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DEVELOPMENT OF LIGHTWEIGHT GRAPHITE/POLYIMIDE SANDWICH PANELS

PHASE II - THIN GAGE MATERIAL MANUFACTURE

H400-12-1-11

February 1972

NASA Contract NAS 9-11368

Prepared by

HERCULES INCORPORATED
SYSTEMS GROUP
Bacchus Works
Magna, Utah

Prepared for

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Houston, Texas

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FOREWORD

This report covers the work performed in Phase II of National Aeronautics and Space Administration (NASA) Contract NAS 9-11368 during the period of January 1971 through January 1972. The work was performed under the sponsorship of the NASA Manned Spacecraft Center R&D Procurement Branch. The program is administered under the direction of Mr. T. J. Dunn, Technical Monitor.

The work is being performed by the Composite Materials Development Department, Hercules Incorporated, Bacchus Works, Magna, Utah.

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ABSTRACT

The work reported herein was accomplished during the thin gage material manufacture (Phase II) on NASA Contract NAS 9-11368. This report is submitted in compliance with Article I, requesting reports at the end of each phase of the contract.

An outline of the total program is presented under paragraph A, Introduction. Basic requirements of the materials selected and the rationale for these requirements are discussed in paragraph B.1.

The resin used in all prepreg manufacture is Monsanto RS-6234 polyimide. The selected fiber for core manufacture is Hercules HT-S, and the selected fiber for face sheets is Hercules HM-S. The technique for making thin gage prepreg was to wind, spread carbon fiber tows into a resin film on a large drum. This technique was found to be superior to others investigated. A total of 22 pounds of 1 to 2 mil/ply prepreg was fabricated for use on the program.

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DEVELOPMENT OF LIGHTWEIGHT GRAPHITE/POLYIMIDE SANDWICH PANELS

A. INTRODUCTION

The objective of this program is to develop the tools and techniques necessary to produce lightweight honeycomb sandwich panels, to fabricate and test these panels, and to develop design data. Carbon fibers are to be used with a resin suitable for a 200-hour, 600° F, exposure. The target core weight is 2.0 lb/cu ft, requiring 0.001 to 0.002 in. per ply thicknesses. This report covers the fabrication of thin prepreg used in core and face sheet fabrication. Honeycomb core in various weights and cell sizes and candidate face sheet orientations are being made and tested using this thin prepreg. Sandwich panels are now being made from the lightest workable gages and a minimum feasible weight configuration is being established. Structural tests will soon be performed to establish design parameters, allowable strengths, and resultant deflections. Test results for the graphite panels will be compared with results to be expected from glass fiber panels of comparable weight and configuration.

The program is being conducted in five phases as follows:

Phase I - Materials Selection

Phase II - Thin Gage Material Manufacture (reported herein)

Phase III - Fabrication Development

Phase IV - Design and Manufacture of Panels

Phase V - Panel Testing and Design Allowables Determination

The program is being conducted in an 18-month time span with reports written at the end of each phase. The program milestone schedule is presented in Table I.

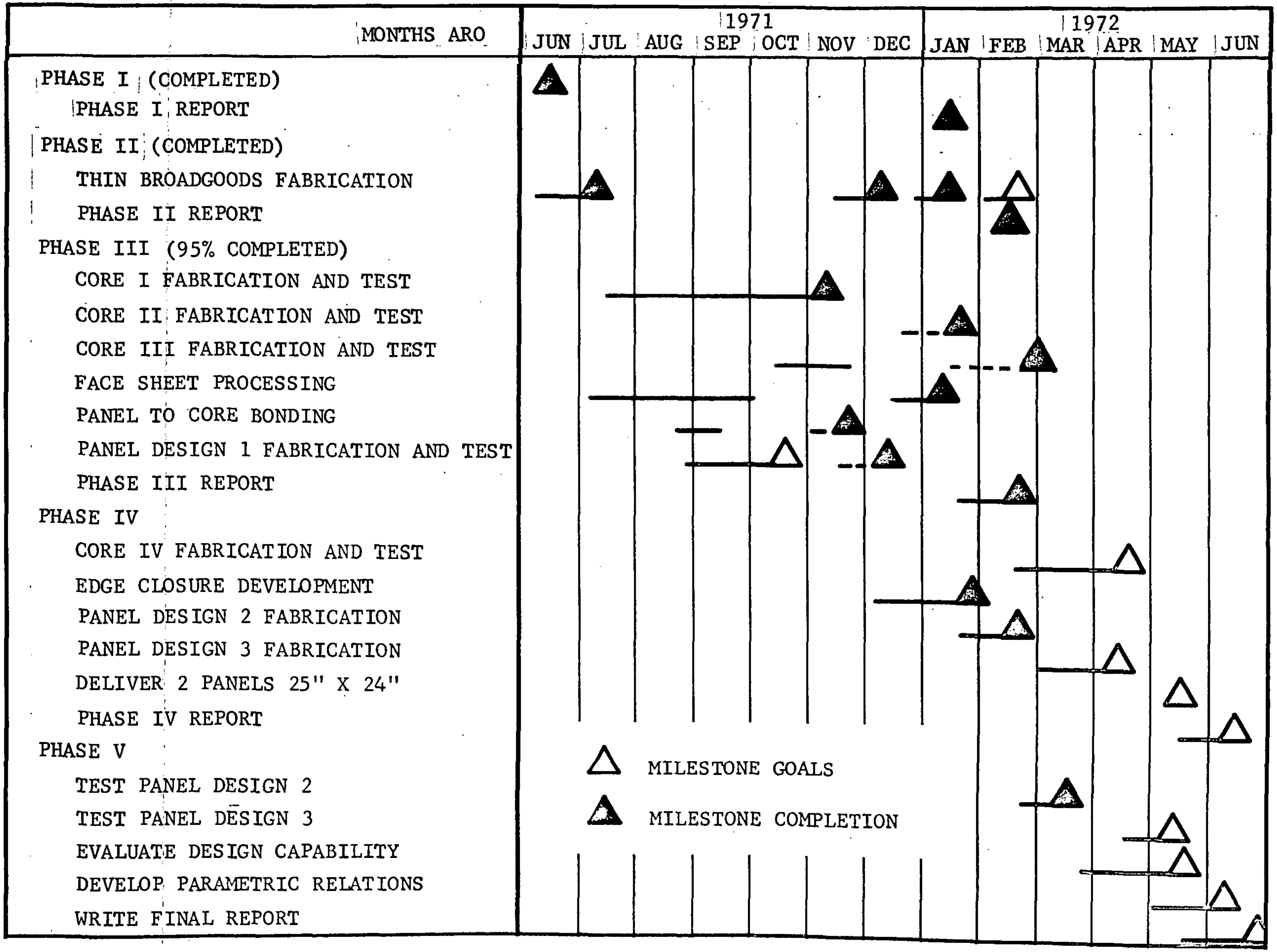
B. PHASE II - THIN BROADGOODS PRODUCTION

1. Material Requirements

The materials were selected during Phase I; this selection was controlled by the intended end use of the lightweight honeycomb sandwich panels. The structures were designed to be support panels for a thermal protection system. The general configuration is shown in Figure 1. The panel is 24-in. wide and is to be simply supported at a distance of 25 in. The load is distributed uniformly over the panel at an ultimate value of 6 psi. The insulation system which the sandwich panel supports has an elongation capability of 0.4 percent (4000 μin./in.). The maximum design temperature is 500° F with a goal of 600° F and an exposure duration of 200 hours.

TABLE I

PROGRAM MILESTONE SCHEDULE



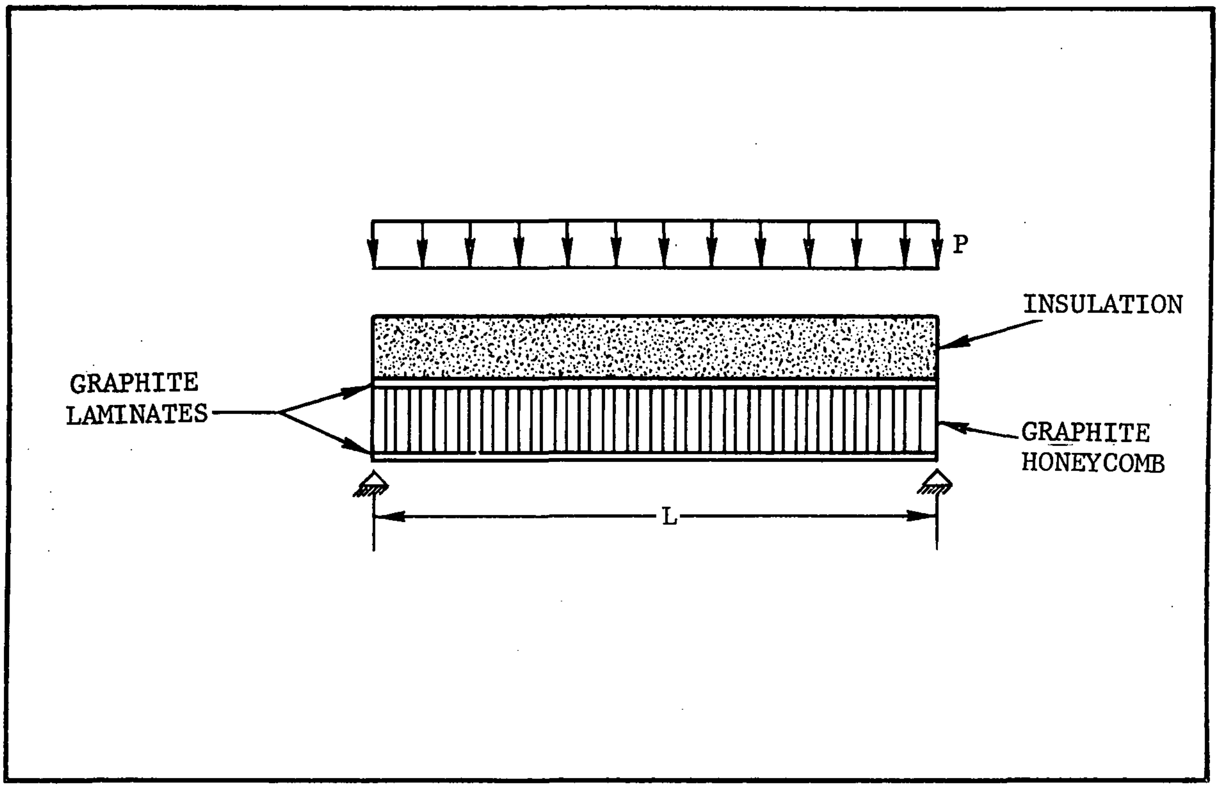


Figure 1. Panel Configuration

a. Core Material Requirements

Based on calculations made during Phase I of the program for a cell size of 0.500 in., the web thickness requirements was determined to be 0.004 in. or less. And, at a cell size of 0.250 in., the web thickness must be less than 0.002 in. Further, the more efficient laminates for combined shear and compressive stress require more than one ply; an example of this is a $\pm 45^{\circ}$ laminate requiring a minimum of two plies. The composite material must, therefore, be capable of achieving less than 0.002 in. per ply.

Hercules HT fiber was selected for the composite core because of its ability to meet these material requirements and its high composite shear strength and modulus. Hercules Type A fiber was substituted for HT fiber in one core, Core III, because of the potential cost savings and better spreading uniformity.

b. Face Sheet Material Requirements

Since the insulation the sandwich panel is to support has a limited elongation capacity (0.4 percent), small panel deflections are required. It was established in the Phase I report that high modulus graphite with an ultimate elongation of 0.4 percent and ply thicknesses of 0.005 in., could be used for the spanwise direction of the face sheet while very thin plies of material, 0.001 to 0.003 in., are required for the transverse direction to permit handling and to carry stresses resulting from the Poisson effect. Because of its high modulus and satisfactory spreading characteristics, Hercules HM fiber was selected as the face sheet fiber with a per ply thickness target of 0.002 in. Monsanto's RS-6234 polyimide resin was selected as the matrix system for both face sheets and core.

2. Fiber Spreading

In order to achieve the required thickness per ply, untwisted bundles of fiber or tows must be spread to 0.66 to 0.75 tows per inch.

There are two primary methods of producing such thin prepregs from continuous carbon tow: (1) Prepreg unspread fiber and then spread the fibers by rolling, (2) spread the fiber band first and then place the fiber into the pregregging resin.

In general, thin prepregging processes employing the unspread fiber principle result in a poor quality product as a result of broken and misaligned fiber. The use of spread tow overcomes these deficiencies and improves reproducibility. Prior to this program, thin (1 and 2 mil) graphite prepregs were developed by Hercules on company funds employing the spread tow principal.

After Hercules submitted the proposal to NASA and before contract award, experimentation, utilizing a spreading technique, disclosed to Hercules under a third party secrecy agreement produced a more satisfactory product and eliminated electrical problems caused by graphite dust released during the spreading process. The principal of creating a tumbling motion of the filaments to produce spreading was the same as the previously developed acoustic spreader.

a. Hercules Acoustic Spreader

The acoustic system is basically a vibratory spreader consisting of a 15-in. diameter speaker driven by an amplifier and audio oscillator. A one mil mylar film is secured across the face of the speaker to provide a vibrating membrane. When the speaker is operating, the two will not actually contact the membrane, but is suspended above it by the pressure pulses caused by its vibration. The oscillator setting used varies with the tightness of the membrane and ranges from 125 cps for a tight membrane to 60 cps as the mylar relaxes.

The input tow is suspended from a bullseye across the mylar as illustrated in Figure 2. Input tow tension is critical and is determined by the height of the speaker face above the tow level in the feed bucket. (See Figure 2.) The tension must be very low to achieve spreading, but should be sufficient to allow the tow to clear the leading edge of the speaker. Any tensioning of the fiber causes a rapid reduction in band width. To avoid this, the fiber was laid loosely in a plastic bucket and pulled through the system by a powered take-up reel.

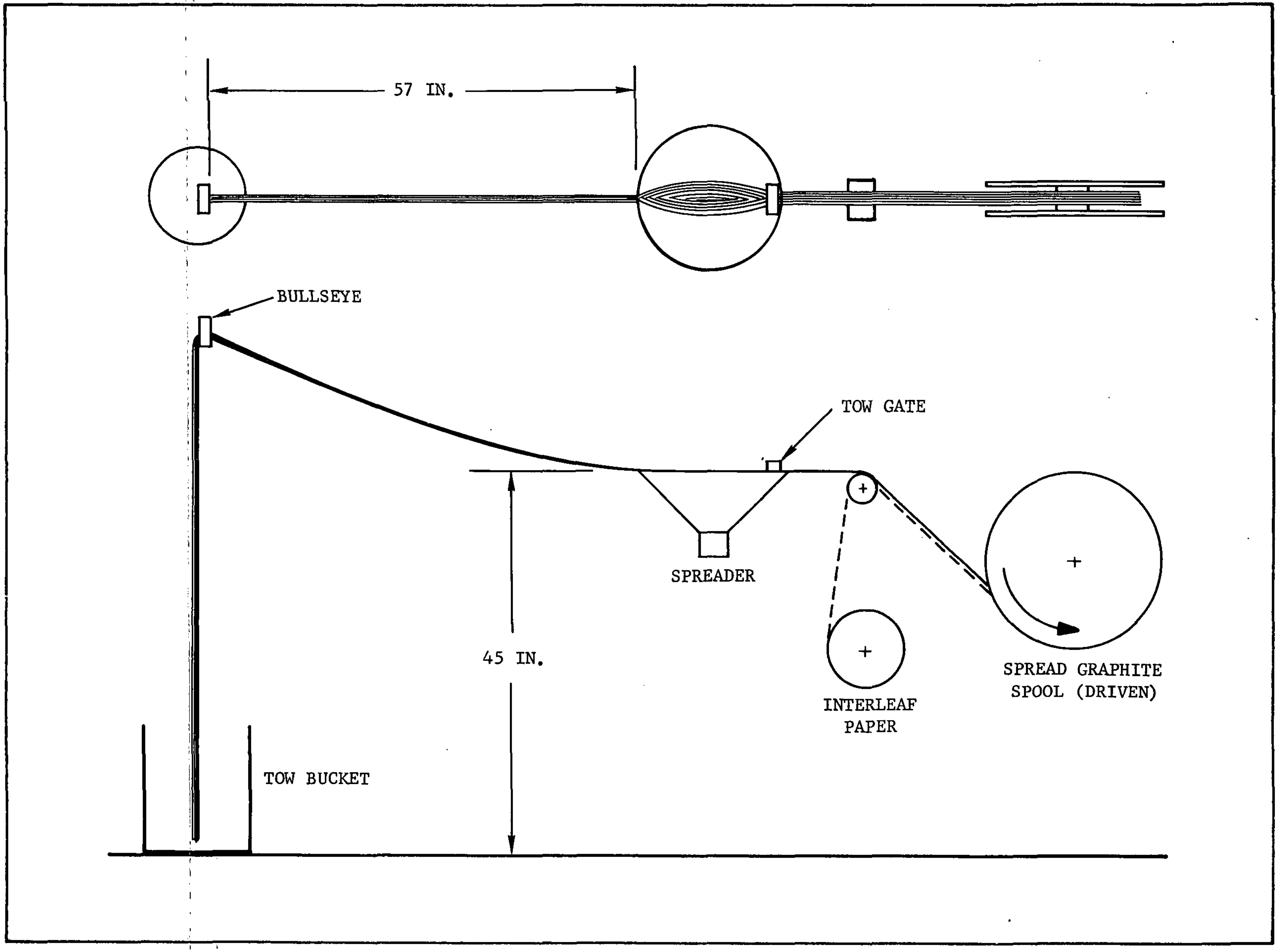


Figure 2. The Acoustic Spreading Setup

A lead-in length (Figure 2) is required to provide a certain amount of tow twist relief. Normally, the two will spread to 1.5 to 3.0 in. on the face of the speaker. The amount of spread can be varied by adjustments to the speaker input by varying fiber tension, and by adjusting the velocity of respooling.

The output tow width is controlled by a gate fastened to the output edge of the speaker. The spread width on the speaker face should be slightly greater than or equal to the gate width. If the gate is too narrow, the gate forces the edges of the spread web to fold in toward the center resulting in a nonuniform filament distribution, since the output web is thick on the edges and thin in the center.

Several passes may be required to achieve the desired output width. The spreading power is inadequate to hold back twists or to open up certain tight fiber areas; the operator must, therefore, be alert to cut out tight fiber areas and twists.

The spread fiber is wound onto a standard plastic spool. Each spool holds up to 400 linear ft of 3-in. spread fiber. Thin interleaf paper prevents entanglement of the spooled fiber. (See Figure 3.) When a defect does occur, the release paper is marked for a minimum of 6 ft prior to the problem. This permits controlled stopping of the machine and placement of the severed fiber on the broadgoods cut line before the splice contacts the resin on the good area. The end of the spool is also marked accordingly. With fiber delivery speeds in excess of 100 linear ft/min, careful preparation of the supply spool is essential.

3. Resin Application

The prepreg is fabricated by a drum winding process. The apparatus is constructed or precision devices capable of depositing resin and fiber onto a release paper covered, rotating mandrel. The mandrel has a 10 ft circumference and is 22 ft long. Figure 4 illustrates the key pieces of equipment. The basic process is summarized as follows:

(a) Spread fiber to 3 in. per tow

(b) Apply release paper to drum

(c) Spray one coat of resin on release paper

(d) Wind fiber into resin

(e) Spray second coat of resin onto fiber

(f) Cut off of drum at 45° angle

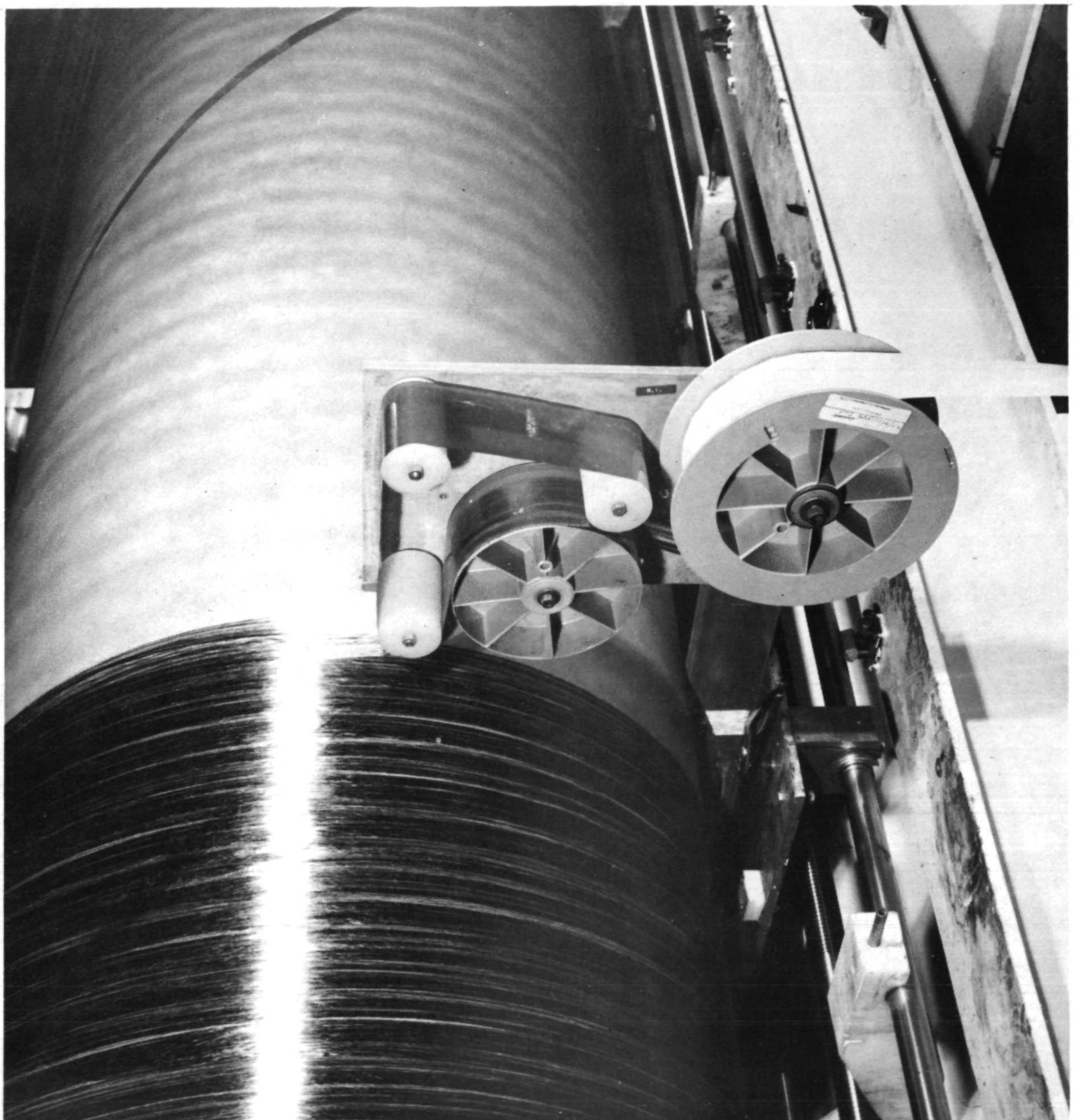


Figure 3. Spread Fiber Delivery System

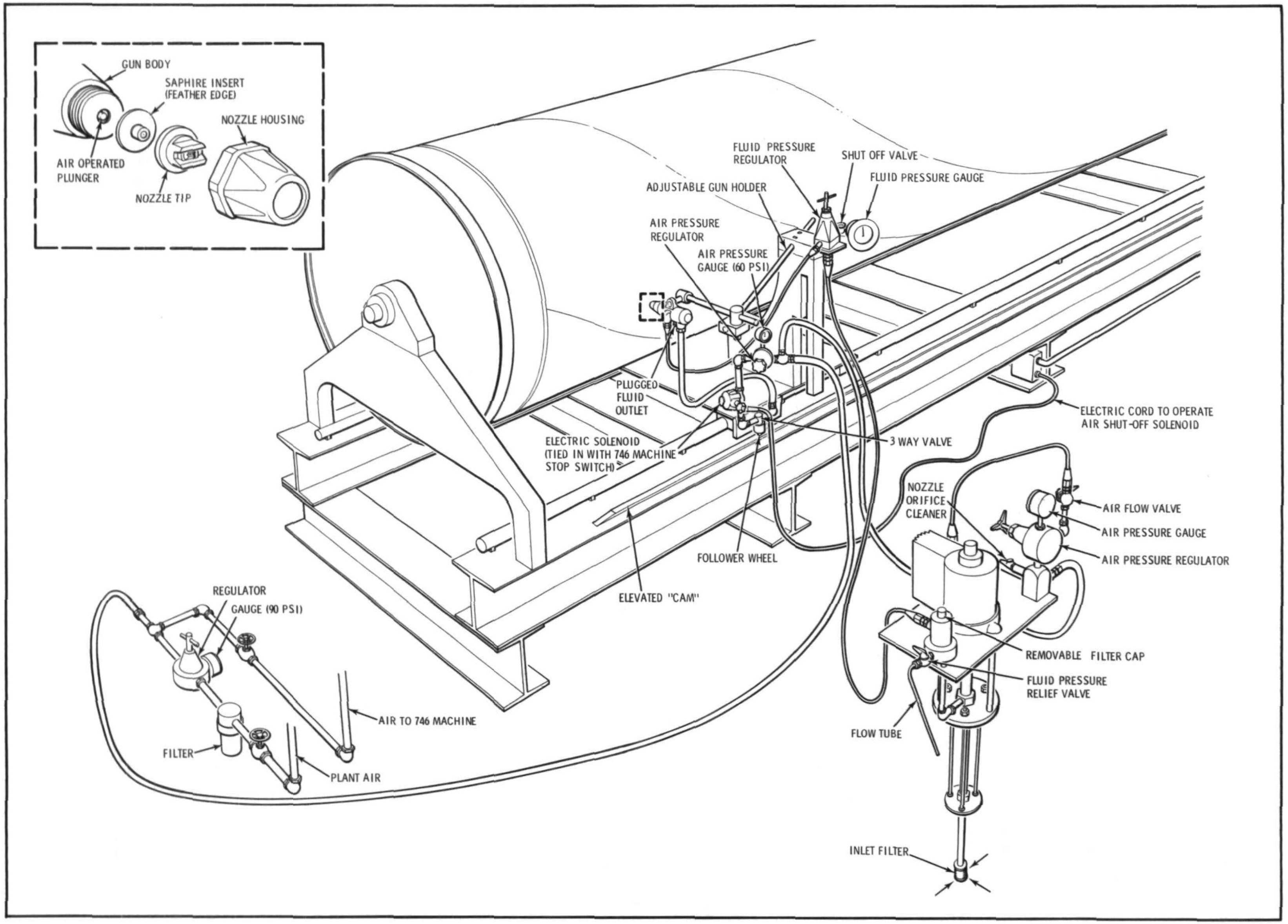


Figure 4. Airless Resin Spray Equipment

An overview of the process is presented in the following paragraphs, and reference to the operational procedures listed below provide more detailed information:

Appendix A - The Thin Prepreg Manufacturing Process

Appendix B - Operation of the Airless Filming Apparatus

Appendix C - M&IR for Thin Broadgoods

The drum winding process was selected primarily as a result of the ease of fabrication of crossplied sheets and the fact that a very low tension can be applied to the spread fiber in the prepregging process. As mentioned in paragraph 2., tensioning of the spread tow causes a rapid reduction in the spread width and is, therefore, a condition to be avoided.

Figure 5 shows the application of silicone-treated kraft release paper to the winding machine drum. Note that the paper is spirally wrapped; this permits the removal of 45^{o} biased prepreg when it is cut from the mandrel. The two sheets are crossplied to yield $\pm 45^{o}$ core web prepreg.

The Binks airless spray gun positioned for the first resin coat is shown in Figure 6. Note the elevated cam on the machine frame; this device automatically starts and stops the resin spray as the carriage traverses the machine.

In Figure 7, the drum is partially covered with sprayed resin. The quality of the spray is dependent on the resin solids content and viscosity, nozzle orifice (exit angle and opening diameter), the fluid pressure, the mandrel speed, the carriage advance, and the distance of the nozzle from the release paper. All of these variables are held constant except the mandrel speed which is varied slightly pending desired changes in prepreg resin content.

Upon completion of the first resin coat, the spread fiber which was stored on spools during the spreading operation is wound into the resin. (See Figure 3.) The fiber spool is slipped onto the delivery system, the fiber fed through a belt tensioning device, and pulled onto the rotating mandrel. The carriage is synchronized with mandrel rotation to assure a constant advance. The separator paper falls freely to the floor and is later discarded. Splices in the fiber are placed on the cut-off line such as shown in the upper righthand corner of Figure 3, thus eliminating splices within the entire sheet of prepreg.

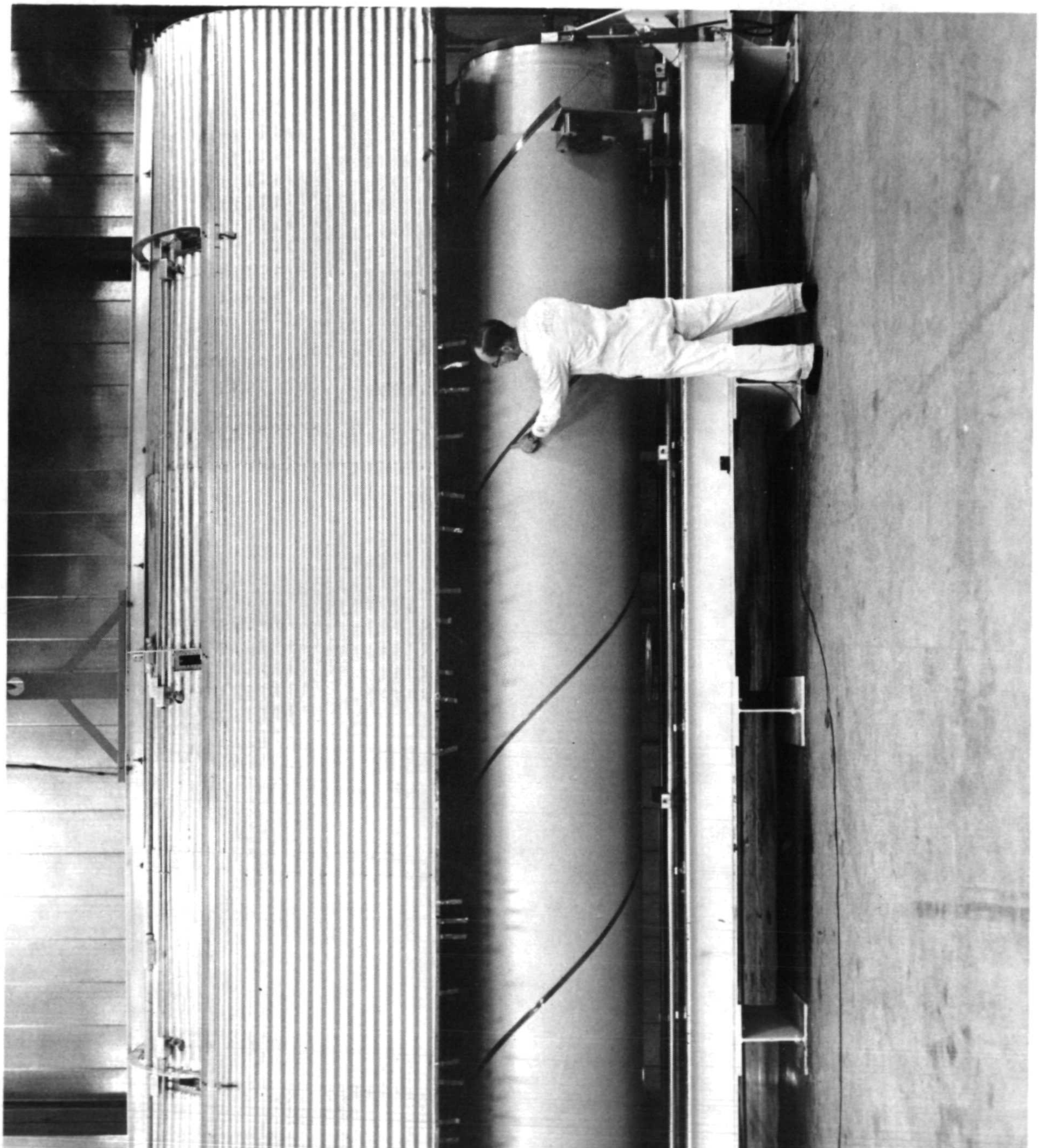


Figure 5. Application of Release Paper

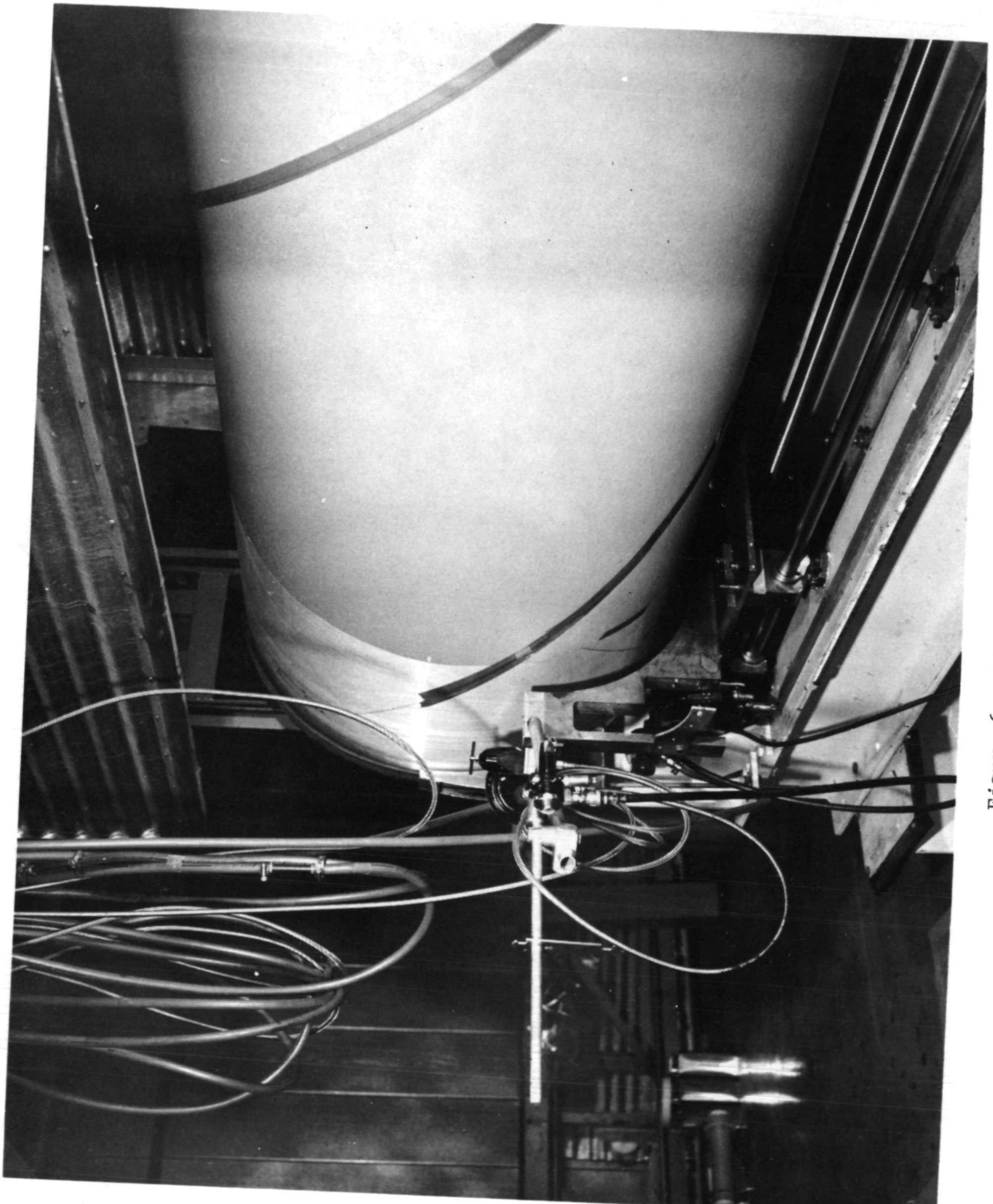


Figure 6. Airless Spray Gun

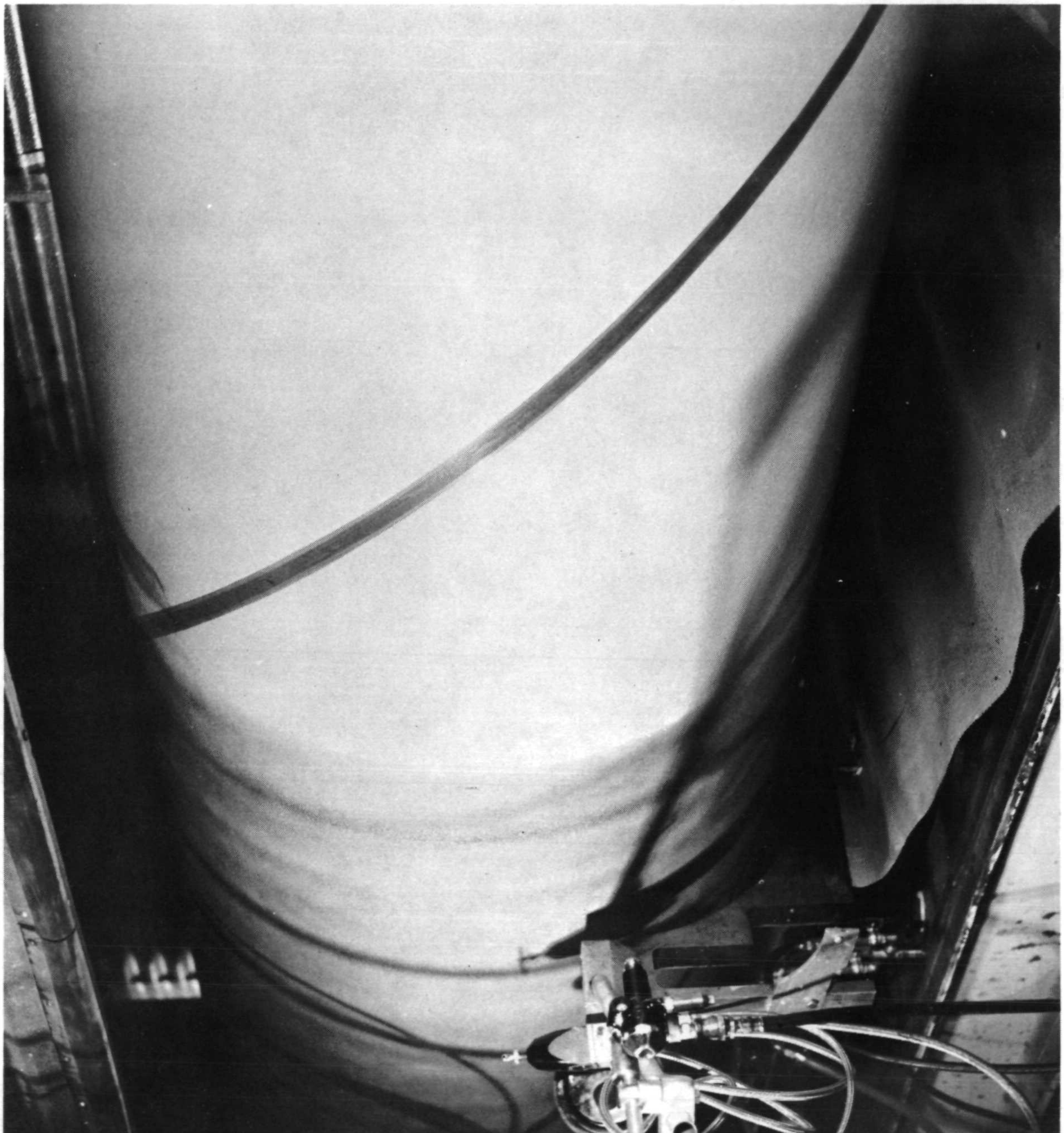


Figure 7. First Resin Coat

Occasionally, fibers are improperly positioned on the drum. These misplaced fibers are repositioned with special knives as shown in Figure 8. The large hood above the machine normally acts as a heat cover for B-staging certain resins; however, it also collects volatiles and overspray from the spraying process. The collected particles are exhausted making the operation safe for most resins.

Once the fiber has been wound on the drum over the first coat of resin a second resin coat is applied directly over the fiber. Figure 9 shows the application of this second coat of resin. The cardboard cover (attached to the carriage below the drum) prevents resin overspray from falling on the precision lead screw and ways. Figure 10 shows the completed winding. The photograph was taken during an early winding and the bands of resin, which may be seen, resulted from loss of solvent between coats. This problem has since been reduced by decreasing the resin viscosity from 380 to 300 cps by the addition of ethanol, a commercial solvent.

The prepreg is not B-staged or heated while on the drum since some movement of the resin during the subsequent pressing operation is desired, and a rather high solvent content is needed.

4. Crossplying and Pressing

The prepreg is supplied to Hexcel in a $\pm 45^{\circ}$ crossplied condition and is pressed to provide good consolidation of the sheets and improve resin distribution.

The prepreg is removed from the drum in two sheets at a 45° bias. A recessed slot, machined into the metal mandrel, provides a guide for quick and accurate cutting. The two large parallelograms are laid flat onto a moving table as the mandrel rotation unwinds the product.

Tape on the edges of the prepreg (which held the release paper to the drum) generally causes wrinkling at the edges after removal. This is removed with sharp shears. If the sheets are to be crossplied, the ends are carefully removed to create two equal rectangles. The ends of one sheet are picked up, flipped over, and then carefully positioned over the other sheet laying face up. This creates a crossplied ($\pm 45^{\circ}$) product. Single sheets used for face sheet construction are covered with release paper. In either case, the prepreg is then pressed.

Pressing is an important step in the thin prepregging process. The spraying process results in the formation of many tiny droplets of resin solution on and within the dry fiber. Pressing both debulks the fiber and forces resin into the fiber, forming an even resin film on the surface next to the smooth release sheet. The pressing equipment is shown in Figure 11.

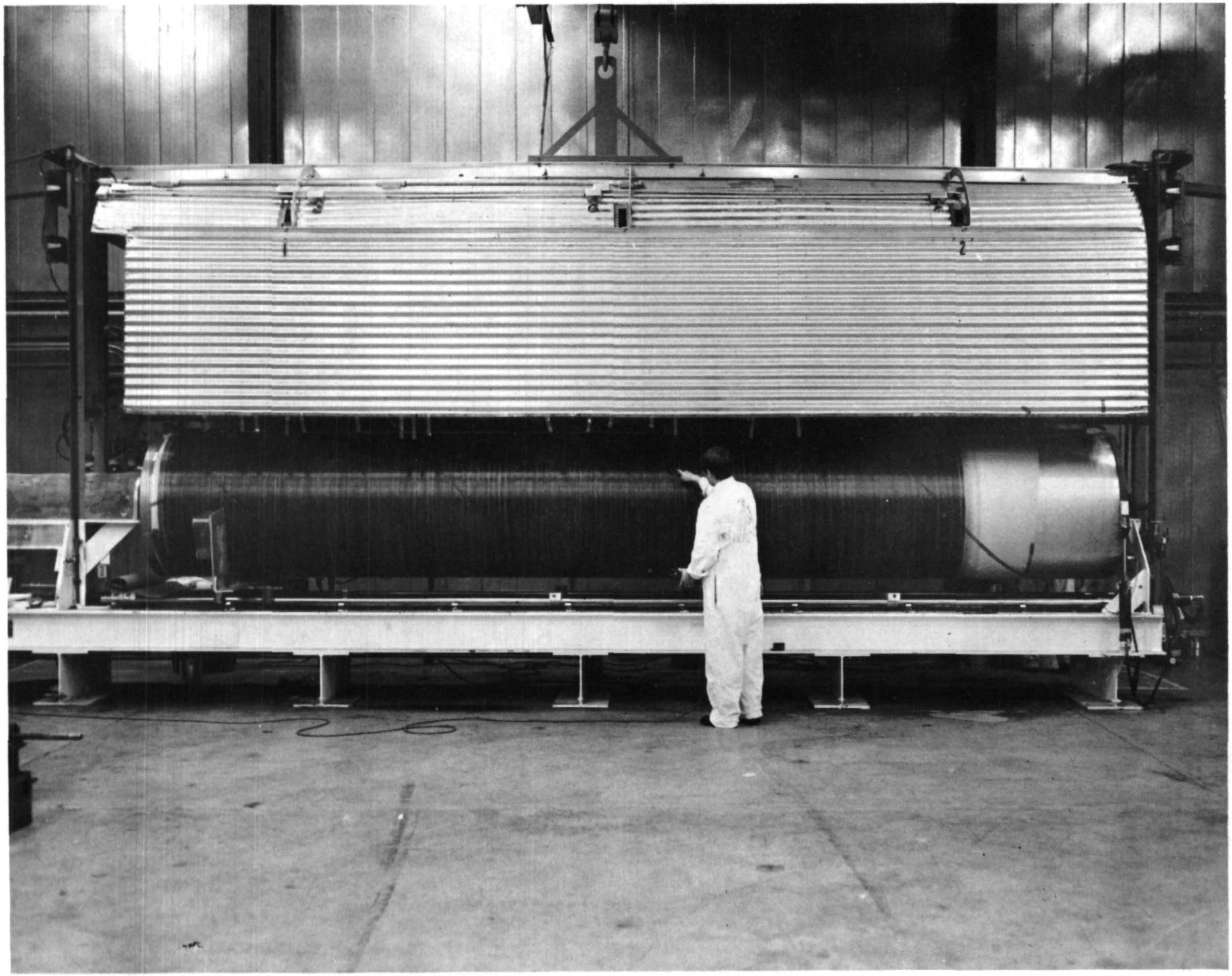


Figure 8. Relocate Poorly Spread Fibers

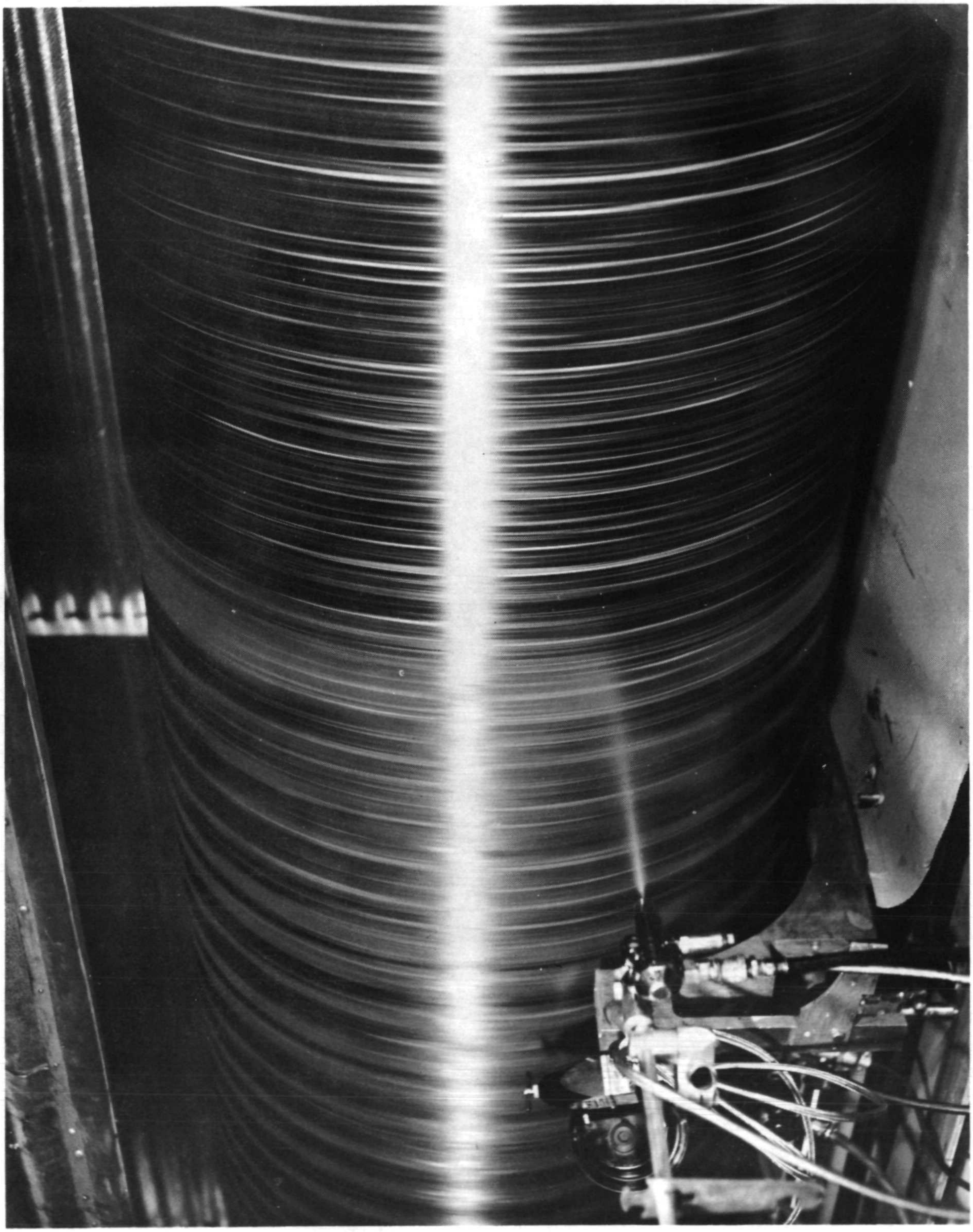


Figure 9. Apply Second Resin Coat onto Fibers

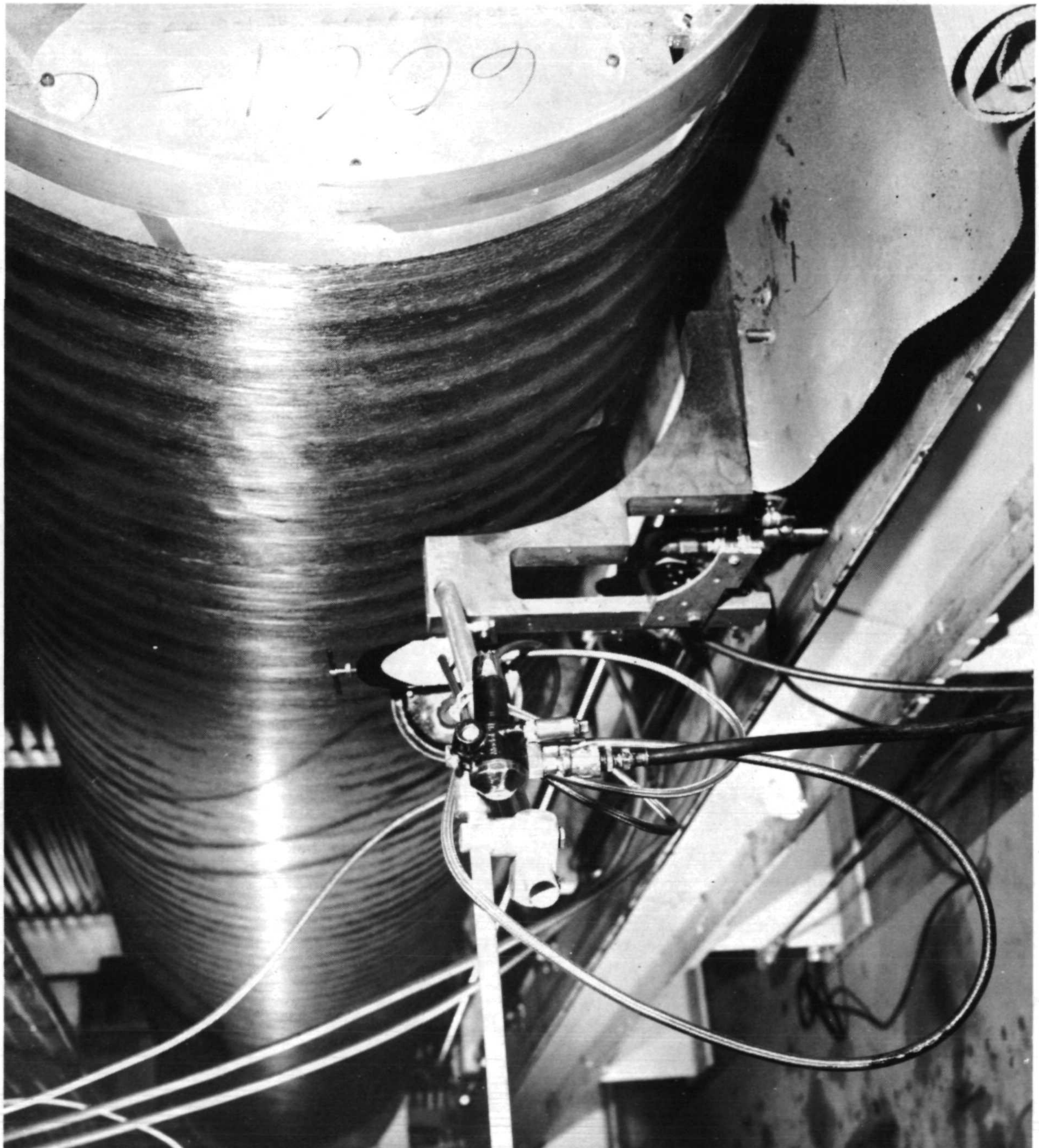


Figure 10. Completed Winding Prior to Removal

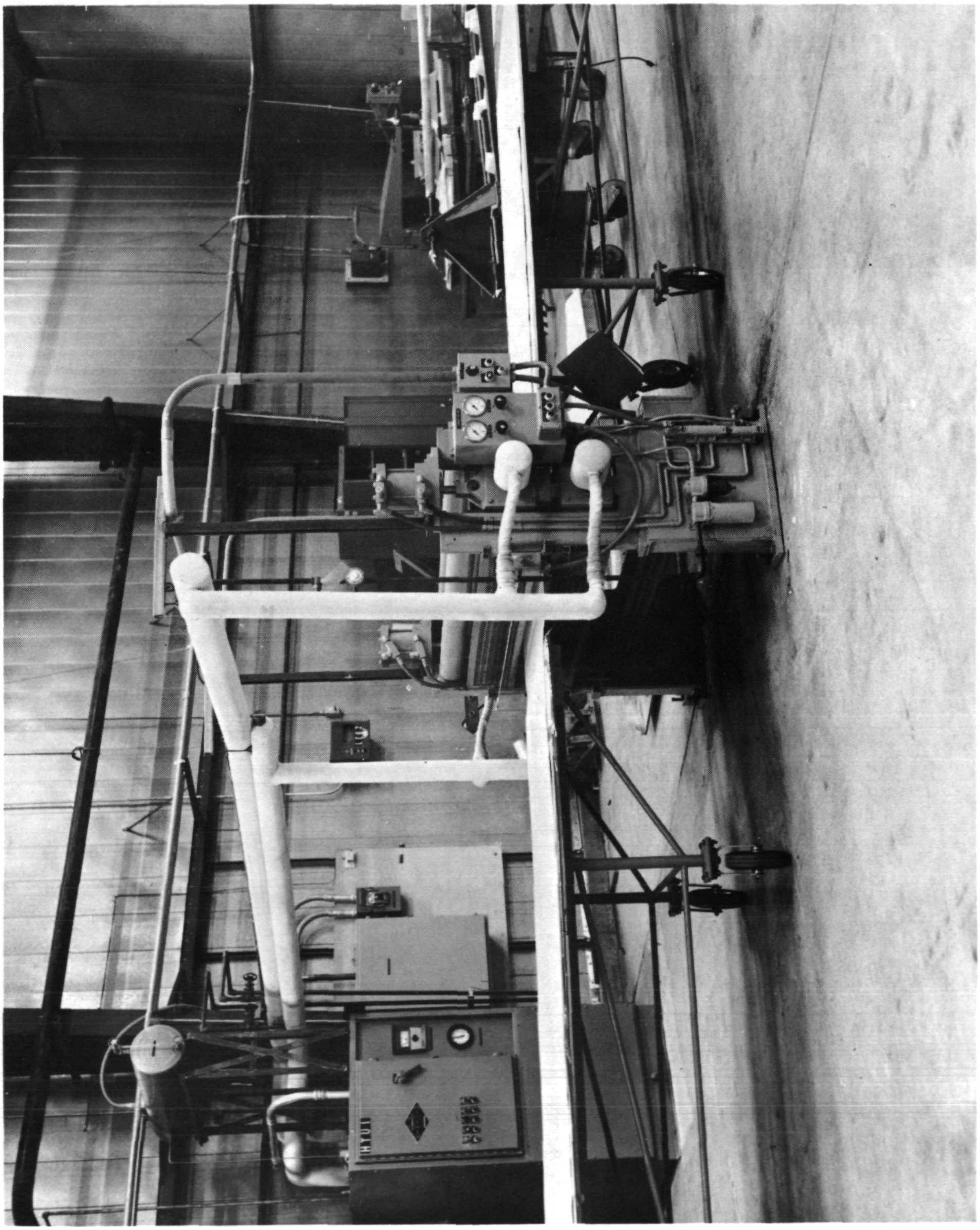


Figure 11. Press Rolls

The two parallel rolls can be finely adjusted for pressure, rate of travel, and temperature for very accurate and reproducible pressing. An elevated temperature is required for the RS-6234 system since it will not flow at room temperature. A rate of 1 ft/min at 100° to 120° F is sufficient to produce acceptable prepreg. Pressing at 200° F resulted in better compaction of the prepreg but tended to initiate a precipitous resin reaction.

After pressing the prepreg, the large sheet(s) are cut into final ply dimensions and then packaged for storage or shipment. Originally, polyethylene film was used as a bagging material (the prepreg must be stored in an airtight bag because moisture condensation during warmup from 0° F storage would damage the uncured prepreg). Later concern over possible loss of important volatiles through the bags during storage resulted in a change to aluminum foil lined polyethylene storage bags which are currently in use.

5. Quality Control

Every lot of thin prepreg manufactured for the program is identified for lot number, fiber and resin type, date of manufacture, and other pertinent data. Table II summarizes the processing information regarding the thin prepreg produced under this contract. A manufacturing and inspection record (M&IR) accompanies each lot of prepreg to check processes and materials. These records are filled out during the prepreg manufacturing process. Standard quality control data including prepreg resin and volatiles content is included in Table II. Table III summarizes the prepreg tests conducted.

The gas chromatograph was used to test samples of aged prepreg (Lot 3, manufactured July 1971) because of insufficient flow during cure. This material had been stored for several months at 0° F in polyethylene bags. A gas chromatograph comparison of this prepreg with a more recent prepreg (Lot 6, manufactured December 1971) showed percentages of solvent for each lot reflected in Table IV and led to the use of the metalized storage bags.

The lesser ethanol content in the earlier lot was unimportant because this solvent does not affect resin flow during the cure cycle. The small drop of NMP was expected because of the high boiling point of the solvent. However, the drop in xylene content was greater than 50 percent, a serious change considering that the xylene controls resin flow at lower temperatures (according to Monsanto, the RS-6234 manufacturer). The xylene probably had evaporated through the polyethylene bag.

Details of the precipitation problem of RS-6234 will be discussed in the Phase III report since the problem is generally considered a cure problem and only indirectly related to prepreg processing.

TABLE II

THIN PREPREG MANUFACTURING DATA SUMMARY

Prepreg Designation	Date Manufactured	Type Fiber	Type Resin	Resin Lot No.	Release Paper Type SRK = Silicone Released Kraft	Fiber Weight (gm/ft)	Fiber Width (in.)	Resin Flow (cc/min)	Pump Range (ma)	Resin Viscosity (cps)	Resin Spray Gears (746 high range)	Fiber Delivery Gears (746 high range)	Fiber Lead (tpi)	Single Sheet or Crossplied (±45°)	Total Number of Passes (resin spray)	Pressing Type (press or rolls)	Pressing Temperature (°F)	Pressure (psi) (lb/in.)	Roll Gap	Time (press) or Rate (rolls)	Roll Rate (percent of full range)	Resin Content (finished prepreg) (%)	Volatiles (finished prepreg) (%)	Quantity of Usable Prepreg Obtained (ft^2)
Experimental 2	6/17/71	HT	6234	3366	SRK	0.2907	2.6	--	--	330	A1	B2	0.765	S	2	Press	200	200	--	1-1/2 min	--	37	20	30
Drum No. 1	7/13/71	HT	6234	3366	SRK	0.2907	2.8	--	--	320	A1	B2	0.765	S	2	Rolls	200	80	0.028	12 ipm	9-1/2	42	21	30
Drum No. 2	7/20/71	HT	6234	3366	SRK	0.2907	2.8	--	--	320	A1	B2	0.765	±45	2	Rolls	200	75	0.028	12 ipm	9-1/2	42	23	180
Drum No. 3	7/21/71	HM	6234	3366	SRK	0.2583	2.7	--	--	330	A1	B2	0.765	S	2	Rolls	200	75	0.028 0.022	12 ipm	9-1/2	41	23	120
Drum No. 4	7/23/71	HT	6234	3366	SRK	0.2907	0.3	--	--	340	A1	E1	5-3/25	S	6	Rolls	200	75	0.032 0.023	12 ipm	9-1/2	34	18	90
Drum No. 4, Exp	9/13/71	HT	P13N	EP4	SRK	0.2907	0.3	41	19.75	400	A1	E1	5-3/25	S	1	Rolls	RT	40	0.026 0.022	12 ipm	9-1/2	39	15	40
Drum No. 5	10/7/71	HT	6234	3366	SRK	--	2.7	--	--	365	A1	B2	0.765	±45	2	Rolls	100	90	0.005	5 ipm	6	43	23	160
Drum No. 6, Exp	11/1/71	HT	6234	3409	SRK	--	--	--	--	2300	A1	--	--	--	--		Could not obtain decent spray							
Drum No. 6	12/7/71	HT	6234	3409	SRK	--	2.9	--	--	320	A1	B2	0.765	±45	2	Rolls	100	90	0.005	5 ipm	6	46	22	200
Drum No. 7	1/25/72	HM	6234	3409	SRK	--	2.9	--	--	340	A1	B2	0.765	S	2	Rolls	110	90	0.000	6 ipm	7	47	26	100+
Drum No. 8	2/16/72	A	6234	3506	SRK	--	3.0	--	--	310	A1	B2	0.765	±45	2	Rolls	110	90	0.002	7 ipm	8	43	26	50+
Drum No. 9	4/3/72	HT	6234	3506	SRK	--	2.9	--	--	310	A1	B2	0.765	±45	2	Rolls	110	60	0.005	7 ipm	8	47	26	180
Drum No. 10	4/3/72	HT	6234	3506	SRK	--	2.9	--	--	310	A1	B2	0.765	±45	2	Rolls	110	60	0.005	7 ipm	8	44	25	180
Drum No. 11	4/5/72	HM	6234	3506	SRK	--	2.8	--	--	310	A1	B2	0.765	S	2	Rolls	110	60	0.004	10 ipm	9	47	24	160
Drum No. 12A	4/13/72	HT	6234	3506	SRK	--	2.8	--	--	310	A1	B2	0.765	±45	2	Rolls	110	60	0.005	7 ipm	8	44	24	80
Drum No. 12B	4/13/72	HM	6234	3506	SRK	--	2.8	--	--	310	A1	B2	0.765	5	2	Rolls	110	60	0.004	7 ipm	8	41	24	80

TABLE III

QUALITY ASSURANCE TESTS FOR EACH LOT OF BROADGOODS

Test	Conditions	Sample Size	No. of Tests per Lot
Volatiles	$420 \pm 15^{\circ}$ F 110 ± 15 min	2 by 10 in.	3
Resin content	Nitric acid digestion Weigh 1 gm sample Add 100 ml conc. nitric acid Boil on hot plate for several hours Filter Boil at 100° C for 3 hr Weigh dry fibers	Use volatiles sample	3
Visual inspection	Gaps Resin rich areas Resin poor areas Fiber alignment		

TABLE IV

GAS CHROMATOGRAPH RESULTS

Test for	Drum 3 (HM/6234)	Drum 6 (HT/6234)
Total volatiles percent of prepreg	21	20
Water percent of prepreg	5.0	6.7
Ethanol percent of prepreg	6.9	8.4
Xylene percent of prepreg	1.5	3.3
NMP percent of prepreg	5.2	5.5

APPENDIX A

1 - 2 MIL BROADGOODS MANUFACTURE

1. SCOPE

1.1 This document describes the general operations for the manufacture of thin (.001" and .002" thick) graphite broadgoods.

2. MATERIALS AND EQUIPMENT

2.1

Material	Description
Hercules Graphite Fiber	Type HM, HT, A
Resin	Whatever specified - resin, however, should have a maximum viscosity of 1000 cps.
Wiping Cloths	Clean, lint free
Solvent	Anhydrous ethanol formula 3A (denatured)
Tape	Assorted types and sizes
Release Paper	Various, selection determined by independent studies
Containers	Assorted pint, quart and gallon cans

2.2

Equipment	Description
Winding Machine	FWM-746
Resin Applicator	"Binks" Airless Spray Equipment
Fiber Deliver System	Suitable for 3" wide spread fiber
Extension Tables	Long hardboard covered
Press Rolls	Black and Clawson
Coveralls	Individually sized
Gloves	Pylox or Butyl #320

3. SAFETY

3.1 Butyl gloves #320 shall be worn when using MEK.

3.2 Safety regulations specified in UOP's shall be followed.

4. GENERAL OVERVIEW

4.1 The objective of this operation is to produce thin broadgoods.

4.2 In order to achieve this end product, several separate operations must be performed.

4.2.1 Dry graphite continuous yarn is first "spread" from its natural circular tow cross-section to a flat band approximately three inches wide.

4.2.2 This spread fiber is received on plastic spools with the fiber wound and separated by white interleaf paper.

4.2.3 The spread fiber is tensioned in a "Witzel wheel" fiber delivery system and wound onto a resin covered release paper.

4.2.4 The resin is coated onto the release material in a thin uniform layer by spraying with an airless spray gun.

4.2.5 The dry fiber which has been wound onto the resin layer is in turn sprayed with a second coat so the fiber is in effect sandwiched between two thin sheets of resin.

4.2.6 The large sheets of prepreg are cut from the winding drum and passed through heated compacting rolls.

4.2.7 In order to compact the thin broadgoods sheet, complete the staging of thes resin, and adhere the release sheets to the prepreg for easier handling, final pressing operation is performed.

4.2.8 This prepreg is then trimmed and cut into sheets of specific dimensions, sealed in airtight bags, and stored in a freezer until use.

5. OPERATIONS

5.1 Mandrel Preparation

5.1.1 Clean the mandrel with a suitable solvent to remove resins, oil, tape, etc., and remove burrs with emery cloth.

5.1.2 Cut the end of the roll of release paper at a 45^{o} angle (use large triangle).

CAUTION: Do not permit paper to fall on floor or other dirty area.

5.1.3 Unroll sufficient paper to cover the mandrel at the 45^{o} angle and reroll the paper.

5.1.4 Tape one end of the paper to one end of the drum being careful to line up the edges so that the one edge just touches the edge of a groove. (The mandrel has 45^{o} cutting grooves to facilitate removal.)

5.1.5 After taping this piece to the drum (using polyester tape), remove wrinkles or loose areas by lifting tape in affected area, smoothing out wrinkle and retaping.

5.1.6 If paper does not cover mandrel completely, add a second or third parallel piece until entire drum is covered with the edges butted and taped.

5.1.7 Go over surface of release paper with clean lint free cloths to remove contaminants.

5.2 Raw Materials

5.2.1 Remove the resin from the freezer. Document the time of removal and identifying numbers from the container.

5.2.2 Permit resin to reach room temperature in closed container. Resin may be placed in sink of warm water to quicken warm-up rate.

CAUTION: *Do not open until room temperature has been reached.*

5.2.3 Document time that resin container was opened.

5.2.4 Mix resin thoroughly. Place a droplet sample in Haake viscometer to determine viscosity. Record.

5.2.5 Reduce viscosity to 330 cps by adding ~12% anhydrous ethanol formula 3. (See supervisor to determine precise quantity of solvent to add.)

5.2.6 Resin solution should be placed in a clean, wide-mouth gallon "paint can". While pouring the solution, strain it through a #100 stainless screen to remove solid particles. These particles must be saved, crushed and returned to the solution and then re-strained until all material is <.006" particle size.

5.2.7 Re-seal the container of undiluted resin and return to refrigerator. Record the tared weight and resin to be used.

5.2.8 Obtain a sufficient amount of spread fiber to wind the prepreg sheets. The three-inch spread fiber should be provided on plastic "boron" spools with interleaf paper.

5.2.9 Record all fiber data for each spool on the M&IR such as fiber type, lot/batch number, weight of ten-foot sample, etc.

5.3 Machine Start-Up

5.3.1 Mount the resin spraying equipment on the 746 filament winding machine if this has not already been done.

5.3.2 Set the gauges and leads as specified for the particular lot of prepreg. Record.

5.3.3 Experiment on a sheet of scrap paper wrapped on the covered drum until this desired resin content is achieved. (These determinations are made prior to the actual winding as follows:)

5.3.3.1 Spray resin onto 10 $in.^2$ sheets of tared aluminum foil at settings estimated from prior experience with similar resin viscosities, solids contents, etc.

5.3.3.2 Weigh this coated sample and reduce or increase the mandrel speed and/or the fluid pressure until precisely one-half the total quantity of resin per unit area is obtained with one or several coats. Record these two important criteria. (Heating the sample is required for removal of volatiles if solids content is desired.)

5.3.4 Spray the entire drum, or the area to be covered by fiber, with the diluted and strained resin. Included in the first winding should be several small aluminum squares located at the seam along the length of the drum. The resin quantity can be checked by weighing these samples and minor adjustments made on the second coat.

5.3.5 Mount the fiber delivery system on the opposite side of the 746 machine if this has not already been done.

5.3.6 Verify that the tensioning belt runs true.

5.3.7 Mount the first spool of dry, spread fiber on the spool holder and feed the fiber through the "Witzel wheel" tensioning device.

5.3.8 Drape the interleaf paper out the back of the delivery system so that the paper falls freely to the floor.

5.3.9 On an unused section of the drum, tape the end of the fiber to the surface and rotate the drum one-half turn.

5.3.10 Check for even tension in the yarn and true tracking (i.e. that the fiber does not wander off the edge of the delivery wheels). If any of the above problems occur, notify supervision to correct the location of the guide rolls or change the angle of the delivery system to the mandrel.

5.3.11 Set the advance of the carriage according to the lead desired with the spread fiber.

5.3.12 Wind over the evenly distributed resin until the entire sheet is covered. Make all splices at the seams in the release paper.

5.3.13 When complete, go over the winding carefully to relocate fibers into bare areas from high fiber concentration.

5.3.14 Complete the application of the resin by spraying the other half into the fiber using the spraying procedure.

6. PRESSING

6.1 Cut the prepreg from the drum with a stanley knife. Follow the 45^{o} slot cut into the drum as a guide.

6.2 Move the cut prepreg sheets onto the long hardboard covered portable tables located in the building.

6.3 Trim six inches of prepreg at each end. Also, trim tape from the sides.

6.4 If the prepreg must be crossplied, cut the prepreg into handleable sizes and crossply. Otherwise, cover with release paper.

6.5 Move the tables to align with the press rolls.

6.6 Press the prepreg sheets using the procedure for that equipment

6.7 Record the temperature, pressure and feed rate used for the prepreg in the M&IR.

7. MARKING AND TRIMMING

7.1 Trim the prepreg and/or cut into finished sizes.

7.2 Obtain QC samples and bag.

7.3 Deliver QC samples for testing. Include data sheet on history and for inclusion of QC test results.

7.4 Mark each sheet in such a manner that each can be identified at a later date.

7.5 Perform visual inspection using fluorescent light if possible.

7.6 Include remarks for each piece in data sheet and on broadgoods surface sheet.

7.7 Package sheets for freezer storage or shipment. Identify packages with sufficient information to permit quick identification in freezer. Use heat sealable aluminized bags to limit volatile loss.

APPENDIX B

OPERATION OF AIRLESS RESIN SPRAY FILMING APPARATUS

1. SCOPE

1.1 The purpose of this procedure is to describe the proper method of operating the Binks airless resin spray filming apparatus and to outline the safety precautions to be followed during the operation of the equipment.

2. REQUIREMENTS

2.1 All persons working with the spray apparatus are responsible for knowing and following this procedure.

3. MATERIALS AND EQUIPMENT

Equipment	Description
Airless Pump	Binks Model Falcon 3A (2/3 GPM)
Fluid Pressure Regulator	Regulates Pressure of fluid up to 2500 psi
Spray Gun	Binks Automatic Type 51H
Various hoses and regulators	Refer to Figure 1
Resin	Refer to specific project procedure
Solvent	Various
Oil	Light vacuum pump type
Gloves	Pylox

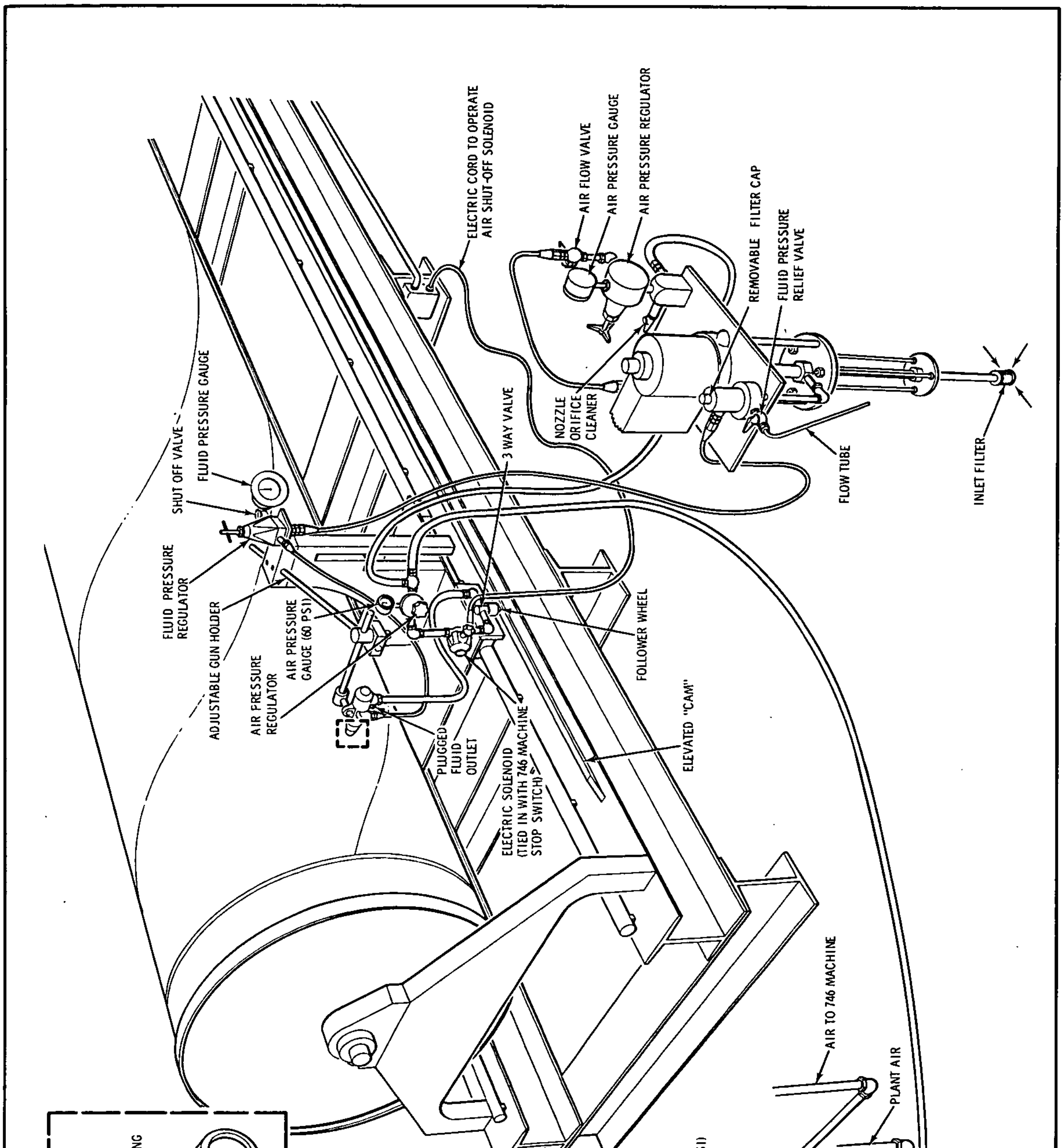
4. SAFETY

4.1 Safe operating methods in the use of the airless spray equipment will be the responsibility of each operator using it.

4.2 At the beginning of each operation, before opening the pressurized air supply valve, check for any irregularities.

4.2.1 Check hoses for cuts, abrasions or other damage.

4.2.2 Assure that all hoses are securely attached to fittings.



ELECTRIC CORD TO OPERATE AIR SHUT-OFF SOLENOID
AIR FLOW VALVE
AIR PRESSURE GAUGE
AIR PRESSURE REGULATOR
REMOVABLE FILTER CAP
FLUID PRESSURE RELIEF VALVE
NOZZLE ORIFICE CLEANER
FLOW TUBE
INLET FILTER
3 WAY VALVE
FLUID PRESSURE GAUGE
SHUT OFF VALVE
FLUID PRESSURE REGULATOR
ADJUSTABLE GUN HOLDER
AIR PRESSURE GAUGE (60 PSI)
AIR PRESSURE REGULATOR
FOLLOWER WHEEL
PLUGGED FLUID OUTLET
ELECTRIC SOLENOID (TIED IN WITH 746 MACHINE STOP SWITCH)
ELEVATED "CAM"
AIR TO 746 MACHINE
PLANT AIR
REGULATOR
GAUGE (90 PSI)
FILTER
GUN BODY
AIR OPERATED PLUNGER
SAPHIRE INSERT (FEATHER EDGE)
NOZZLE TIP
NOZZLE HOUSING

4.2.3 Assure that all gauges are set at zero and that calibration date has not expired (where applicable).

4.3 During normal operations, the toxicity levels of solvents such as Ethanol in the room should be below the minimums set forth by the Safety Department. Several solvents, however, such as DMF, require special handling procedures. Check with your supervisor to determine if any hazards exist when handling a specific material.

4.4 Use pylox gloves when using resins in the spray system.

5. HISTORY

5.1 Since airless spray resin filming is a new operation at Bacchus, a brief history is included to acquaint the operator with the system and the function of some of the key components. Refer to Figure 1.

5.1.1 Acquisition of the equipment came about from a need to uniformly distribute a controlled quantity of resin onto a sheet of release paper prior to laying fibers in the conventional prepregging process.

5.1.2 The main advantage to this system over others is that the resin filming is performed with the release paper right on the mandrel rather than filming on a separate filming machine, thus eliminating the handling of sticky sheets of resin coated paper. Simply, the gun mounted to the carriage of the 746 machine is forced to travel from headstock to tailstock at a specific rate while the rotating mandrel exposes new surface area to the gun spray. The resin, applied in a spiral pattern, can achieve very evenly distributed and highly automated placement.

5.1.3 A wide range of resin thicknesses are obtainable with most resins by either increasing the drum rotation velocity or inserting different gun nozzle orifices. Also, filming can be improved by varying the gun distance from the mandrel, selecting nozzle tips which give wider or narrower spray bands or by varying the viscosity of the resin either by diluting with solvent or heating the resin in a recirculating heater setup (not yet available). Variation in fluid pressure by adjusting a fluid pressure regulator located in the fluid delivery line just before the spray gun is another way of affecting the filming behavior of a resin.

5.1.4 A brief summary of the functions of several major components of the system follow.

5.1.4.1 Pump - The air operated Falcon 3A pump is found mounted on the east wall of the building in back of the 746 machine. The pump causes pressurization of the resin supplied underneath the pump. The airless pump features teflon seals which make it possible to clean the pump without disassembly.

5.1.4.2 Automatic Spray Gun - The airless spray gun is designed to direct an even flow of pressurized resin, received from the pump, through an orifice (nozzle tip) and to locate this point of delivery at a variable distance from the surface being sprayed. The gun is a recirculating type so that in the future heated resin may be circulated through the gun. One side is currently plugged (dead end system), i.e. all the resin solution that enters the gun must exit through the nozzle tip.

5.1.4.3 Nozzle Tip - Various nozzles are available for a wide range of spray patterns.

CAUTION: The nozzles are machined from tungston carbide. It is very brittle material and will break if dropped on the floor.

A sapphire insert is also available. This insert causes a finer edge (feathering) of the elliptical spray pattern.

5.1.4.4 Fluid Regulator - The fluid regulator permits fine control of fluid pressure for repeatedly sprayed resins under production conditions. This regulator and gauge are located just before the gun and are mounted on the adjustable gun holder base.

5.1.4.5 High Pressure Hose - A teflon hose overwrapped with steel wire delivers the high pressure resin from the pump to the gun via the fluid pressure regulator. Since the pressures in this hose exceed 2000 psi, it is essential that this hose be straight (free from kinks and sharp angles).

5.1.4.6 Three-Way Valve - The orifice of the gun is normally sealed by a movable plunger. Pressurized air (60 psi maximum) is required to open the plunger and permit flow of resin through the orifice. Air at 90 psi enters the air control center mounted onto the 746 machine movable carriage. (See Figure 1.) Air at this pressure goes to the pump. The remainder is reduced to 60 psi by a regulator. An electric solonoid located after the gauge closes (the plunger) when the winding machine is stopped in the event of a problem. Normally, however, the air to the gun is opened and closed automatically by a three-way valve (bolted to the carriage) which conveniently opens and shuts off at the beginning and end of a spray run by a movable follower wheel following a flat cam (elevation) located on the base of the 746 machine.

5.1.5 A fine film of resin cannot be obtained with every resin. The release properties of the material sprayed upon tend to bead up the resin. This is not a serious problem; however, since during a follow-up pressing operation, most beaded resins will spread out evenly. What is important, however, is the resin be distributed evenly.

6. OPERATION

6.1 Attach a sheet of cardboard to the carriage of the winding machine in such an area so as to protect the ways and lead screw from resin overspray. Also, cover the floor area around the pump with poly film.

6.2 Select a lead suitable for the particular nozzle being used. Try the greatest lead (approximately three inches per revolution) first. Record in M&IR.

6.3 Cover the section of the 746 winding machine mandrel to be sprayed with the desired release material. The paper must be taped tightly against the mandrel and free of wrinkles.

6.4 Using cork-type Coraprene or a similar tape, make a straight line cam with tapered (30° angle) ends. The entire strip should be slightly shorter than the width of the release paper to prevent overspray onto the bare mandrel surface. Two thicknesses of Coroprene will be required to completely open the three-way valve.

6.5 Solvent clean the surface of the 746 machine I-beam base upon which the self-adhering Coroprene will be attached. Using the 746 machine procedure, move the spray gun to the position desired for starting the resin spray.

6.6 Place the end of the strip at the base of the three-way valve actuating wheel. Measure the distance to the edge of the I-beam and locate the rest of the strip on a straight line on the beam. Tape the ends down with tape and check that the wheel travels on the top of the elevated strip.

6.7 Prepare the resin to be sprayed. Filming is influenced by viscosity. If the viscosity is too high (over 600 cps), the sprayed resin will be too dry and will not wet the surface of the release paper. On the other hand, if the resin is too thin (under 100 cps) and thinned with solvent, the sprayed resin will film well but will quickly break up and migrate into large droplets (a minimum surface tension phenomenon). The exception to this latter rule is the spraying of heated resins, a condition which has not yet been investigated completely. Each resin behaves differently and experimentation is required to establish speeds, pressures, etc., most suitable for each particular resin. Resin should be strained to remove large particles before spraying resin.

6.8 Push the "stop" button on the portable SCR controller.

6.9 Open the valve releasing plant air into the spray gun lines (located on South wall). Set regulator at 90 psi.

6.10 At the winding machine carriage, regulate the air pressure to the gun at 55 - 60 psi.

CAUTION: Exceeding this pressure may damage the spray gun.

6.11 Cover the hoses and gauges under the airless gun spray with plastic film.

6.12 Remove the nozzle tip and insert from the spray gun. Clean the two items in solvent (MEK) and clean orifices with air blast from air gun mounted on the airless pump. Set aside to dry.

CAUTION: Hold parts carefully in your finger tips as air blast may blow nozzle tips out of your fingers.

CAUTION: Nozzle is carefully machined from tungston carbide, a very hard and brittle material. Parts will break if dropped to the floor.

6.13 Remove the spray gun from its holder and aim into a liquid waste container. The pump contains oil to prevent drying of the seals when not in use. This oil must be removed prior to pumping resin through the system. Triclene solvent will cut the oil followed by a solvent which is compatible with the resin to be pumped (example, MEK for 3002 or methyl alcohol for RS-6234). Pumping air through the lines will remove the solvent.

6.13.1 Move the three-way valve onto the cam to open the three-way valve.

6.13.2 Place a one-gallon container of solvent under the pump.

CAUTION: The intake screen must be completely immersed in the solvent for proper pumping of solvent or resin.

6.13.3 Increase air pressure to the pump to 15 psi.

6.13.4 Open pump valve to activate pump. Pump will stop when solvent fills system and is under pressure.

6.13.5 Increase fluid pressure to gun by turning regulator next to gun. Increase to 100 psi. Increase pump pressure if required.

6.13.6 With speed control on zero, push either forward or reverse button.

CAUTION: High pressure spray may splatter if not aimed into large container.

6.13.7 Add additional solvent to intake until all traces of oil are removed from system (visual). The pump may vibrate when pumping solvent; reduce pump pressure to minimum.

6.13.8 Turn off pump (close valve).

6.13.9 Release air pressure to pump (regulator).

6.13.10 Push stop button on SCR portable controller.

6.13.11 Release pressure to pump (fluid regulator).

6.13.12 Remove pressure (solvent) from lines and filter using relief valve next to filter.

6.13.13 Remove supply of solvent from under pump.

6.14 Pump air through system to remove solvent. (Follow steps 7.14.3 through 7.14.12.)

6.15 Place container of resin under pump.

6.16 Increase pump pressure to in excess of 60 psi (60 to 90 psi). Fluid will be pumped into lines.

CAUTION: Level of resin must be above top of intake filter at all times.

6.17 Increase fluid pressure to between 1000 and 2000 psi. (Exact reading requires experimentation; start with 1500 psi.)

6.18 Push "forward" button. Spray about one pint of resin to remove residuals from fluid lines. Push stop button to cut off spray.

6.19 Release fluid pressure and move valve off cam with sufficient time to set carriage speed before spray starts (2 feet).

6.20 Clean airless gun and mount on winding machine gun holder.

6.21 Adjust distance of gun from mandrel as desired. Increasing distances of gun tip from the mandrel surface gives wider spray band but increases chances of resin beading with thicker resin solutions. Decreased distances improves filming but increases splattering, overspray and dripping.

6.22 Move three-way valve off track.

6.23 Locate nozzle tip and sapphire insert onto spray gun and tighten using special wrench. Examine tip so that slot is horizontal.

6.24 Push "forward" button on controller if going toward tailstock or vise versa.

6.25 Increase mandrel speed to desired setting. Use tachometer for accurate reading. (This second variable influences quantity of resin sprayed and required experimentation to establish proper speed.)

6.26 Spray will start when moving carriage causes three-way valve to open on elevated cam.

6.27 Spray will stop when valve roller drops off the elevated track.

6.28 If nozzle clogs during spraying:

6.28.1 Cover spray with empty gallon can.

6.28.2 Turn fluid pressure regulator to 0 psi.

6.28.3 Slowly stop mandrel rotation.

6.28.4 Remove splattered resin from paper (if any) with clean lint-free cloth.

6.28.5 Rotate drum/carriage back to about 2 feet in front of where spray stopped (or where resin was removed back to).

6.28.6 Remove nozzle tip and insert from gun and remove clog from orifice with brush, solvent and air blast.

6.28.7 Clean surface of gun with solvent and replace inserts.

6.28.8 Holding empty can in front of gun, push forward button and increase speed to desired amount.

6.28.9 Increase fluid pressure to desired amount.

6.28.10 When lead brings gun to position where spray left off, remove can collecting resin and continue spraying of release paper.

7. <u>MACHINE SHUTDOWN</u>

7.1 Remove supply of resin from pump intake.

7.2 Remove resin from lines.

7.2.1 Remove nozzle tip and insert from gun and clean.

7.2.2 Place end of gun in solvent waste container.

7.2.3 Place container of compatible solvent under pump and follow procedure for cleaning lines (Steps 7.14 through 7.15).

7.3 Remove pump filters and clean in solvent.

7.4 Place a gallon can of light vacuum pump oil under pump and fill lines with oil using procedure Steps 7.14.3 through 7.14.6.

7.5 Follow steps 7.14.8 through 7.14.12.

7.6 Clean airless gun and mount on winding machine gun holder.

7.7 Move carriage assembly as required to permit cleaning of resin from 746 machine. Check and re-oil ways and lead screw if there is evidence of resin on either. Remove resin from carriage and rest of machine with solvent.

7.8 Clean floor around 746 machine and area around pump.

7.9 Shut off power to 746 machine.

7.10 Shut off air supply to both the 746 machine and airless spray equipment.

APPENDIX C

MANUFACTURE AND INSPECTION RECORD FOR THIN PREPREG

COMPOSITE MATERIALS DEVELOPMENT MANUFACTURING RECORD
HERCULES INCORPORATED, BACCHUS, PLANT 3

Schd. Date: ______
Date Comp.: ______

A.O. Number ______
A.O. Mod. Number ______
Serial Number ______
Lot/Batch Number ______

Broadgoods 1-2 Mil Fabircation Record
Procedure Nos ______ Airless Resin Spray, Manufacure of 1-2 Mil Broadgoods

Charge Code ______
Labor ______
Material ______

Sign-Off A/R

Operation Started ______ Time ______ Date ______

Mandrel Preparation

Mandrel cleaned (6.1.1) manufacture of 1-2 mil broadgoods
Type release paper selected ______
Paper cut and applied to mandrel snugly and wrinkle free (6.1.2) 1-2 mil broadgoods.

Resin removed from freezer and allowed to come to room temperature in closed can.
Record time removed ______.
Record method used to bring resin to room temperature ______ (6.2.2) 1-2 mil broadgoods
Record resin temperature prior to use ______. Do not open can until resin has reached room temp.

Raw Materials

Resin
Resin type ______
Can No. ______ (6.2.1) 1-2 mil broadgoods
Record time resin container was opened ______ (6.2.3) 1-2 mil broadgoods
Gross Wt. Can Before Start (6.2.4) ______ ______ ______ ______ ______
Determine viscosity by Haake Method. Record ______ (6.2.4)
Reduce viscosity to 330 cps by adding solvent, (see supervisor) (6.2.5)
Strain resin to remove solid particles. (6.2.6)

Number of stops caused by clogged nozzle ______
Gross wt. can after first coat or resin ______

Approvals:
Operations ______
Project ______

COMPOSITE MATERIALS DEVELOPMENT MANUFACTURING RECORD
HERCULES INCORPORATED, BACCHUS, PLANT 3

Schd. Date: ______
Date Comp.: ______

A.O. Number ______
A.O. Mod. Number ______
Serial Number ______
Lot/Batch Number ______

Broadgoods 1-2 mil fabrication Record
Procedure Nos: airless resin spray
manufacture of 1-2 Mil Broadgoods

Charge Code ______
Labor
Material ______

Sign-Off A/R

Fiber (6.2.9) 1-2 Mil

	S. F. spool	S. F. spool	S. F. Spool	S. F. Spool	S. E. Spool
Fiber type					
Lot/Batch No.					
Weight of 10 Foot lenth tow					
Spool Weight Start					
Spool Weight End					
No. of Stops Due to Poor Fiber Quality					
No. of splices; fiber					
paper					

	S. F. spool	S. F. Spool	S. F. Spool	S. F. Spool	S. F. Spool
Fiber type					
Lot/Batch No.					
Weight of 10 Foot Lenth tow					
Spool Weight Start					
Spool Weight End					
No of stope due to poor Fiber quality					
No. of splices: fiber					
paper					

* S. F. indicates spread fiber spool.

Approvals:
Operations ______
Project ______

Page 2 of ____

COMPOSITE MATERIALS DEVELOPMENT MANUFACTURING RECORD
HERCULES INCORPORATED, BACCHUS, PLANT 3

Schd. Date: ______
Date Comp.: ______

.O. Number ______
.O. Mod. Number ______
erial Number ______
ot/Batch Number ______

Broadgoods Fabrication Record
Procedure No. ______

Charge Code ______
Labor ______
Material ______

Sign-Off A/R

Winding Setup

Mount resin spraying equipment on 746 machine per UOP (6.3.1) Date ______ Time ______
Place cam on machine base and check on-off operation of nozzle for mandrel length.
Calculate quantity of resin per unit area (6.3.2) ______
Carriage Lead ______

1. Mandrel speed ______ rpm. (ma) 2. Fluid Press ______ (at pump)
Nozzle tip size ______ mils, Ø__ Fluid pressure (guage) ______ psi (at gun)
Spray Area to be Covered by fiber ______ in. (6.3.3.3) using ½ of resin. (6.3.4) Time ______
Distance of gun tip from mandrel ______ in.
Check resin coating of first layer.
Mount Fiber delivery system on 746 machine (6.3.5)
Check tension of yarn & true tracking of belt and fiber ______ (6.3.10)
Set advance of carriage to desired lead. (6.3.11) ______ Record wind spread fiber over entire sheet,
Make splices at seams (6.3.12) Finish time ______
Relocate fibers into bare areas -(6.3.13) time spent ______ Minutes. Time ______
Complete resin application by spraying over fiber covered drum (6.3.14) Time ______
Check resin coating of second layer, add additional thin coat if total resin solids are below requirement.

pprovals:
perations ______
roject ______

Page 3 of ______

COMPOSITE MATERIALS DEVELOPMENT MANUFACTURING RECORD
HERCULES INCORPORATED, BACCHUS, PLANT 3

Schd. Date: ______
Date Comp.: ______

A.O. Number ______ A.O. Mod. Number ______ Serial Number ______ Lot/Batch Number ______	Broadgoods Fabrication Record Procedure No. ______	Charge Code ______ Labor ______ Material ______	Sign-Off A/R

Pressing

Remove prepreg from drum (Time ______)
Trim large sheets of prepreg to remove tape, etc. Cover with release paper (Time ______)
Pass prepreg through press rolls or cut and cross ply if this is called for.
Press rolls @ ___ °F temperature (2 hours to stabilize ______)
Nip pressure in(left ______ right ______)
Roll speed (% of 100) ______ (in/min)
Rolls gap ______ (include calculated thermal expansion)

Pressing complete; record time ______.

Approvals:
Operations ______
Project ______

COMPOSITE MATERIALS DEVELOPMENT MANUFACTURING RECORD
HERCULES INCORPORATED, BACCHUS, PLANT 3

Schd. Date: ______
Date Comp.: ______

A.O. Number ______
A.O. Mod. Number ______
Serial Number ______
Lot/Batch Number ______

Broadgoods Fabrication Record
Procedure No. ______

Charge Code ______
Labor ______
Material ______

Sign-Off A/R

Marking and Cutting

Wide goods cut and identified per CMD manufacturing record

Record width of starting edge scrap ______. /Area of sheets ______.

Cutting and Q.C. Sample Preparation

Q.C. samples cut and bagged per CMD MFG. Record

Deliver test samples to Q.C. for preparation of test request.

Inspection

Q.C. to prepare request for testing in accordance with CMD MFG. Record

Perform visual inspection using fluorescent light

Packaging

Package panels for freezer storage, or shipment and identify CMD MFG. Record instructions.

Documentation Review

Approvals:
Operations ______
Project ______

Page 5 of ______

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