NASA Contractor Report 181780

(NASA-CR-181780) MANUFACTURING DEVELOPMENT OF PULTRUDED COMPOSITE PANELS Final Report (Lockheed Aeronautical Systems Co.) 58 p CSCL 11D

N92-22637

Unclas G3/24 0085731

Manufacturing Development of Pultruded Composite Panels

L. E. "Roy" Meade

LOCKHEED AERONAUTICAL SYSTEMS COMPANY
BURBANK, CALIFORNIA 91520

PREPARED FOR
LANGLEY RESEARCH CENTER
UNDER CONTRACT NAS1-15069
APRIL 1989

RELEASE WILL BE THREE (3) YEARS FROM DATE INDICATED ON THE DOCUMENT

NASA
National Aeronautics and
Space Administration

Langley Research Center
Hampton, Virginia 23665-5225

NASA Contractor Report 181780

Manufacturing Development of Pultruded Composite Panels

L. E. "Roy" Meade

LOCKHEED AERONAUTICAL SYSTEMS COMPANY
BURBANK, CALIFORNIA 91520

PREPARED FOR
LANGLEY RESEARCH CENTER
UNDER CONTRACT NAS1-15069
APRIL 1989

NASA
NATIONAL AERONAUTICS AND
SPACE ADMINISTRATION

National Aeronautics and
Space Administration

Langley Research Center
Hampton, Virginia 23665-5225

FOREWORD

This report was prepared by the Lockheed Aeronautical Systems Company, Lockheed Corporation, Burbank, California, under Contract NAS1-15069. This contract was sponsored by the National Aeronautics and Space Administration (NASA), Langley Research Center. The Program Manager for Lockheed is Mr. C. F. Griffin. The Technical Representative for NASA, Langley is Mr. M. Dow.

The following Lockheed personnel were principal investigators to the program:

Engineering Manager
C. F. Griffin

Design
R. Bernard

Materials
G. Hull and M. Paul

Final Report
L. E. "Roy" Meade

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF FIGURES (CONTINUED)

MANUFACTURING DEVELOPMENT OF PULTRUDED COMPOSITE PANELS

L. E. "Roy" Meade

Lockheed Aeronautical Systems Company
Burbank, California

SUMMARY

A detailed design and analysis was conducted for a stiffened wing cover panel. The design had three "J" shaped stiffeners spaced on 6.6 inch centers. Damage containment features developed in NASA contracts were incorporated within the design. Structural analysis of the design was made using Lockheed's computer programs and NASA's "PASCO" computer program. Three types of graphite fiber fabrics and Shell 9310 resin were selected for construction of the stiffened panel. The selected materials were characterized in pultruded laminates and the data compared to test results available on laminates made from preimpregnated graphite/epoxy materials.

A shaping/curing die was designed and fabricated for the stiffened panel. Stiffened panels were pultruded and post-cured. Tests were conducted which showed less than desired laminate quality due to excessive porosity. Sections of panels were shipped to NASA/LaRC for structural testing and results are presented in this report.

Material and process improvement studies were undertaken to improve the pultruded laminates. Autoclave cured laminates were compared with pultruded laminates. Results indicated entrained air in the resin as being the chief source of laminate porosity.

INTRODUCTION

The weight savings potential of graphite/epoxy composites for secondary and medium primary aircraft structures has been demonstrated. One of the greatest challenges facing the aircraft industry is to reduce the acquisition costs for composite structures to a level below that of metal structures. The pultrusion process, wherein reinforcing fibers, after being passed through a resin bath, are drawn through a die to form and cure the desired cross-section, is an automated low cost manufacturing process for composite structures. The Lockheed Aeronautical Systems Company (LASC) Composites Development Center designed, characterized materials, fabricated and tested a stiffened cover concept compatible with the continuous pultrusion process. The procedures used and the results obtained therefrom are presented in this report.

NOTE: Use of commercial products or names of manufacturers in this report does not constitute official endorsement of such products or manufacturers, either expressed or implied, by either the National Aeronautics and Space Administration or the Lockheed Aeronautical Systems Company.

PULTRUDED J-STIFFENED PANEL DESIGN

General

The program objective was to pultrude a panel designed for the C-130 center wing box structure. This article was particularly suitable to pultrusion since it has a constant cross-section. The effort was to investigate the potential of pultrusion to produce complex aerospace quality parts.

Several process driven constraints were encountered during the design that are unique to the pultrusion process. Since each ply requires a unique width as seen in Figure 1 due to the changing radius through the part's thickness, a part quickly became unmanageable due to the number of creels required to feed the material. The process also required a particular percentage and location of 0 degree plies in the laminate since they were used to pull the rest of the material through the die. This limited the use ply layups that were optimum for structural requirements.

The material vendor supplied several material plies of similar width stitched together so that the number of creels required could be reduced, see Figure 2. The pultrusion tool was designed to accommodate the extra width of the plies so that more plies could be stitched together to further reduce the creeling problem. In the "J" stiffened panel originally there were 117 identified different widths of material. By working with the material supplier and the tool geometry, this number was reduced to a much more manageable 13 creels of stitched material shown in Figure 3. The overall layup orientation of the panel was only slightly modified to reduce the number of different widths. The material vendor also varied the thickness of each 0 degree ply (by varying the yarn ends/inch) stitched into a group. This allowed some control over the location of the 0 degree plies for structural and damage tolerance efficiency. This was done by using thinner plies of 0 degree material where they were needed to pull other plies through the die and by placing thicker plies in the areas where they were structurally efficient.

The program also investigated the capability of ultrasonically examining a part as it exits the curing die. This examination avoided pulling an expensive large length of material only later to find out that the part was not acceptable. It was determined that the NDI information taken during the pultrusion was representative of that taken by a conventional machine.

The test and fabrication details of the pultruded panel are included herein. Figure 4 shows the pultruded panel.

Panel Design

Design of the J-stiffened panel derived from one developed for the Composite Wing Development Program, was initiated using NASA's Panel Analysis and Sizing Code (PASCO) computer program to synthesize the panel. The Lockheed-developed program, Integrally I-Stiffened Panel Stability and Strain Analysis (ITERI), was adapted to develop the analytical methods used to size the pultruded "J"-stiffened cover panels.

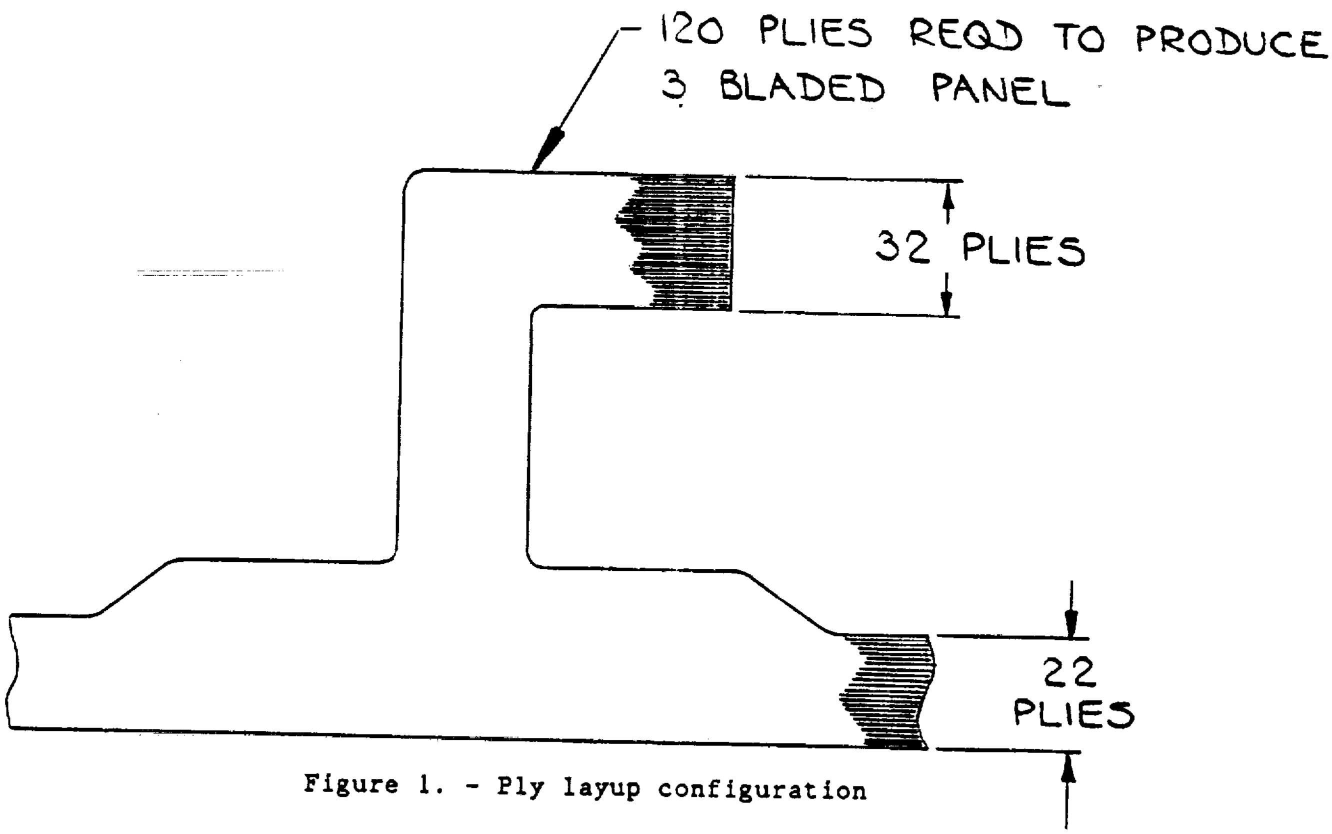


Figure 1. - Ply layup configuration

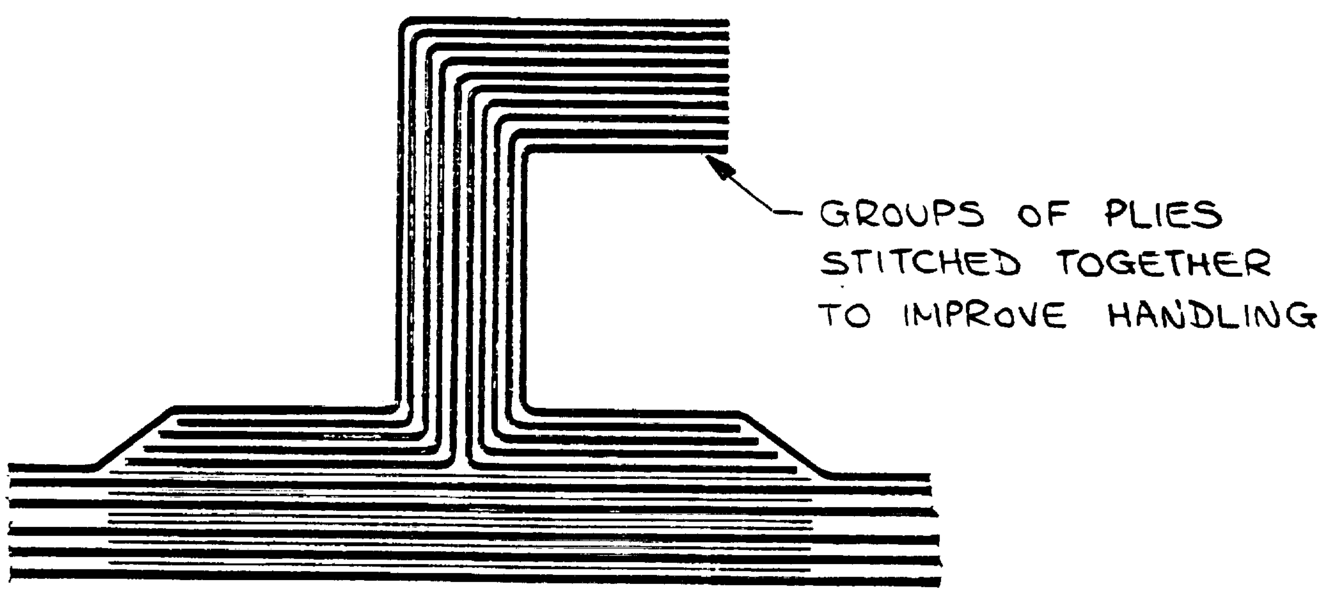
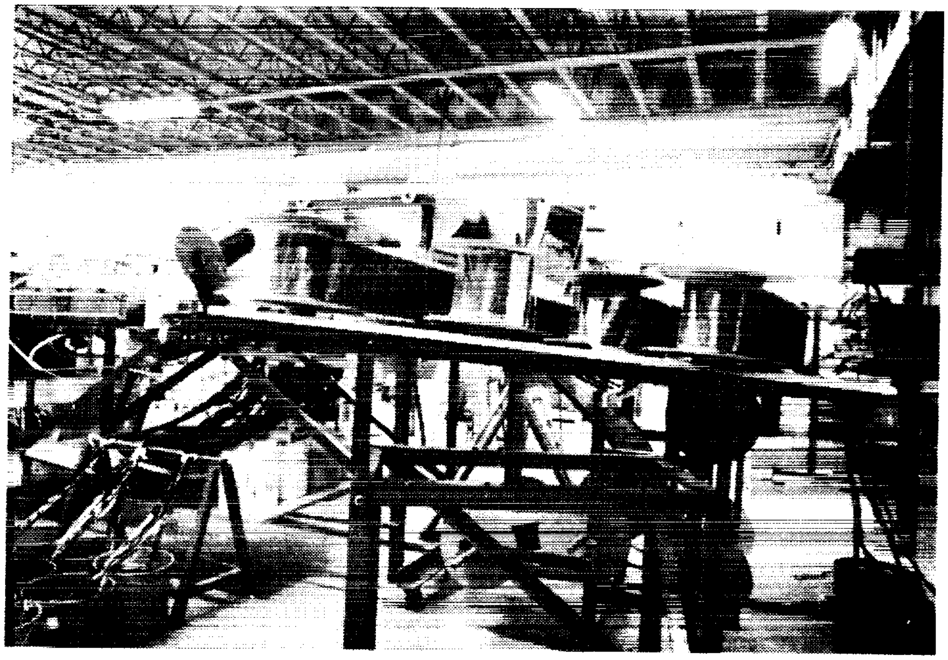


Figure 2. - Ply grouping arrangement to reduce number of creels



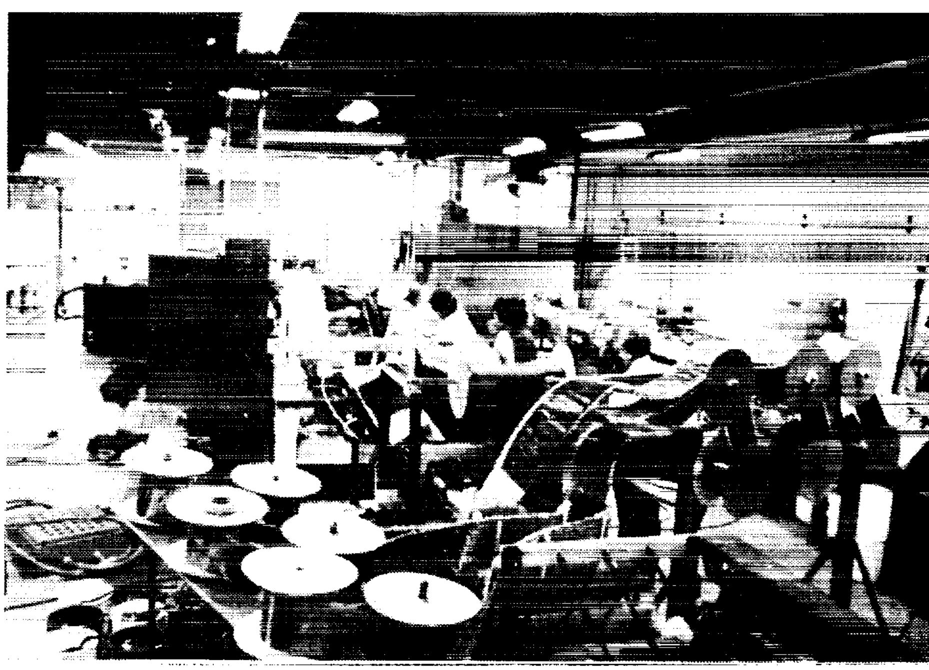


Figure 3. - Prestitched widths of material on reduced number of creels

[70]M0925.AA0

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

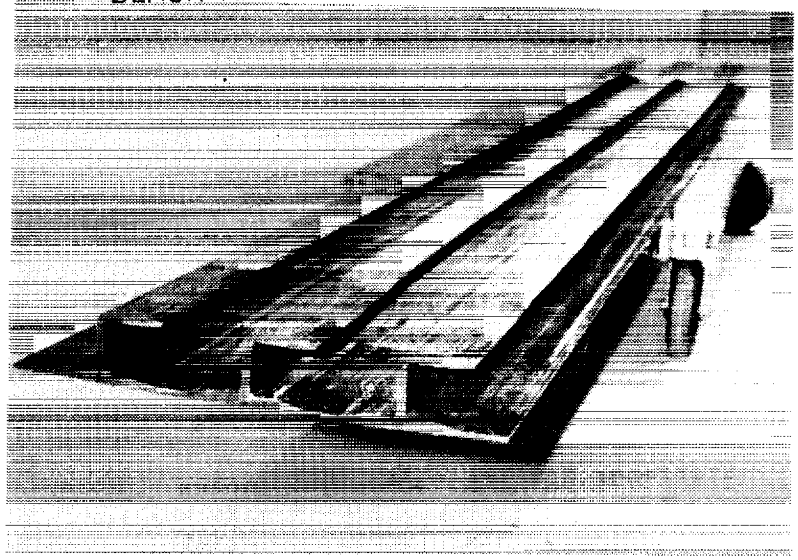


Figure 4. Pultruded panel

Multiple design iterations were conducted to obtain a minimum weight configuration having a predicted strength which met or exceeded the design requirements shown in Figure 5. The final design is shown in Figure 6. This design has a predicted compression buckling load of 33 kips/inch. For the combined compression, shear, and pressure loading, the maximum compression strain was 4100 μin/in. No panel eccentricities were included in this analysis. With a panel eccentricity of 0.040 inches, the maximum compression strain was approximately equal to the design value of 4500 μin/in.

This design was furnished by LASC's subcontractor, Pultrusion Technology, Inc., (PTI) for their review and critique. In discussions with PTI concerning the J-stiffened panel design, recommendations were made:

a. Minimize the number of different widths of fabric used within the panel.

b. Use small radii on the inside corners to reduce tooling costs.

c. Use large outside corner radii to prevent fiber bridging.

d. Place bias ($\pm$45 degrees) plies on the surface to increase pulling ease and improve surface quality.

e. Incorporate complete fiber coverage (no gaps) to minimize skin pad migration during pultrusion.

f. Use typical fabric width tolerances of +0.060 inch - 0.000 inch

g. Trim the upper flange of the J-stiffener after the panel has been pultruded.

PANEL DESIGN REQUIREMENTS

Loads	Axial	Shear	Pressure
	-22.0 kips/inches	1.9 kips/inches	-6.82 psi
	16.3 kips/inches	2.3 kips/inches	--
Stiffness	Shear Stiffness (Gt) =	0.68×10^6 pounds/inches	
Geometry	Rib Spacing =	40 inches	
	Stiffener Spacing =	6.6 inches	

Figure 5. Stiffened panel design criteria

After incorporating the PTI recommendations, the design contained 117 pieces of fabric with 6 different widths. The structural analyses were again performed using PASCO. For the combined loads of 22,000 pounds/inch compression, 1900 pounds/inch shear, 6.82 psi pressure acting inward and an assumed eccentricity (bowed inward) of 0.040 inches, the maximum axial strain was 4400μ in/in. For pure axial compression loading, the predicted panel buckling load was 37,650 pounds/inch. The panel weight was 0.0316 lb/in.^{2}, which represents a predicted weight savings of 26.5 percent compared to the aluminum baseline design.

PROCESS DEVELOPMENT

Pultrusion Technology, Inc. (PTI) was placed under contract to pultrude flat plates using ply orientations and materials specified by Lockheed. A 12-inch wide 20 ply plate (25% 0°/50% ±45°/25% 90° AS4 graphite ply orientation) was pultruded by PTI using the Lockheed-developed and selected resin system designated T3AO which uses a Dow Chemical Co. resin as the base epoxy. Previous Lockheed pultrusion tests with this resin indicated superior processing characteristics compared to Shell 9310. Two different types of graphite fiber fabric were used - knitted biased material from Xerkon and a 5-harness satin weave bidirectional fabric from Fiberite. The fabrics were hand slit to 12 inch widths. The 5-harness satin weave presented a problem in that the warp yarns nearest the slit edge were not held tightly and fell out of the fabric easily. During the pultrusion process, the loss of warp yarns caused an excess of resin to build up at the edges of the die. As a result resin cured and locked the fabric in the die after about 14 feet of plate had been pultruded.

The cured plate was sent to Lockheed for examination. Ultrasonic inspection indicated a highly porous part. This result was confirmed by photomicrographs of a typical section of the plate as shown in Figure 7. The causes of the porosity were deemed due to the unfilled die problem and improper wet-out of the fabric in the dip tank. These problems had to be

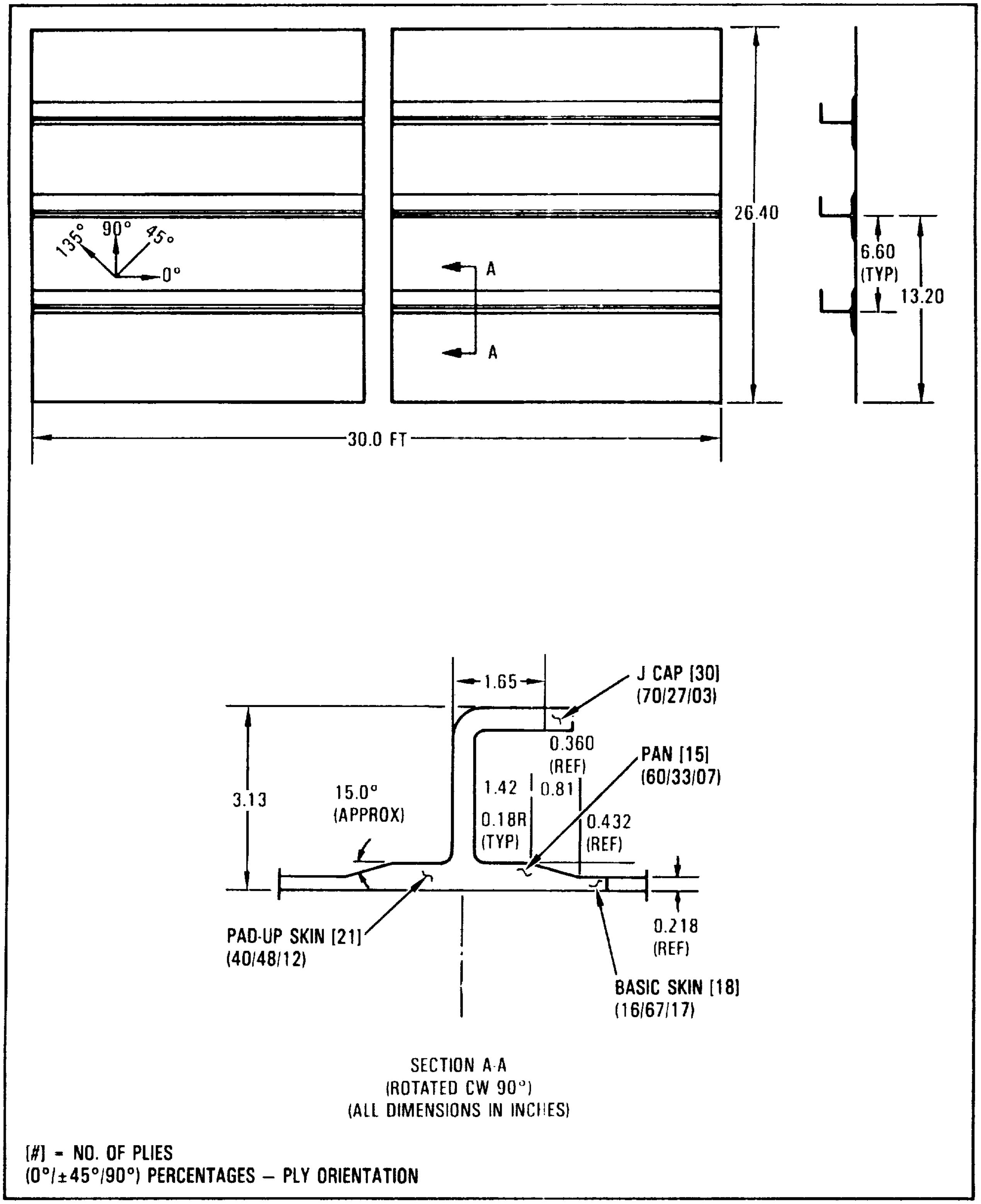


FIGURE 6. BASELINE DESIGN OF PULTRUDED PANEL

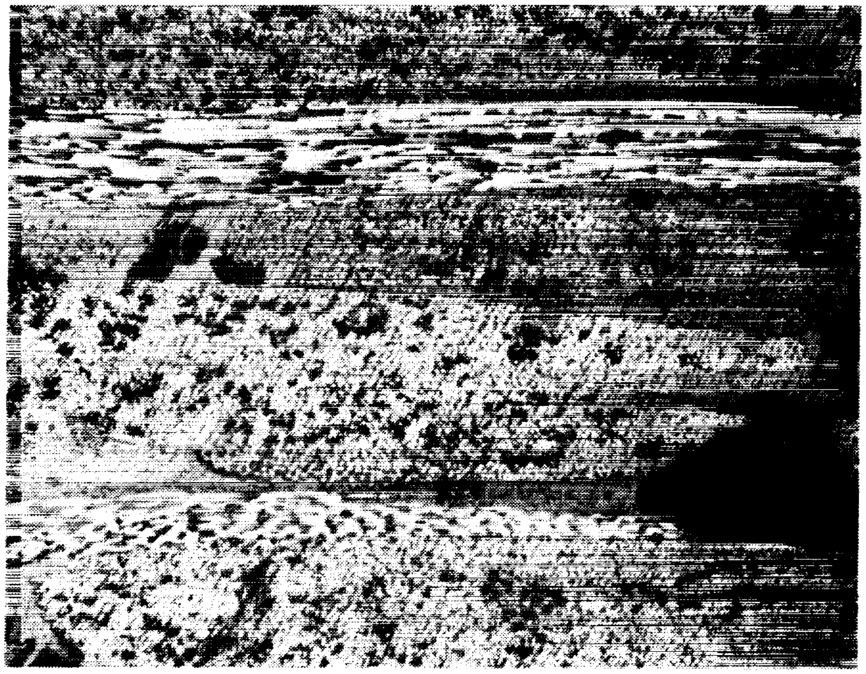


Figure 7. Micrograph of pultruded plate

resolved before another plate was pulled. Fabric was ordered woven to a 12-inch width with a leno edge (two twisted glass warp yarns) to hold the warp yarns in place. A larger dip tank was needed plus means to heat the resin to reduce its viscosity to ensure thorough wetting of the fabric.

Lockheed received its Pultrusion Technology, Inc. pultrusion machine at this time, therefore it was decided to continue the pultrusion of flat plates at Lockheed rather than at PTI.

After a larger resin dip tank and revised creeling system for the flat plate were completed, a second attempt was made at LASC using the PTI machine. As in the PTI process, the 20 plies of fabric were placed on a creeling frame and passed through a dip tank and then through the die. The pull was begun by pulling dry fabric through the die. A very high (1,000 pounds) pulling force was required. The cause appeared to be the oversized fabric. In an effort to avoid the apparent underfilled die condition at PTI, fabric had been ordered at 12 inch width with a tolerance of +1/8, -0 inches. The 5-harness satin weave material had a 12 inch width from the edges of the warp yarns. However, the fill yarns extended 1/4 inch beyond the warp yarns. Thus, additional fabric had to be pulled through the die. The biased fabrics measured 12-1/2 inches on the roll. Some decrease in the width was expected when the fabric was stretched during the pulling operation. To lower the dry pulling force, the fill yarn of the woven fabric was trimmed to be flush with the warp edge. The biased fabrics were then slit to 11-3/4 inches. Even with these modifications, the dry pulling force was larger than desired. The die opening was then increased from 0.250 to 0.289 inches with shims. Dry pulling force was then deemed acceptable.

After modifications to the fabric and die were completed, the run was begun by heating the die to operating temperature of 360°F to 370°F. While the die was heating, the wet-out tank was filled with T3A0 resin. The dip tank was designed to ensure thorough wetting of all the fabrics as seen in Figure 8. The fabric webs are brought in individually allowing the resin to contact both sides of the fabric. The fabrics are then brought together through two rollers, then separated; all within the dip tank. Each fabric was immersed for at least 4-1/2 feet of its length. The dip tank was heated to maintain 150°F, the temperature of minimum viscosity for the resin.

When the die reached temperature and the tank was full of resin, pulling began. The first 1 or 2 feet of fabric was pulled dry through the die, then material which had been in the dip tank entered the die. At a pulling speed of 12 inches per minute, the resin wet fabric took 3 minutes to emerge partially cured from the die. The first 4 feet of laminate seemed to be at a higher temperature than expected. A thermocouple was inserted between plies 10 and 11 and was pulled into the die. A maximum temperature of 440°F was measured. After 17 feet of plate had been pulled, the laminate jammed in the die and the top and bottom plies of biased fabric broke. Pulling was stopped and the die opened for examination. A large amount of cured resin was found in the center half of the die, both top and bottom, extending across the width.

An examination of the plate revealed the first 5 feet to be visually acceptable. Beyond this point, surface irregularities became visible. One side of the plate developed a depression about 1/2 inches from the edge. The surface of the plate developed a wavy character across the width. Near the point where the plate jammed in the die, the biased fabric was severely distorted on both the upper and lower surfaces. This distortion was severe enough that 45 degree fibers were bent to 90 degrees at several locations.

The 17-ft plate was postcured at 400°F for 4 hours then C-scanned. Numerous indications of delaminations and porosity were found, however, the first 5 feet appeared to be free of voids. Photomicrographs of both "good" and "bad" areas indicate problems throughout the laminate. Figure 9 shows a section of a "good" area while Figure 10 is from a "bad" area. Microcracking is apparent in both photos. Severe fabric distortion is seen on Figure 10.

Twenty tension and compression coupons were machined from the first 5 feet of the panel. The test results from these coupons are compared in Figure 11 with data from AS4/1806 prepreg material autoclave cured laminates and AS4/T3A0 laminates (hand layup and autoclave cured, respectively). The mechanical properties of the pultruded plate and hand laid-up laminates fabricated with AS4/T3A0 are inferior to those fabricated of AS4/1806. This is particularly true for compression loading. The facts that the hand laid-up laminate had a high resin content and that it is difficult to maintain fiber alignment when handling the dry fabric may account for part of the differences in compression strength and modulus. The pultruded plate had extensive microcracking which could be responsible for the reduced compression strength of the pultruded plate. However, the compression strength of pultruded unidirectional rods of AS4/T3A0 was also much lower than that measured on coupons manufactured with materials such as AS4/1806 or AS4/2220. This could indicate that the modulus of the T3A0 resin is too low to support the fibers. The tensile modulus of the neat resin has been measured at 380 ksi. Resins such as 3502 and 2220 have moduli of 530 ksi and 430 ksi, respectively.

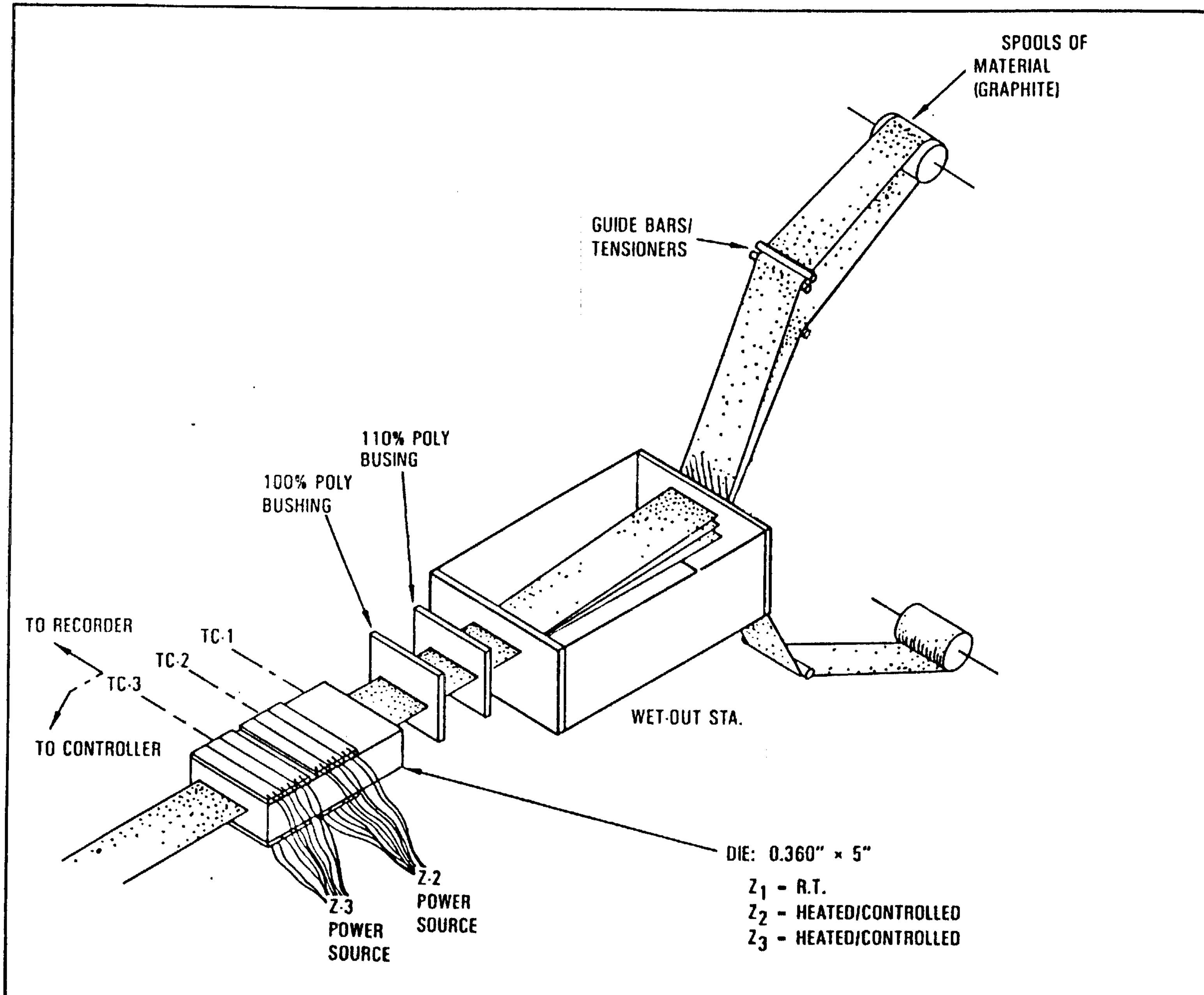


Figure 8. - Pultrusion set-up for 12 inch wide plate

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

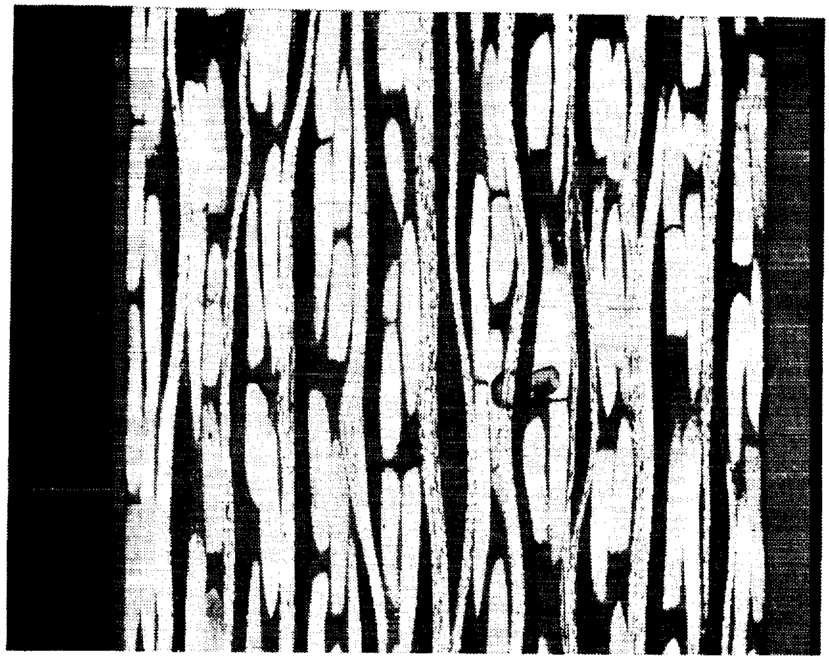


Figure 9. Photomicrograph of a "good" area of the plank

Figure 10. Photomicrograph of a "bad" area of the plank

		AS4/1806 BI-DIR. FABR.	AS4/T3A0 HAND LAYUP	AS4/T3A0 PULTRUSION
0° TENSION UNNOTCHED RTD	RESIN CONTENT (%) FAILURE LOAD (kip) FAILURE STRESS (ksi) FAILURE STRAIN (E-06 in/in) INITIAL MODULUS (msi)	36.90 12.52 83.45 12033 6.94	44.00 11.95 64.66 11934 5.61	35.80 30.28 73.64 12405 6.03
0° TENSION NOTCHED RTD	RESIN CONTENT (%) FAILURE LOAD (kip) FAILURE STRESS (ksi) FAILURE STRAIN (E-06 in/in) INITIAL MODULUS (msi)	36.90 7.07 47.03 7740 6.83	44.00 7.34 41.07 6874 6.04	35.80 17.27 41.87 6856 6.01
90° TENSION UNNOTCHED RTD	RESIN CONTENT (%) FAILURE LOAD (kip) FAILURE STRESS (ksi) FAILURE STRAIN (E-06 in/in) INITIAL MODULUS (msi)	N/A N/A N/A N/A N/A	44.00 12.51 67.27 12231 5.57	35.80 24.99 60.24 10993 5.53
90° TENSION NOTCHED RTD	RESIN CONTENT (%) FAILURE LOAD (kip) FAILURE STRESS (ksi) FAILURE STRAIN (E-06 in/in) INITIAL MODULUS (msi)	N/A N/A N/A N/A N/A	N/A N/A N/A N/A N/A	35.80 16.61 40.04 7485 5.39
0° COMPRES. UNNOTCHED RTD	RESIN CONTENT (%) FAILURE LOAD (kip) FAILURE STRESS (ksi) FAILURE STRAIN (E-06 in/in) INITIAL MODULUS (msi)	36.90 47.03 77.35 13671 6.15	N/A N/A N/A N/A N/A	35.80 46.60 56.04 12203 4.99
0° COMPRES. NOTCHED RTD	RESIN CONTENT (%) FAILURE LOAD (kip) FAILURE STRESS (ksi) FAILURE STRAIN (E-06 in/in) INITIAL MODULUS (msi)	36.90 28.93 47.55 7811 6.13	44.00 5.79 33.14 6951/7381* 4.47/4.49*	35.80 31.85 38.67 7644 5.10
0° COMPRES. NOTCHED 180 DEG, WET	RESIN CONTENT (%) FAILURE LOAD (kip) FAILURE STRESS (ksi) FAILURE STRAIN (E-06 in/in) INITIAL MODULUS (msi)	N/A N/A N/A N/A N/A	44.0 4.71 26.94 5090 5.11	35.80 27.12 32.89 5909 5.41
COMPRESSION 1000 in-lb/in IMPACT RTD	RESIN CONTENT (%) FAILURE LOAD (kip) FAILURE STRESS (ksi) FAILURE STRAIN (E-06 in/in) INITIAL MODULUS (msi)	36.90 39.40 39.01 6202 6.22	N/A N/A N/A N/A N/A	35.80 42.08 30.49 5662 5.28

*AVERAGING EXTENSOMETER/DSST

Figure 11. - Comparison of AS4/T3A0 fabric pultrusion and hand layup, and AS4/1806 bi-directional fabric test data

As a result of the tests conducted on the T3A0 resin, Shell 9310 epoxy was selected for the pultrusion of the J-stiffened panel. Pultrusion processing data were available for this resin and the mechanical properties of composites made with this resin were acceptable.

STIFFENED PANEL PULTRUSION

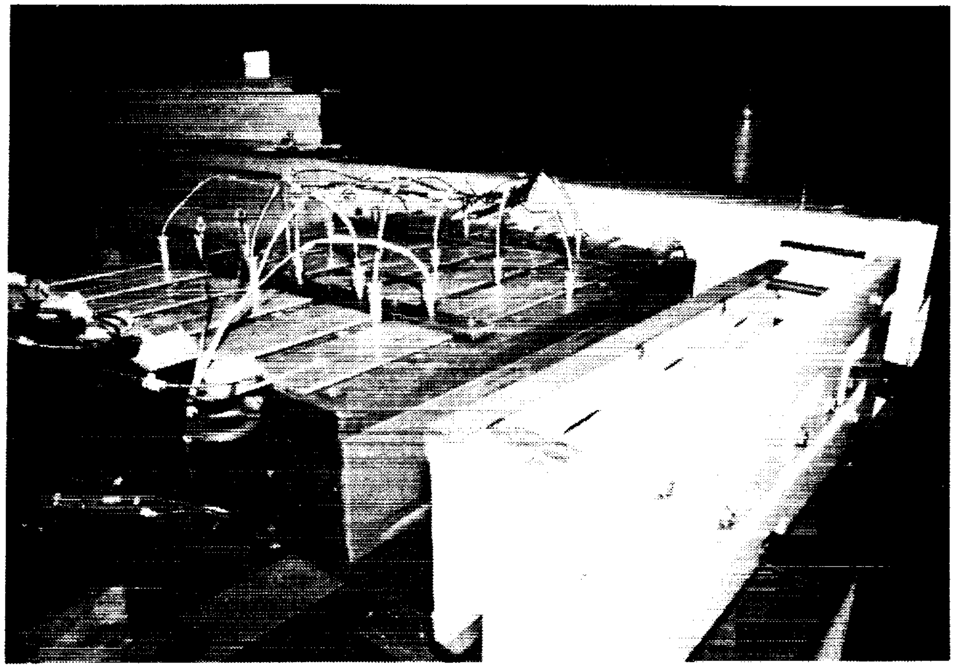
Progressive Tool and Die, Inc., Gardena, California was issued a contract to design and fabricate a curing die for pultruding the three bladed J-Stiffened panel. Progressive Tool and Die designed and fabricated a 24-inch long die made of 4130 steel with commercial grade chrome plating on the pultrusion surfaces. The die, as shown in Figure 12, is configured to the net nominal dimensions of the J-stiffened panel design. Teflon guide plates were provided at the entrance side of the die to gather and feed the fabric material layers into the die.

A contract was made with Goldsworthy Engineering, Inc. to pultrude the J-stiffened panel. The pultrusion die was shipped to Goldsworthy. Goldsworthy shortened the die to reduce pulling loads and reviewed the Lockheed drawing of the pultrusion J-panel. They recommended removal of the outer ±45 and ±45 plies from the panel to aid in pulling the part. Also, they recommended removal of two 0 degree plies from each stiffener. Lockheed agreed to their recommended design modifications.

The AS4 graphite fabric was sent to Hi-Tech Composites of Reno, Nevada to be stitched into ply sets. By combining the plies into the stitched groups of plies, ply fabric handling problems could be reduced. In order to impregnate the ply sets with resin, Goldsworthy constructed pressurized wet-out stations.

A test of the resin system/impregnation system was completed at Goldsworthy. Five-inch wide 50-foot long plate was pultruded. Figure 13 shows the pressurized impregnation system and Figure 14 is an overall view of the pultrusion process. The 50 foot long plate was cut into 5 foot pieces and post-cured at Goldsworthy. The parts were then sent to Lockheed for ultrasonic inspection which revealed numerous indications of porosity. Previous experience at Lockheed with a similar pultrusion suggested that improper wetting of the fabric, particularly the knitted 0/90 degree fabric, was the cause of the porosity.

A tool try panel was pultruded using fiberglass fabric to assess the operation of the folding tools, wet-out system, and curing die. Two days were spent "stringing" the die which consisted of pulling the individual pieces of fabric through the folding tools, the wet-out vessel, and finally the curing die. The fabric was tied to a pulling bar seen in Figure 15 which was connected to the start-up chain of the pultruder. Because of the short length of the anticipated pull (less than 50 feet), the clamping mechanism was not employed. Figure 16 shows some of the glass fabric used in the tool try on the creeling racks. This fabric, available from a previous LASC pultrusion program, was slit to the same width, as the graphite fabric to be used in the final part, but was not the same thickness. As a result, the volume of fiberglass fabric was less than had been used in the design of the wet-out vessel and curing die. The resin was polyester, selected for its low cost and ease of pultrusion.



a. Inlet end of die

b. Exit end of die

Figure 12.- Curing Die for "J" Stiffened Panel

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

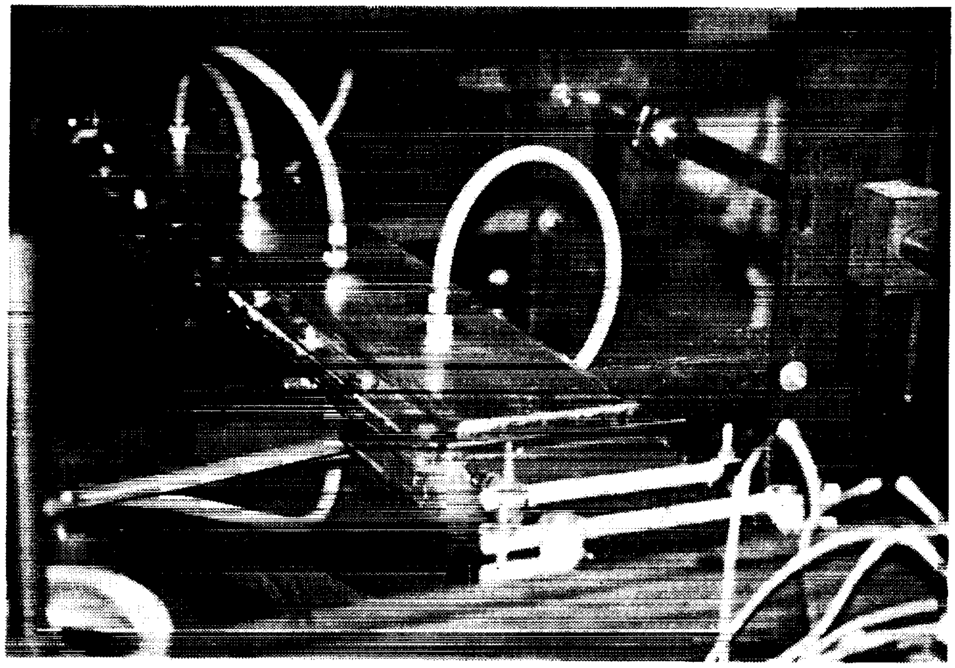


Figure 13. - Pressurized impregnation system

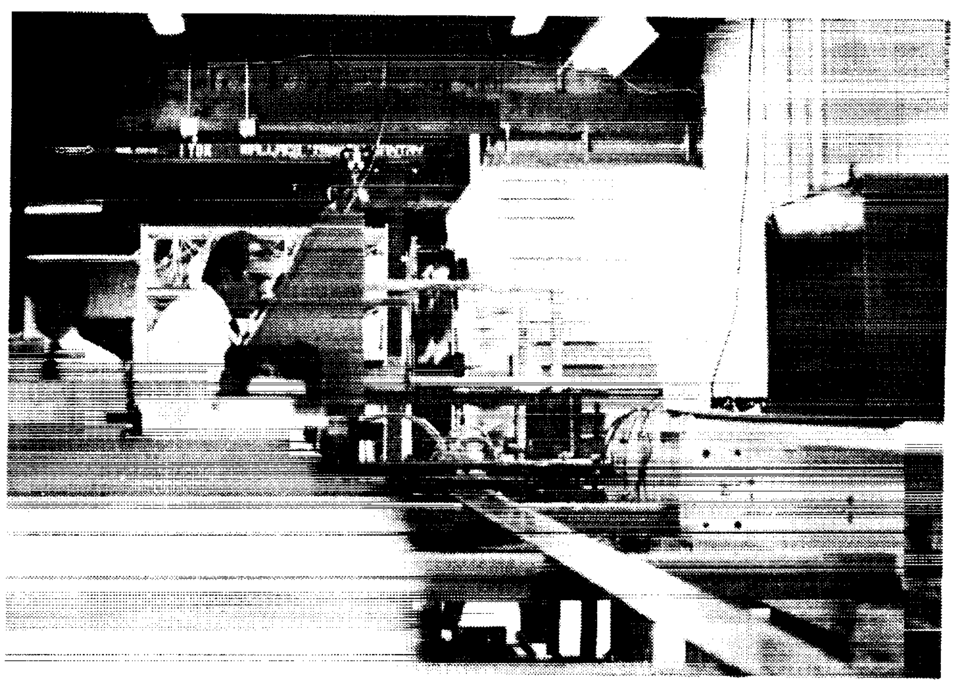


Figure 14. - Pultrusion of stitched graphite fabrics

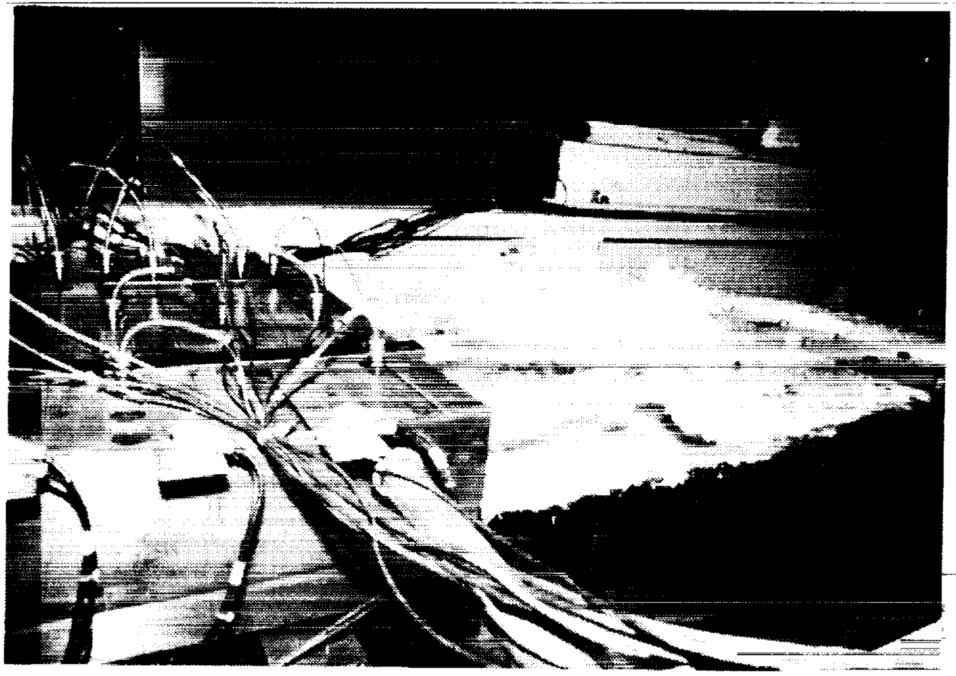


Figure 15. - Attachment of the glass fabric to the pulling bar after being strung through the curing die

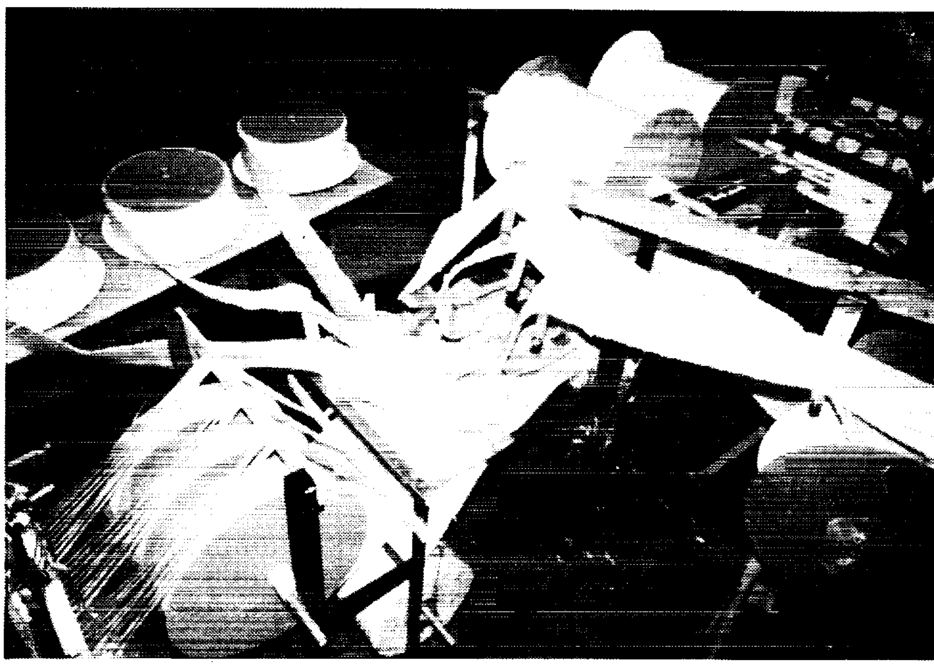


Figure 16. - Glass fabric on the creeling racks. The fiberglass folding tools used to form the J-stiffeners can be seen in the center of the photograph.

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

The tool try pultrusion proceeded very smoothly except for one major problem. No pressure could be developed in the wet-out vessel due to the reduced volume of glass used. Resin flowed freely from the entrance and exit of the wet-out vessel and due to the lack of pressure, the fabric was not thoroughly wetted. A cross-section of the part revealed a shell of composite surrounding dry fiberglass fabric.

The tool try demonstrated that the folding tools and creeling mechanism worked well and ensured that the pultruder was operating properly. The problem with the wet-out system was corrected prior to the pultrusion of the graphite/epoxy part. Net sized steel bushings were machined from a remnant of the curing die and were attached to the entrance and exit of the wet-out vessel. The bushings prevented resin leakage and allowed pressure to be developed.

The creeling system was modified to hold the graphite fabric rolls and the system made ready to pultrude. Because of the wet-out problems experienced with wetting of the glass fabric, two quality assurance tests were incorporated in the graphite/epoxy pultrusion. Plugs were to be drilled from the composite using a small hole saw as the plank exited the die and an ultrasonic A-scan was also performed as the composite exited the die. Insufficient wetting could be corrected either by increasing the wet-out pressure or by slowing the pultrusion.

The graphite was creeled and dry fabric was pulled through the die as shown in Figure 17. The fabric was tied to a start-up bar. Before pulling began, the wet-out vessel was pressurized to 20 psi. Numerous leaks occurred but pressure could be maintained. Pulling started at about 1.5 inches/minute. after approximately 1 foot of composite was pulled through the die, a plug was drilled from the laminate. Three more plugs were drilled in other sections of the laminate. All plugs indicated thorough wetting of the fabric. Ultrasonic inspection (Figure 18) revealed some porosity, however, a rough surface finish made this inspection difficult. Pultrusion continued at 1.5 inches/minute using a pulling force of about 6000 pounds. Sloughing was observed on almost all surfaces of the plank, caused by insufficient fabric, resulting in excess resin on the surface (Figure 19). After approximately 10 feet of plank had been pulled, severe distortion of one outer J-stiffener occurred. The distortion was caused by resin curing on the die surface creating a ball. The pull was stopped for a few minutes to allow the ball to adhere to the composite which upon re-start of the pull would then be pulled from the die. However, the machine could not generate sufficient force to re-start. Thus the run ended with about 13 feet of plank having been pultruded.

When the die was opened, a jam was found in the central J-stiffener. The jam was created by tows of graphite fabric which were added to the tip of the J-stiffener cap or flange to provide additional tool filling. The tows had wandered into a die seam. This jam was probably the cause of the stoppage rather than the suspected resin ball on the outer stiffener.

The pultrusion run produced 10-1/2 feet of pultruded J-stiffened panel (shown in Figure 20). Ultrasonic inspection revealed numerous indications of porosity. These indications were confirmed by photomicrographs.

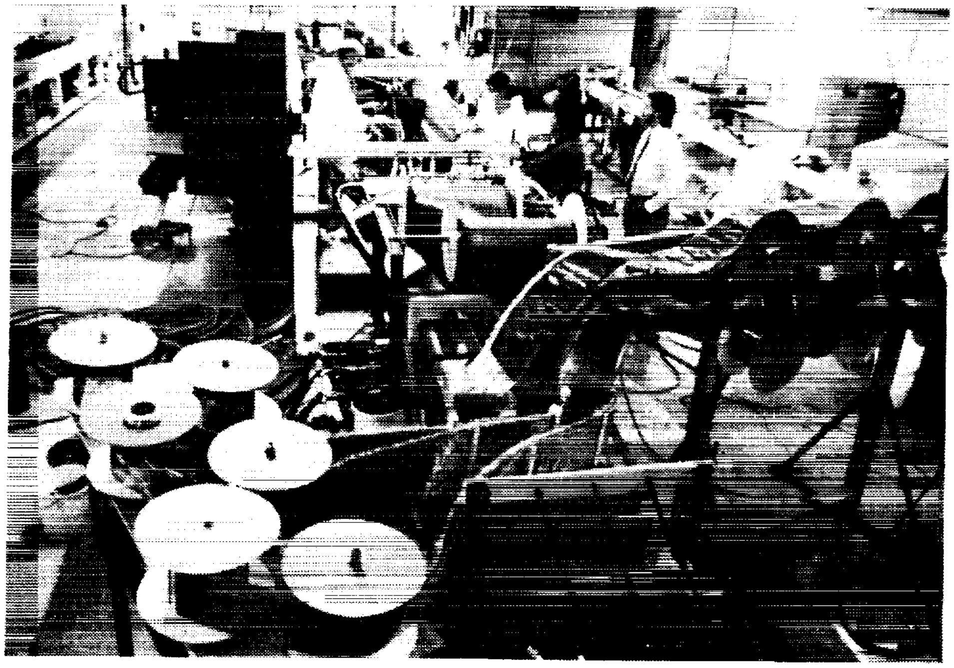


Figure 17. Graphite fabric creeling arrangement and folding tools

Figure 18. Ultrasonic A-scan of pultruded plank

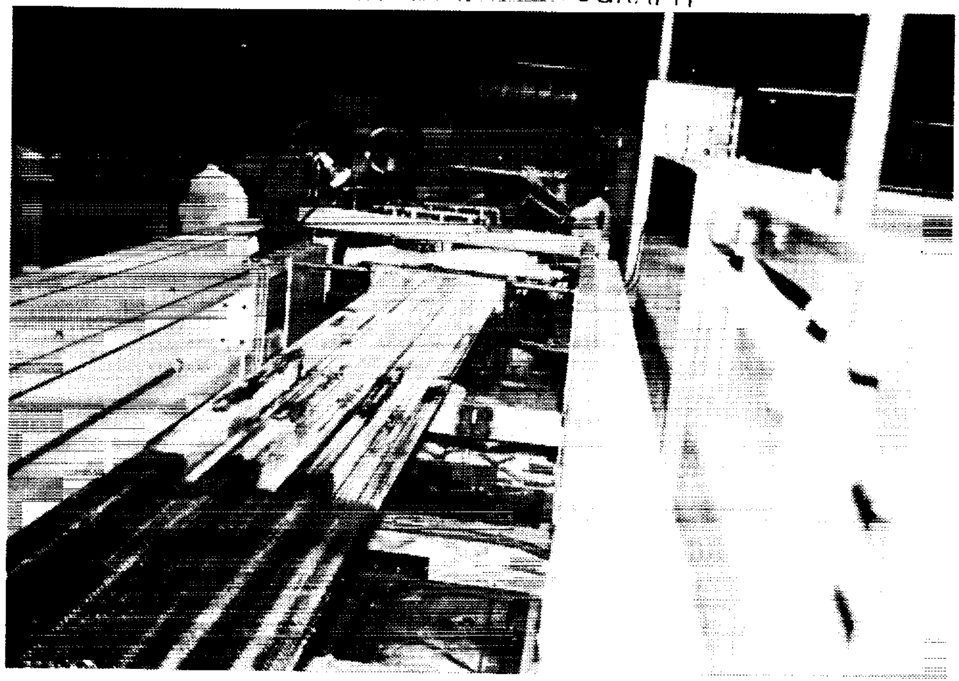


Figure 19. Pultruded plank showing sloughing (white resin) condition. The black area in the foreground is fabric used for start-up. The dark areas show where A-scans were made

Figures 21 and 22 show typical porosity found in the panel. Most of the voids occur between tows but these are indications of porosity in some of the tows. Resin content measurements were made in the skin and blade areas. The skin areas had resin contents of 36 to 39 percent while the blades were between 30 and 32 percent. This result suggests that additional plies should be included in the skin.

The upper flanges of the stiffener were machined to the correct width; and one 78-inch long section was shipped to NASA Langley Research Center for testing.

As a result of the run, several modifications were identified to be made prior to continuing the program. The wet-out vessel required redesign and fabrication to alleviate leaking. Proper filling of the die was required to eliminate sloughing. The major question to be answered prior to pultruding another J-panel was how to eliminate the porosity. Goldsworthy Engineering stated that an increased fiber loading might significantly decrease porosity; also, allowing the mixed resin to stand overnight to allow removal of entrained air might help.

The design for the pultruded J-stiffened panel was modified to allow the use of all knit fabrics rather than a combination of woven and knit fabrics. Thicknesses and percentages of 0 degree, ± 45 degree, and 90 degree material remained the same as the original design. Goldsworthy ordered the knit fabrics from Hexcel/Hi Tech. Goldsworthy also made the modifications to the pressurized wet-out tank and installed it on the pultrusion machine.

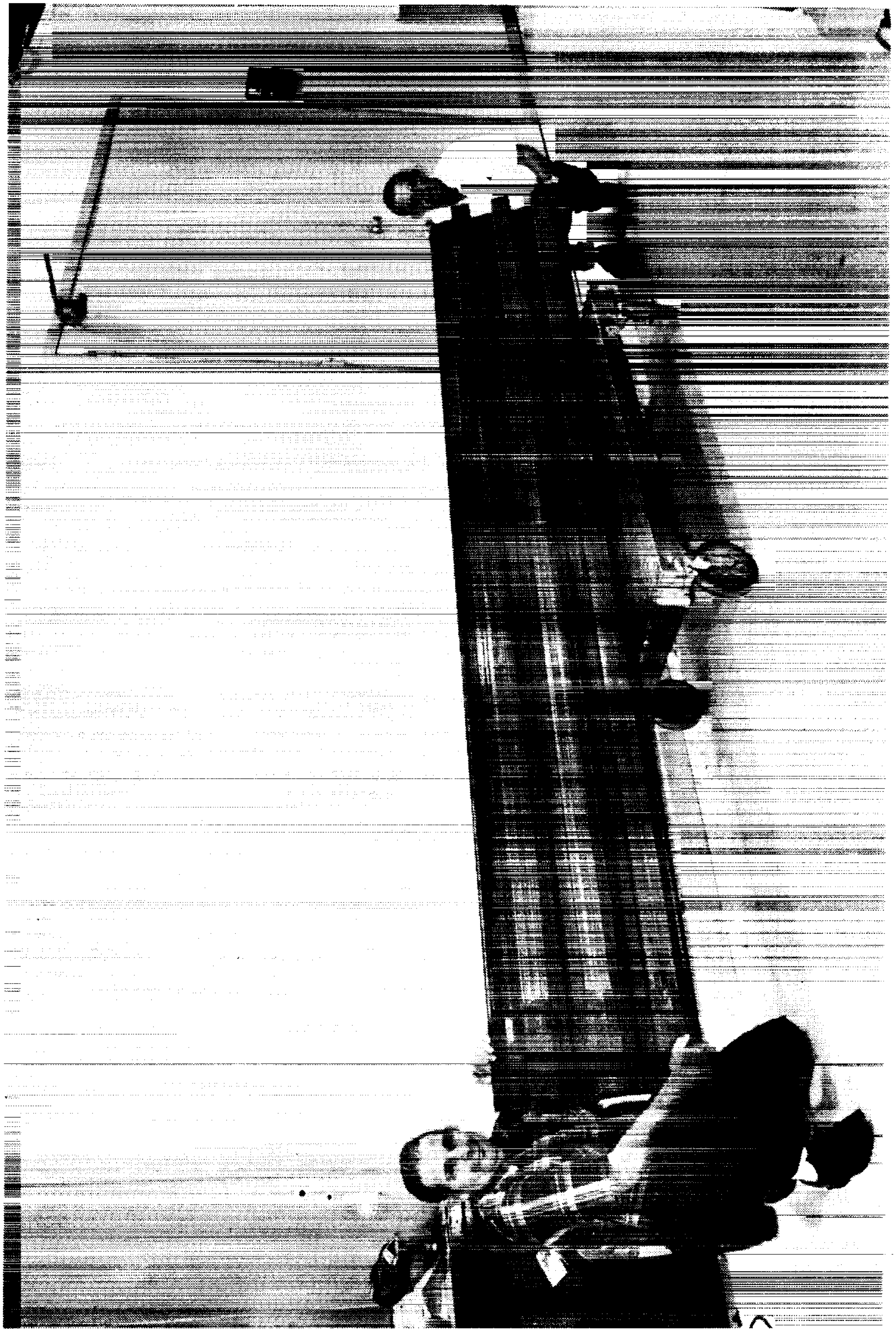


Figure 20. - Pultruded J-stiffened panel (1986)

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

Figure 21. - Photomicrograph through skin area directly under a stiffener

Figure 22. - Photomicrograph through a stiffener

Goldsworthy Engineering was ready to pultrude the second J-stiffened panel when all of the Hexcel/Hi Tech manufactured fabric had been pulled through the folding horns, the wet out vessel, the curing die, and tied to the puller head. These fabrics were a significant improvement over those previously used. Fewer and narrower gaps were seen in the material. The stitching used to incorporate several fabric layers into a ply set was much improved.

The Shell 9310 resin for the pultrusion was mixed the day before, except for the catalyst in an attempt to reduce the amount of air mixed into the resin. The catalyst was added just before it was pumped into the wet out vessel.

The curing die was heated to its curing temperature of 350°F. The resin was then pumped into the wet out vessel. Almost immediately, resin began to leak around the fabric both toward the folding horns and toward the curing die. Pressure in the vessel was limited to 10 psi or less. The leaks suggested that the fabrics were less than the proper thickness. Furthermore, the lack of pressure in the wet out vessel could lead to insufficient wetting of the fabric. A core sample was taken from a cured piece of the pultrusion as it exited the die. Results were inconclusive, but the core's appearance was not significantly different from cores taken from the previous pultruded panel. Ultrasonic inspection via hand scans of the pultrusion showed the part to be worse than the previously pultruded part. A second ultrasonic inspection system was provided by Amdata, Inc. of Milpitas, California. This computerized scanning technique confirmed the results of the hand scan. With these results indicating problems, the pultrusion effort was terminated.

Following clean-up of the wet out vessel and the curing die, Goldsworthy began an investigation of the fabric. The ply sets were compressed in a press and their thickness was measured. The ply sets were found to be about 20 percent undersized. To correct these problems, Goldsworthy suggested doubling the thickness of the pan plies and adding one additional ply set to the skin. An attempt was made to pultrude this configuration, but during start up the bolts holding the fabric to the pulling head broke. The dry pulling forces, that is, the friction generated by pulling the fabric through the die, were too great.

Preparations were made for another pultrusion. This time only one set of pan plies was used between stiffeners, but the additional skin ply was retained. Also, stronger bolts were used to attach the fabric to the pulling head. During start-up the pulling forces remained low, about one-fifth of the force measured before breaking of the bolts in the previous attempt. Pressure in the wet out vessel of 17 psi was quickly achieved and maintained throughout the run. Twenty-three feet of panel were pultruded. Pulling forces near 7000 pounds were measured. External appearance was good, however, no on-line ultrasonic inspection was attempted. The pultruded panel was shipped to LASC for post curing, inspection and testing.

The 23 foot pultruded panel, shown in Figure 23, was received by LASC and was postcured for two hours at 350°F. Following the postcure, the panel was inspected on a large gantry robot to determine the flatness of the panel. Results are shown in Figures 24, 25, and 26. The panel exhibited the same warping observed in previous panels whether autoclave cured or pultruded. Three pieces were cut from the panel for ultrasonic inspection and tag end

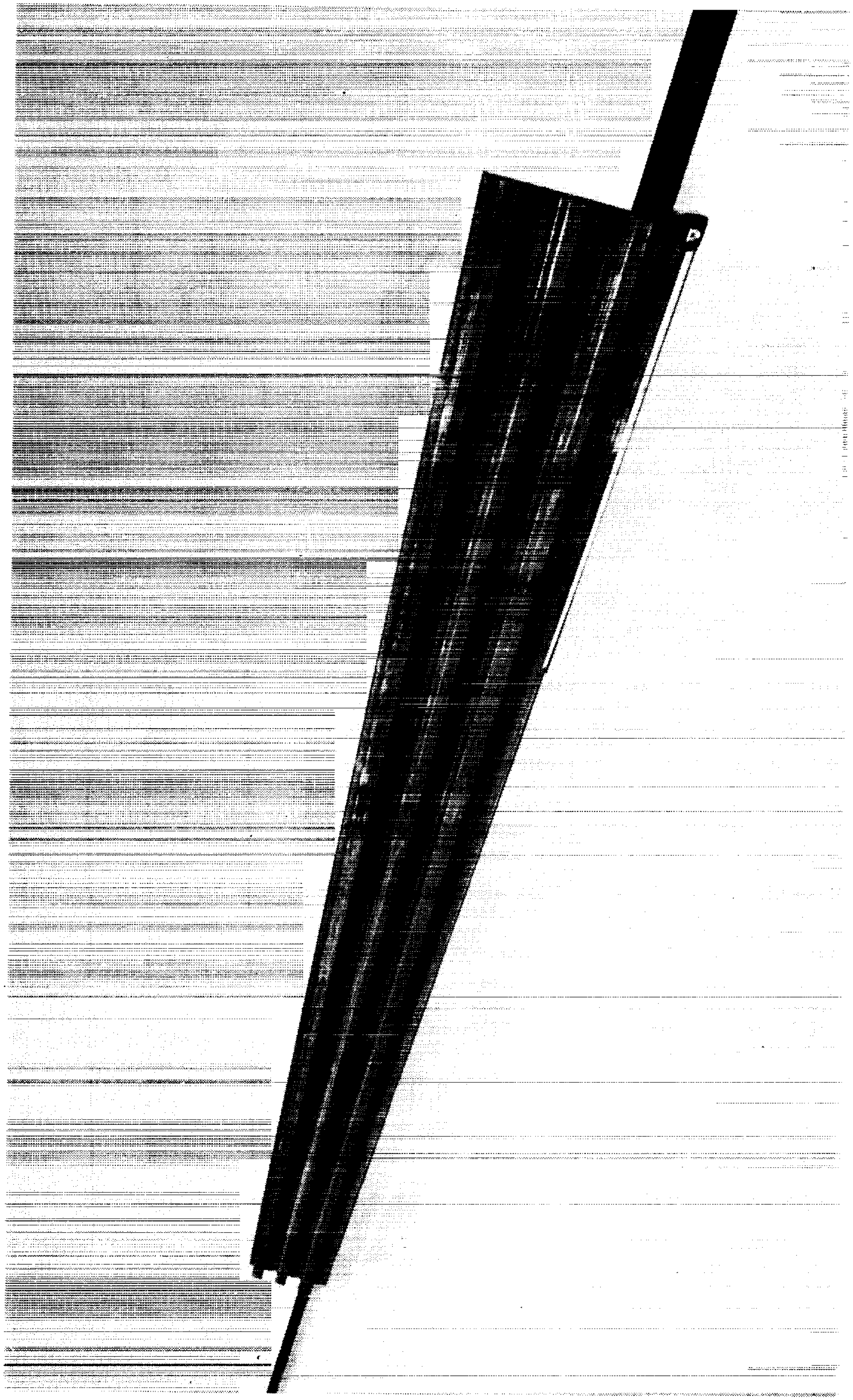


Figure 23. - Twenty-three foot long pultruded panel

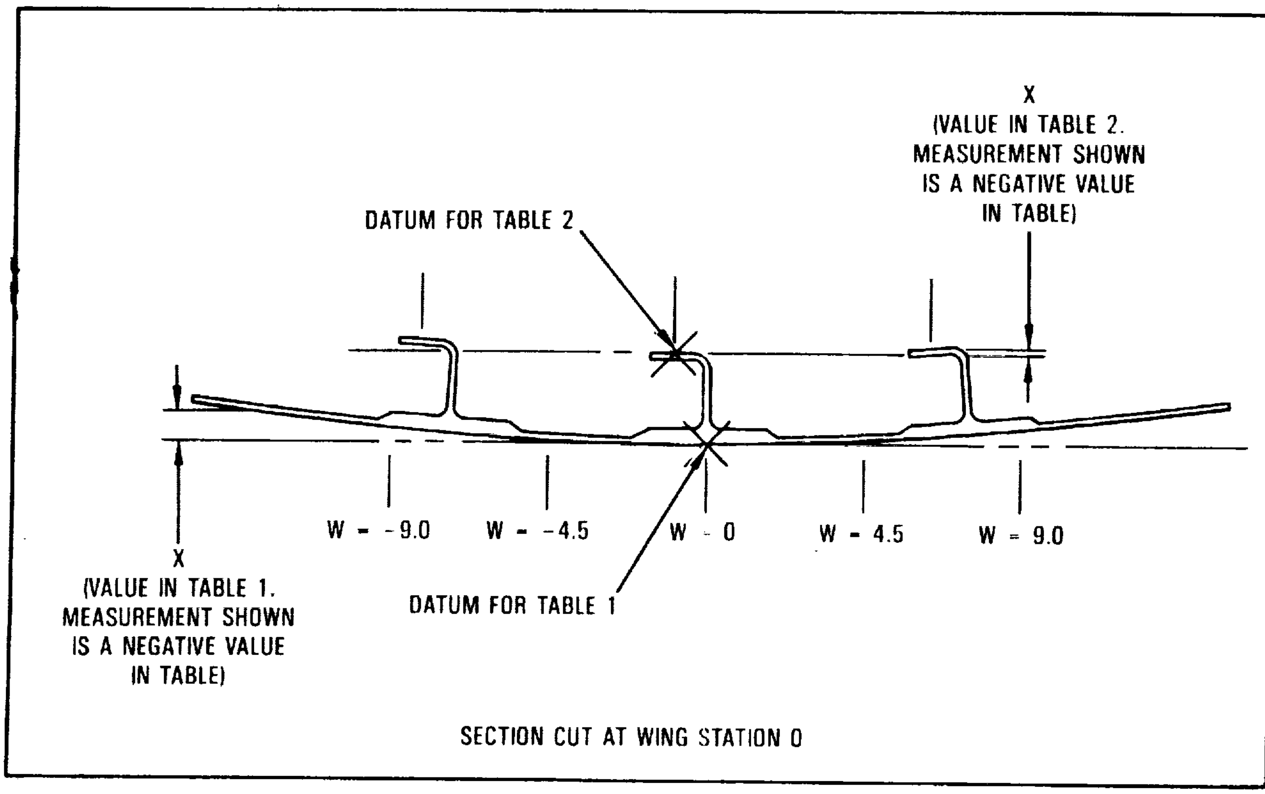


Figure 24. - Key to flatness data in figures 27 and 28

LENGTH/WIDTH	-9.0"	-4.5"	0	+4.5"	+9.0"
-120	0.040	0.0135	0.188	0.188	0.154
-108	0.001	0.090	0.138	0.136	0.103
-96	-0.032	0.055	0.098	0.094	0.053
-84	0.054	0.027	0.064	0.054	0.012
-72	-0.072	0.008	0.041	0.025	-0.023
-60	-0.083	-0.009	0.022	0.003	-0.048
-48	-0.086	-0.019	0.009	-0.012	-0.067
-36	-0.090	-0.026	0.000	-0.022	-0.078
-24	-0.092	-0.028	-0.003	-0.027	-0.083
-12	-0.092	-0.028	-0.005	-0.028	-0.087
0	-0.092	-0.028	0.000	-0.027	-0.088
12	-0.088	-0.022	0.006	-0.022	-0.084
24	-0.083	-0.012	-0.016	-0.015	-0.080
36	-0.075	-0.007	0.022	-0.010	-0.075
48	-0.075	-0.018	0.006	-0.023	-0.078
60	-0.077	-0.022	-0.002	-0.033	-0.087
72	-0.072	-0.016	0.006	-0.028	-0.086
84	-0.052	0.006	0.018	-0.008	-0.068
96	-0.020	0.037	0.057	0.006	-0.060
108	-0.130	0.006	0.070	0.070	0.030

Figure 25. - Pultruded plank flatness - skin side

LENGTH/WIDTH	LEFT J	CENTER J	RIGHT J
-108	-0.108	-0.125	-0.054
-96	0.060	-0.007	-0.004
-84	-0.018	-0.018	0.034
-72	0.013	0.000	0.062
-60	0.034	0.018	0.080
-48	0.047	0.032	0.092
-36	0.055	0.032	0.097
-24	0.046	0.024	0.093
-12	0.046	0.012	0.083
0	0.042	•0.000•	0.070
12	0.032	-0.010	0.058
24	0.012	0.008	0.040
36	-0.006	-0.007	0.014
48	-0.017	-0.047	0.006
60	-0.022	-0.039	-0.006
72	0.003	-0.040	-0.027
84	-0.017	-0.066	-0.057
96	-0.058	-0.106	-0.100
108	-0.096	-0.162	-0.152

Figure 26. Pultruded plank flatness - stiffener side

testing. Ultrasonic inspection revealed numerous porous areas. This result was confirmed by photomicrograph. Figures 27 through 27E show typical areas of the panel. Unwetted fiber could be seen as well as voids within the resin itself. Tag end tests on the panel were made. Upon completion of the inspection, a 10 foot section was shipped to NASA for structural testing. Results of the NASA testing are presented later in this report.

To compare the quality of the pultruded panel fabricated in 1987 to that panel made in 1986, photomicrographs of similar areas were taken at the same magnification. These photomicrographs were then digitized using an Automatix computer vision system. The hardware consisted of a solid state video camera, a frame buffer having 340 x 512 pixel resolution with 64 gray levels per pixel, and a host computer. The camera was focused such that the photograph filled the entire frame. The resolution, then, would be 1/512 of the photograph's longer dimension, or 1/340 of its shorter dimension; this corresponds to the smallest void distinguishable.

The Automatix system software was used to enhance the contrast of the image and set a threshold for the purpose of distinguishing voids: areas darker than the set threshold were considered as voids. This threshold was set individually for each image. The system flashes points at or below the threshold, which can then be adjusted to obtain best agreement of flashing points with actual voids while minimizing false selection of non-void areas. Only those points in the photograph which included the laminate material were considered in this selection process. The area of the photo considered voids was divided by the total area to yield a percent void.

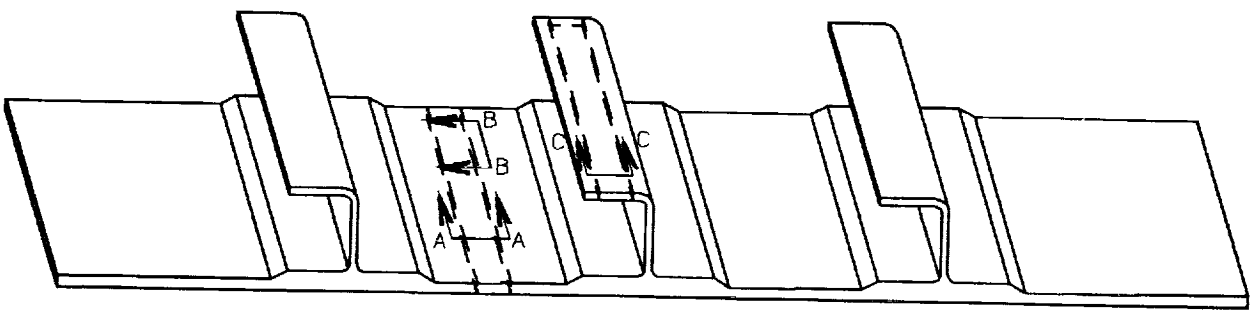


Figure 27. Key to photomicrograph locations

Figure 27A. Photomicrograph A-A (25X)

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

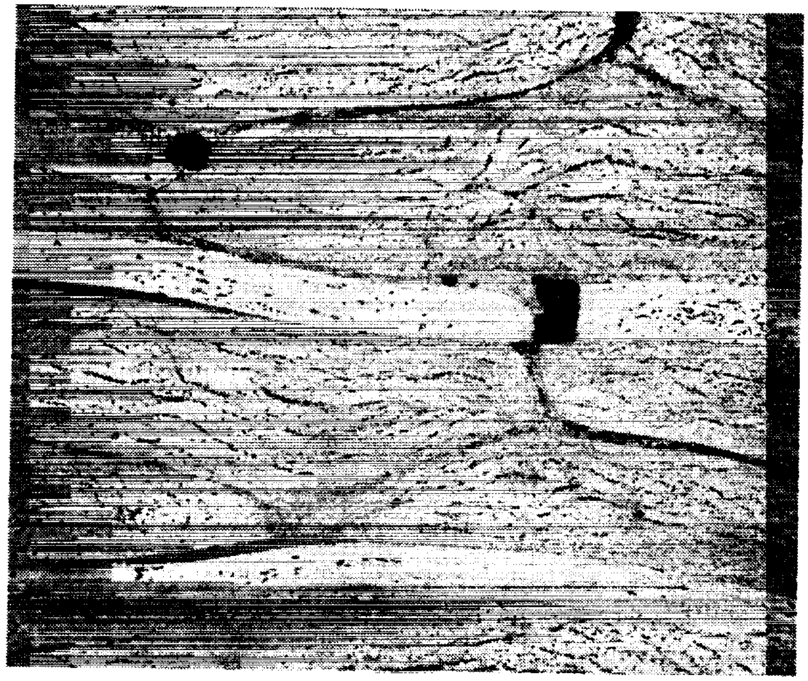


Figure 27B. Photomicrograph C-C (25X)

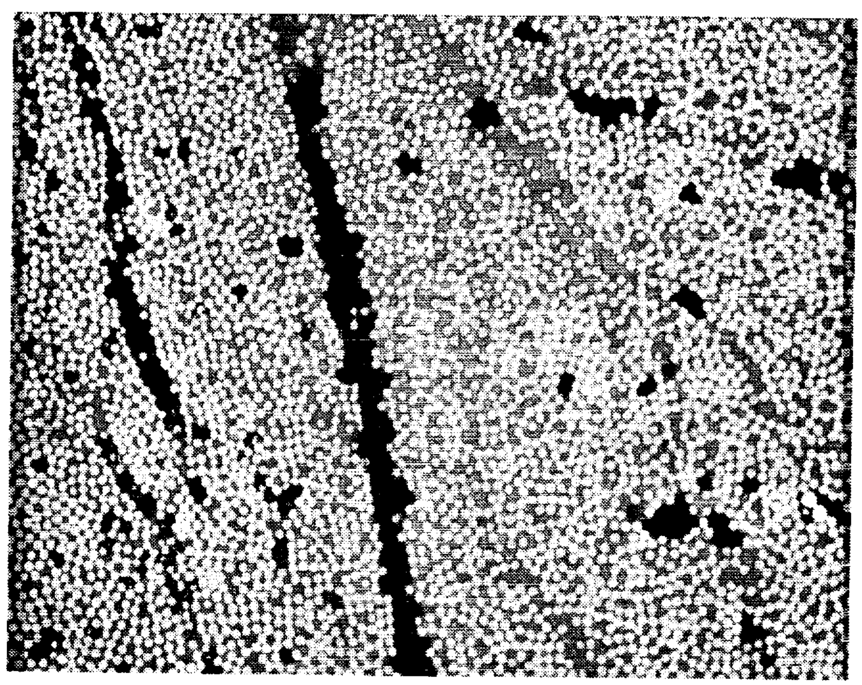


Figure 27C. Photomicrograph C-C (200X)

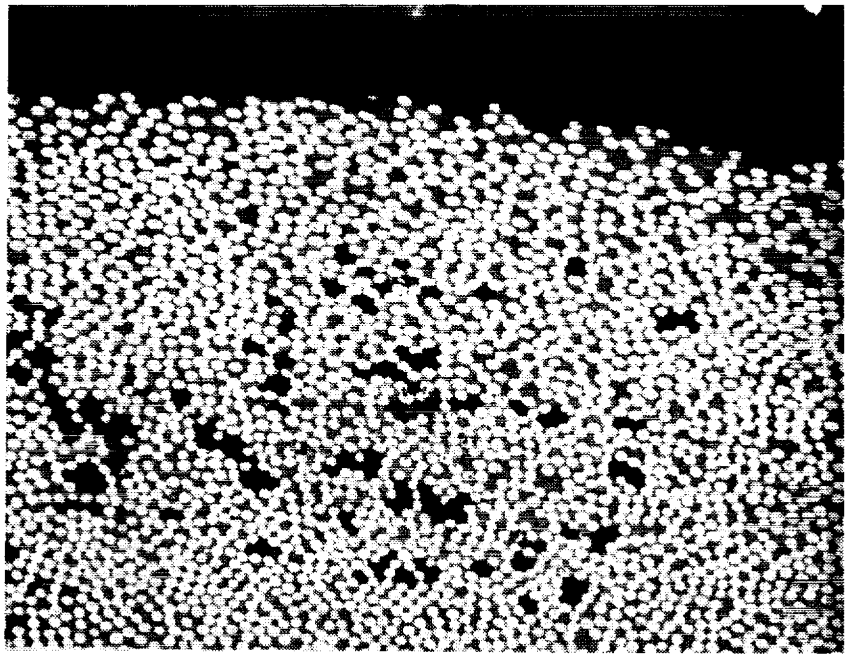


Figure 27D. Photomicrograph A-A (200X)

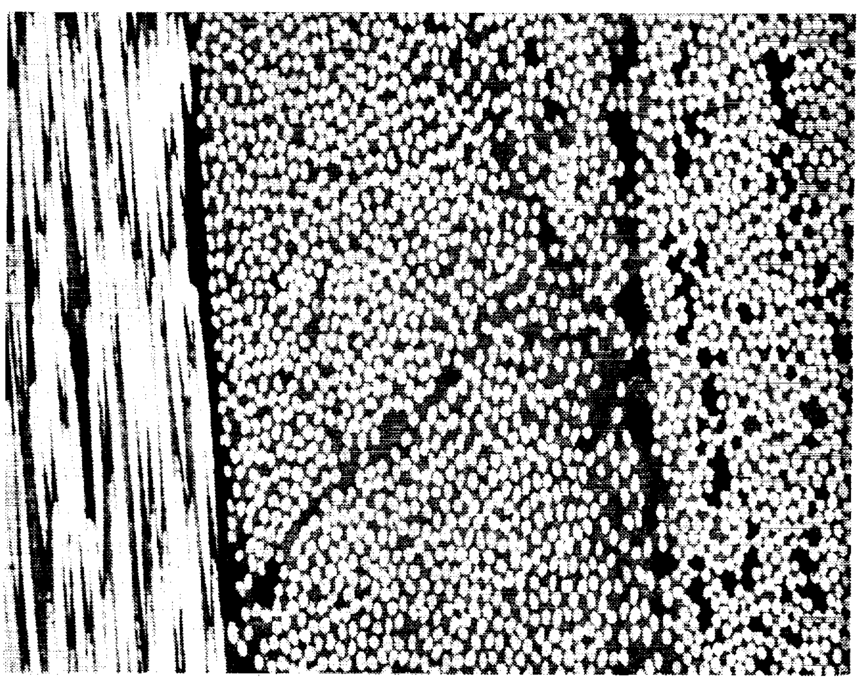


Figure 27E. Photomicrograph B-B (200X)

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

[70]M0925.AA0

Preliminary measurements showed the first 1986 panel to have 1.5 percent voids in the skin section compared to 0.75 percent voids in the 1987 panel. Measurements at the base of the stringer indicated 2.0 percent in the 1986 panel and 1.9 percent in the 1987 panel. However, the resolution of the camera may exclude a significant void area. A higher resolution system was sought to better measure the void content.

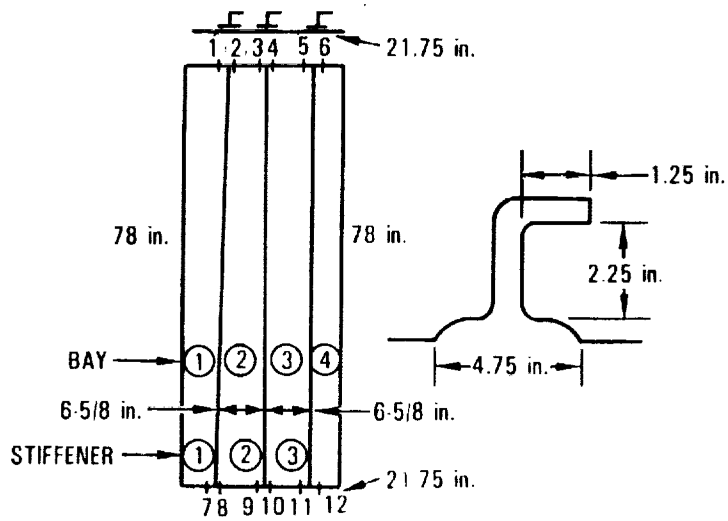
The photomicrographs were then analyzed on a second imaging system the Dapple Image Analysis System which uses an Apple Computer based system with 256 x 256 pixel resolution. This was less than the resolution from the previous system used. Results from the Dapple system indicated an average void area of 8.01 percent in the 1986 pultruded panel and a void area of 4.96 percent in the 1987 panel. These values are much higher than those reported previously; however, the comparative levels (8:5) were about the same. The differences were largely operator based. The operator of either imaging systems selected a gray scale level below which was considered good and above which was considered void.

PULTRUDED J-STIFFENED PANEL STRUCTURAL TESTS

Measurements were made on a 78 inch section of the 1986 panel shown in Figure 22 and are listed in Figure 28. From the J-stiffened panel pultruded in 1986, three axial compression panel specimens (1, 2 and 3) were machined; and from the panel pultruded in 1987, specimens 4, 5 and 6 were obtained. The loaded ends of each specimen were potted as shown in Figure 29 and then ground flat, square and parallel.

Each of the panel specimens was extensively instrumented with strain gages. The 42 gages were installed on panels 1, 2, 5, and 6. To simulate panel-to-rib cap attachment, 0.25 inch diameter holes were drilled in the skin and stiffeners of specimens 3 and 4. All the holes were located along the lateral centerline of the specimens. Specimens 3 and 4 were more extensively strain gaged than the specimens without holes. Direct current differential transducers (DCDT) were used to measure specimen load shortening and lateral displacements during testing. Prior to testing, specimens 1 and 6 were impacted. The impactor was a 0.5 inch diameter aluminum sphere fired from an air gun. Specimen number 1 was impacted at 534.7 feet/sec (equivalent to 29.4 feet-pounds) and specimen number 6 was impacted 516.5 feet/sec (27.4 feet-pounds). Figure 30 shows panel specimen 4 after failure. The pultruded test panel results are summarized in Figure 31. Despite the process improvements incorporated in the 1987 pultrusion, the test results were similar to those obtained in 1986. Both sets of panel specimens contained significant regions of porosity and voids which severely degraded structural efficiency, particularly in specimens with holes or impact damage. NASA CR 4177 reports data for J-stiffened panels fabricated by conventional means using AS4/1806 material. After impact, the AS4/1806 panel carried a compression load of 524.9 kips, about 100 kips more than the impact damaged AS4/9310 pultruded panels.

VIEW FROM STIFFENER SIDE



I. **STIFFENER CAP THICKNESS: (INCHES) AT VARIOUS LOCATIONS**

STIFFENER NO. 1: 0.3544, 0.3521, 0.3465, 0.3465, 0.3361, 0.3387, 0.3375
STIFFENER NO. 2: 0.3472, 0.3412, 0.3381, 0.3366, 0.3337, 0.3373, 0.3358
STIFFENER NO. 3: 0.3451, 0.3407, 0.3344, 0.3322, 0.3320, 0.3326, 0.3341

II. **WEB THICKNESS: (INCHES)**

STIFFENER NO. 1: 0.3579, 0.3387
STIFFENER NO. 2: 0.3489, 0.3434
STIFFENER NO. 3: 0.3582, 0.3477

III. SKIN THICKNESS (INCHES)

BAY NO. 1: 2184", 0.2166, 0.2154, 0.2125, 0.2052, 0.2069, 0.2138
BAY NO. 2: 2300, 0.2176
BAY NO. 3: 0.2302, 0.2133
BAY NO. 4: 0.2202, 0.2211, 0.2197, 0.2210, 0.2231, 0.2212, 0.2216

J-STIFFENED PANEL (CALAC) PULTRUDED (3 STIFFENERS)

COMBINED SKIN/ATTACHMENT FLANGE THICKNESS

1. 0.4384	7. 0.4278
2. 0.4442	8. 0.4344
3. 0.4411	9. 0.4297
4. 0.4462	10. 0.4327
5. 0.4437	11. 0.4250
6. 0.4359	12. 0.422[illegible]

FIGURE 28 THICKNESS MEASUREMENTS ON STIFFENED PANEL PULTRUDED IS 1986

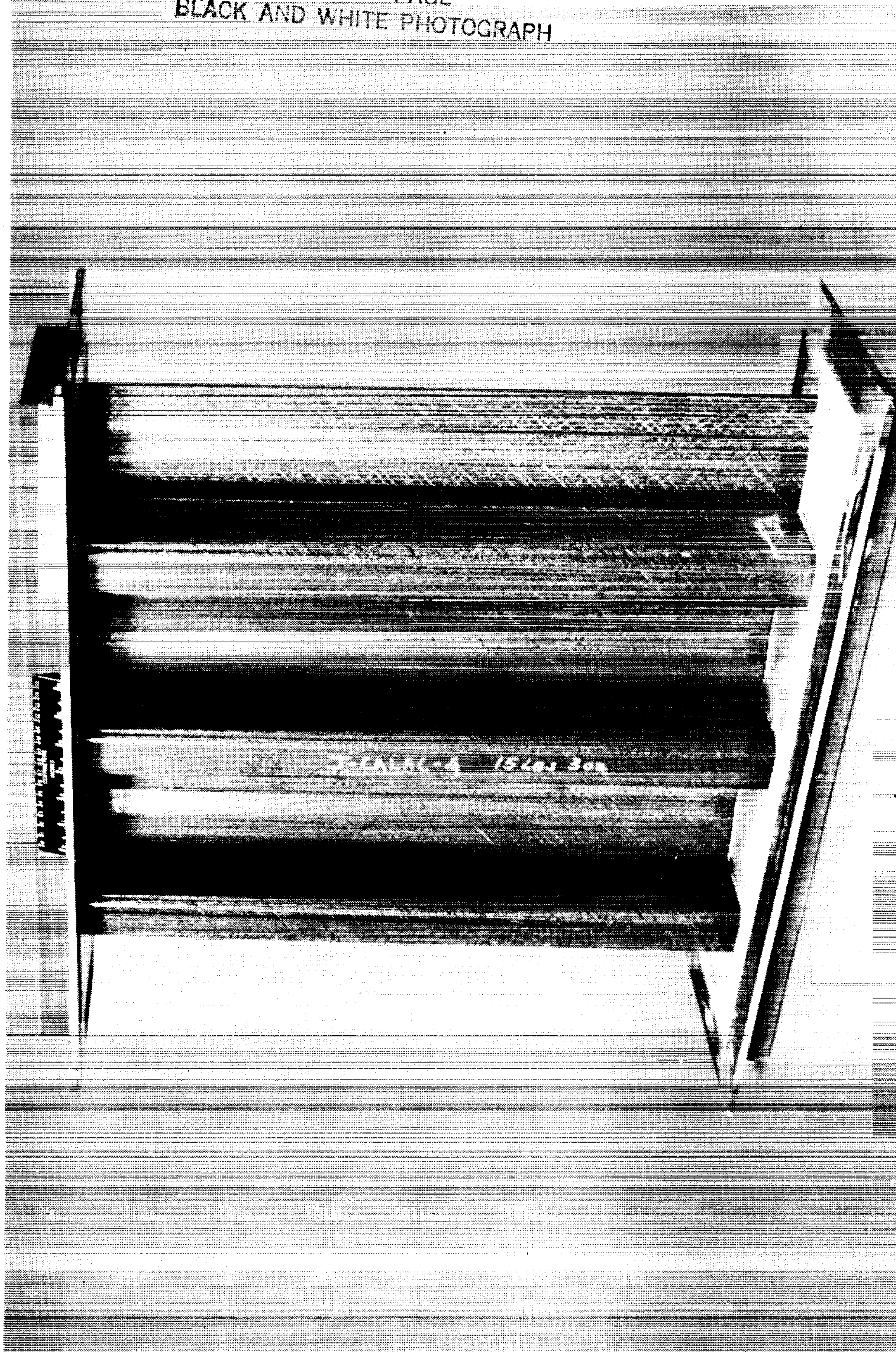


Figure 29. View of compression test panel with loaded ends potted, ground flat, square and parallel

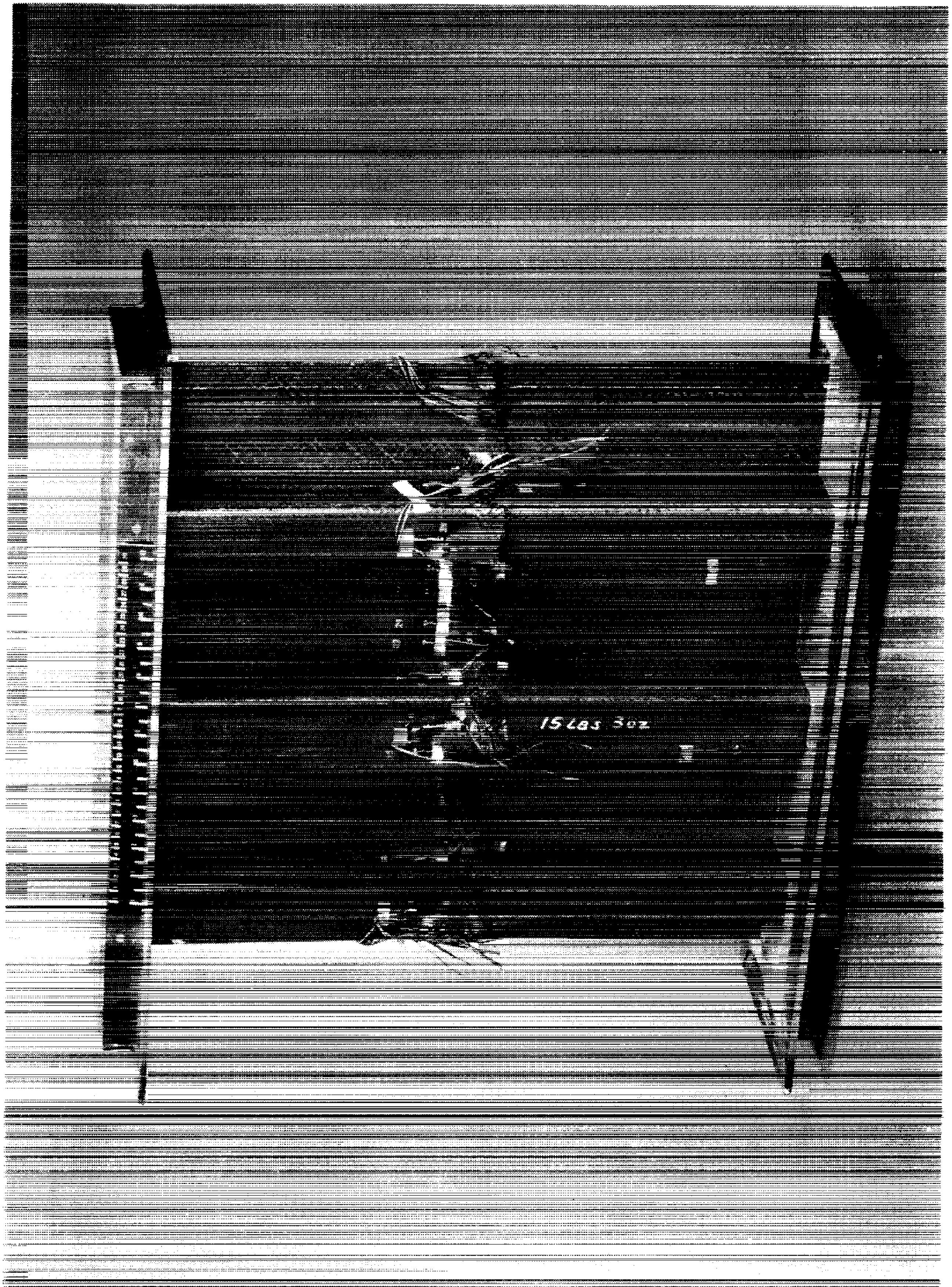


Figure 30. - Typical compression specimen after failure

	TEST CONDITION	FAILURE LOAD (KIPS)	FAILURE STRAIN* (10^{-6} IN/IN)
1986 PULTRUDED PANEL	UNNOTCHED	621	0.0058
	0.25 INCH DIAMETER OPEN HOLES	538	0.0057
	IMPACT DAMAGE (29.4 FT-LB) 534.7 FPS	406	0.0036
1987 PULTRUDED PANEL	UNNOTCHED	600	0.0050
	0.25 INCH DIAMETER OPEN HOLES	506	0.0048
	IMPACT DAMAGE (27.4 FT-LB) 516.5 FPS	420	0.0035

* AVERAGE FAR FIELD STRAIN

Figure 31. Pultruded J-stiffened panel compression test results

PROCESS INVESTIGATION FOR THICK SECTIONS

In light of the results from the J-stiffened panel tests, it was concluded that additional process development was required for thick sections. Process parameters investigated included pulling rate, die and resin temperatures, and die and resin bath configurations. To determine the effect of process variations, laminates were hand laid-up and autoclave cured to provide baseline data.

The laminates to be pultruded were 5 inches wide with a nominal thickness of 0.36 inches. The fiber-resin system chosen for this program was a knitted AS4 dry fabric and Shell's 9310, an untoughened epoxy. The lay-up of these laminates consisted of +45°, -45°, and 0° plies which were stitched together in ply packages of three.

The pultrusion die was fabricated and shims were made to provide means to modify the fiber volumes of the pultruded parts and in case of variations in fiber areal weight of the knitted broadgoods. The pultrusion resin baths were designed and constructed.

Hi-Tech knitted the graphite material. The +45°, 0°, -45°, and the -45°, 0°, 45° ply sets were completed without problems. Problems were encountered however in making the 0°/0°/0° ply sets. The quality of the carbon veiling used as a carrier was very poor and inhibited the knitting operation. Hi-Tech's request for a change to a Dacron veiling was approved.

For the plate pultrusion, the wet out station, die, and dry fiber rolls were assembled per Figures 32, 33, and 34. Quality assurance testing for the dry fabric was completed. Figure 35 lists the fiber areal weights of the three types of ply sets used (0_3°, 0°/+45°/-45°, and 0°/-45°/+45°) as well as the percentage by weight of each ply in the ply set. Quality assurance analyses on the resin were conducted as was simulation of several pultrusion runs with differential scanning calorimetry (DSCs). A one ply wet out experiment was also used to study the time required in the resin bath for complete fiber wetting.

A successful pultrusion run was completed. There were no jam ups in the die or "sloughing" of the resin during the run. Die temperature ranged between 310°F and 350°F from inlet beginning to exit of the die, respectively. Pulling rates were varied from 1 to 5 inches/min and no problems were encountered except that at 5 inches/min; the resin was still tacky as it left the pultrusion die.

Non-destructive evaluations, resin contents and photomicrographs were performed on specimens cut from areas within each pultrusion speed. Differential Scanning Calorimetry (DSC) was performed on all specimens to determine the degree of cure. The C-scan results indicated porosity in all panels; photomicrographs verified the C-scan results. A typical photomicrograph is shown in Figure 36. Resin contents ranged between approximately 28 and 30 percent by weight for all specimens.

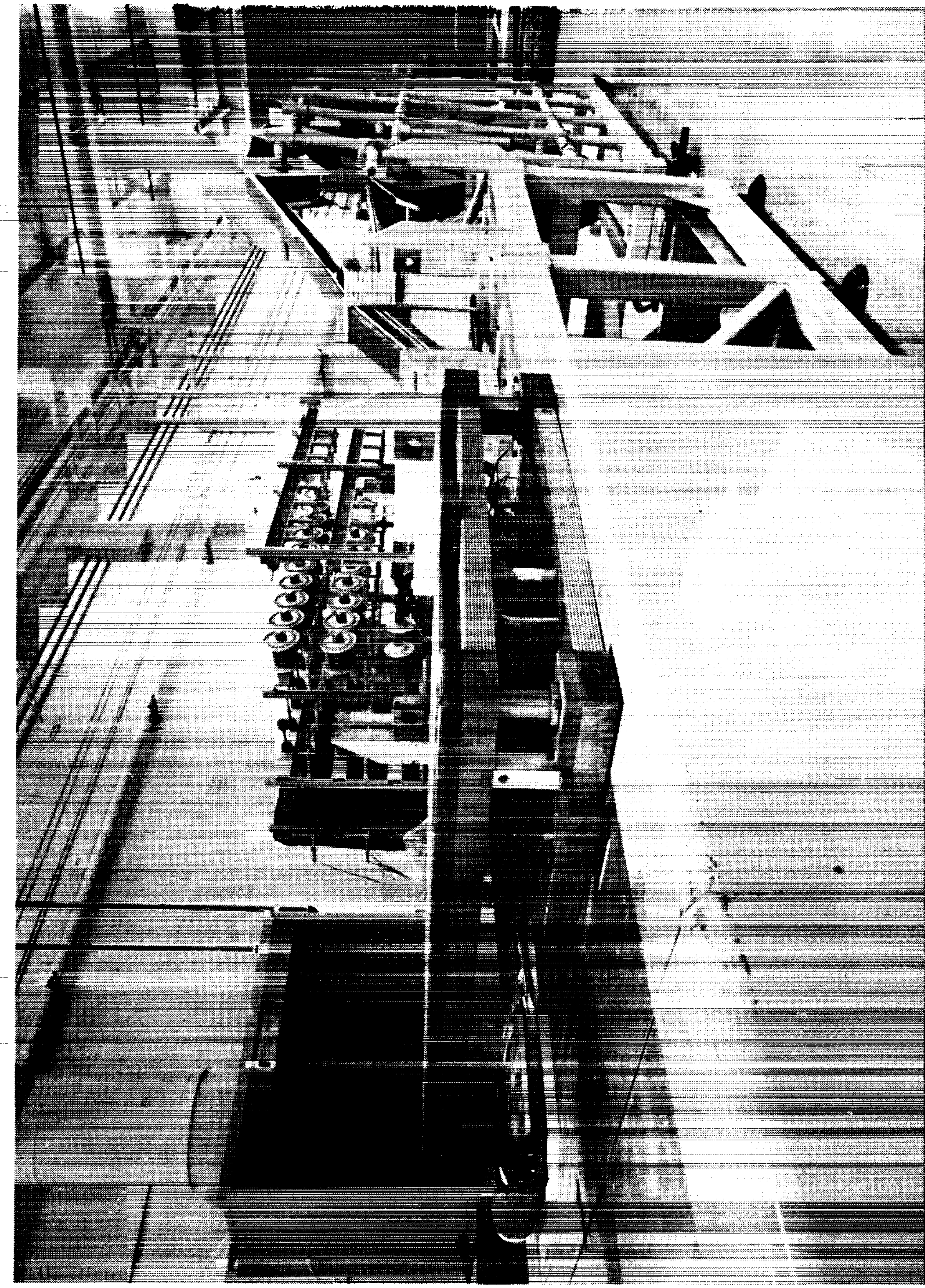


Figure 32. - Pultrusion die

Figure 33. - Dry fibers in place to start run

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

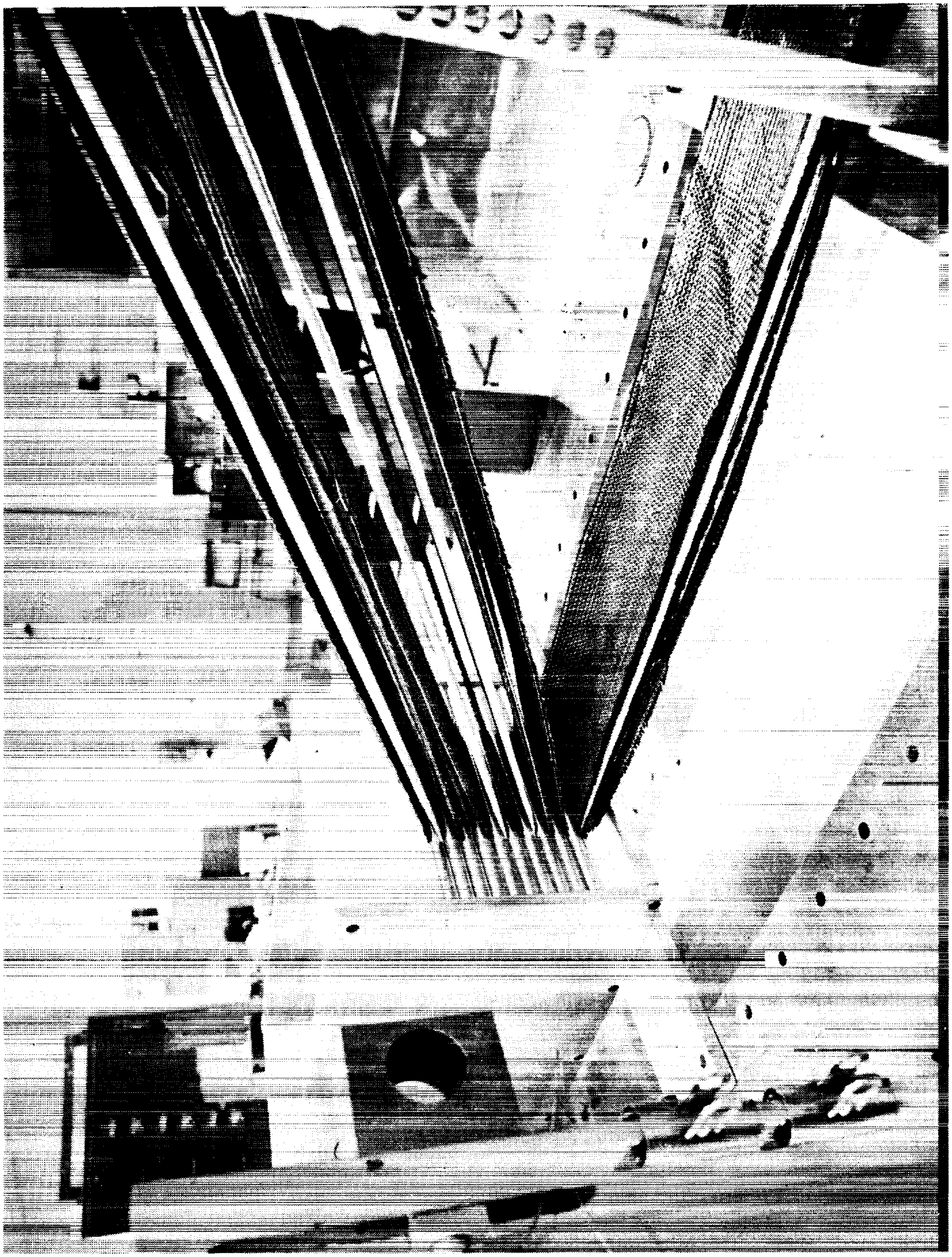


Figure 34. - Dry fiber rolls

MATERIAL	AREAL WEIGHT g/m^2	WEIGHT PERCENTAGE IN PLY SET
UNI-ON POLYESTER* VEILING 0$_3$°	500.8	--
UNI-ON CARBON* VEILING 0$_3$°	485.8	--
0°, +45°, -45°	448.2	0° - 34 45° - 33 -45° - 33
0°, -45°, +45°	450.2	0° - 33 -45° - 34 45° - 33

Figure 35. Quality assurance test results for the carbon fabric

As part of a Lockheed funded program on pultrusion, a small laminate was pultruded using unidirectional tows. The purpose of this experiment was to determine if the textile forms used previously were stitched too tightly to permit proper wet-out. Photomicrographs of the unidirectional fiber part showed relative freedom from porosity unlike that seen in the fabric parts. Based on this experiment, it was concluded that the fabric forms would be acceptable if the resin was degassed prior to pultrusion. Compression tests were conducted on the pultruded unidirectional specimen. The results listed in Figure 37, show that the strength of the void free pultruded laminate are less than a laminate made with 1806 preimpregnated material.

The investigation included several discussions with and visits by Shell technical personnel. Also performed were three processing studies using a wet layup of 9310 in a press. The purpose of the processing studies was to establish a baseline for the 9310 resin. It was not evident whether poor consolidation and subsequent poor mechanical performance was a function of the pultrusion process creating the porosity or the resin system itself volatilizing to form the porosity.

The first wet layup panel was made using a "dip" method. Plies of the dry broadgoods were dipped into a bath of the 9310 resin system. The layup was then placed in the autoclave and cured. Unfortunately, the caul plate hung up on the tooling and the laminate did not consolidate. Photomicrographs from this laminate showed very good fiber wetting with a minimum of microporosity. However, the resin content was 60+ percent.

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

25x mag.

Figure 36. - Typical cross section of 2 in/min pultruded panel edge

Material	Resin Content (%)	Compression Strength (ksi)
AS4/1806 Tape (1)	27.8	190.8
AS4/9310 Pultruded (2)	28.0	147.9

(1) Average of 3 coupons
(2) Average of 4 coupons - void free

Figure 37. 0-Degree compression strength comparison

The second wet layup was made using a "puddle" method with a press and vacuum bag. To obtain the correct resin content, resin was poured (or puddled) onto each ply. The layup was then placed under vacuum and into a 300°F press at 200 psi for 30 minutes to simulate the pultrusion process. The resin content of this laminate was in the desirable range, however, photomicrographs indicated porosity levels similar to that of the pultruded panels. Figures 38 and 39 show photomicrographs of the puddle method and pultruded plank, respectively.

After making the second wet layup panel, Shell personnel visited LASC to discuss what they had been doing to optimize the use of the 9310 resin system. They had found that a new combination of release agents (antifoaming agents) were effective. Shell also indicated that about 50 percent of the entrapped air in the resin occurs during mixing. Based on this input and some preliminary data obtained from a Lockheed-funded program, it was decided to degas the resin after mixing as a standard procedure.

Based on the results of degassing studies, the resin was degassed for another wet layup trial. The "dip" method was used to impregnate the fibers for this trial and so about a half gallon of resin was degassed. The resin was degassed for two hours and there was still a 1-inch head of foam on the resin. Since it was not desired to detrimentally stage the resin, the layup was started after the two hour degassing step. The panel was cured in a press under vacuum, at 170 psi and 300°F for 30 minutes. Photomicrographs of this panel are shown in Figure 40.

Based on the photomicrographs of the wet layup panels as well as the fact that the half gallon containers of resin did not degas after 2 hours, an investigation was performed on the resin itself. (Note that small quantities of resin have been successfully degassed.) Small cups of completely degassed resin were placed in an oven at 300°F for half an hour. The resin in these

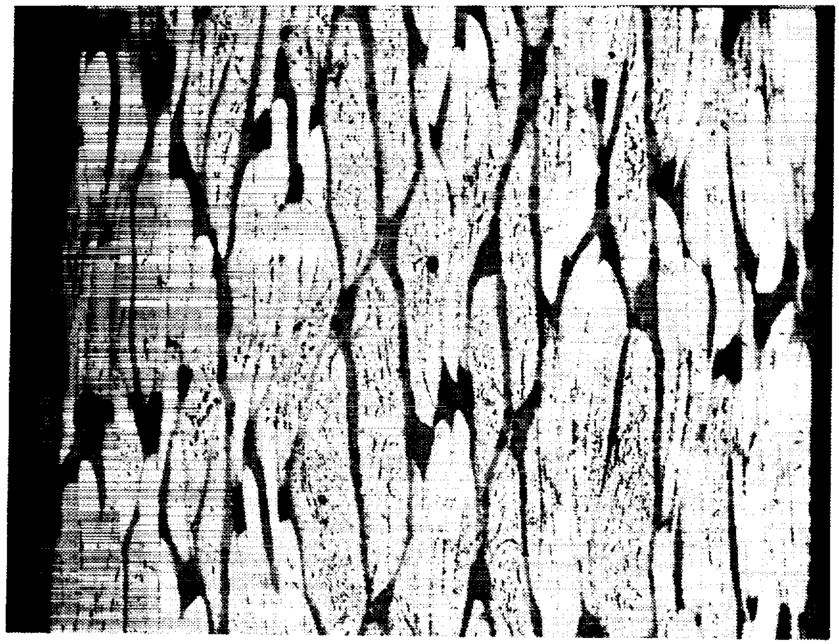


Figure 38. 9310 "puddle method" wet layup 10x magnification

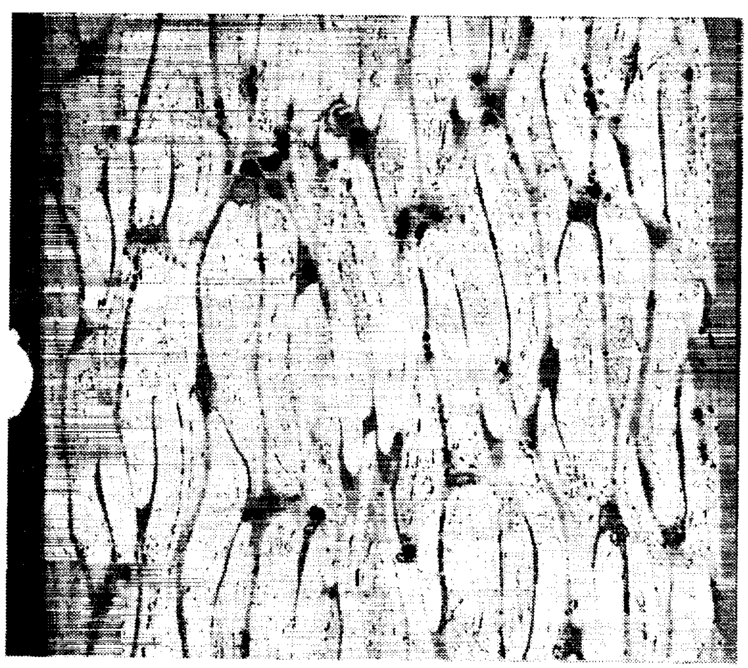


Figure 39. 9310 pultruded laminate 10x magnification

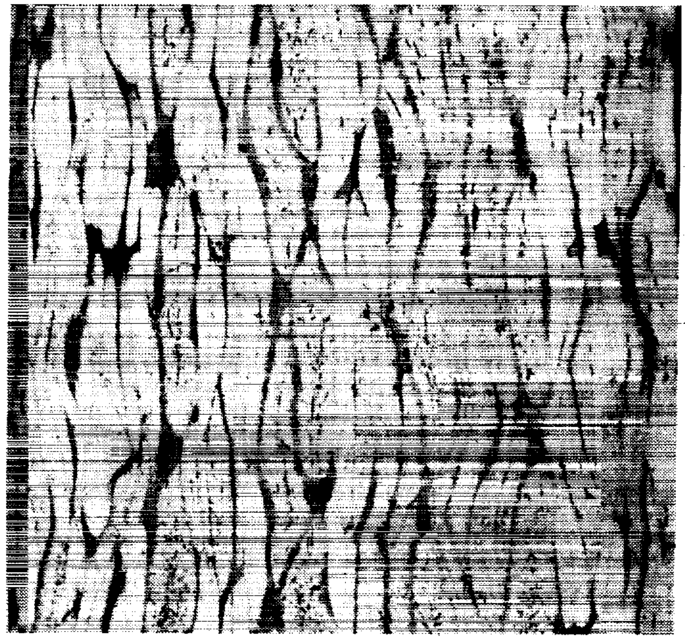


Figure 40. Third "wet layup" trial panel 10x magnification

cups foamed to almost double its original volume. This result indicated an agent in the resin which is highly reactive or volatile. According to the Material Safety Data Sheet (MSDS) sheet for 9310, the resin is loaded to 10.34 percent by weight with stryrene. Figure 41 is the vapor pressure diagram for stryrene for pressures up to 1 atmosphere. (Data is from the Chemical Engineer's Handbook.) Two significant points were noted from this chart. First, boiling temperature is logarithmic with respect to pressure, that means the boiling point of 293°F at 1 atm does not increase significantly even at 10 atmospheres. The second point noted was that the processing temperatures of 300-325°F are above or extremely close to the boiling temperature of stryrene. Dialogue was continued with the vendor to determine how to combat this problem prior to the last pultrusion.

In the final pultrusion trials, knowledge, gained from earlier work and from the resin vendor's trial effort results was incorporated. The tooling, material, and process modifications and justification for their incorporations are summarized below.

- Die opening reduced from 0.36 inches to 0.33 inches

  Justification:

  a. Increased pressure during cure will squeeze out voids better

  b. Vendor recognized better consolidation with higher volume fraction of fibers

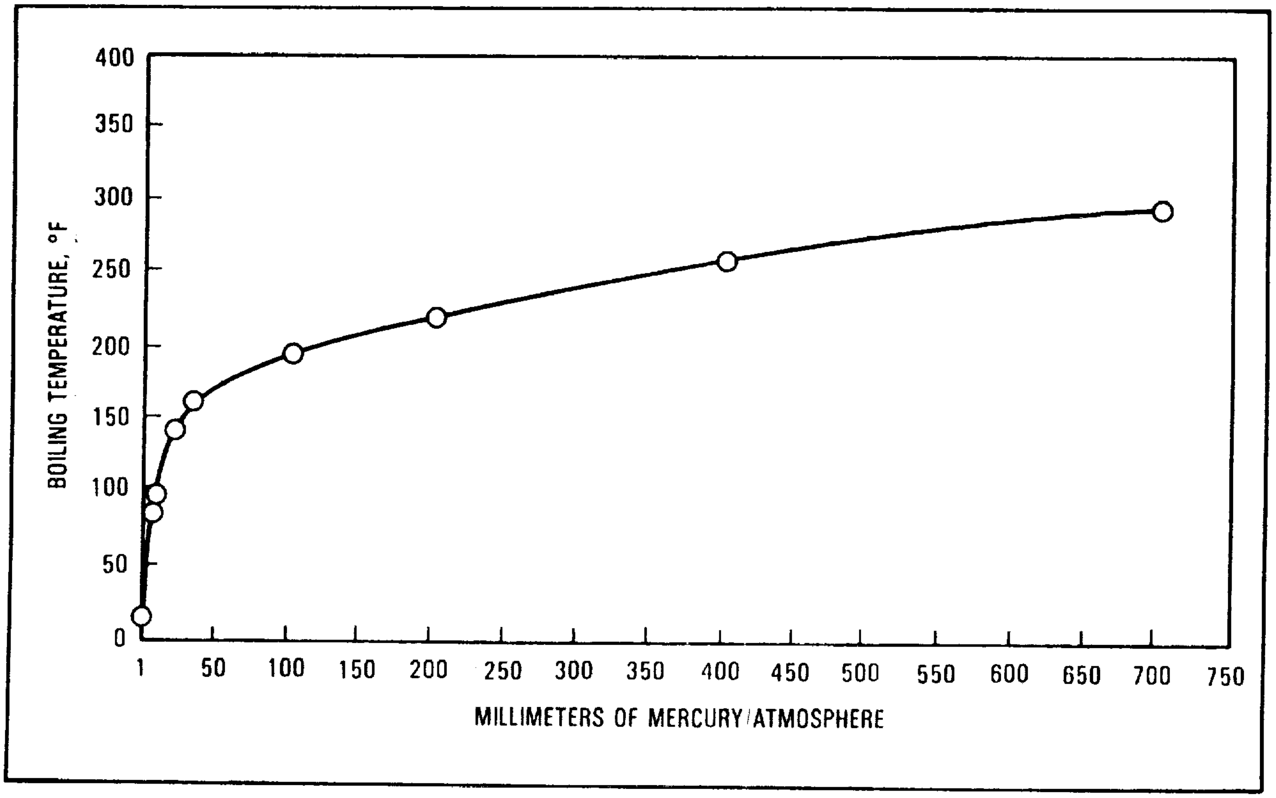


Figure 41. Stryrene vapor pressure curve (Source: Chemical Engineer's Handbook)

- Die length increased to 29 inches; pultrusion speeds increased to range from 5 ipm to 13 ipm.

  Justification:

  a. Other LASC independent programs produce 1 inch x 0.05 inches unidirectional "stuffer" pultrusions that are void free

  b. These are standard die lengths and speeds used by vendor to develop processing parameters

- Different amounts and types of air release agents used

  a. Vendor studies show this combination as best for consolidation; qualitatively determined from photomicrographs.

- Resin system degassed between mixing and pultrusion

  Justification:

  a. Wet layup studies revealed much air was whipped into resin during mixing

  b. Resin used for void-free "stuffer" was degassed.

The pull speed, die temperature, and number of ply sets were varied in these final pultrusion trials, to assess the effect of these parameters on processibility and consolidation. The processing parameters varied included speeds of 5-13 ipm (inches per minute), both 19 and 20 ply set thicknesses, and temperatures of 305, 315, 325, and 335°F. A 29 inch stainless steel die was used with a 0.33 inch opening heated by two zone platens having zone temperatures of 315° and 335°F. The Material system based upon 9310 resin contained 1) ASP 400 Clay filler/ 20 parts per 100 parts resin, 2) Axel 1846 Release Agents/0.67 parts per 100 parts resin, 3) BYK A 501 Air Release/0.27 parts per 100 parts resin, and 4) BYK A 515 Air Release/0.40 parts per 100 parts resin.

At speeds above 11 ipm, excessive pulling forces were encountered. Therefore, the maximum speed investigated was 11 ipm. The best results were obtained using 7 ipm, 19 sets of ply stacks and platens temperatures of 305° and 325°F. Figures 42 and 43 through 49 exhibit resin contents and photomicrographs results, respectively. Noting the 32.95% resin content and the relatively void free condition of this well consolidated pultrusion seen in Figure 50, much improvement can be seen when compared to the earlier pultrusion trials as depicted in Figure 38 pultruded laminate. The 7 ipm panel was selected for mechanical testing. The strength and modulus of the pultruded panels was compared with that of AS4/1806 panels with similar layups in Figure 50. The 1806 resin, like the 9310 resin system is an untoughened epoxy. The coupon configuration for the 1806 was 3 inches wide and 0.25 inches thick. The modulus values of the pultruded panel were the same as the AS4/1806 panels while the strengths were slightly higher. Based on these results, the intrinsic properties of the 9310 resin do not seem low for an untoughened epoxy.

It appears that the better consolidation of the second set of pultrusion trials was due to changes in two specific processing parameters. Better air release agents were used in the resin formulation and the resin was degassed after mixing.

Although the degassing procedure used was far from optimal, a significant reduction of air was seen in the resin. The resin was degassed in 2 gallon quantities in bell jars for 2 hours. After this time there were still considerable amounts of foam on the resin surface. Because the resin could not be degassed for longer periods of time due to the outlife of the resin, the foam was manually removed from the surface. In a production operation, a vacuum mixer or a high shear mixer which does not whip air into the resin could be used.

From this brief processing study, it was concluded that there are several parameters needed to make pultrusion a viable manufacturing process for aircraft structures. Although not new information, reduction of voids was critical in improving mechanical performance. From microscopic examination of all laminates made, it was found that the voids were not inside the fibers bundles (that is the resin wetted the fibers well) but in the resin itself between the bundles. Several sources were found as void entrapment causes in the resin:

SAMPLE	RESIN CONTENT % WT
5 IPM 20 PLY SETS DIE TEMP: 315,335°F	34.8
20 PLY SETS DIE TEMP: 305,325°F	32.07
19 PLY SETS DIE TEMP: 305,325°F	32.1
7 IPM 20 PLY SETS DIE TEMP: 315,335°F	32.95
9 IPM 20 PLY SETS DIE TEMP: 315,335°F	33.4
11 IPM 20 PLY SETS DIE TEMP: 315,335°F	32.79

Figure 42. Resin content measurements for second set of pultrusion trials

1) air whipped in during mixing

2) low boiling components in the resin vaporizing

3) air entrapped during the wetting process, i.e., air being carried into the bath by the fibers.

4) volatilization or degradation of the polyester stitching or sizing or water attached to the stitching yarns or clay filler particles.

From the second session of pultrusion trials, and the internal research study, it was found that degassing the resin significantly reduces the voids in the resin. However, it was not determined whether the void reduction was caused by eliminating items 1), 2) or both. Pultrusion of a resin mixed in a high shear mixer would be able to determine whether the air or low boiling compound removal was critical in the degassing procedure.

The residual porosity entrapped in the resin in the second pultrusion session could either be caused by items 3) or 4). To eliminate air being carried into the pultrusion bath by the fibers, the bath could be pressurized slightly to push the air out as the fibers enter the bath.

Photomicrographs from 2nd pultrusion trials

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

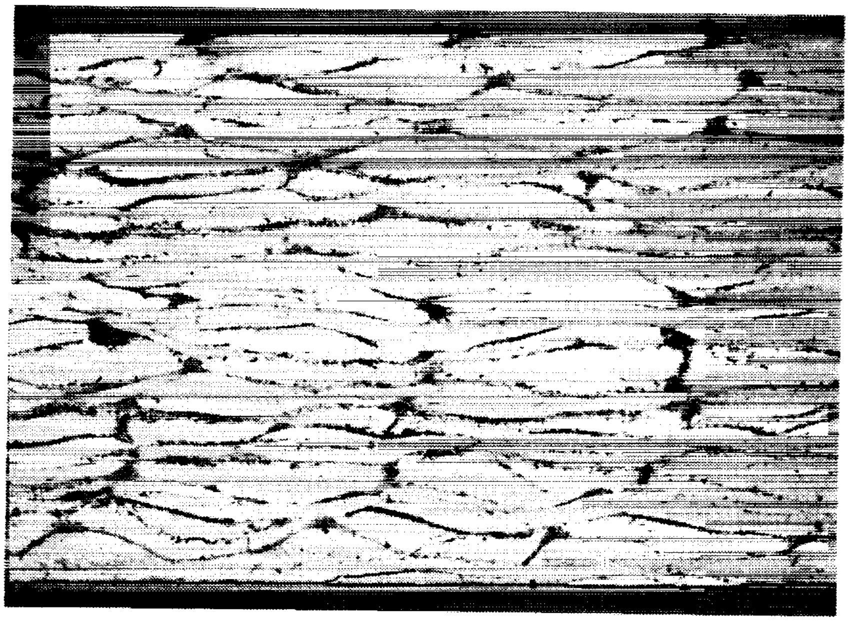


Figure 43. Pull speed 5 ipm, 20 ply sets die temp: 315, 335°F

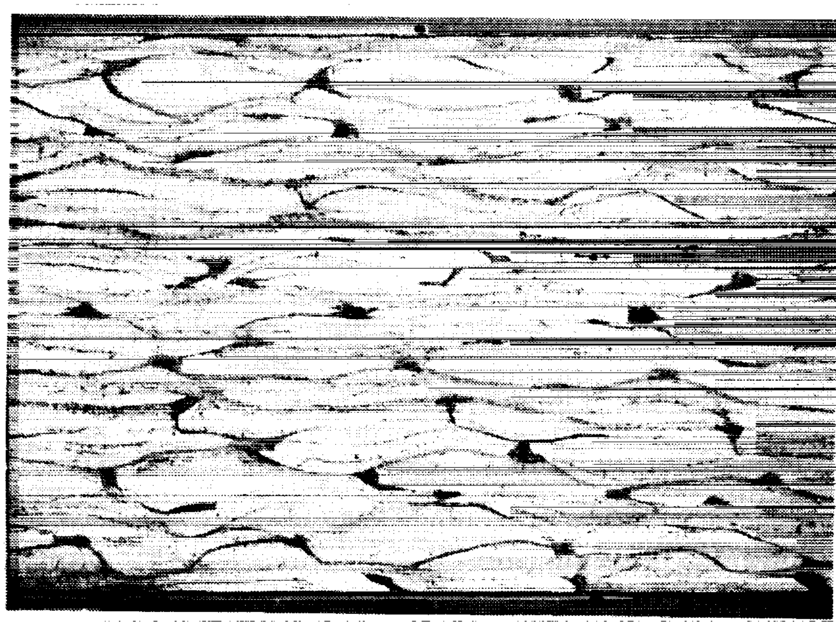


Figure 44. Pull speed 5 ipm, 20 ply sets die temp: 305, 325°F

Photomicrographs from 2nd pultrusion trials

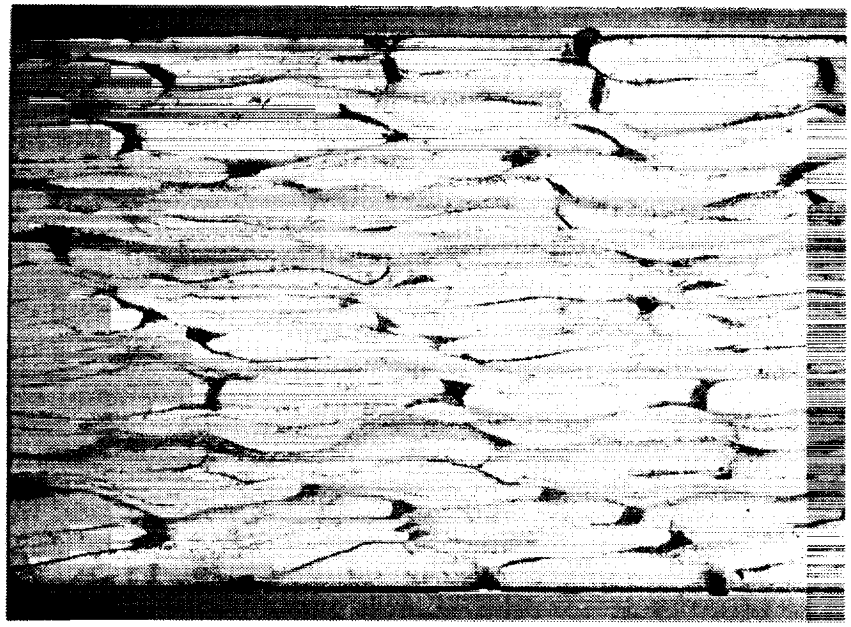


Figure 45. Pull speed 5 ipm, 19 ply sets die temp: 305, 325°F

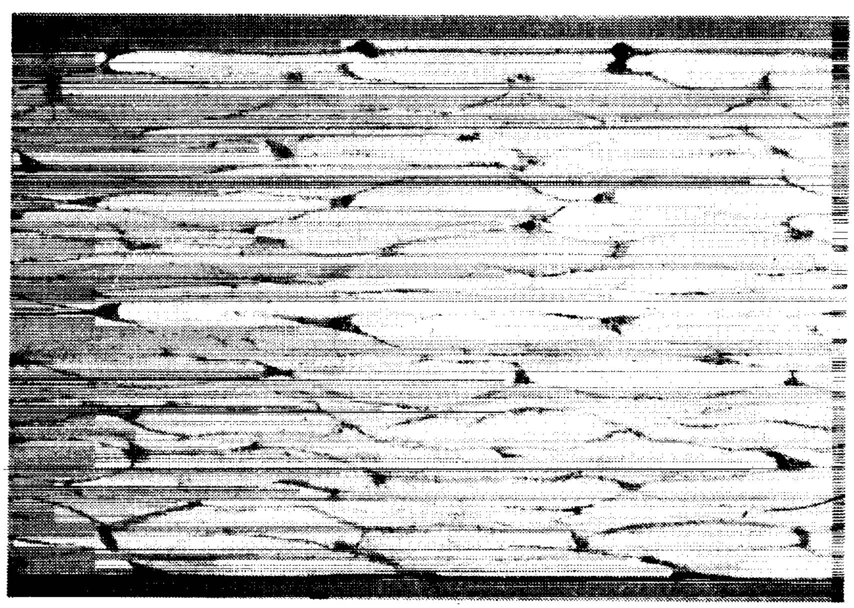


Figure 46. Pull speed 7 ipm

[70]M0925.AA0

Photomicrographs from 2nd pultrusion trials

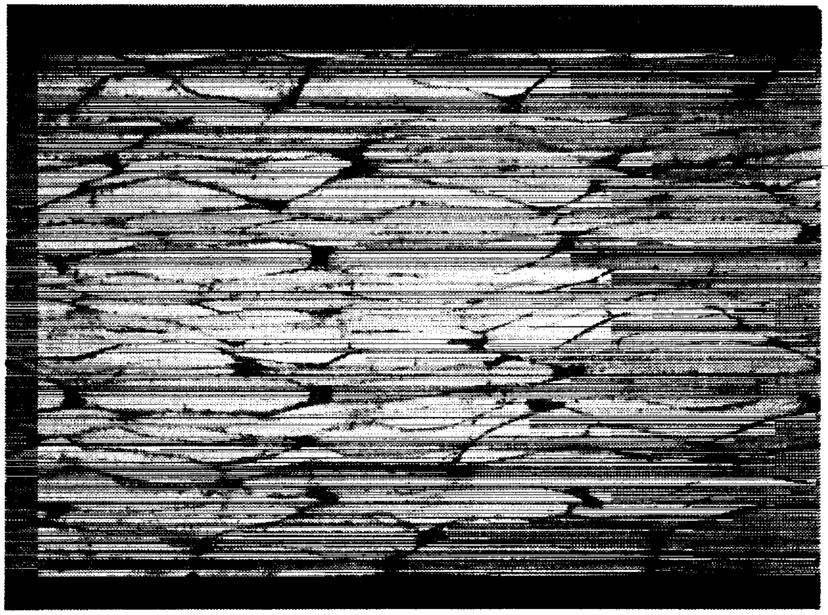


Figure 47. Pull speed 9 ipm

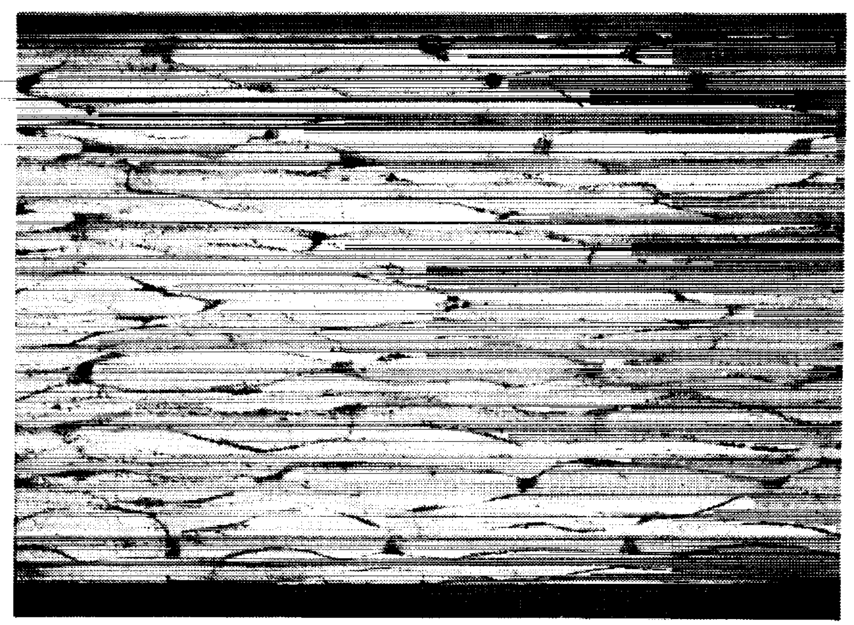


Figure 48. Pull speed 11 ipm
8.5 x magnification

40 X magnification

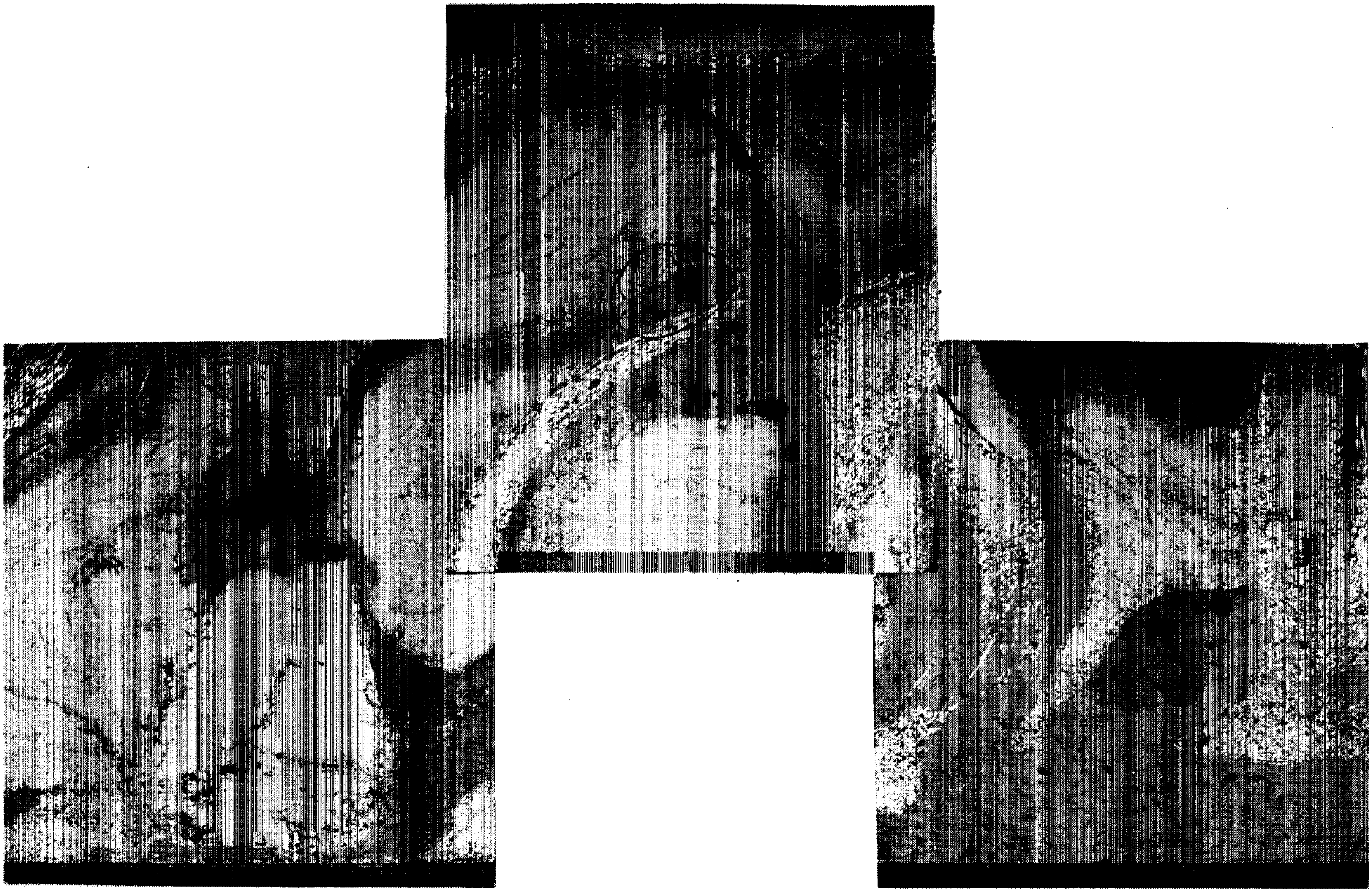


Figure 49. - Photomicrographs showing void locations near polyester stitching yarns and clay filler particles

COMPARISON OF STRENGTH AND MODULUS OF PREPREG VERSUS WET LAYUP AND PULTRUSION

Property	✝AS4/1806 Prepreg	AS4/930 Pultrusion
Resin (%)	38.3	32.95
Compression (KSI) Open Hole	65.8	51.6

LAM. ORIENT + (55% 0/45/+45)
✝LAM. ORIENT. = (50% 0/50%/+45)

Figure 50. Comparison of AS4/9310 "wet" layup and pultrusion with AS4/1806 "prepreg"

After analyzing the photomicrographs of the second pultrusion trials, it was noted that a lot of the residual porosity was "attached" to the polyester stitching yarns and the clay filler particles as described by the photomicrographs in Figure 55.

The voids near the Dacron stitching yarn and the clay filler particles could have several causes. Both the Dacron and the clay (Kaoline) are hydrophillic. They could have absorbed small amounts of water which would evaporate during the pultrusion process. Pre-drying both the clay and the pultrusion fabric would eliminate this problem. In addition, the Dacron or a sizing used with the Dacron could be degrading just enough to cause the porosity seen in Figure 53. A solution to this problem would be to use carbon stitching thread. When this program was initiated, the vendor (Hi-Tech/Hexel) was questioned about using a carbon stitching yarn. At that time, the a carbon yarn was a developmental product and could not be delivered during this program. Today, however, the carbon stitching yarns could replace the Dacron.

In summary, a process was developed which eliminated the majority of the porosity in our pultruded panels. It was also shown that elimination of this porosity greatly enhanced mechanical performance. Although the majority of voids were eliminated, approximately 1% residual porosity remained. Several possible causes of this residual porosity were located and several methods to eliminate the phenomena were proposed.

CONCLUSIONS

The pultrusion process is one of several manufacturing methods which have the potential to fabricate composite parts at a cost much lower than autoclave molding using hand laid-up preimpregnated materials. However, given the current state of the art of pultrusion technology and the limited availability of aerospace grade pultrusion resins, additional process development is required to successfully fabricate a pultruded part as compared to an autoclave molded part. The research discussed within this report has resulted in the following conclusions.

When designing a part that is to be pultruded, the designer must work closely with the materials engineer, tool designer and the pultrusion specialist. Design configurations and fabric constructions must be selected that minimize the complexity of the creeling system and wet-out tanks. Laminate designs must be selected to minimize possible fabric distortion due to the pulling forces during part pultrusion. Part configurations should be selected which minimize the complexity of the die. This may require some reduction in structural efficiency to enhance manufacturability. For example: a blade stiffened panel design would have been much easier to pultrude that the J-stiffened configuration which was selected.

Development of a satisfactory process for pultruding complex parts takes more time and funds than an equivalent development cycle for an autoclave molded part. Each pultrusion process development trial requires extensive set-up time and consumes a relatively large amount of material because of the location of the creeling system and grippers relative to the die. If refinements in the fabric design are required, as was the case in the development reported herein, then several weeks are needed for the weaving of the new fabric pre-form.

Based on the results of the pultrusion development done within this program and work conducted with Lockheed funding , it is concluded that two items need additional research for the successful pultrusion of complex parts, wet-out tank designs and resins supported with a specific pultrusion process specification. Within this program, both free standing and pressurized wet-out tank designs were used with limited success. Additional research is required to develop tanks which adequately wet-out the fibers in the multi-ply stitched fabrics required for a complex pultruded part. With regards to resin development, it should be noted that neither of the epoxy systems used in this program enabled the fabrication of parts having strengths comparable to preimpregnated materials. This was true even when the pultruded part was well wet-out and void free. Based on the tests conducted on the resins used within this program, it is concluded that additional research is required to develop an aerospace grade pultrudable epoxy resin which when pultruded produces low-void/porosity free pultrusion laminates.

The activities reported herein have demonstrated that a very complex panel can be successfully pultruded. However, additional research in the areas of wet-out tank design and resin formulation along with very specific pultrusion process parameter controls are required to transform the pultrusion process from its current status to a production ready process.

[redacted]

Report Documentation Page

National Aeronautics and Space Administration

1. Report No. NASA CR-181780	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle: Manufacturing Development of Pultruded Composite Panels		5. Report Date: April 1989
		6. Performing Organization Code
7. Author(s): L. E. "Roy" Meade		8. Performing Organization Report No.
		10. Work Unit No. 505-63-01-06
9. Performing Organization Name and Address: Lockheed Aeronautical Systems Company, P. O. Box 551, Burbank, California 91520		11. Contract or Grant No. NAS1-15069
		13. Type of Report and Period Covered: Contractor Report
12. Sponsoring Agency Name and Address: National Aeronautics and Space Administration, Langley Research Center, Hampton, VA 23665-5225		14. Sponsoring Agency Code

15. Supplementary Notes

Langley Technical Monitor: Marvin Dow
Final Report

16. Abstract

The weight savings potential, of graphite,epoxy composites for secondary and meduim primary aircraft strucuters, has been deomonstrated. One of the greatest challenges facing the aircraft industry is to reduce the acquisition costs for composite structures to a level below that of metal structures. The pultrusion process, wherein reinforcing fibers, after being passed through a resin bath are drawn through a die to form and cure the desired cross-section, is an automated low cost manufacturing process for composite structures. The Lockheed Aeronautical Systems Company (LASC) Composites Development Center designed, characterizated materials for, fabricated and tested a stiffened cover concept compatible with the continuous pultrusion process. The procedures used and the results obtained therefrom are presented in this report.

17. Key Words (Suggested by Author(s)): Pultrusion, Test, Stiffened panel, Analysis, Epoxy	18. Distribution Statement: [redacted] Subject Category 24

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of pages	22. Price
unclassified	unclassified	60	