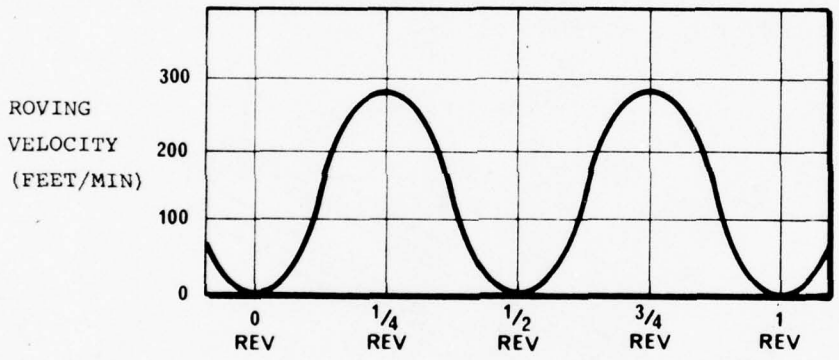


Figure 26. Roving Velocity vs Mandrel Rotation at 200 fpm (RMS)

This type demand required the impregnating system to accelerate and decelerate between these rates at a frequency of 318 cycles per minute when operating at 200 fpm (RMS) roving velocity.

A spring-loaded compensating device (Figure 27) was fabricated to smooth out the variable roving velocity.

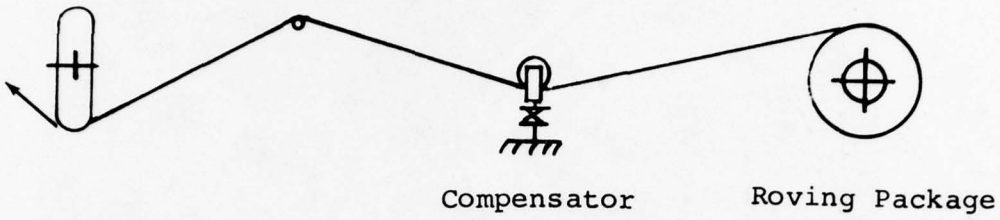


Figure 27. Compensation Device Schematic

The tension created by the compensating device combined with the drag required on the roving spools to prevent over-spin during deceleration resulted in high roving tension through the system.

After consideration of the problems associated with producing race-track-style specimens, it was decided to discard this type in favor of the short-beam shear specimen configuration per ASTM Standard D2344 that is produced on a circular mandrel (Figure 28).

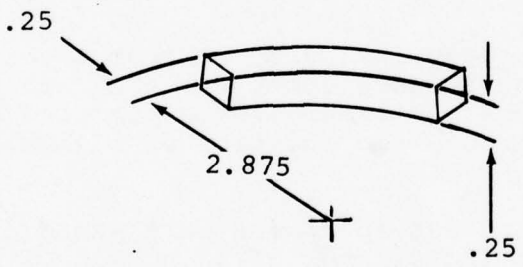


Figure 28. Short Beam Shear Specimen Per ASTM D2344

Multiple fixtures for winding and curing circular short-beam shear specimens were fabricated to expedite specimen preparation. (Refer to Figure 29 for a comparison of fixtures.)

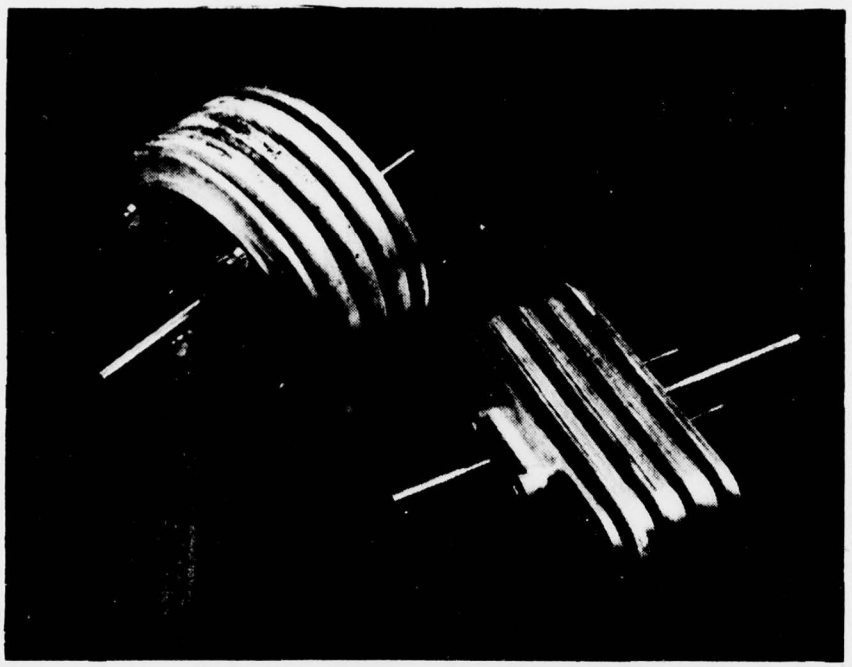


Figure 29. Comparison of Winding Mandrels

The short-beam shear samples were cut from a series of four rings wound adjacent to each other for each set of process variables evaluated. The samples were produced on the impregnation equipment through the addition of a leveling device (Figure 30).

The short-beam shear test specimens were wound on circular mandrels using 4 roving strands of each type of fiber. The 4 strands were impregnated separately through the impregnator and subsequently wound together to make a common specimen. This approach was used because it was representative of a production situation wherein all roving is applied to a common component. Four strands were selected for processing because this number was considered sufficiently representative of a production situation, and the processing of additional strands would only increase test specimen fabrication difficulty and material costs.

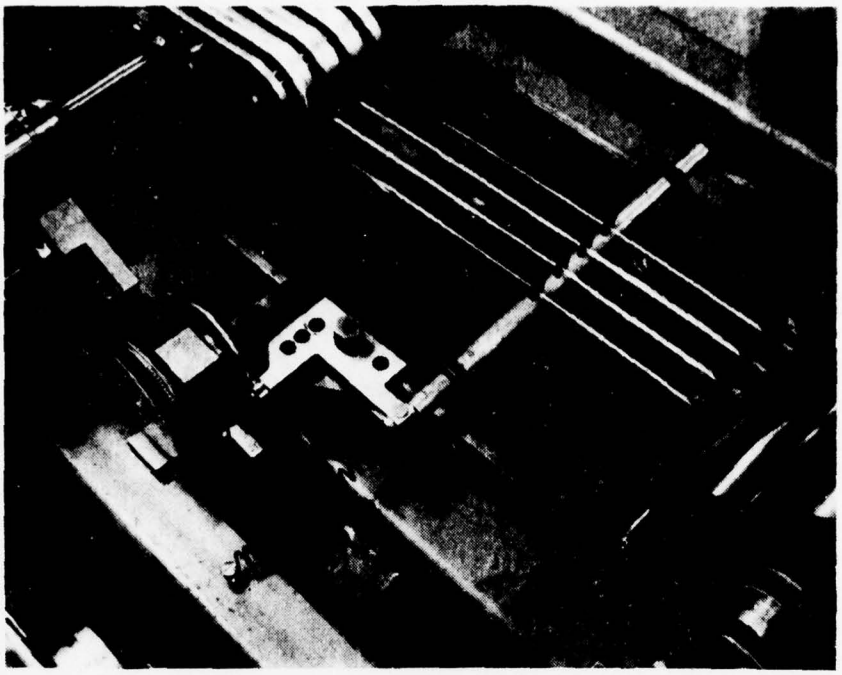


Figure 30. Roving Leveling Device

The wound samples were vacuum bagged and oven cured. With the exception of graphite specimens, the cure process was monitored through the use of dynamic dielectric cure analysis equipment and the point of vacuum application determined from monitoring the relative molecular dissipation of the specimens.

The short-beam shear tests were conducted on a Tinius Olsen UEH test machine with a load capacity of 30,000 pounds. Figure 31 represents the short-beam shear test arrangement.

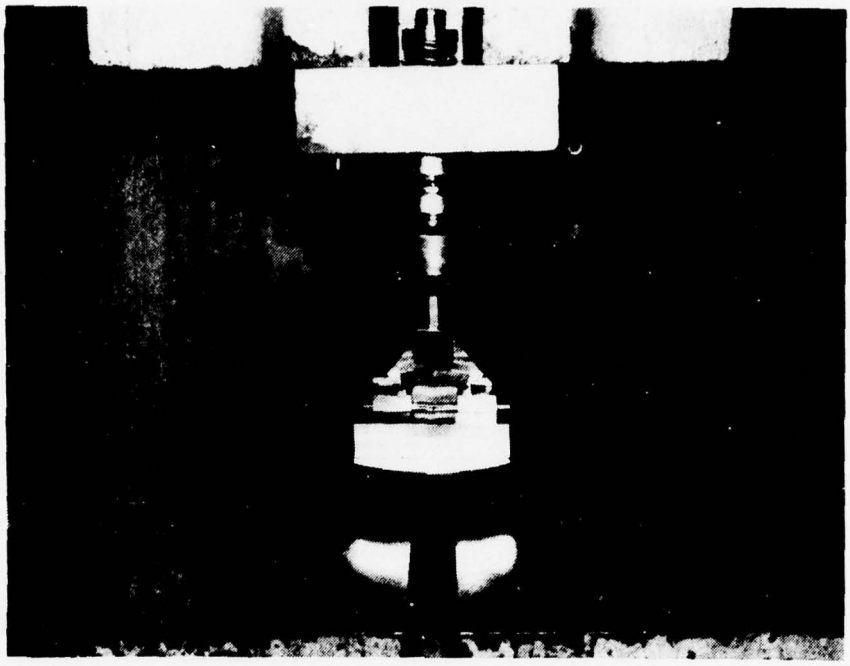


Figure 31. Short-Beam Shear Test Arrangement

The resin-to-fiber ratio of uncured roving was determined by weighing 30-foot lengths of dry and wet roving.

Due to the amount of resin bleed-out experienced during cure of the specimens, it was necessary to determine the "as-cured" resin content.

The resin content of cured fiberglass specimens was determined by the "burn-off" method per ASTM D 2584. The resin content of Kevlar and graphite specimens was determined by the nitric acid extraction method per ASTM D 3171.

The scanning electron microscope (SEM) was used to make qualitative evaluations of void content, resin/fiber wetting, and resin/fiber distribution. This procedure was used on specimens selected on an engineering judgmental basis wherein a qualitative evaluation was deemed necessary to evaluate results or explain process and test anomalies.

The test specimens for SEM evaluation were prepared by cutting and polishing sections transverse to the fiber direction from the ends of areas adjacent to the 1/4 x 1/4-inch beam shear specimens.

This method of evaluation, although it could not be quantified, provided a visualization of the resin/fiber structure. Evaluation of a cured transverse section provides a means of observing resin/fiber distribution and void location, both of which provide an insight to wetness or wetness consistency. Irregular or nonuniform fiber distribution in a specimen or voids adjacent to the fibers are both indications of poor wetting or impregnation.

4.2 PHASE II - PROCESS EVALUATION

This program phase addressed the evaluation of the impregnation process parameters of resin viscosity, impregnation speed, fiber temperature, and resin temperature as applicable to fiberglass, Kevlar, and graphite fibers. Short-beam shear tests were used as the primary test medium.

This phase consisted of three task areas corresponding to the materials evaluated.

4.2.1 Task 1 - Evaluation of Equipment and Process Variables Using S-2 Glass. The initial test results obtained using low viscosity resin were lower than anticipated. Initial experimentation consisted of varying the cure cycle and determining the shear strength. However, subsequent resin content tests conducted on the cured specimens revealed low resin content to be the problem rather than the cure cycle. The data from these tests are presented in Table 12.

The mean short-beam shear results presented in this section are the mean value of 6 to 8 specimens.

TABLE 12. SHORT-BEAM SHEAR RESULTS - CURE CYCLE VARIATION

Run No.	Sample Code	Speed (fpm)	Shear Strength Mean (psi)	Coefficient of Variation (%)	Resin Content (%)
1 Cure Cycle: 2 hours @ 150°F + 40 hours @ 250°F					
	AA2	50	5824	5.5	-
	AA3		4999	9.5	-
	AB2	100	5423	7.4	-
	AB3		5374	15.3	-
	AC2	200	2119	23.5	-
	AC3		6098	4.3	-
2 Cure Cycle: 2 hours @ 175°F + 3.0 hours @ 350°F					
	AA2	50	6998	5.7	-
	AA3		6473	5.4	-
	AB2	100	6316	4.4	-
	AB3		5917	7.0	-
	AC2	200	6934	4.0	-
	AC3		5848	9.0	-
3 Cure Cycle: 2 hours @ 150°F + 2.0 hours @ 175°F + 2.0 hours @ 250°F					
	AA2	50	5792	3.5	16.7
	AA3		5822	2.7	17.2
			6082	3.4	*
	AB2	100	5624	6.2	18.1
	AB3		5620	8.7	18.4
			5849	5.9	*
	AC2	200	5718	4.1	16.2
	AC3		5864	2.0	17.9
			6234	3.5	*
(Starting Resin Content = 28% by wt)					

* Post cured for 4.0 hours @ 350°F

Although the impregnated roving delivered by the equipment was within the target range of 28 percent resin (±2 percent), the tendency of the resin to migrate under the lowest laminating and curing pressures resulted in resin-starved specimens with unacceptable low-shear values.

Additional tests were conducted wherein the pressure was applied at various stages in the cure cycle in order to determine a suitable point for pressure application where sufficient polymerization had already occurred to restrict resin migration. As anticipated, the application of pressure during the latter cure stages increased the resin content. The optimum point of pressure application, however, was unknown.

In order to obtain the general cure characteristics of this resin system, Dynamic Dielectric Cure Analysis (DDA) tests were conducted on 3 x 3-inch patches of hand-impregnated woven glass fabric. From these tests, the nature of the molecular dissipation curve (Figure 32) was determined which provided a measure of relative resin viscosity during cure. Following these tests, one of the circular short-beam shear specimen winding mandrels was modified to provide the electrical isolation necessary to enable DDA to be conducted directly on the specimens.

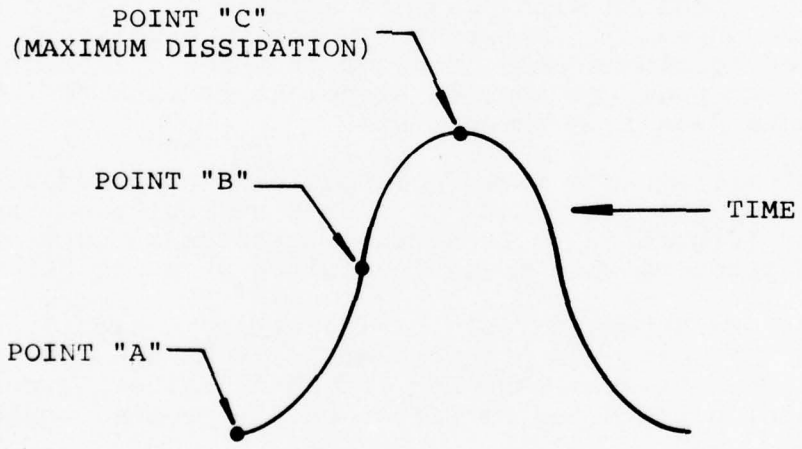


Figure 32. Typical DDA Dissipation Curve APCO 2434 Resin/2330 Hardener

The initial samples were produced at 50 rpm and pressure applied at points "A" and "B" of the DDA dissipation curve per Table 13.

TABLE 13. TEST RESULTS WITH PRESSURE APPLIED AT POINTS "A" AND "B"

Sample Code	Winding Speed (FPM)	Point of Pressure Appl.	Mean Short Beam Shear	Resin Content (%)
AA-2	50	A	6578	27.86
AA-3	50	A	6544	27.13
AA-2	50	B	7794	25.80
AA-3	50	B	8027	24.73

(Starting Resin content = 28%)

Based on the above test results, point "B" was selected for the test series for investigating the effects of winding speed on impregnation quality. Subsequent SEM photomicrographs of test specimens produced with pressure applied at this point revealed excessive porosity. Due to the porosity experienced, another series of specimens were produced at three different winding speeds with pressure applied at points "B" and "C". Table 14 represents data from these tests.

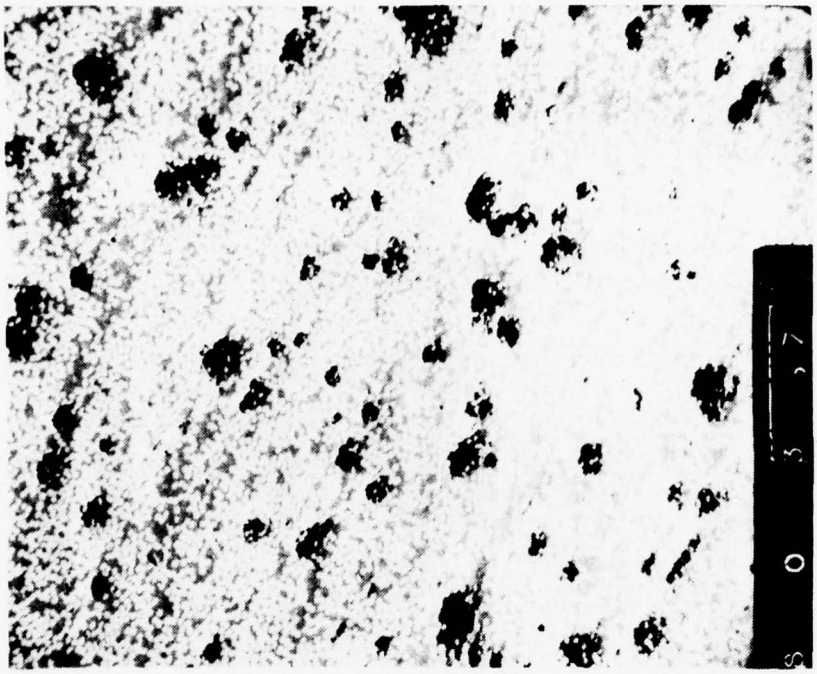
SEM photomicrographs made from specimens produced with the pressure applied at point "C" showed an equivalent amount of porosity (Figure 33). Predictably, the resin content of the samples produced with pressure applied at point "C" was low.

The data presented in Table 14 indicates no significant difference in the specimens produced at 50, 100, and 200 fpm. However, since all specimens had low shear values, a conclusion that winding speed had no effect on the process would be invalid.

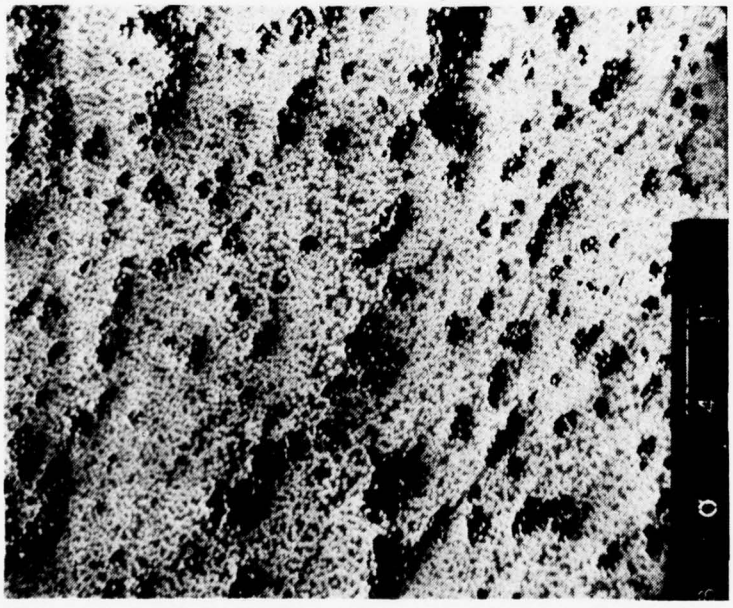
TABLE 14. TEST RESULTS WITH PRESSURE APPLIED AT POINTS "B" AND "C"

Sample Code	Winding Speed (FPM)	Point of Pressure Appl.	Mean Short Beam Shear (PSI)	Resin Content (%)
AA2	50	B	6285	24.91
AA3	50	B	7552	26.98
AB2	100	B	7128	25.25
AB3	100	B	6564	28.38
AC2	200	B	6755	26.00
AC3	200	B	7646	25.63
AA2	50	C	5896	22.98
AA3	50	C	7250	22.32
AB2	100	C	6738	19.55
AB3	100	C	7361	22.38
AC2	200	C	7209	21.07
AC3	200	C	6932	20.12

(Starting Resin Content = 30%)



Specimen with Pressure Applied at Point "B"



Specimen with Pressure Applied at Point "C"

Figure 33. Comparison of Shear Specimen Microstructure (SEM 100x)

TABLE 15. EFFECT OF RESIN TEMPERATURE ON IMPREGNATION OF GLASS FIBER

Sample Code	Resin Temp °F	Hardener Temp °F	Mean Short Beam Shear (psi)	Coefficient of Variation (%)	Resin Content (%)
BA2	100	150	7739	3.8	28.0
BA3	100	150	7485	5.7	27.3
BB2	150	150	6664	3.9	27.2
BB3	150	150	7372	8.6	28.1
BC2	200	200	7207	4.5	27.31
BC3	200	200	7314	5.9	26.63

(Starting Resin Content = 30%)

The above specimens were produced under what is considered to be suboptimum conditions (room temperature resin and glass). The porosity condition was noted and development continued with evaluation of the next variable - resin temperature.

The effect of resin temperature on impregnation quality was evaluated using Epon 828 resin and APCO 2330 hardener. Due to the higher viscosity of this resin (11,000 centipoise at room temperature), cure pressure was applied at the peak of the dissipation curve with satisfactory resin retention.

As can be determined from the data in Table 15, there is no significant difference between the samples produced with the various resin temperatures. All SEM photomicrographs of these specimens exhibited porosity similar to the porosity shown in Figure 33. The similarity of short-beam shear values was contrary to expectations. One possible explanation is that the effects of the hot resin alone becomes insignificant when applied to the room temperature glass and processed through the unheated rollers.

The next test series consisted of evaluating the effects of fiber temperature on impregnation quality using the optimum resin temperature from the preceding tests. A 200°F resin temperature was selected. Samples were produced at a winding speed of 50 feet per minute with fiber temperatures of 110°F, 150°F, 200°F and 250°F. Prior to running these tests, the glass was dried for 16 hours at 150°F and maintained in a

plexiglass enclosure at approximately 100°F. Epon 828 resin and APCO 2330 hardener were used. The results of the fiber temperature evaluation are presented in Table 16.

TABLE 16. EFFECT OF FIBER TEMPERATURE ON IMPREGNATION OF GLASS FIBER

Sample Code	Fiber Temp °F	Mean Short Beam Shear (PSI)	Coefficient of Variation (%)	Resin Content (%)
CA2	100	7427	3.0	28.8
CA3	100	7834	2.6	29.5
CB2	150	7656	11.6	27.1
CB3	150	7870	5.4	-
CC2	200	7910	2.6	27.6
CC3	200	7935	1.7	28.1
CD2	250	8216	2.9	27.3
CD3	250	8292	2.1	27.6

(Starting Resin Content 30%)

The results of these tests are presented graphically in Figure 34.

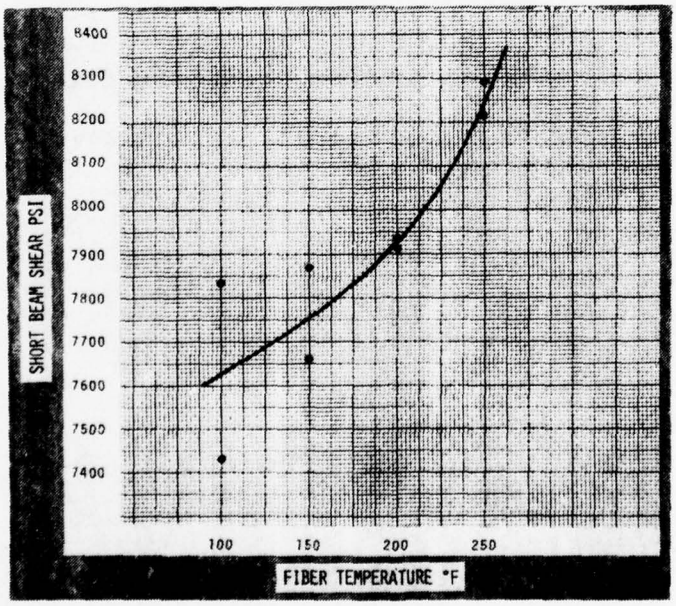


Figure 34. Shear Strength vs Fiber Temperature

The short-beam shear strength of the specimens produced from vacuum impregnated roving indicated no significant improvement over the corresponding specimens produced from roving impregnated in air. The reason for the lack of anticipated improvement is not totally known, but one explanation is that exposure time was too short to enable migration and removal of the entrapped air. Due to time constraints of the test schedule, this concept was not explored to the fullest extent.

The evaluation of the impregnation characteristics of the hot-melt resin was conducted using DOW DEN 438, an epoxy Novolac resin, and APCO 2330 hardener. The resin and hardener were preheated and delivered through the heated lines and manifold at a temperature of 200°F. Samples were produced with fibers preheated to 110°F (residual glass chamber temperature), 200°F, and 250°F. The short-beam shear test results of the samples produced are presented in Table 17. These samples were produced at a winding speed of 50 feet per minute and cured for two hours at 150°F, and two hours at 250°F with pressure applied prior to initiating the cure cycle.

TABLE 17. EFFECT OF FIBER TEMPERATURE ON IMPREGNATION WITH HOT-MELT RESIN

Sample Code	Fiber Temp (°F)	Mean Short Beam Shear (psi)	Coefficient of Variation (%)	Resin Content (%)
DA2	110	6361	12.2	30.5
DA3	110	6521	9.3	28.9
DA4	110	6085	9.4	-
DC2	200	6610	7.6	28.6
DC3	200	5763	12.9	29.5
DC4	200	6331	7.3	-
DD2	250	5657	12.6	31.5
DD3	250	5477	13.4	31.2
DD4	250	5845	12.2	-

(Starting Resin Content = 30%)

The increased shear values with increased temperature should be noted. A qualitative evaluation of the SEM photomicrographs taken of samples CD2 and CD3 also indicated less porosity. Figure 35, representing the microstructure of Sample CD3, is contrasted with Figure 36 that represents the microstructure of a specimen produced from preimpregnated roving. The absence of porosity in the preimpregnated sample should be noted. It was speculated that the porosity obtained is due to a lack of thorough wetting of the fiber.

In an attempt to improve fiber wetting, the roller portion of the impregnator was enclosed in order to produce a vacuum chamber, as shown in Figure 37. Specimens were fabricated using:

- 110°F glass and 150°F resin, and
- 250°F glass and 200°F resin.

The glass was impregnated under 20 inches of Hg.

Figure 35. Cured Roving Microstructure - Direct Impregnation (SEM 100x)

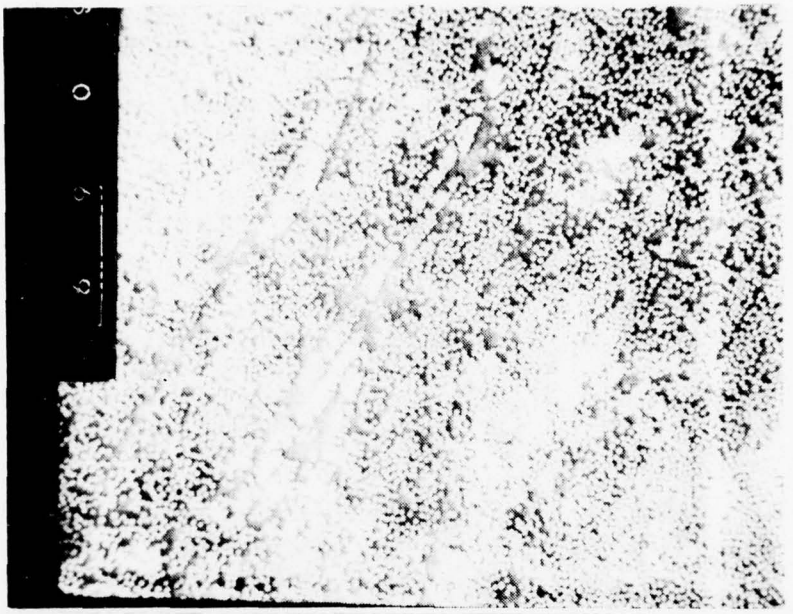


Figure 36. Cured Roving Microstructure, Preimpregnation (SEM 100x)

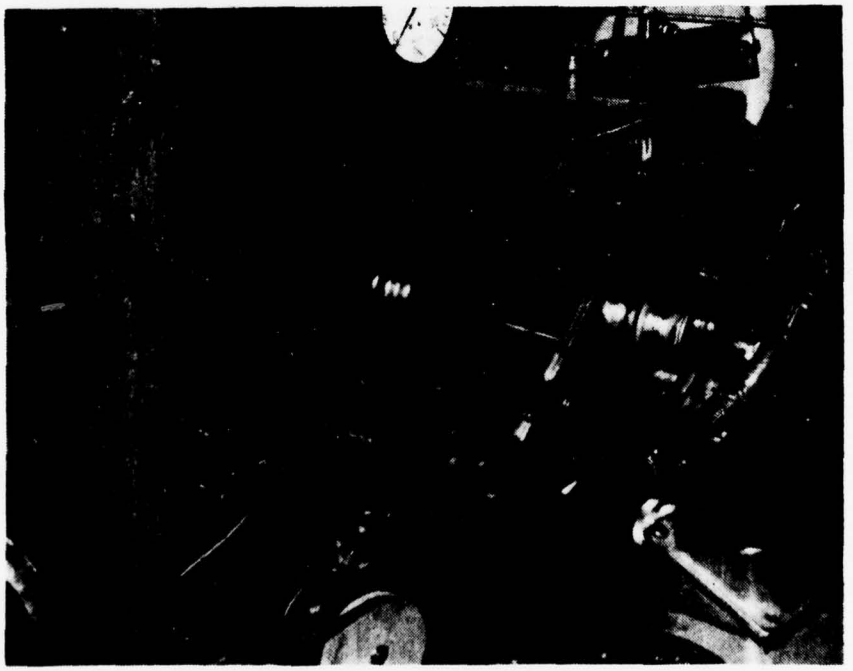


Figure 37. Vacuum Chamber Modification to Impregnator

The DEN 438 resin is solid at room temperature and has a viscosity of approximately 400-500 CPS at 200°F, the delivery temperature. The resin was pumped and delivered to the glass without significant difficulty; however, problems were experienced during impregnation. The resin, upon reaching the glass, had a tendency to congeal on the glass surface and on the rollers. The congealing was alleviated slightly by preheating the rollers to 150-160°F; however, poor impregnation was still realized.

Impregnation was slightly improved at the higher fiber temperatures but not to an acceptable level. Impregnation was characterized by resin adhering to the roving surface with very little penetration of the roving strand. The definite lack of resin around some of the fibers can be seen in the photomicrographs in Figure 38.

The ability of the impregnation system to impregnate S-2 glass of 60-end count was evaluated. As anticipated, the short-beam shear results were lower than experienced with 20-end glass impregnated under the same conditions. The roving was impregnated at 50 fpm using 200°F resin/hardener. The fibers were preheated to 250°F. The short-beam shear test results from this evaluation are presented in Table 18.

TABLE 18. SIXTY-END S-2 GLASS IMPREGNATION RESULTS

Sample Code	Mean Short Beam Shear (psi)	Coefficient of Variation (%)	Resin Content (%)
EA3	7759	4.2	27.27
EA4	6821	6.9	28.94
EA5	7054	7.2	24.93
EA6	7094	4.0	26.33
EA7	6732	3.0	25.45
	(Starting Resin content = 30%)		
Results Obtained with 20-End Glass (Reference Table 16)			
CD2	8216	2.9	27.3
CD3	8292	2.1	27.6

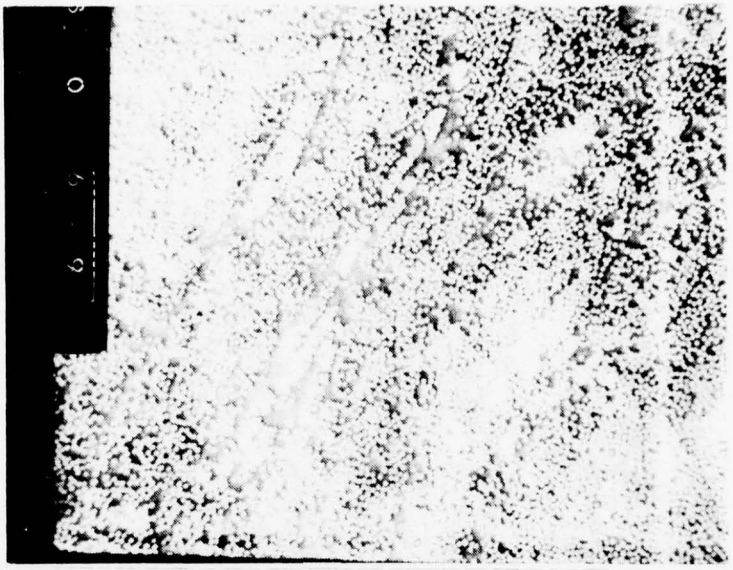
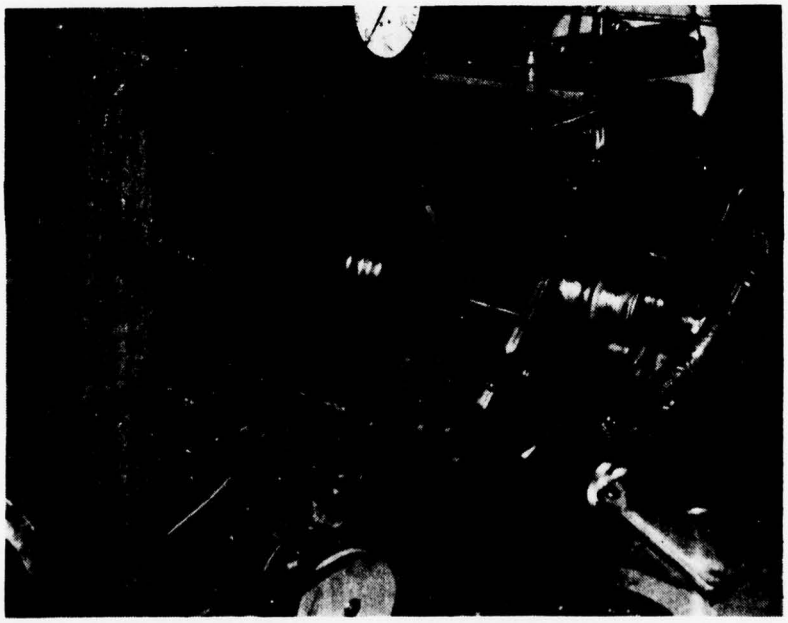


Figure 36. Cured Roving Microstructure, Preimpregnation (SEM 100x)



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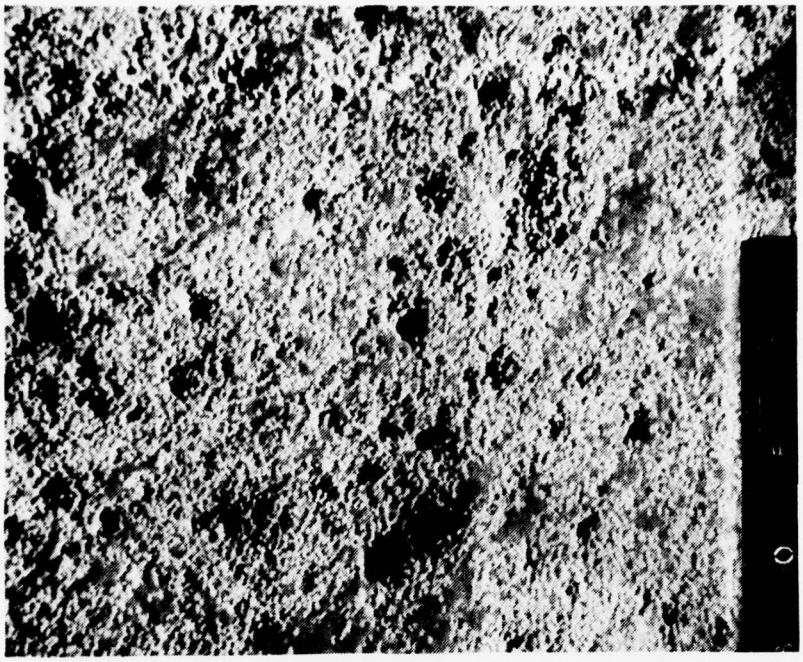
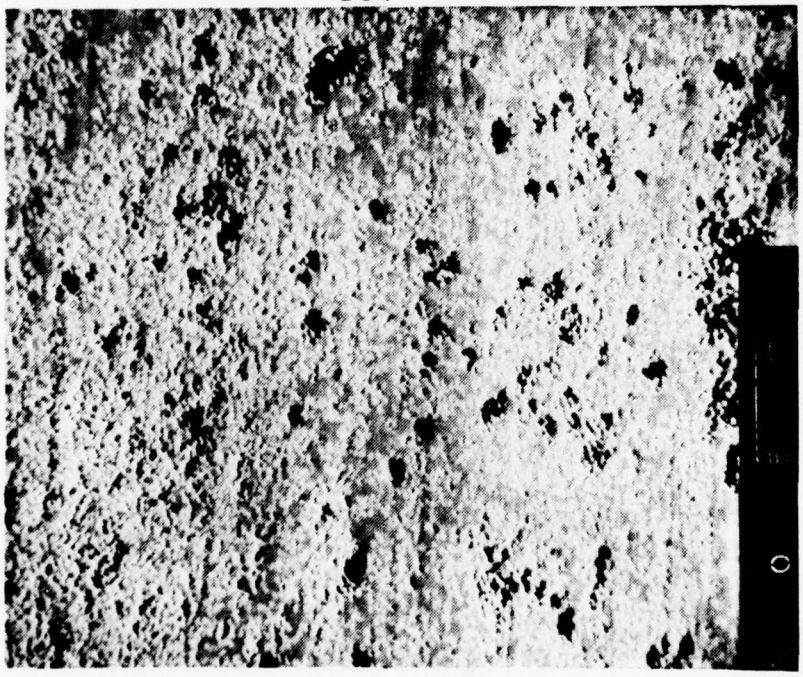
EA7	6732	3.0	25.45
	(Starting Resin content = 30%)		
Results Obtained with 20-End Glass (Reference Table 16)			
CD2	8216	2.9	27.3
CD3	8292	2.1	27.6

The compression tests were conducted on a SATEC Universal



DC2



DC3

Figure 38. Photomicrographs of Hot Melt Resin Specimens DC2 and DC3 (SEM 100x)

Based on the visual appearance of the 60-end glass, lower results were anticipated. The material was poorly wet as indicated by a resin-rich surface and a streaked white appearance (as compared to the amber translucence of thoroughly impregnated roving). The resin had a tendency to diffuse through the test specimen after winding, and this probably improved the results.

The lower short-beam shear results (as compared to 20-end glass) is attributed to poor wetting of the fiber mass. This lack of wetting is evident in Figure 39. The uneven resin-to-fiber distribution should be noted, as well as the lack of resin around some of the fibers.

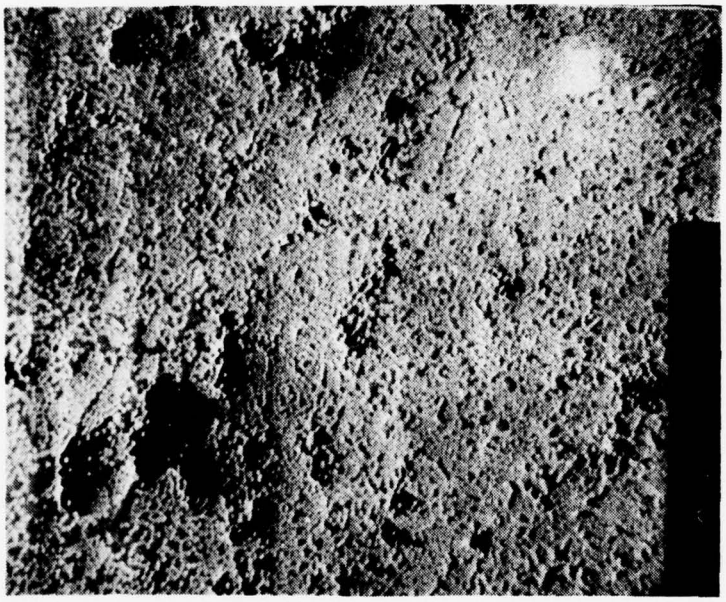


Figure 39. Typical Microstructure 60-End Glass Roving Specimens (SEM 100x)

Based on these tests, it is concluded that trying to impregnate 60-end roving with this equipment without spreading the fibers or otherwise facilitating impregnation cannot be recommended.

Based on the evaluation conducted completely on S-2 glass, one could conclude that direct impregnation is not affected by roving speed or resin temperature. This is generally contradictory to experience in the industry. The reason for the lack of difference in shear results is not known. One explanation is that the variables have minimal effect in the range evaluated. Since the results obtained were suboptimum, the true effect of these variables could not be determined from the data obtained in these tests.

The positive effect of fiber temperature on shear results was significant with the best results obtained using 200°F resin and 250°F glass.

The use of hot-melt resins with this equipment is not recommended. The congealing of the resin on the rollers and the lack of thorough fiber impregnation indicates that the total resin, fiber, and equipment system would have to be maintained at an elevated temperature until thorough impregnation is obtained.

The resin system selected for use in the succeeding Task II and III evaluations was Epon 828 resin and APCO 2330 hardener applied at 200°F. This resin was selected primarily due to the reduced resin migration experienced during cure and the satisfactory shear results obtained. The mixing and application of this resin system with a diamine hardener at 200°F does have some drawbacks however. The time at temperature reduces the resin's pot life. Also, any winding process delays introduce the potential for resin cure within the mixer and manifold. It should be recognized that the problem of resin migration during cure is also a function of the component being cured as well as the resin viscosity. Components consisting of a large roving mass and components configured or processed to restrict resin flow could be successfully fabricated using the lower viscosity resin applied at a lower temperature.

4.2.2 Task 2 - Evaluation of Equipment and Process Variables Using Kevlar. This task consisted of evaluating the ability of the impregnation system to impregnate Kevlar fiber. The evaluation consisted of determining the effect of winding speed and fiber temperature on impregnation characteristics. The tests were conducted using Epon 828 resin and APCO 2330 hardener applied at 200°F. The fiber was DuPont's Kevlar 49. All wound samples were cured for 2 hours at 150°F and 2 hours at 250°F with pressure applied at the peak of the dissipation curve.

Initial tests were conducted using 4560 Denier roving. The shear values obtained were lower than anticipated. These low values were partially attributed to poor impregnation due to the high-fiber mass/unit length of the 4560 Denier roving.

To validate this assumption, the tests were repeated using the equivalent of 2280 Denier roving (obtained by separating the 4560 Denier roving). The data from these tests are presented in Tables 19 and 20.

The starting resin content for the Kevlar evaluation was 42.6 percent by weight, which is equivalent to a fiber volume of 51.7 percent. The percent fiber volume is related to the resin percent by weight according to the following formula:

$$V_f\% = \frac{1}{1 + \frac{W_r\%}{(1-W_r\%)} \times \frac{\rho_f}{\rho_r}}$$

where: $V_f\%$ = Volume percent fiber (expressed as a decimal)
$W_r\%$ = Weight percent resin (expressed as a decimal)
ρ_f = Density of fiber
ρ_r = Density of resin

Prior to evaluation the Kevlar fibers were dried for 16 hours at 150°F. The fibers were stored during testing in an enclosure maintained at a temperature of 100-110°F.

The data from winding speed evaluation are presented in Table 19.

TABLE 19. EFFECT OF WINDING SPEED ON IMPREGNATION OF KEVLAR

Sample Code	Roving Size (Denier)	Winding Speed (ft/min)	Mean Short Beam Shear (psi)	Coefficient of Variation (%)	Resin Content (%)
FA2	4560	50	2481	5.9	27.2
FA3	4560	50	3137	4.1	25.6
FA4	4560	50	3022	8.7	51.1
FB2	4560	100	3371	5.4	51.7
FB3	4560	100	2705	12.0	51.2
FB4	4560	100	2778	11.7	26.8
FA2	2280	50	3206	4.1	29.9
FA3	2280	50	3262	5.9	29.8
FA4	2280	50	3205	6.4	33.0
FB2	2280	100	3883	4.1	34.0
FB3	2280	100	3602	4.5	30.8
FB4	2280	100	3863	1.8	31.3
FC2	2280	200	3914	6.7	29.2
FC3	2280	200	3781	2.3	27.9
FC4	2280	200	3239	4.9	29.8

(Starting Resin Content = 42.6%)

As in the previous winding speed tests, a deterioration of short-beam shear results with increasing speed was not realized. In fact, samples FA wound at 50 fpm had slightly lower short-beam shear strengths than the samples wound at higher speeds. This anomaly is attributed to test error.

The wide variation in the as-cured resin content results of the samples produced with 4560 Denier roving was recognized. This variation is attributed to a combination of sampling error and poor distribution. The resin contents were determined using the nitric acid extraction method. The samples for this method are smaller than the samples for the burn-off method, and some sampling error is undoubltedly present. The 4560 Denier roving was wet-out very poorly during impregnation as evidenced by visual inspection, as well as by the low and erratic shear strengths. The poor wetting may have caused the poor distribution of resin indicated by the resin content results.

The next series of evaluation tests consisted of determining the effects of fiber heat on Kevlar impregnation. The samples for this evaluation were all wound at 50 fpm.

The results of this evaluation are presented in Table 20.

TABLE 20. EFFECT OF FIBER TEMPERATURE ON IMPREGNATION OF KEVLAR

Sample Code	Roving Size (Denier)	Fiber Temp. °F	Mean Short Beam Shear (psi)	Coefficient of Variation (%)		Resin Content (%)
GA2	4560	110	3389	3.4		31.98
GA3	4560	110	3875	3.0		38.42
GA4	4560	110	3793	2.3		40.95
GB2	4560	150	3480	2.7		34.65
GB3	4560	150	3969	1.4		37.38
GB4	4560	150	3690	1.5		33.98
GC2	4560	200	4112	6.3		35.26
GC3	4560	200	4058	4.5		35.51
GC4	4560	200	3634	11.5		39.17
GD2	4560	250	3360	4.0		33.05
GD3	4560	250	3672	2.1		35.39
GD4	4560	250	3585	2.2		34.10
					Avg.	35.82
GA2	2280	110	4041	9.8		42.91
GA3	2280	110	4023	8.7		40.65
GA4	2280	110	4192	3.8		38.38
GB2	2280	150	4026	7.3		40.38
GB3	2280	150	4026	8.2		38.23
GB4	2280	150	3980	5.1		39.14
GC2	2280	200	3946	6.4		38.63
GC3	2280	200	4100	5.2		37.30
GC4	2280	200	3970	4.4		39.97
GD2	2280	250	4143	3.2		36.25
GD3	2280	250	4120	3.5		37.42
GD4	2280	250	3995	6.0		39.31
					Avg.	39.09

(Starting Resin Content = 42.6%)

In this evaluation, as well as in the preceding winding speed evaluation (Refer to Table 19), the 2280 Denier roving wetted better and provided better short-beam shear results than the 4560 Denier roving.

Preheating the Kevlar fibers did not improve impregnation characteristics. There was very little difference in the short-beam shear results of the samples produced with 110°F fiber and 250°F fiber.

Photomicrographs taken of the Kevlar samples revealed very little porosity in both the 4560 and 2280 Denier samples. Figure 40 compares photomicrographs of the two structures. The only discernible difference in photomicrographs of the two structures appears to be better fiber resin distribution in the 2280 Denier samples. This improved distribution, indicative of better impregnation, is partially responsible for the improved shear results. Another factor contributing to higher values is the higher resin content of the 2280 Denier samples; 39.09 percent average vs 35.82 percent average for the 4560 Denier samples (refer to Table 20). These resin contents provide fiber volume percentages of approximately 55 and 59 percent, respectively, with the higher fiber volume contributing to the lower shear values. The fact that the as-cured 4560 Denier samples had a lower resin content is significant since both samples were impregnated at 42.6 percent. Poor impregnation results in resin migrating to the surface of wound components, thus increasing the propensity for resin loss during cure.

The short-beam shear results obtained in the Kevlar evaluation were significantly lower than the results obtained with S-2 glass. Low-shear values are typical of Kevlar with values between 4,000 and 6,000 psi expected. The low results obtained are also attributed to the combined effects of the resin/hardener system, resin/fiber compatibility, and impregnation quality. The relative difference in the shear results obtained from the Kevlar specimens, however, provides an indication of the effects of the process variables investigated.

4.2.3 Task 3 - Evaluation of Equipment and Process Variables Using Graphite. This program phase evaluated the ability of the system to impregnate graphite fibers.

Prior to conducting this evaluation, impregnator modifications were required to eliminate the hazard of minute graphite filaments to electrical equipment and electronic systems. The plexiglass enclosure previously fabricated to house the fiber spools was evacuated by an industrial dust collector through a port in the bottom. The enclosure inlet was positioned at the fiber entrance to the impregnator producing an air flow by the impregnator, thus gathering any filaments which become airborne during processing (refer to Figures 22 and 23).

4560 Denier

2280 Denier

Figure 40. Comparison of 4560 and 2280 Denier Kevlar Roving Microstructure (SEM 100x)

The parameters evaluated were impregnation speed and fiber temperature. The evaluation was conducted using Epon 828 resin and APCO 2330 hardener applied at 200°F. The starting resin content for this series of tests was 38 percent or approximately 52 percent fiber by volume.

The data from these tests are presented in Tables 21 and 22.

TABLE 21. EFFECT OF WINDING SPEED ON IMPREGNATION OF GRAPHITE

Sample Code	Winding Speed (fpm)	Mean Short Beam Shear (psi)	Coefficient of Variation (%)	Resin Content (%)
HA2	50	8281	12.8	38.76
HA3	50	8053	9.9	38.1
HA4	50	7696	8.5	41.6
HB2	100	6657	7.2	36.5
HB3	100	6790	11.8	37.0
HB4	100	7222	9.5	38.2
HC2	200	6068	10.9	36.6
HC3	200	6724	5.4	34.5
HC4	200	6302	5.1	37.5

(Starting Resin Content = 38%)

TABLE 22. EFFECT OF FIBER TEMPERATURE ON IMPREGNATION OF GRAPHITE

Sample Code	Fiber Temp. (°F)	Mean Short Beam Shear (psi)	Coefficient of Variation (%)	Resin Content (%)
JA2	110	7077	8.8	35.81
JA3	110	6727	16.5	33.79
JA4	110	6627	12.3	34.10
JB2	150	7912	11.0	38.09
JB4	150	7777	8.9	36.94
JC3	200	8221	3.6	37.03
JC4	200	8620	5.3	37.25

(Starting Resin Content = 38%)

As can be determined from the data in Table 21, the short-beam shear values decreased with increasing winding speed. The decrease in shear values is attributed to a corresponding decrease in the quality of fiber wetting.

Comparable to the results experienced with S-2 glass, the short-beam shear results improved with fiber temperature (refer to Table 22). This increase is attributed to improved wetting.

The short-beam shear results obtained from both the speed and temperature evaluations were lower than expected. Shear strengths of 11,000 to 12,000 psi were anticipated. These generally low values are attributed to two factors - porosity or voids in the structure and some fiber damage. Figure 41 presents photomicrographs that illustrate the typical porosity condition found in the graphite short-beam shear specimens. This condition was found in all specimens including samples adjacent to specimens exhibiting higher shear values.

A considerable amount of fiber fraying occurred prior to impregnation. The graphite fibers were found to be readily susceptible to breakage. These characteristics are typical of unimpregnated graphite fiber.

Problems were encountered when using the orifice-type manifold. The loose or frayed fibers tended to accumulate at the orifice entrance causing a constriction that in turn produced more fraying. Frequent clearing of the manifold was required. The damage to the fibers from the manifold is considered to be one of the major contributors to the low-shear values.

The relative difference in the short-beam shear values experienced with graphite are in accordance with industry experience, wherein slower winding speed and higher fiber temperature are both conducive to better impregnation.

4.3 PHASE III - PROCESS VERIFICATION

This phase consisted of fabricating and testing short-beam shear, tubular compression, and unidirectional tension specimens using the optimum processes established in Phase II. Ten specimens of each specimen type were fabricated and tested according to the test matrix presented in Table 11.

Following completion of Phase II tasks, the impregnating equipment was combined with the laboratory winding equipment for fabrication of the tubular compression and tensile specimens. The winding equipment has an adjustable roving distribution capability that was not available on the impregnation equipment. The specimens were fabricated by impregnating the

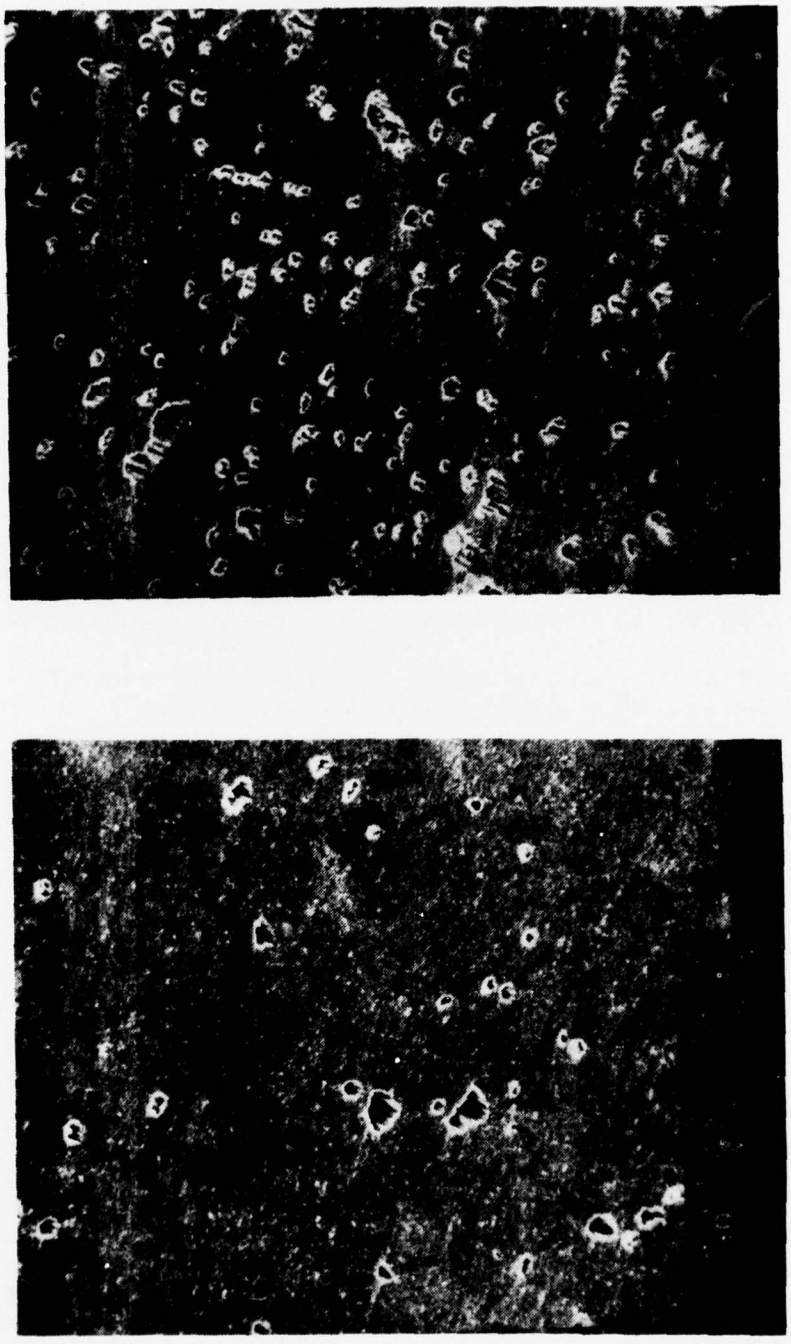


Figure 41. Typical Microstructure of Graphite Specimens (SEM 100x)

fiber on the roller impregnator and transferring the fibers directly to the winding equipment (refer to Figure 42).

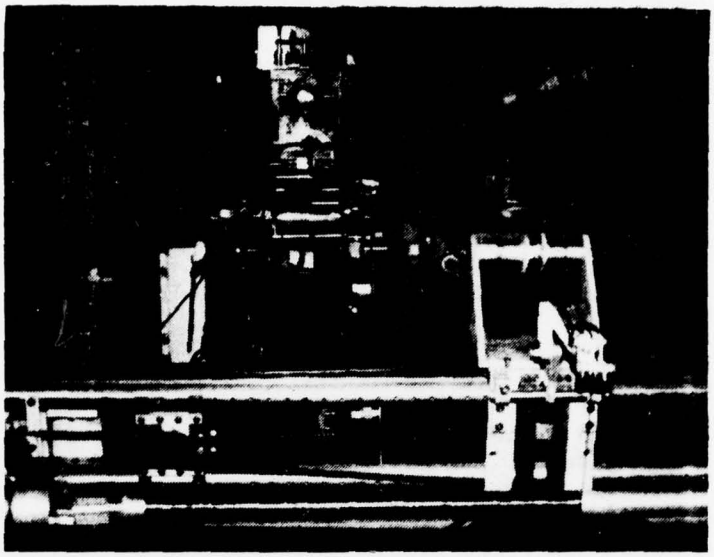


Figure 42. Impregnator Supplying Winding Equipment

The short-beam shear samples produced and tested were per ASTM D2344 as were previous specimens.

The tubular compression samples were fabricated based on designs by BHT material engineers because no applicable test standards could be located. The compression specimens had a 2-inch I.D. and .15-inch thick wall. The specimen length was machined to equal the specimen O.D. in order to provide an aspect ratio of 1:1. These specimens were cut from a tube produced on an aluminum mandrel with the fibers wound 90 degrees to the tube axis. The specimens were cured with a caul sheet over the windings and vacuum bagged for pressure. The cure cycle for all samples was 2 hours at 150°F plus 2 hours at 250°F with vacuum applied at the peak of the DDA dissipation curve. Following cure, the specimens were cut to approximate length, the O.D. machined, and the length trimmed to final length.

The tensile specimens were fabricated by wet filament winding the fibers over a flat aluminum plate to a thickness of .030-.040 inch. Caul sheets were applied to the sides of the plate and the fibers were cured under vacuum producing two cured flat sheets of unidirectional fibers. The cure cycle was two hours at 150°F plus two hours at 250°F with vacuum applied at the peak of the DDA dissipation curve. Strips .5 inch wide by 9.5 inches long were cut from the flat fiber sheets. Pull tabs 2.25 inches long were bonded on each end of the strips creating a tensile specimen with a 5-inch gage length.

Figure 43 depicts the test specimens as fabricated from graphite fiber.

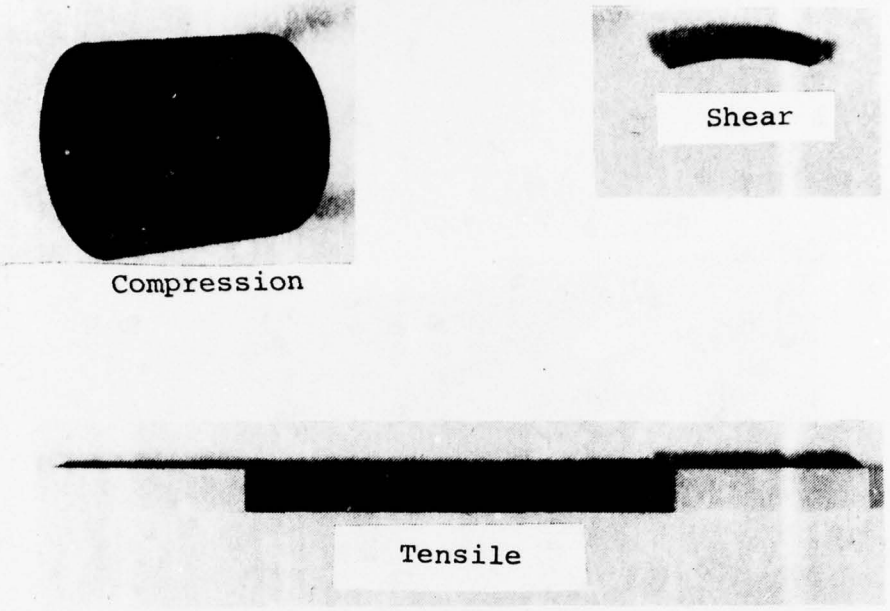


Figure 43. Test Specimens

The short-beam shear and tensile testing was performed on a Tinius Olsen UEH test machine with a load capacity of 30,000 pounds. Figure 44 represents the tensile test arrangement.

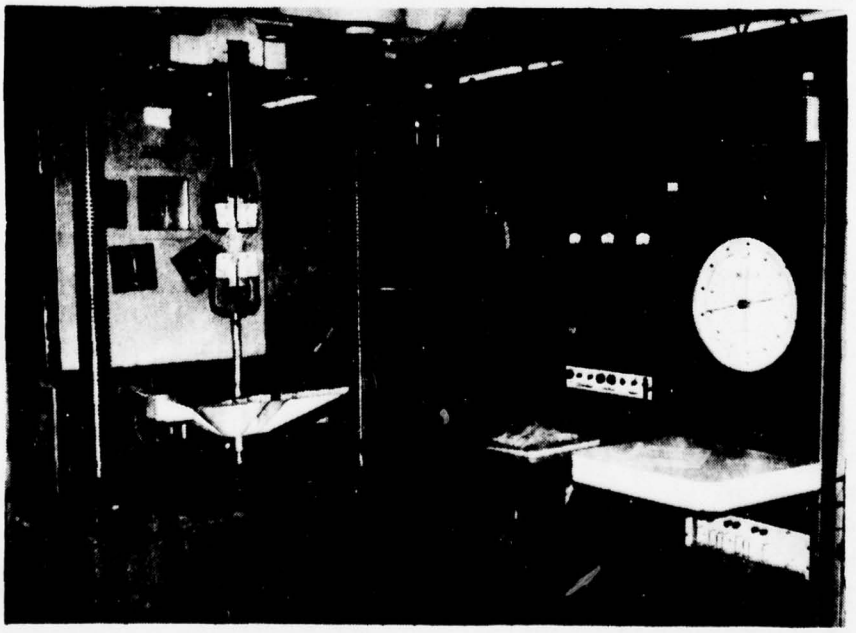


Figure 44. Tensile Test Arrangement

The compression tests were conducted on a SATEC Universal Testing Machine, Model 500 HVL, with a capacity of 500,000 pounds. The test arrangement is shown in Figure 45.

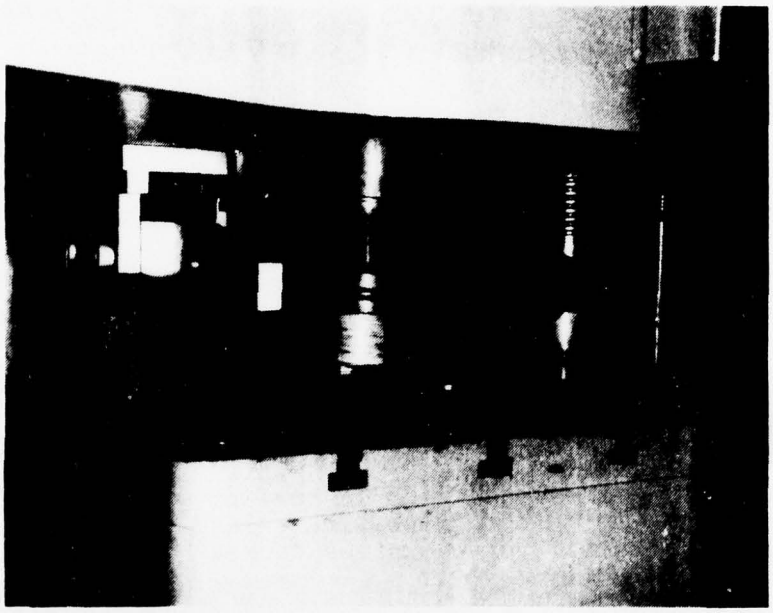


Figure 45. Compression Test Arrangement

Summaries of these test results are presented in Tables 23, 24, and 25 which represent the results of S-2 glass, Kevlar 49, and graphite, respectively. The values presented are the mean of 8-10 test specimens. The complete results from these tests are presented in the appendix.

TABLE 23. S-2 GLASS TEST DATA SUMMARY

Sample Code	Mean Value (psi)	Coefficient of Variation (%)	Expected Values (psi)
	Tubular Compression		
KA	15,889	18.5	30,000
	Unidirectional Tension		
KB	253,666	8.1	185,000
	Short Beam Shear		
KC1	6733	8.2	9,000
KC2	6342	11.8	
KC3	6135	8.7	

TABLE 24. KEVLAR TEST DATA SUMMARY

Sample Code	Mean Value (psi)	Coefficient of Variation (%)	Expected Values (psi)
	Tubular Compression		
LA	11,229	8.0	20,000
	Unidirectional Tension		
LB	157,468	16.3	160,000
	Short-Beam Shear		
LC1	3143	3.9	5,000
LC2	3228	3.0	
LC3	3177	5.2	

TABLE 25. GRAPHITE TEST DATA SUMMARY

Sample Code	Mean Value (psi)	Coefficient of Variation (%)	Expected Values (psi)
	Tubular Compression		
MA	15,086	13.3	30,000
	Unidirectional Tension		
MB	189,975	13.5	210,000
	Short-Beam Shear		
MC2	6944	4.3	12,000
MC3	6986	13.4	
MC4	6685	9.8	

The tubular compression and short-beam shear values are lower than expected. Since the fiber direction in the tubular compression samples is 90 degrees to the test axis, matrix-dominant properties are measured comparable to short-beam shear testing. These data are generally commensurate with data gathered in Phase II indicating that optimum matrix properties have not been obtained.

The unidirectional tension results obtained were generally satisfactory. Very good results were obtained from S-2 glass. The Kevlar results are slightly lower than expected; however, the results obtained are higher than results obtained from some preimpregnated Kevlar rovings evaluated at BHT. The graphite tensile results were also lower than expected. Values of 200,000 to 220,000 psi were anticipated. These lower results are attributed to porosity and to fiber damage incurred from the orifice-type manifold used.

In general, the results from this program phase confirm the results in Phase II indicating that further development is required to obtain better matrix properties.

5. CONCLUSIONS

Based on the development and evaluations conducted on roller impregnation equipment during this program, the following conclusions have been reached.

- Resin and hardener can be applied to fibers within ±2 percent by weight for a given fiber yield. This accuracy is attainable using metering pumps and pressure balance control.

- The use of beta ray measurement for determining, in process, roving mass is not feasible with current technology due to geometric characteristics of roving.

- The premixing of resin and hardener prior to fiber application is required to obtain thorough mixing.

- Additional equipment development is required to obtain thorough fiber wetting.

The development conducted toward meeting primary program objectives has resulted in the secondary benefit of equipment improvements. These improvements, which consist of electronic ratio control of metering pumps, pump pressure balancing (required for ±2 percent resin delivery control), and static premixing of resin/hardener have been incorporated into new equipment currently available to industry.

6. RECOMMENDATIONS

There is a need for further equipment development and evaluation. Specifically, the following areas should be addressed.

- A delivery manifold capable of applying resin/hardener uniformly to both sides of roving.
- A roving spreading device to spread out roving for improved wetting.
- Evaluation of alternate delivery pumps with reduced sensitivity inlet/outlet pressure balance.
- Conduct a study of fiber measurement methods capable of determining the weight or volume of dry and impregnated roving.

In conjunction with the above, a parallel program of resin characterization is recommended. This program should address the viscosity, mechanical properties, pot life, and hygroscopic characteristics of resins considered as candidates for direct impregnation.

TABLE A-1. S-2 GLASS COMPRESSION AND TENSILE DATA

Tubular Compression Strength		Unidirectional Tensile Strength	
Sample	Strength (psi)	Sample	Strength (psi)
KA-1	20,373	KB-1	270,408
KA-2	10,312	KB-2	265,700
KA-3	12,092	KB-3	263,888
KA-4	16,612	KB-4	263,987
KA-5	16,154	KB-5	255,868
KA-6	18,075	KB-7	292,201
KA-7	14,480	KB-8	230,593
KA-8	16,377	KB-9	219,502
KA-9	17,966	KB-10	243,362
KA-10	16,453	KB-13	248,113
		KB-14	236,714
Expected	30,000 psi	Expected	185,000 psi
Mean	15,889 psi	Mean	253,666 psi
Standard Deviation	2,941 psi	Standard Deviation	20,615 psi
Coefficient of Variation	18.5 %	Coefficient of Variation	8.1 %

Resin Content
(% by weight)

Sample	Percent
KB-1	25.28
KB-2	25.45
KB-3	24.93
KB-4	24.43
Mean	25.02%

(Starting Resin Content = 30%)

TABLE A-2. S-2 GLASS SHORT-BEAM SHEAR DATA

Sample	Shear Strength (psi)	Sample	Shear Strength (psi)	Sample	Shear Strength (psi)
KC1-1	5725	KC2-1	7041	KC3-1	5992
KC1-2	6591	KC2-2	7122	KC3-2	5952
KC1-3	6221	KC2-3	6329	KC3-3	5327
KC1-4	7140	KC2-4	6117	KC3-4	6511
KC1-5	6653	KC2-5	4809	KC3-5	6711
KC1-6	7152	KC2-6	6028	KC3-6	6529
KC1-7	7364	KC2-7	6921	KC3-7	5472
KC1-8	7038	KC2-8	6371	KC3-8	6590
Expected	9000 psi				
Mean	6733 psi		6342 psi		6135 psi
Standard Deviation	552 psi		750 psi		532 psi
Coefficient of Variation	8.2 %		11.8 %		8.7 %

TABLE A-3. KEVLAR COMPRESSION AND TENSILE DATA

Tubular Compression Strength		Unidirectional Tensile Strength	
Sample	Strength (psi)	Sample	Strength (psi)
LA-1	10,985	LB-1	110,185
LA-2	12,231	LB-2	128,877
LA-3	11,141	LB-3	135,675
LA-4	11,806	LB-4	159,872
LA-5	11,184	LB-5	167,832
LA-6	9,302	LB-8	176,056
LA-7	10,269	LB-9	163,703
LA-8	11,729	LB-11	164,444
LA-9	11,434	LB-13	198,473
LA-10	12,208	LB-14	169,565
Expected	20,000 psi	Expected	160,000 psi
Mean	11,229 psi	Mean	157,468 psi
Standard Deviation	900 psi	Standard Deviation	25,608 psi
Coefficient of Variation	8 %	Coefficient of Variation	16.3 %

Resin Content
(% by weight)

Sample	Percent
LB-1	41.45
LB-2	36.07
Mean	38.76%

(Starting Resin Content = 42.6%)

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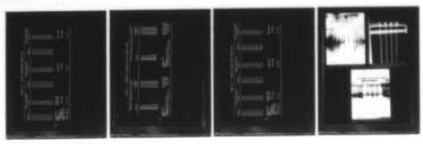

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TABLE A-4. KEVLAR SHORT-BEAM SHEAR DATA

Sample	Shear Strength (psi)	Sample	Shear Strength (psi)	Sample	Shear Strength (psi)
LC1-1	3188	LC2-1	3375	LC3-1	3267
LC1-2	3050	LC2-2	3167	LC3-2	3085
LC1-3	3143	LC2-3	3112	LC3-3	3156
LC1-4	3067	LC2-4	3302	LC3-4	3363
LC1-5	3087	LC2-5	3248	LC3-5	2950
LC1-6	2988	LC2-6	3149	LC3-6	3231
LC1-7	3334	LC2-7	3148	LC3-7	3390
LC1-8	3293	LC2-8	3323	LC3-8	2971
Expected	5000 psi				
Mean	3143 psi		3228 psi		3177 psi
Standard Deviation	121 psi		97 psi		166 psi
Coefficient of Variation	3.9 %		3.0 %		5.2 %

TABLE A-5. GRAPHITE COMPRESSION AND TENSILE DATA

Tubular Compression Strength		Unidirectional Tensile Strength	
Sample	Strength (psi)	Sample	Strength (psi)
MA-2	18,413	MB-1	135,185
MA-3	17,297	MB-4	169,518
MA-4	16,466	MB-5	181,162
MA-5	16,971	MB-7	213,245
MA-7	15,923	MB-8	198,701
MA-8	15,151	MB-9	220,129
MA-9	19,263	MB-14	192,982
MA-10	15,940	MB-15	194,382
MA-11	15,010	MB-16	204,469
MA-12	15,514		
Expected	30,000 psi	Expected	210,000 psi
Mean	15,086 psi	Mean	189,975 psi
Standard Deviation	2,012 psi	Standard Deviation	25,650 psi
Coefficient of Variation	13.3 %	Coefficient of Variation	13.5 %

TABLE A-6. GRAPHITE SHORT-BEAM SHEAR DATA

Sample	Shear Strength (psi)	Sample	Shear Strength (psi)	Sample	Shear Strength (psi)
MC2-1	6871	MC3-1	7823	MC4-1	6439
MC2-2	7105	MC3-2	6151	MC4-2	7546
MC2-3	6780	MC3-3	5743	MC4-3	6438
MC2-4	7255	MC3-4	7495	MC4-4	5904
MC2-5	7208	MC3-5	7176	MC4-5	6962
MC2-6	6530	MC3-6	7463	MC4-6	7292
MC2-7	7242	MC3-7	8173	MC4-7	5761
MC2-8	6558	MC3-8	5864	MC4-8	7141
Expected	12000 psi				
Mean	6944 psi		6986 psi		6685 psi
Standard Deviation	301 psi		936 psi		652 psi
Coefficient of Variation	4.3 %		13.4 %		9.8 %

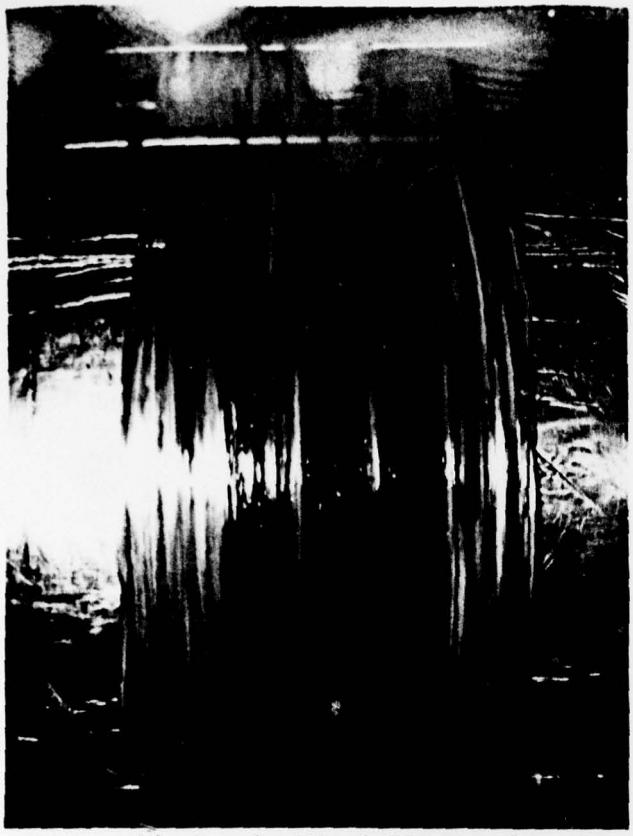

Figure 16. Poorly Mixed Pigmented Resin

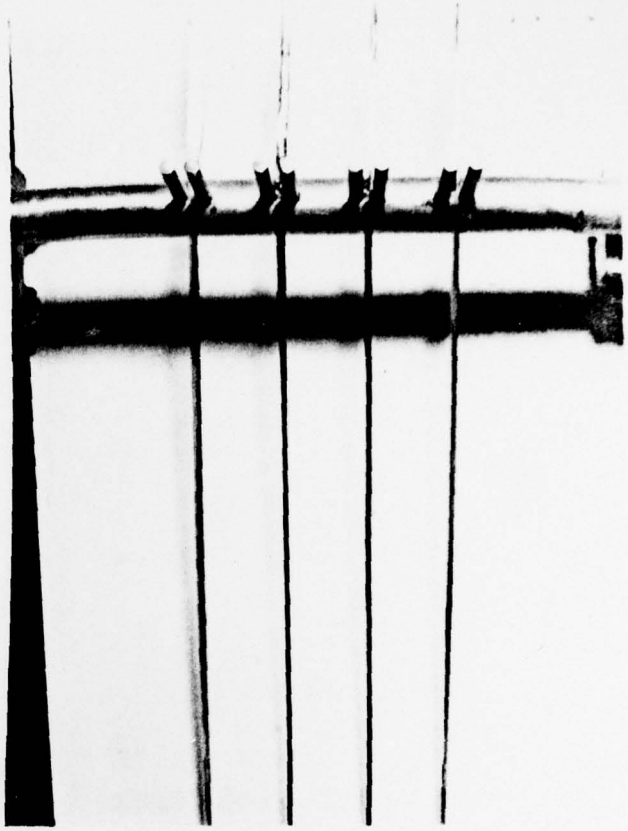

Figure 17. Poorly Mixed Resin Component Color Distribution Along Roving

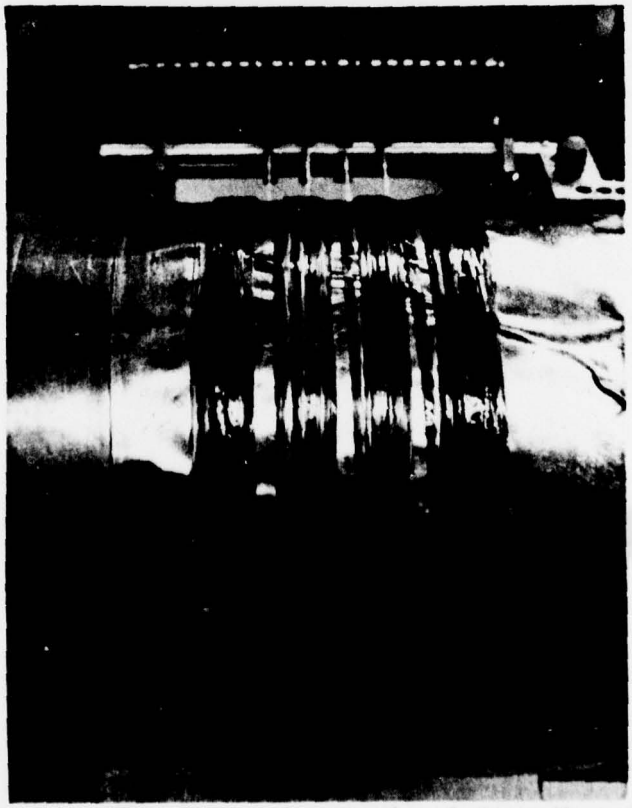

Figure 19. Uniformaly Mixed and Distributed Resin