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DEVELOP FABRICATION/PROCESSING TECHNIQUES FOR HIGH TEMPERATURE ADVANCED COMPOSITES FOR USE IN AIRCRAFT STRUCTURES

E. B. Birchfield and R. Kollmansberger

MCDONNELL AIRCRAFT COMPANY

TECHNICAL REPORT AFML-TR-72-91

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Air Force Materials Laboratory
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

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FOREWORD

The following final report describes work performed on Air Force Contract F33615-70-C-1546, "High Temperature Advanced Composites", by the McDonnell Aircraft Company (MCAIR), St. Louis, Missouri. The work accomplished and reported herein was performed by MCAIR, St. Louis, Missouri, in conjunction with Whittaker Research and Development Corporation (WRD), San Diego, California. The program was administered under the direction of the Air Force Materials Laboratory, Advanced Composites Division, by Mr. R. M. Neff, Project Engineer.

The program was conducted by the Structural Research Department at MCAIR, St. Louis, and was managed by J. M. Finn, with E. B. Birchfield and R. Kollmansberger as principal investigators.

The primary contributors to the program include:

Materials and Process Department	R. O. Ahrens and K. L. Kremer
Materials Laboratory	J. F. Harrell
Advanced Fabrication	K. R. Kreder and H. D. Rex
Strength	T. O. Glenn
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WRD, San Diego	V. A. Chase, R. Van Auren, D. Beeler

This report covers the entire program contract period from June 1970 to July 1972. The first draft of this report was released by the authors in May 1972.

This technical report has been reviewed and is approved.

R. C. Tomashot
Acting Chief
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ABSTRACT

The objectives of this program were to place polyimide matrix advanced composite processing technology on a production basis, and to demonstrate its acceptability for structural applications by fabrication and test of representative structure. Under this program polyimide matrix advanced composites were cured in a one shift autoclave operations (~8 hours) at 350°F and 200 psi maximum while maintaining epoxy matrix-type properties.

These process parameters were confirmed by the successful fabrication and test of an F-4 polyimide rudder, which demonstrated the flight worthiness of thin gauge boron and graphite/polyimide in this type structure. Design allowable data for tension, compression, and inplane shear were developed at both room temperature and 550°F.

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1. INTRODUCTION AND SUMMARY

This is the final report on the High Temperature Advanced Composites program, Contract F33615-70-C-1546. The High Temperature Advanced Composites (HITAC) Project was a program to develop boron and graphite polyimide composite materials and fabrication techniques required to successfully apply these materials to primary aircraft structure. Fabrication methods were developed for use with conventional production equipment. Original plans were to design, analyze, fabricate, and test, both at room temperature and 550°F, an F-15 horizontal stabilator torque box. Because of problems with fabricating thick laminates a revised program was undertaken, directed toward fabrication and test of the polyimide F-4 rudder, establishment of boron and graphite/polyimide design allowables, and fabrication and test of an F-15 composite wing compression panel utilizing polyimide materials. The work performed as described in this report, and summarized in this section, was divided into the following tasks.

1.1 Materials Development

The objective of this task was to evaluate and select (a) the boron and graphite/polyimide prepregs, (b) the polyimide adhesives, and (c) the high temperature honeycomb core materials to be used in the program. Prepreg processing objectives were a cure cycle compatible with current production schedules, autoclave pressure not to exceed 100 psi, and cure temperature not to exceed 450°F. In addition, matrix properties similar to those obtained for epoxy matrix composites were necessary for the structural components. Adhesive and honeycomb core requirements were basically that the selected materials perform satisfactorily in the F-4 rudder and F-15 horizontal stabilator torque box originally planned.

Eight candidate polyimide resins were evaluated with boron and graphite fibers, for mechanical and physical properties and prepregging capability. Based on the results obtained, Monsanto's Skybond 703 (SB703) and E.I. du Pont's (PI4707) polyimide resins were selected for final evaluation. The SB703 resin demonstrated higher mechanical properties with less scatter and exhibited more consistent fabrication results with both boron and graphite fibers, and was selected for use in the program.

Several candidate polyimide adhesives were evaluated for structural bonding applications. Bloomingdale's FM-34 and FM-29 adhesives were selected for skin-to-honeycomb core and edgemember-to-honeycomb core applications,

respectively. Narmco Metlbond 840 was picked for all primary and secondary metal-to-metal type applications.

The following honeycomb core materials were evaluated: HRH-327 fiberglass/polyimide, 301 stainless steel, PH15-7 Mo stainless steel, and Ti-75A titanium. Based on mechanical properties, cost, and machinability the HRH-327 fiberglass/polyimide honeycomb core was selected for all program applications.

1.2 Fabrication Scale-Up and Evaluation

The objective of this task was to perform the following scale-up efforts prior to structural component fabrication: (a) boron/SB703-titanium splice joint fabrication, (b) graphite/SB703 substructural shape demonstration, (c) large area polyimide composite processing development, (d) large area polyimide composite honeycomb sandwich panel fabrication, and (e) polyimide composite sandwich beam fabrication and evaluation.

Initial fabrication of 40-44 ply boron/SB703 laminates (simulating the F-15 Stabilator Torque Box skin) was unsuccessful. Processing changes that affected "B" stage, cure and post cure cycles were instituted after extensive analysis and process development efforts. The resultant modified fabrication techniques were demonstrated for 15 through 40 ply laminates with satisfactory interlaminar shear strengths of 12-16 ksi at R.T. and 6.5 - 8.5 ksi at 550°F. The cure cycle used was acceptable for production considerations.

Due to the extensive effort required to solve the thick laminate fabrication problem and the redirection of the program, items a, c, and e above, associated with the major structural demonstration component - the F-15 Stabilator Torque Box, were deleted. However, the fabrication and test of the F-4 rudder were retained in the program as originally planned. The major structural component selected for fabrication and evaluation was changed from the F-15 Stabilator Torque Box to an F-15 Wing Compression Panel using thinner boron/SB703 skins with 18 and 17 plies for the panel and stiffeners, respectively.

Graphite/SB703 substructural shape scale-up was demonstrated by the fabrication and evaluation of several "C" channel sections. Tensile specimens machined from one "C" channel failed at 190-208 ksi when tested at R.T. Maximum thickness required for graphite/SB703 development was 17 plies and no major problems were encountered.

This task was concluded by the fabrication of a two foot by three foot honeycomb sandwich panel consisting of 6 ply boron/SB703 skins, HRH-327

fiberglass/polyimide honeycomb core and FM-34 polyimide adhesive. NDT and dissection of the panel revealed a satisfactory bonding process.

1.3 Structural Analysis/Design

The purpose of this task was to perform a design and analysis on the F-4 rudder, an F-15 compression wing panel, and the stabilator torque box. The detailed design and analysis of these items are discussed in Section 4.0 of this report.

1.4 Design Mechanical Properties

The purpose of this task was to establish design allowable strengths and elastic constants for design and analysis of polyimide structures. Included were room temperature and 550°F tests of boron and graphite/polyimide specimens in tension, compression, and in-lane shear. "B" basis design allowables have been calculated and are reported in Section 5.0 of this report.

1.5 Component Fabrication/Test

The purpose of the component fabrication/test task was to demonstrate the adequacy of polyimide materials for use in aircraft structures. An F-4 polyimide rudder was successfully fabricated and tested. The fabrication of the F-15 horizontal stabilator torque box had to be discontinued because of difficulties encountered in fabricating thick section boron/polyimide laminates, as discussed in Section 1.2 above. In place of the stabilator torque box, an F-15 composite wing compression panel was fabricated and static tested to failure.

2. MATERIALS DEVELOPMENT

2.1 Introduction

The objective of the Materials Development task was to: (1) select and develop an acceptable prepreg for both boron/polyimide and graphite/polyimide, (2) select polyimide adhesives for high temperature honeycomb sandwich structure, and (3) select a high temperature honeycomb core material. Prepreg to be developed was to require a cure not to exceed 450°F, a cure pressure not to exceed 100 psi, and a cure-time cycle compatible with heating and cooling rates attainable in conventional autoclaves. This task was a joint effort between McDonnell Aircraft Company (MCAIR) and Whittaker Research and Development Company (WRD), with MCAIR concerned primarily with adhesive and honeycomb core selection and WRD involved in polyimide prepreg development, under MCAIR direction.

2.2 Boron/Polyimide and Graphite/Polyimide Prepreg Development

2.2.1 Literature/Information Survey - A literature/information survey was performed by MCAIR and WRD for candidate polyimide resins. Government reports, vendor literature, and in-house information revealed that Brunswick BPI-373, General Electric Gemon resin, Monsanto Skybond 703 and Skybond 709, TRW P13N, and DuPont PI4707 and PI5077 were potential candidates for this program.

The Brunswick BPI-373 prepreg, although exhibiting good flexure and shear properties (Reference (1)), was eliminated because it was available only in the form of fabricated hardware. The General Electric Gemon polyimide resin did not have sufficient thermal stability and had not been sufficiently developed for autoclave operations. The P13N resin was selected for comparison purposes only with the autoclave grade resins. It is a compression molding resin that was used extensively for advanced composite engine applications.

Monsanto Company, Plastic Products and Resins Division, was approached to determine if resins other than Skybond 703 and 709 were available for this program. Information was forwarded on three additional resins that offered potential processing and/or temperature improvements over the Skybond 703: MS6264, MS6270, and MS6271.

DuPont felt that PI4707, a controlled flow version of the PI 4701, was the best polyimide resin they had available. It was expected to result in an improved cure cycle, with thermal stability essentially the same as

that exhibited by the PI4701. The PI5077 was also included because of successful prepreg experience with it at WRD.

2.2.2 Candidate Resin Evaluation/Selection - MCAIR and WRD jointly selected the Skybond 703, Skybond 709, RS 6264, RS 6270, RS 6271, PI4707, PI5077, and P13N for evaluation based on the literature survey, resin supplier survey, and in-house experience. The P13N, while not considered a candidate because of its high cure temperature and pressure requirements, was included for comparison purposes using cure pressures of 100 psi and cure temperatures of 600°F. PI5077 was added because it showed promise of developing into an acceptable prepreg resin for graphite filament and also because of the experience WRD had with this resin.

WRD evaluated all the above, except PI5077, with boron filament, and the same group plus PI5077 with graphite filament. The data obtained are presented in Tables I and II. In addition, MCAIR also evaluated boron/Skybond 703 prepreg, with the data presented in Table III. The graphite filament utilized throughout the program was Morganite Type 2 meter length. The boron filament was standard 4 mils in diameter from Hamilton Standard. Each data point shown in Tables I and II was an average of six individual specimens.

The new Monsanto resins (RS 6264, RS 6270, and RS 6271) did not perform with consistency. The RS 6270 and RS 6271 did not wet the graphite filament well enough to make an acceptable laminate. In most property comparisons they were 25% lower than Skybond 703 although they had much lower void contents. Further work, beyond the scope of this program, would be required to make these three resins more acceptable for boron and graphite prepregs.

Skybond 709 and PI 5077 were rejected because their matrix dependent properties (90° flexure and interlaminar shear) were poor. The Skybond 709 properties were generally 25-50% lower than those for Skybond 703. The PI5077 had very low 90° flexure properties when prepregged with graphite filament. In addition, both Skybond 709 and PI 5077 composites using graphite filament had low 0° flexure properties.

A numerical rating system used in the evaluation demonstrated the superiority of the Skybond 703, P13N, and PI4707 resin systems. The rating system is applied to the 0° flexure, 90° flexure, and interlaminar shear values at 75°F and 550°F. The boron/polyimide systems are rated 1 to 7 and the graphite/polyimide systems are rated 1 to 8, where the lowest number reflects the poor-

TABLE I
EVALUATION OF BORON/POLYIMIDE LAMINATES AT WRD

Property	Temp (°F)	Resin System						
		RS 6264	RS 6270	RS 6271	SB 703	SB 709	P13N	PI 4707
Resin Content (% by Weight)		27.60	19.30	18.50	19.10	17.20	34.30	29.30
Specific Gravity		1.72	2.02	2.01	1.90	1.91	1.80	2.01
Void Content (% by Vol.)		14.70	6.10	7.20	11.80	12.70	6.20	0
0° Flexure (ksi)	RT	111.40	223.00	225.80	220.60	192.40	159.10	206.90
	550	89.90	136.60	180.60	134.10	148.00	135.80	196.80
90° Flexure (ksi)	RT	7.90	4.70	5.70	8.40	6.10	8.30	6.80
	550	5.90	2.70	3.60	5.10	5.80	5.10	4.20
Short Beam Shear (ksi)	RT	6.60	6.70	6.30	8.30	3.80	5.60	9.80
	550*	9.50	9.00	4.90	10.60	4.20	5.30	7.80

* Thermoplastic Behavior

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TABLE II
EVALUATION OF GRAPHITE/POLYIMIDE LAMINATES AT WRD

Property	Temp (°F)	Resin System							
		RS 6264	RS 6270	RS 6271	SB 703	SB 709	P13N	PI 4707	PI 5077
Resin Content (% by Weight)		32.10	21.10	33.10	36.40	28.20	38.50	33.90	32.10
Specific Gravity		1.47	1.49	1.46	1.43	1.43	1.49	1.50	1.47
Void Content (% by Vol.)		6.40	5.20	6.80	7.70	10.00	3.20	4.00	6.40
0° Flexure (ksi)	RT	118.10	24.70	–	158.10	85.70	212.40	177.90	115.50
	550	88.30	34.30	29.70	81.80	72.00	146.70	125.20	76.90
90° Flexure (ksi)	RT	4.10	5.10	3.40	5.70	2.10	5.80	1.70	0.90
	550	4.60	3.30	3.30	4.30	2.50	4.30	3.40	0.90
Short Beam Shear (ksi)	RT	6.80	9.60	9.50	12.50	5.40	10.70	5.80	6.60
	550	8.70	5.30	5.10	5.80	5.90	5.70	5.10	4.60

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est performance and the highest number indicates superior performance. Using this system, the candidate resins are rated in Table IV. Skybond 703 and PI 4707 were selected for further development and evaluation under this task.

2.2.3 Skybond 703 and PI 4707 Prepreg Development - Prepreg development consisted of the following detailed efforts for each resin system.

(a) Use of analytical techniques for selection of cure cycles.

(b) Evaluation of prepregging method (hot melt vs. solvent coat resin application).

(c) Fabrication of boron and graphite reinforced laminates using selected processing methods.

(d) Evaluation of each material system for mechanical and physical properties.

Material evaluation was to be performed at both WRD and MCAIR.

Initial prepreg development for Skybond 703 and PI 4707 involved the application of infrared spectroscopy (IR), thermogravimetric analysis (TGA) and gas chromatography (GC) to determine the extent of polyimide resin imidization, and volatile release and identification during cure cycle. The analytical data was correlated with prepreg handling characteristics and the resultant laminate properties (using boron or graphite fibers) to help select the optimum prepreg fabrication methods.

The infrared spectrums showed increases in imidization with higher "B" staging temperatures and resultant decreases in the polyamic acid content. However, these spectrums were of minimal assistance since reactions that occur during "B" staging could be masked by spectrums of the resin solvents when using the Beckman IR-7. The TGA plot of weight loss versus increasing temperature (see Figure 1) is helpful in identifying temperatures at which large amounts of volatiles are released. In addition, the outgassing products given off at various temperatures were then identified by gas chromatography. As a result of the above infrared spectrums, TGA plots, and gas chromatography studies "A" and "B" stagings and a cure cycle were selected. The "A" staging is at 195°F for 10-14 hours. The "B" staging is at 200°F for 2 hours. The cure cycle contains a "stop" at 200-225°F for 2 hours followed by heating slowly through 250°F to the cure temperature of 350°F for another 2 hours curing.

The hot melt process was successfully used with both PI 4707 and Skybond 703 resins, with application temperatures on the drum ranging from

TABLE III
MCAIR EVALUATION OF BORON/SKYBOND 703

Property	Test Temperature (°F)	Strength (PSI)	
		Post Cure "C"	Post Cure "A"
90° Flexure	RT	10,000	9,600
Shear	550	6,200	7,040
Interlaminar	RT	12,800	10,700
	550	9,300	9,300

Notes:

Post Cure "C" - 2 hours at 400°F, 2 hours at 500°F, and 2 hours at 600°F.
Post Cure "A" - Heat to 600°F at 1/4 - 1°F/min slow constant rate, Hold at 600°F for 2 hours.

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TABLE IV
RATING OF CANDIDATE POLYIMIDE RESINS

Resin System	Numerical Rating		Total
	Boron/PI	Graphite/PI	
Skybond 703	25	33	58
P13N	17	37	54
PI4707	25	23	48
RS 6264	18	28	46
RS 6270	17	20	37
RS 6271	20	14	34
Skybond 709	18	12	30
PI 5077	–	13	13

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Sample:	X-Axis	Y-Axis	Run No. ____ Date 7-13-70
Skybond 703	Temp. Scale 100°C / Inch	Scale 20 mg / Inch	Operator ____
	Shift 0 Inch	(Scale Setting x 2)	Heating Rate 5 °C / Min
Size 101 mg		Suppression 0 mg	Atm. Vacuum
			Time Constant 1 Sec

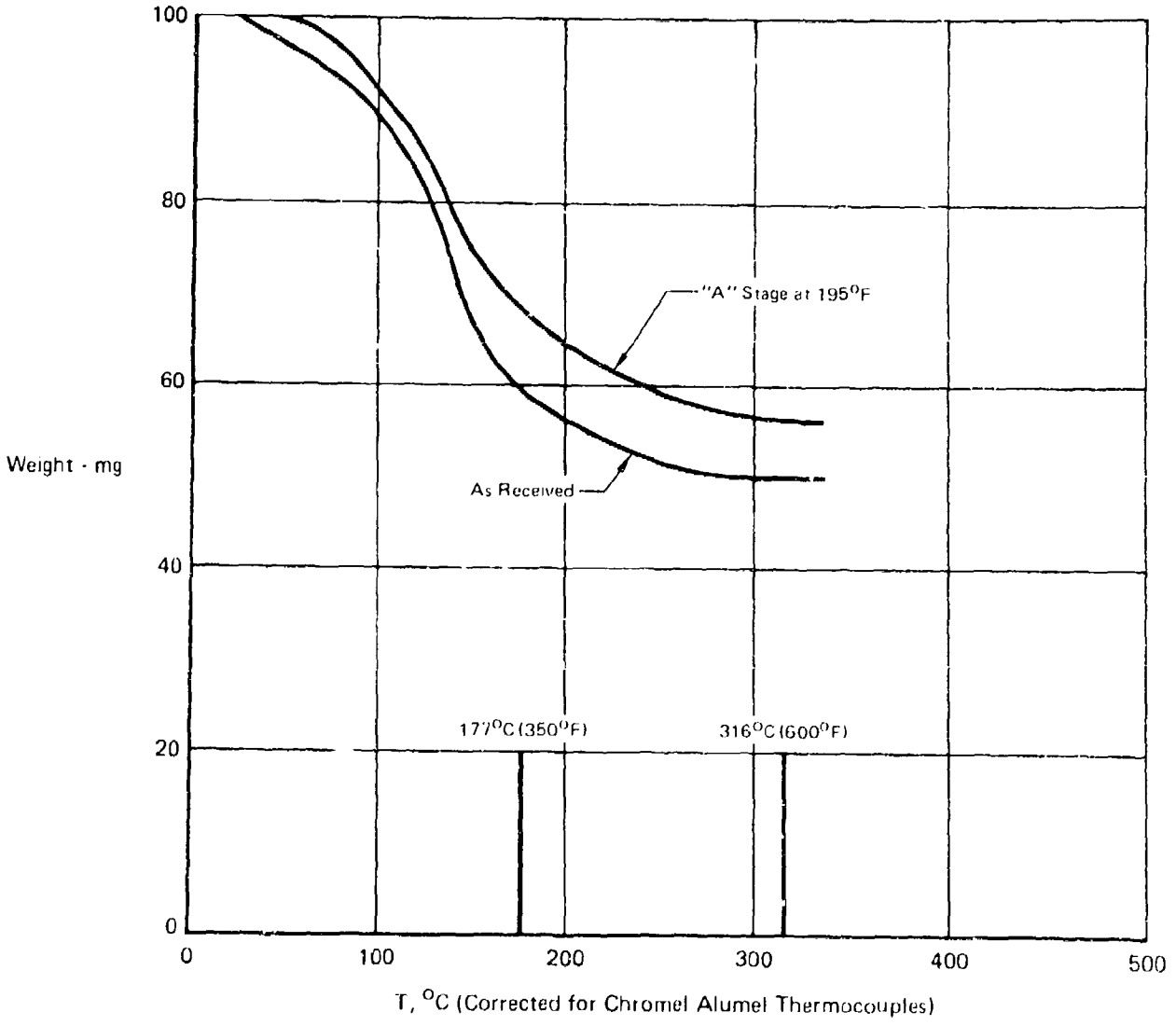

FIGURE 1
TGA CURVES ON SKYBOND 703 POLYIMIDE RESIN

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180°F to 210°F. The hot melt application method is necessary because of the low solvent content remaining in both resins after the "A" staging process. The handling characteristics of the prepregs were satisfactory since tack was apparent when prepreg was pressed against itself. The draping characteristics were also satisfactory so that contoured configurations could be fabricated without difficulty. WRD established boron/SB703 and graphite SB703 laminate cure cycles that resulted in acceptable mechanical properties. The void content was typically 3-5 percent and resin content was 27-35 weight percent.

WRD experience with the PI4707 polyimide resin was less successful. The reproducibility of mechanical properties was a continual problem for both boron and graphite reinforced laminates, and resin precipitation during the cure cycle could not be controlled.

WRD supplied MCAIR with boron/PI 4707 and Modmor II graphite/PI 4707 prepregs for evaluation, and 15 ply laminates were made with each prepreg using the WRD recommended cure cycles. The boron/PI 4707 laminate was excessively thick (.008 in/ply) as a result of very little resin bleed during cure. It also exhibited resin precipitation. The graphite/PI 4707 laminate averaged .0095 - .0103 in./ply (also too thick) and the resin did not wet completely through the graphite tow, with delamination as the result. Neither of the laminates was considered satisfactory for mechanical property testing.

The better performance of the SB703 resin with both boron and graphite resulted in more effort expended on its development than on PI 4707 development. Table V shows a comparison of SB703 and PI 4707 prepreg/laminate properties developed at WRD.

<u>boron/SB703</u> - MCAIR and WRD, however, were not obtaining consistent laminate results with boron/SB703. Whereas WRD reported acceptable mechanical properties and consistent fabrication results, MCAIR did not succeed in fabricating acceptable laminates using material from the identical batch and process cycle. It was suspected that the prepreg was changing with increase storage time at MCAIR even though it was packaged with dessicant in heat sealed plastic bags. To resolve these problems two approaches were investigated. First, an optimum "B" stage had to be established that would sufficiently stabilize the prepreg prior to lay-up. Second, the prepreg itself would be evaluated to determine if a low flow (mini-flow) type of prepreg would perform better than the high flow prepreg.

TABLE V
WRD POLYIMIDE COMPOSITE DATA

Material	0° Flexure (ksi)		90° Flexure (ksi)		Shear (ksi)	
	RT	550°F	RT	550°F	RT	550°F
Boron/Skybond 703	145.6		4.4			
	237.1		5.1		7.5	
	188.2		5.6		7.3	
	245.4		7.5		10.2	
	164.8		4.9		8.4	
	204.6		9.6		11.1	
	216.5		8.6		13.0	
	172.6		7.1		12.8	
	204.6		6.9		11.3	
	161.7	138.1	8.0	4.9	13.5	8.6
	248.1				13.4	
	225.4				14.3	
Boron/PI 4707			6.1		8.1	
	122.7		6.9		8.8	
	271.0		7.7		10.5	
			6.6		6.0	
	204.2		9.9		7.3	
			1.5		3.5	
	190.0		4.9		9.1	
	165.6				8.7	
	0° Flexure (ksi) @ RT		90° Flexure (ksi) @ RT		Interlaminar Shear (ksi) @ RT	
Graphite/Skybond 703	173.0		5.0		10.3	
	151.3		6.7		10.1	
	172.1		6.8		11.3	
	162.4		6.7		12.7	
	160.4		7.2		11.7	
	200.0		10.0		11.2	
Graphite/PI 4707			3.4		6.9	
	165.2		3.2		7.4	
	159.0				5.8	

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Water analysis of the boron/SB703 prepreg by the Karl Fisher method revealed that prepreg "B" staged 1 hour at 200°F (or less) had variable water content vs. time. However, prepreg "B" staged 2 hours or more at 200°F exhibited considerable water content stability. Figure 2 shows a comparison of prepreg water content vs. time. The 2 hour at 200°F "B" stage stabilized the prepreg, but did so at the expense of prepreg handleability. It was decided however, to proceed with Boron/SB703 prepreg "B" staged 2 hours at 200°F at some sacrifice of prepreg tack to obtain the desired reproducibility.

To investigate a mini-flow system, prepreg was manufactured by WRD with 30 percent instead of the normal 38 to 40 percent of total resin solids, and "B" staged 2 hours at 200°F instead of 1 hour at 200°F. It was intended that this prepreg would flow very little, and the resin content would be approximately correct since 28 to 30 percent is required in the cured laminate to obtain a thickness of 5.0 to 5.5 mils/ply. Prepreg "B" staged 1.5 hours at 200°F with 30% resin solids content was also manufactured. Laminates fabricated with prepreg "B" staged 1.5 hours at 200°F did not appear any better than prepreg "B" staged 1 hour at 200°F. Laminates fabricated with prepreg "B" staged 2 hours at 200°F consistently gave acceptable results. There was no evidence of resin precipitation or voids, and there was minimum resin bleed into the bleeder cloth. Table VI summarizes the typical and mechanical properties of laminates fabricated from prepreg "B" staged 2 hours at 200°F.

The results with the mini-flow boron/SB703 prepreg were encouraging and it was selected for further use on this program. The mini-flow prepreg selected was "B" staged 2 hours at 200°F after hot melt prepregging with 30 percent of total weight resin solids. The cure cycle selected was: apply full vacuum and heat to 200°F, hold for 1 hour heat to 250°F, hold for 1 hour and apply 100 psi, heat to 350°F and hold 2 hours. Postcure was a stepped heat up to 600°F with a 6 hour hold at 600°F. It have the most acceptable mechanical properties in addition to consistency of fabrication.

Graphite/Skybond 703 - MCAIR evaluated several batches of Modmor II graphite/Skybond 703 prepreg with the WRD recommended cure cycle. Laminates cured to an acceptable thickness of .007 inch/ply. Mechanical property data was generated at room temperature and 550°F for 0° flexure, 90° flexure and interlaminar shear. The cure cycle selected was:

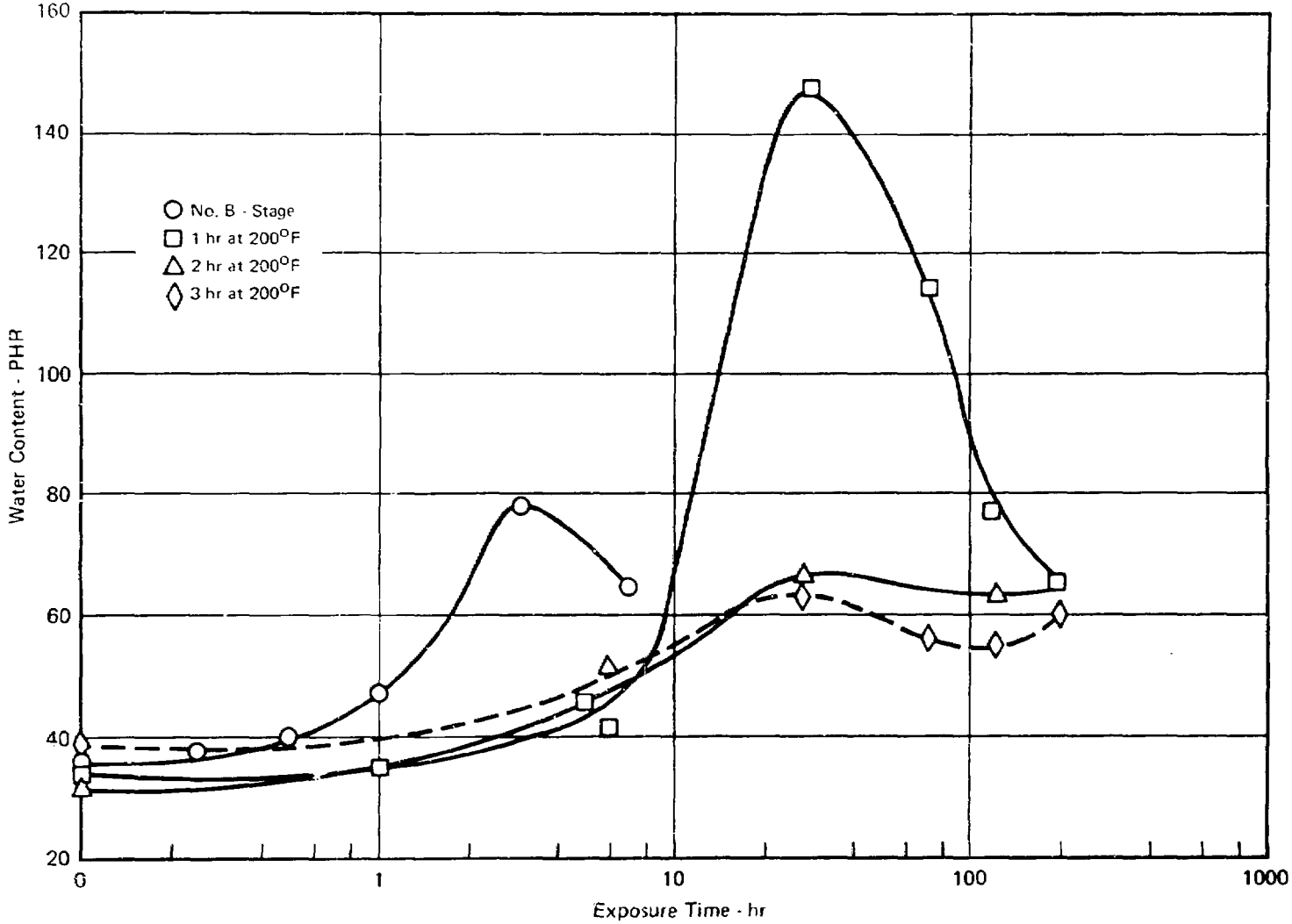

FIGURE 2
EFFECT OF TIME AT ROOM TEMPERATURE ON THE WATER CONTENT OF POLYIMIDE PREPREGS

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TABLE VI
MCAIR/WRD DATA FOR BORON/SKYBOND 703 LAMINATES

Prepreg Resin Solids Content and Remarks		Laminate Resin/Void Content	Test Temp	Average Test Values		
				0° Flex (ksi)	90° Flex (ksi)	Shear (ksi)
30%	Made with 2 hr at 200°F "B" stage prepreg. No resin precipitation.	28.3/2.1	RT	242.1	10.0	12.4
			550	103.0	6.1	10.7
38%	Made with 2 hr at 200°F "B" stage prepreg. No resin precipitation.	37.6/2.4	RT	258.1	9.4	12.7
			550	109.5	6.1	8.9
38%	Made with 2 hr at 200°F "B" stage prepreg. No resin precipitation.	39.5/2.6	RT	–	–	13.2
32%	Made with 2 hr at 200°F "B" stage prepreg. No resin precipitation.	35.3/3.5	550	–	–	8.8
30%			RT	–	–	13.9
			550	–	–	4.0*

*Thermoplastic Behavior

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Heat to 200°F under full vacuum and hold 1 hour.

Heat to 250°F and apply 100 psi.

Continue heating to 350°F and hold 2 hours.

Post cure by heating slowly to 600°F and holding for 6 hours.

Table VII summarizes data generated at MCAIR for graphite/SB703.

TABLE VII
GRAPHITE/SKYBOND 703 MECHANICAL PROPERTY DATA

Panel No.	Test Temp (°F)	0° Flexure (ksi)	90° Flexure (ksi)	Interlaminar Shear (ksi)
1023	RT	105.0	10.4	11.1
	550	25.8	1.8	*
1116	RT	–	10.3	7.3
	550	–	2.1	*
1117	RT	–	6.3	9.6
	550	–	1.8	4.0*
1124	RT	189.0	7.1	9.7
	550	103.0	5.7	5.7

*Thermoplastic behavior

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3.3 Adhesive Development

A MCAIR survey of high temperature adhesives resulted in the selection of Bloomingdale FM-34 and FM-39, DuPont PI 5505, and Narmco Metlbond 586 for program evaluation. All these adhesives, except for the FM-39 Foaming Adhesive, were of the supported film type. These adhesives were evaluated to 550°F for metal-to-metal, skin-to-honeycomb core, and/or edgemember-to-honeycomb core bonding applications. Since it was initially planned to evaluate several 7 ft sandwich beams after a 500 hour soak at 550°F, it was necessary to evaluate adhesives under the same conditions. All subsequent adhesive data is given as an average of at least 5 specimens.

The commercially available supported film adhesives gave satisfactory bonding for metal-to-metal (or metal-to-composite) type applications, but initial skin-to-honeycomb core results showed poor filleting. As a hedge

against continued poor performance of the commercially available polyimide film adhesives, MCAIR and WRD initiated development of a polyimide film adhesive based on the SB703 resin used in the prepreg studies.

2.3.1 Honeycomb Core Bonding - FM-34, Metlbond 840, PI5505 and the MCAIR SB703 were evaluated for honeycomb core bonding capability. Figure 3 shows the lack of filleting of the commercial adhesives, and the improved filleting obtained with the SB703 adhesive developed at MCAIR. The commercial adhesives were cured per the vendor's recommended cure cycle and with the appropriate vendor recommended primer. The FM-34 evidenced slightly better filleting Metlbond 840 or PI5505, but still, when used to bond HRH-327 polyimide core plate shear specimens, it resulted in premature failures in the adhesive rather than in the core as expected. Flow studies were performed with both the commercial adhesives and MCAIR developed SB703 adhesive to determine when optimum flow occurs. Utilizing this information, the cure cycles for the candidate adhesives were modified and an attempt was made to bond aluminum sheet to aluminum honeycomb to investigate flow and filleting. The MCAIR SB703 polyimide adhesive and FM-34 were then selected for honeycomb sandwich panel bonding studies. The MCAIR SB703 adhesive had the best filleting characteristics but its void content was excessively high and resulted in lower properties. At this stage, development of the SB703 adhesive was transferred to WRD from MCAIR where more adhesive manufacturing technology was available. It was then designated WRD SB703 and was used in conjunction with either BR-34 or an SB703 resin primer. Additional studies of the FM-34 adhesive resulted in improved flow properties through cure cycle modification and changing the BR-34 primer application method.

The BR-34 primer was selected based on lower apparent void content and better honeycomb core wetting characteristics. Flatwise tension specimens were fabricated with FM-34 and WRD SB703 (specimens are shown in Figures 4 and 5) and tested at room temperature and at 550°F with either no exposure or after a 500 hour soak at 550°F. Table VIII shows the values obtained. The FM-34/BR-34 system was selected for use in the program because it exhibited adequate bond strengths and is a readily available commercial product.

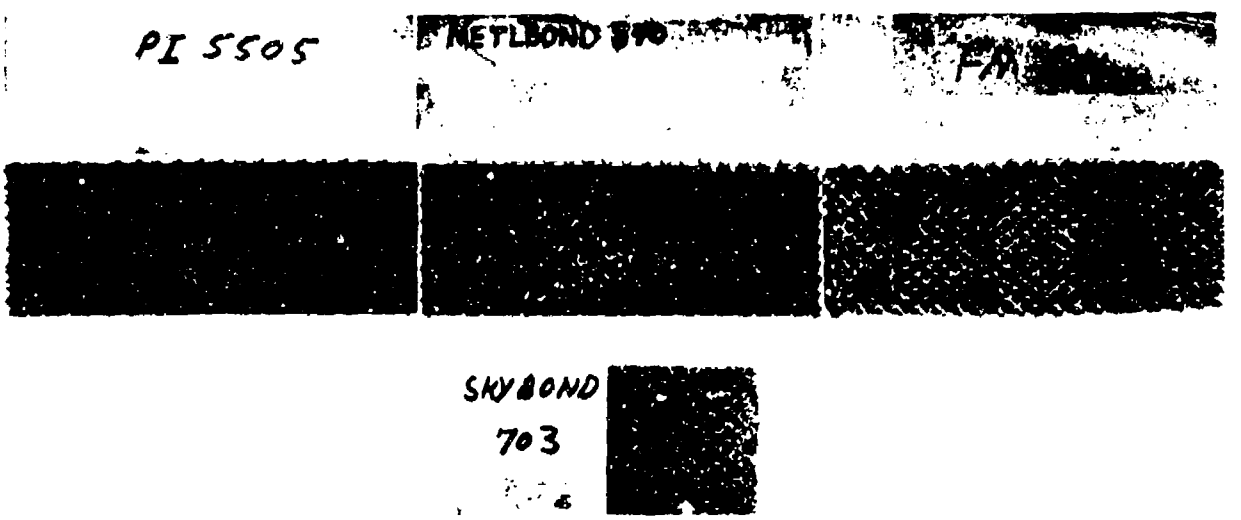

FIGURE 3
SANDWICH BONDING COMPARISON OF POLYIMIDE ADHESIVES
(Before Improvements)

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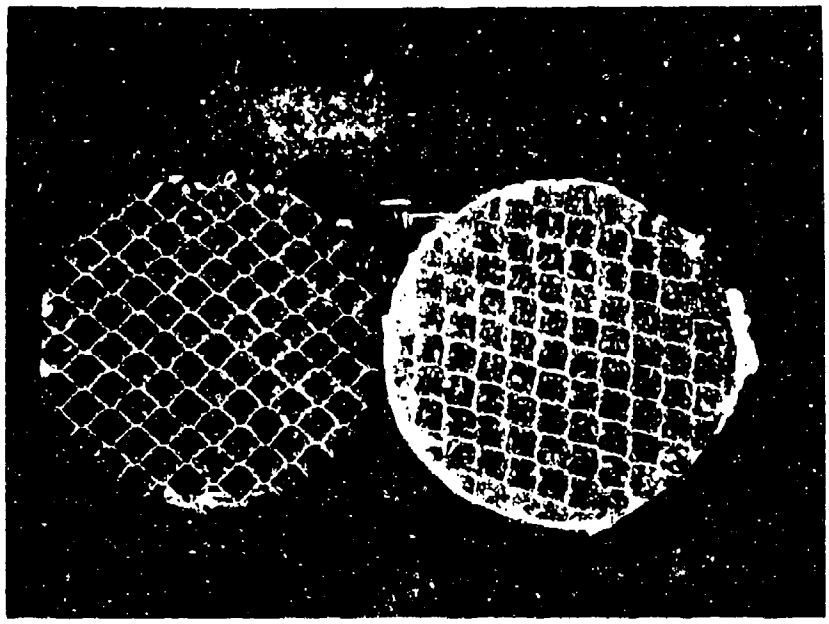

FIGURE 4
SB703 POLYIMIDE ADHESIVE FLATWISE TENSION SPECIMEN

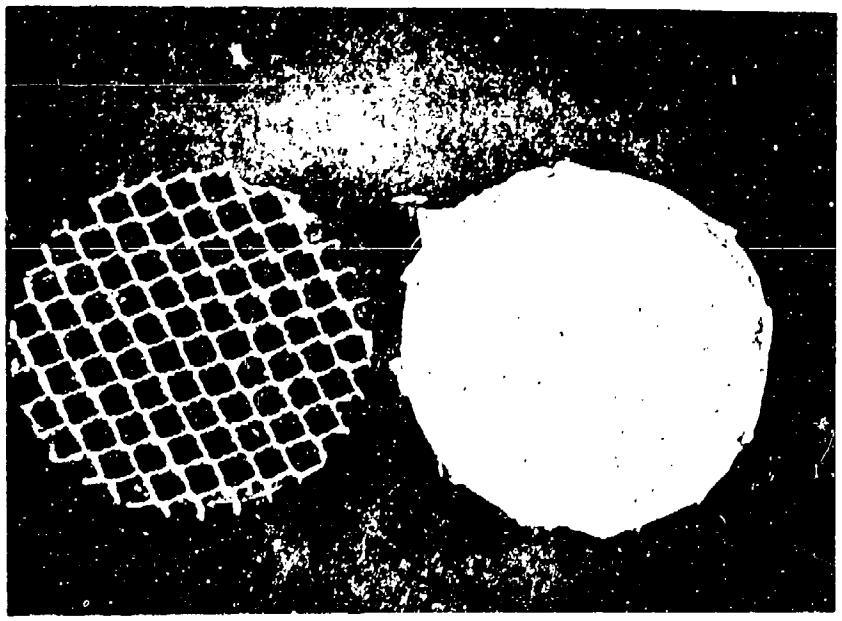

FIGURE 5
FM-34 FLATWISE TENSION SPECIMEN
(After Improvements)

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TABLE VIII
POLYIMIDE ADHESIVE HONEYCOMB FLATWISE TENSION DATA

Adhesive System	Exposure Condition	Flatwise Tension (psi)	
		RT	550°F
WRD SB703/SB703 Primer	None	745	401
	500 hrs @ 550°F	318	287
WRD SB703/BR-34 Primer	None	797	385
	500 hrs @ 550°F	427	328
FM-34/BR-34 Primer	None	568	308
	500 hrs @ 550°F	174	275

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2.3.2 Metal-to-Metal Adhesive - Primary and secondary bonded boron/polyimide - titanium double lap shear specimens illustrated in Figure 6 were evaluated to 550°F for selection of an (metal-to-metal type skin to edgemember) adhesive bonding. FM-34, Metlbond 840, PI5505 and the MCAIR SB703 were all selected as candidates for evaluation. The primary bonded specimens consisted of the test adhesive sandwiched between boron/polyimide prepreg and 6Al-4V titanium sheet, and cured per the boron/polyimide prepreg cure cycle. The secondary bonded specimens had the test adhesives bond boron/polyimide laminates to 6Al-4V titanium using the adhesive cure cycle. All specimens were post cured to 600°F using the boron/polyimide cycle or the commercially recommended adhesive cycle, depending on whether the specimens were primary or secondary bonded.

Initial screening consisted of tests at room temperature and 550°F. Adhesives that successfully passed these tests were tested further by exposing specimens to 100 hours or 500 hours at 550°F prior to test at 550°F. Table IX

TABLE IX
DOUBLE LAP SHEAR STRENGTH OF CANDIDATE POLYIMIDE ADHESIVES

Adhesive	Type of Bond	RT	550°F (30 min)	550°F After 100 hrs @ 550°F	550°F After 500 hrs @ 550°F
Metlbond 840	Secondary	4325	2135	2750	2580
	Primary	3820	2055	2015	1975
FM-34	Secon[illegible]	4910	1525	2370	2380
	Primary	1505	870	–	–
PI 5505	Secondary	2750	1930	–	–
MCAIR SB 703	Secondary	3450	1675	–	–
	Primary	2320	1205	–	–

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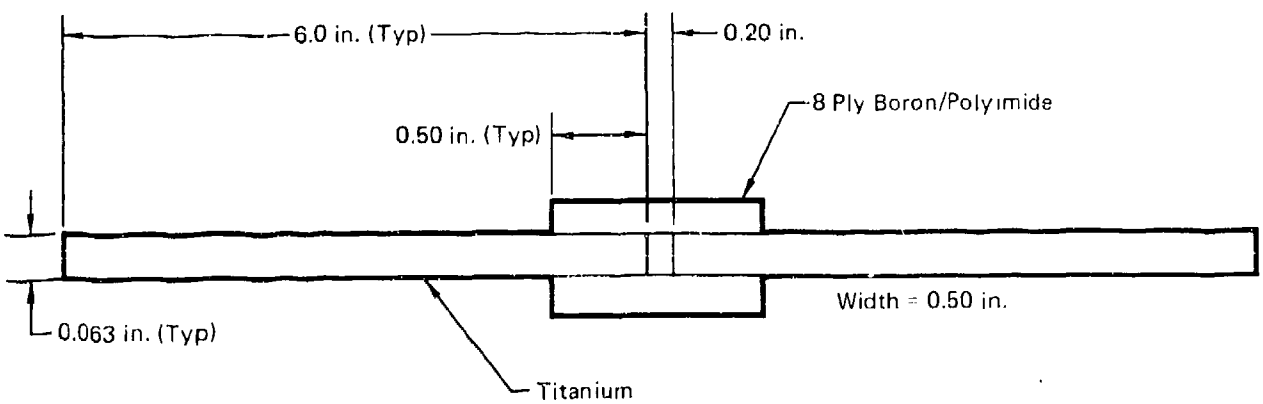

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FIGURE 6
DOUBLE LAP SHEAR SPECIMEN

lists the data developed for all the adhesives. The Metlbond 840 adhesive was superior to the other candidates and was selected for further use on the program. The FM-34, although not quite as effective, was considered the back-up adhseive for secondary bonding applications.

T-Peel specimens per Federal Specification MMM-A-132 were fabricated and tested using the three commercial adhesives plus the SB703 polyimide adhesive. The adhesive was sandwiched between two 0.010 inch thick by 1.0 inch wide by 12.0 inch long sheets of 6Al-4V titanium. FM-34 and Metlbond 840 both resulted in low values at 75°F and 550°F when compared to currently used high temperature epoxy adhesives. Values at 75°F were 2.0-4.2 lbs/in. (peel initiation) and 1.5-1.7 lbs/in. (peel continuation). At 550°F typical values were 1.8-3.6 lbs/in. (peel initiation) and 0.5-1.2 lbs/in. (peel continuation). Three specimens were tested for each adhesive for each condition.

3.5.3 Core Splice Adhesive - The foaming adhesive candidate for core splice applications was Bloomingdale FM-29. A potential back-up was multiple layers of FM-34 or Metlbond 840 supported film adhesives. A beam shear specimen, shown in Figure 7, was used to evaluate the FM-29 foaming adhesive.

The first beam shear tests were performed at R.T. and 550°F with 3.5 lb/ft^3 titanium honeycomb core (spliced as shown in Figure 7), titanium skins, and Bloomingdale FM-29 polyimide core splice adhesive. Half of the six specimens had an 0.010 inch thick titanium shim in the bondline. These were included to simulate the more difficult edgemember bond of honeycomb core to titanium spar/ribs. The vendor's recommended cure cycle was followed and adhesive expansion (~ 100 percent) was satisfactory. The skins were then bonded to the spliced honeycomb with FM-34. In all cases the honeycomb core failed rather than the FM-29. Another series of tests were performed using the higher strength HRH-327 4.5 lb/ft^3 honeycomb core. These tests also caused failure of the honeycomb core rather than the FM-29 as desired. Finally, 7.0 lb/ft^3 stainless steel honeycomb core was used so that the shear strength of the FM-29 could be established. Table X summarizes the data generated for FM-29 evaluation.

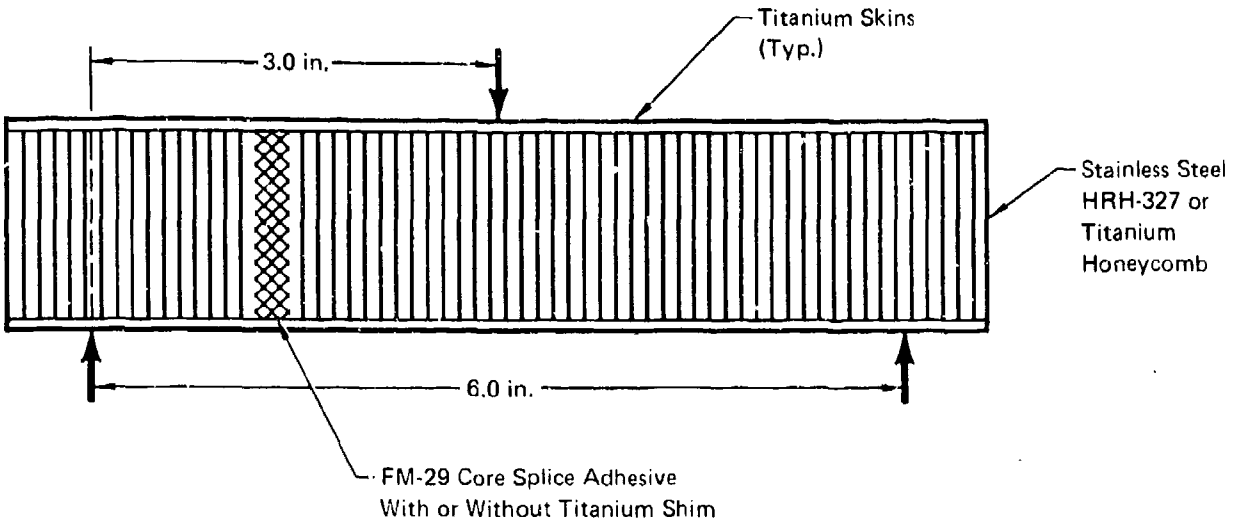

FIGURE 7
CORE SPLICE ADHESIVE BEAM SHEAR SPECIMEN

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TABLE X
FM-29 SHEAR STRENGTH DATA

Honeycomb Core	Exposure Conditions	Beam Shear (PSI)	
		R.T.	550°F
*Titanium w/Shim	None	126	82
*Titanium w/o Shim	None	126	81
*HRH-3[illegible] Polyimide	None	296	240
	100 hr @ 550°F	300	228
	500 hr @ 550°F	263	220
Stainless Steel (12 lb/ft^3)	None	377	291

*All failures in honeycomb - not FM-29.

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Honeycomb Core Evaluation

Four types of high temperature core were evaluated in this program:

1. 301 stainless steel honeycomb core (4.2 lb/ft^3)
2. PH 15-7 Mo stainless steel honeycomb core (3.5 lb/ft^3)
3. Ti-75A titanium honeycomb core (3.5 lb/ft^3).
4. HRH-327 polyimide honeycomb core (4.5 lb/ft^3).

Flatwise compression tests were performed at room temperature and at 550°F after 30 min., 100 hour and 500 hour soaks at 550°F as shown in Table XI. Plate shear tests have been performed for the same conditions. The long term tests were performed only with honeycomb core materials that looked promising after initial room temperature and 550°F tests.

The initial HRH-327 tests used a core that had not been post cured prior to testing. However, on subsequent tests all HRH-327 honeycomb core was post cured to 600°F prior to testing. All honeycomb panels for this evaluation were bonded with FM-34/BR-34. Modifications to the adhesive and primer application procedures (as previously discussed) provide adequate honeycomb core bonding. There were no adhesive failures while performing honeycomb core tests.

Table XI shows the test data generated for all the candidate honeycomb core materials selected for the program. Figure 8 provides a structural efficiency comparison of the two leading candidate materials, HRH-327 and PH15-7Mo (TH1050).

These data indicate the structural advantages of HRH-327 honeycomb core in both core shear and flatwise compression. The core shear data is the more significant for structural considerations. The HRH-327 is also superior from a machining viewpoint. It can be machined with the same type equipment as aluminum honeycomb core, while stainless steel and titanium honeycomb require special tooling and/or machining procedures. For these reasons HRH-327 fiberglass/polyimide honeycomb core has been selected for use in this program.

TABLE XI
MECHANICAL PROPERTY DATA FOR CANDIDATE
HONEYCOMB CORE MATERIALS

Honeycomb Core Material	Density (lb/ft^3)	Flatwise Compression (psi)				Core Shear (psi)			
		RT	550°F After 30 min	550°F After 100 hr at 550°F	550°F After 500 hr at 550°F	RT	550°F After 30 min	550°F After 100 hr at 550°F	550°F After 500 hr at 550°F
HRH-327	4.5	375	206	220	180.0	403	240	386	307
PH15-7Mo (TH1050)	3.5	210	190	235	183.8	158	90	115	112
301 St. Steel	4.2	149.4	125.4	–	–	96	53	–	–
Ti-75A Titanium	3.5	193.2	144.8	–	–	147.3	80.4	–	–

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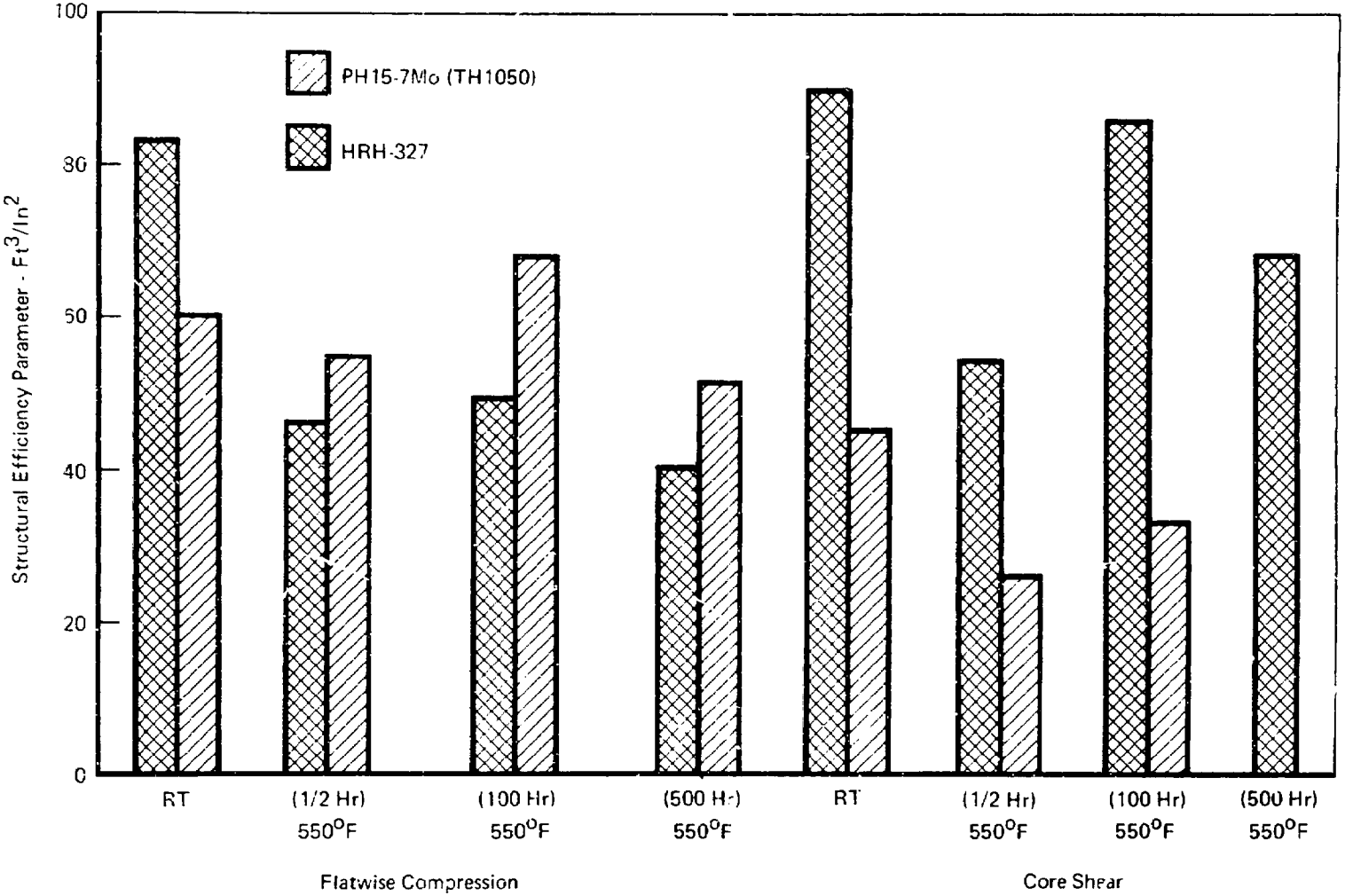

FIGURE 8
STRUCTURAL EFFICIENCY COMPARISON OF CANDIDATE HONEYCOMB CORE

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3. FABRICATION SCALE-UP AND EVALUATION

3.1 Introduction

The objective of this task was to demonstrate that large scale structural elements and panels could be fabricated for evaluation. Fabrication scale-up and evaluation were planned for the following items:

(1) Optimization of polyimide composite lay-up procedure and cure cycle.

(2) Boron/polyimide-titanium splice joint section fabrication and test to simulate both the stabilator and edgemember attachment areas, the primary load transfer area.

(3) Graphite/polyimide channel element for the demonstration of substructure (spars and ribs) fabrication capability.

(4) Joint specimens utilizing titanium boron/polyimide and graphite/polyimide to evaluate adhesive bonding, mechanical fastening, and a combination thereof for verification of joint designs.

(5) Large area polyimide composite laminates to demonstrate confidence in fabricating large area panels and to provide design allowable specimens.

(6) Large area polyimide composite honeycomb sandwich panel to evaluate size effects and process control for full depth honeycomb fabrication.

(7) Polyimide composite sandwich beams which encompass the technology developments of items (1), (2), and (6).

Considerably more effort was required to scale-up the boron/SB703 prepreg for fabrication of thick (40 ply) laminates, item (1), than was originally planned. As a result of this delay plus the lead time required for component fabrication, the Stabilator Torque Box component (which requires 40 ply fabrication technology) was replaced with an F-15 Wing Compression Panel requiring, at most, 20 ply fabrication technology. Fabrication Scale-Up and Evaluation items (2), (4), (5) and (7) were deleted as they were no longer necessary. Development of 40 ply boron/SB703 fabrication technology continued along with items (3) and (6) above to complete the requirements of this task necessary to support the fabrication of both the F-4 rudder and wing compression panel.

Difficulties were encountered with "B" stage temperature control when the sheet size was increased from 12 in. x 40 in. used in Task I to 15 in. x 62 in. Also prepreg variability as evidenced by poor thickness/ply control and

delamination of 40 ply laminates was encountered. Changing the "B" stage, autoclave cure and post cure cycles resolved these problems after considerable effort. Results showed that small 40 ply laminates can be made with the selected process that have shear strengths of 12.3 ksi at room temperature and 6.5 ksi at 550°F. Boron/SB703 15 ply laminates give shear strengths of 13 - 16 ksi at room temperature and 8.6 - 10.2 ksi at 550°F using the same processing cycle. Scale-up to large panel sizes has not been demonstrated.

The graphite/SB703 prepreg system scaled up with minimum difficulty. Prepreg formability was improved with a light coat of solvent applied immediately prior to lay-up of spar/rib configurations. Graphite/SB703 mechanical properties were satisfactory with 0° tension strength of over 200 ksi, and 90° tension strain well above 4,000 microinches/inch.

A boron/SB703 skin - HRH-327 polyimide fiberglass honeycomb core sandwich panel was bonded with FM-34 to demonstrate sandwich panel bonding capability. The boron/SB703 skins used were very porous resulting in premature failure of the test specimens. However, the adhesive bonding passed NDT and visual inspection after dissection, and the bond line did not fail in any of the tests. As a result, the adhesive bonding procedures were deemed acceptable.

3.2 Boron/SB703 Scale-Up and Evaluation

Efforts in this area were directed at resolution of problems that occurred during scale-up of prepregging operations and processing difficulties experienced with 40 ply laminates. The effort is broken down into the following areas:

- o Prepreg scale-up
- o Material/process optimization
  - (a) Analytical techniques for material/process control
  - (b) Optimum prepreg resin content
  - (c) Processing cycle modifications.
- o Fabrication scale-up.

Two overall considerations in this effort were to: (1) establish fabrication procedures that are production oriented (cycle time, temperature, pressure, etc.), and (2) obtain mechanical properties comparable to those of boron/epoxy at room temperature, with a high retention at 550°F.

3.2.1 Prepreg Scale-Up - Boron/SB703 was used initially for fabrication of a 3 in. x 6 in. x 15 ply and a 6 in. x 12 in. x 44 ply stepped splice plate specimen similar in cross-section to the stabilator torque box skin root splice. These panels were cured with the following cure cycle:

- o Apply full vacuum and heat to 200°F
- o Hold at 200°F and then heat to 235°F
- o Hold at 235°F, apply 100 psi and then heat to 350°F for a 2 hour hold
- o Cool down under pressure
- o Post cure 2 hours at 400°F, 2 hours at 450°F, 2 hours at 500°F, 2 hours at 550°F, and 6 hours at 600°F.

The prepreg was from batch 121570B-1. The 44 ply panel was delaminated, and both the 15 ply and 44 ply panels were badly precipitated. The prepreg was analyzed for resin, volatile, and water content for comparison with a previously successfully used batch (112970B). That comparison is given in Table XII.

TABLE XII
MCAIR PREPREG BATCH ANALYSIS

Batch Designation	Total Resin (Solids + Volatiles) –% of Prepreg	Resin Solids –% of Prepreg	Resin Volatiles		Water-PHR (Parts per Hundred of Resin Solids)
			% of Prepreg	% of Total Resin	
112970B	38.0	28.3	9.7	24.5	4.2
121570B-1	34.6	23.8	10.8	31.2	6.4
121570B-2	34.6	25.6	9.0	29.6	5.5
122170B-1	39.6	28.8	10.8	28.0	5.0
122170B-2	39.6	27.9	11.7	29.5	5.4
122970B	34.5	23.8	10.7	31.2	6.5

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The volatile percent of total resin was much higher for the new batches (28.0 - 31.2%) compared to the 24.5% of batch 112970B. Investigation of the prepreg manufacturing operation revealed a change in temperature control capability during the "B" stage operation. The "B" stage operation had previously been performed in a small laboratory oven (capable of holding

[illegible] in. x 40 in. sheets) at 200 ± 2°F. When the sheet size was increased to [illegible] in. x 60 in., a larger oven was required. The large oven operated at 195 ± 9°F when set at 200°F. This change of "B" stage temperature resulted in less advancement of the resin and higher volatile retention in the prepreg.

A series of 16 ply panels were fabricated at WRD to substantiate these findings. "B" stage cycles evaluated were: (1) 200 ± 2°F for 2 hours, (2) 195 ± 9°F for 2 hours, and (3) 213 ± 10°F for either 1, 2 or 3 hours. The results are given in Table XIII which compares the visual appearance and shear strength of several test panels. The desired thickness for these panels was 5.0 - 5.6 mils/ply because of stabilator torque box skin splice plate tolerances, since the plans were to utilize existing boron/epoxy tooling.

TABLE XIII
COMPARISON OF LAMINATES FABRICATED AT WRD

Laminate Number	Prepreg Conditions	Remarks	Shear Strength (ksi)	
			RT	550
010671D	2 hrs @ 195 + 9°F	Proper thickness. Precipitated.	13.3	10.6
010671E	2 hrs @ 200 ± 2°F	Too thick. Good appearance.	13.6	11.3
010671F	2 hrs @ 195 ± 9°F	Too thick. Precipitated.	No test	No test
010671G	6 hrs @ 195 ± 9°F	Voids. Too thick.	12.9	11.6
010771B	2 hrs @ 213 ± 10°F	Too thick. Good appearance.	14.6	10.6
010771C	3 hrs @ 213 ± 10°F	Too thick. Excellent appearance.	16.8	8.6
010771F	6 hrs @ 195 ± 9°F	Slightly thick. Mild precipitation.	15.0	6.7

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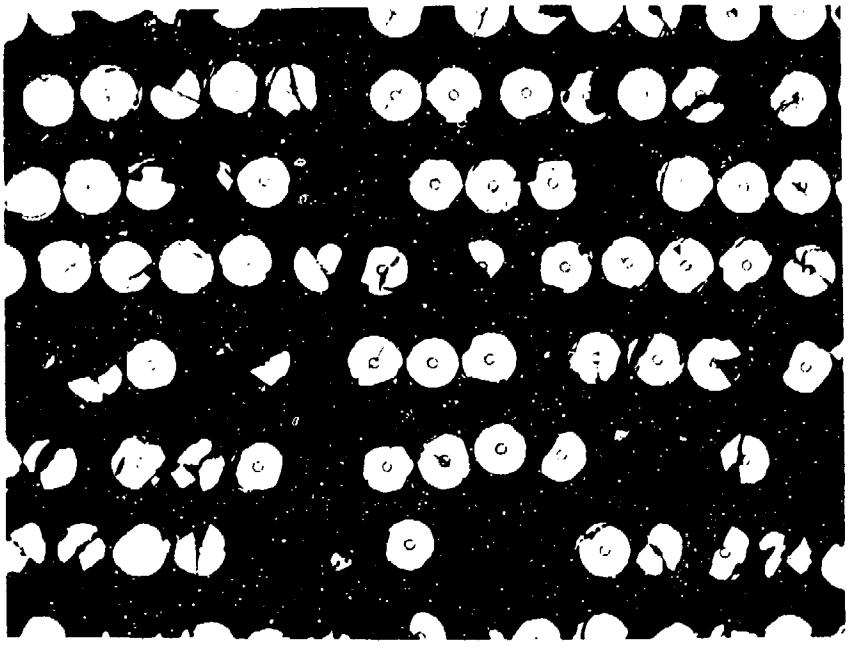
As a result of this study, it was determined that tight control of "B" stage temperature was necessary. The tightest temperature control (± 1-2°F) was possible in a 12 ft. x 12 ft. oven at MCAIR, and it was decided to "B" stage the prepregged material at MCAIR. Individual prepreg sheet packaging (with dessicant) in seal plastic bags proved satisfactory for the "A" staged prepreg during shipment and storage at MCAIR. It was then decided that all future material would be "B" staged at MCAIR to maintain the required temperature control.

3.2.2 Material/Process Optimization - After resolution of the "B" stage difficulties, 28.0% resin solids content prepreg was used to fabricate several 15 and 40 ply laminates. Two additional problems were readily apparent. First, the laminate thickness/ply range of .0057 - .0072 inches encountered was unacceptable as previously mentioned for the planned stabilator skin splice plate. Prepreg that started with 28% resin solids resulted in laminates of 32 - 34% apparent resin solids content. First the thickness/ply problem was the result of solvents (e.g., n-methyl pyrrolidone) retained after the cure/post cure cycle. The titanium splice plate required .0050 - .0056 inches/ply to meet tooling requirements. Second, the majority of 40 to 44 ply specimens delaminated after either the cure or post cure cycle.

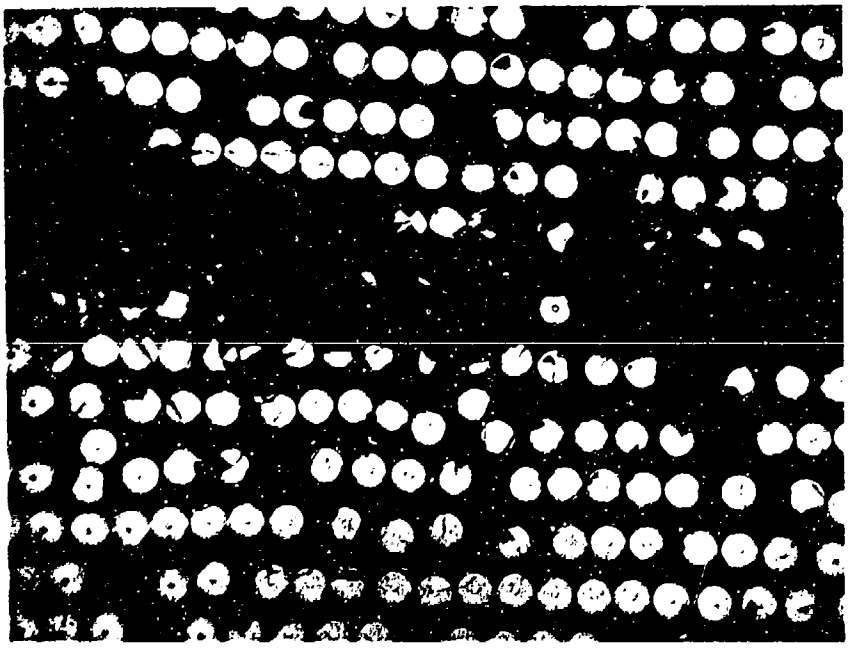
Figure 9 shows a comparison of laminates fabricated during initial scale-up of boron/SB703 prepreg. "A" depicts a high void content laminate, "B" shows a delaminated laminate, "C" is a highly precipitated through low void content laminate, and "D" is considered optimum for meeting the requirements of this program. As the data of Table XIII shows, there is no clear indication that static properties are seriously affected by resin precipitation; however, effects have not been thoroughly documented and the material suppliers strongly recommended against a precipitated resin condition.

Three different approaches were considered to resolve these two problems:

- Analytically evaluate the polyimide resin and prepreg throughout the manufacture cycle at WRD and MCAIR to select approximate material controls
- Vary the resin solids content of the prepreg so that the thickness/ply of the cured laminate would be in the acceptable range of 0.0050 - 0.0056 in.



"A" - High Void Content



"B" - Delaminated

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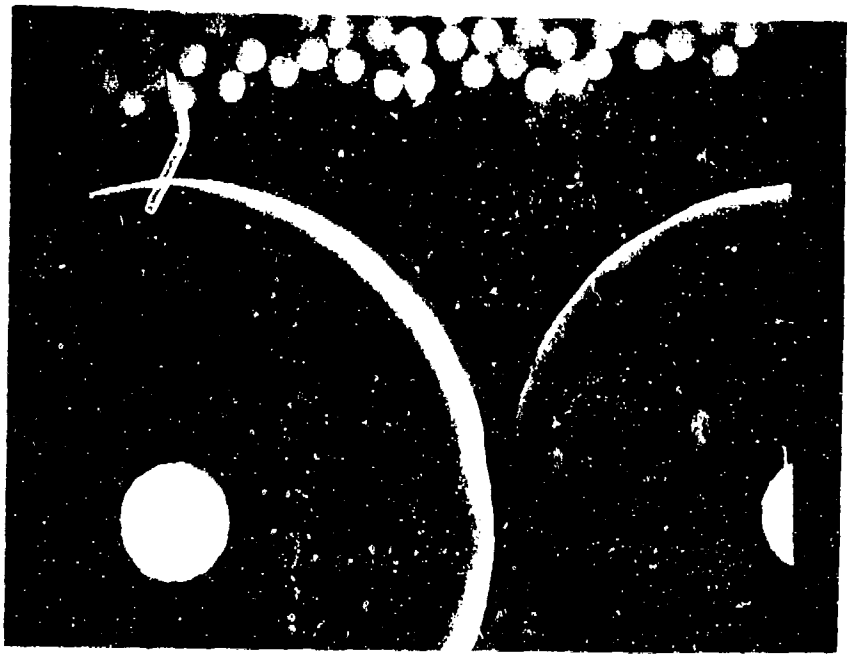
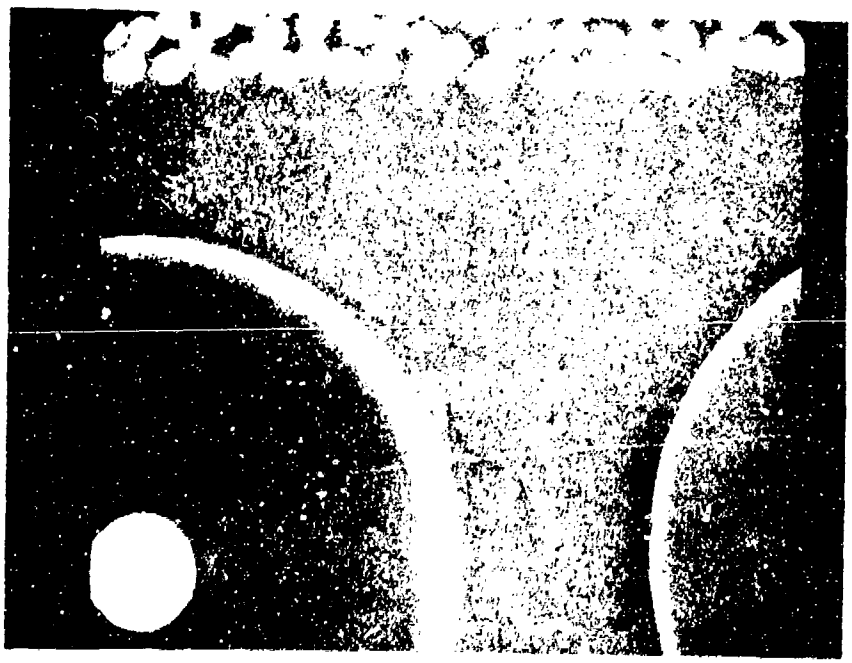
FIGURE 9
VISUAL COMPARISON OF VARIOUS TYPES OF LAMINATES



"C" - Precipitate Resin



"D" Vitreous Resin

FIGURE 9
COMPARISON CLOSE-UP OF PRECIPITATED AND VITREOUS POLYIMIDE RESIN (Continued)

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- Alter the processing cycles ("B" stage, cure, and post cure) to eliminate the delamination problem and establish satisfactory properties for 40 to 44 ply laminates.

3.2.2.1 Analytical Techniques for Material/Process Control - This effort was directed at determining how the various ingredients in the polyimide prepreg resin affected the laminate processing and which ingredients would subsequently require improved controls.

Three different approaches were investigated:

- Gas chromatography
- Infrared spectroscopy
- Thermal-gravimetric analysis with mass spectrometry.

In general, the gas chromatography tests revealed that detailed knowledge of prepreg constituents and their ratios was of little value in resolving the processing problems. Only the total volatile content gave indications of a value to control the prepreg. Infrared spectroscopy and thermogravimetric analysis with mass spectrometry were helpful in identifying degree of resin cure vs. cure cycle and volatile release patterns during critical portions of the cure cycle (between 250°F and 350°F).

Gas Chromatography - Detailed gas analysis of the prepreg resin was performed throughout the processing cycle at WRD and MCAIR. Variables recorded were resin solids, resin volatiles, and volatile constituents - water, ethanol and n-methyl, pyrolidone. Basically, the gas analysis revealed that the starting resin, as supplied by Monsanto to WRD, was well controlled insofar as resin solids, resin volatiles, and volatile constituent percentages were concerned. The material did change during the resultant processing, and these changes were detected and recorded by the gas analysis. Variations occurred in total resin volatile, water, ethanol, and n-methyl pyrrolidone contents. A sampling of the data gathered is presented in Table XIV. The "A" stage cycle used for all samples in Table XIV is 195°F for 10 to 14 hours at full vacuum. The "B" stage cycle used was 2 hours at 200°F. This evaluation was performed on 12 separate WRD batches of boron/SB703 prepreg. Attempts to correlate this data with predictions of laminate acceptability were inconclusive. Other conclusions possible from the data are:

- The only prepreg variable that appears to influence laminate processability is total resin volatile content.
- "B" stage temperature cycles did not significantly alter the resin volatile content.

TABLE XIV
PREPREG ANALYTICAL TESTS
RESIN BATCH ANALYSIS

WRD Batch 115								
	WRD Analysis				MCAIR Analysis			
	As Rec'd. at WRD	"A" Stage Resin	"A" Stage Prepreg		"A" Stage Prepreg		"B" Stage Prepreg	
			26%	28%	26%	28%	26%	28%
Resin Solids %	50.1	57.0	25.6	29.4	24.2	26.6	24.0	26.3
Resin Volatiles %	49.9	43.0	40.0	39.6	40.3	43.0	32.4	32.8
NMP % of Volatiles	70.4	77.5	73.0	73.4	65.8	62.7	65.4	62.4
Ethanol % of Volatiles	25.5	20.0	21.2	20.9	22.1	21.4	23.5	24.2
Water % of Volatiles	–	2.4	5.8	5.7	4.9	5.7	3.2	2.8

WRD Batch 116					
	WRD Analysis		MCAIR Analysis		
	As Rec'd. at WRD	"A" Stage Resin	"A" Stage Prepreg	"B" Stage Prepreg (2 hr)	"B" Stage Prepreg (3 hr)
Total Resin		37.8	37.8	34.1	29.3
Resin Solids %	50.1	26.0	26.7	26.9	26.2
Resin Volatiles %	49.9	42.2	40.5	30.1	33.9
NMP % of Volatiles	70.4	79.4	61.4	61.0	57.0
Ethanol % of Volatiles	25.5	17.3	22.1	26.3	23.7
Water % of Volatiles	4.9	3.3	5.0	3.2	3.6
Prepreg Volatiles %			15.3	10.3	10.0

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- The water content of the prepreg was not a significant prepreg control factor insofar as curing operations are concerned, although it did affect prepreg handleability.
- The ratio between, and total amount of, ethanol and n-methyl pyrolidone in the volatile content did not have a clear effect on laminate processing.
- Volatile release from the laminate during cure was not significant until temperatures over 235°F at full vacuum were reached.

Gas analysis at MCAIR was performed on the equipment shown in Figures 10 and 11.

Infrared Spectroscopy - Improved infrared spectroscopy analysis methods were utilized for polyimide resins, resulting in definition of degree of imidization from "A" stage through post cure (without interference from n-methyl pyrrolidone peaks),and volatile content as a function of cure cycle.

Figure 12 shows several of the spectrograms developed for boron/SB703. As can be seen, the relative quantity of volatiles (ethanol and n-methyl pyrrolidone)does not decrease until the temperature is increased from 275°F to 340°F under full vacuum. During the "B" stage operation, infrared spectrograms indicate that the prepreg is changing primarily in degree of imidization, not volatile content. The imide ring formation is indicated by the arrow labeled No. 1, while the n-methyl pyrolidone is designated No. 2 and ethanol is No. 3.

From this data, four conclusions are made. First, the volatile content of the prepreg is controlled by the "A" stage operation. Second, the "B" stage cycle advances the imidization cycle to control the resin flow during cure. Third, volatile removal from the laminate occurs at temperatures above 250°F, probably in the range of 275°F to 320°F. Fourth, it is confirmed that imidization is continuing into the post cure cycle at 600°F. This information is useful in selecting a cure cycle for this material.

Imidization advancement data is presented in Figure 13, and it reveals that the bulk of polymerization occurred between 275°F and the end of the 2-1/2 hour hold at 340°F. In addition, it can be seen that polymerization continued at a nearly constant rate during the post cure. The fact that a constant absorbance value was never reached indicated that longer post cure periods would be required to achieve maximum polymerization.

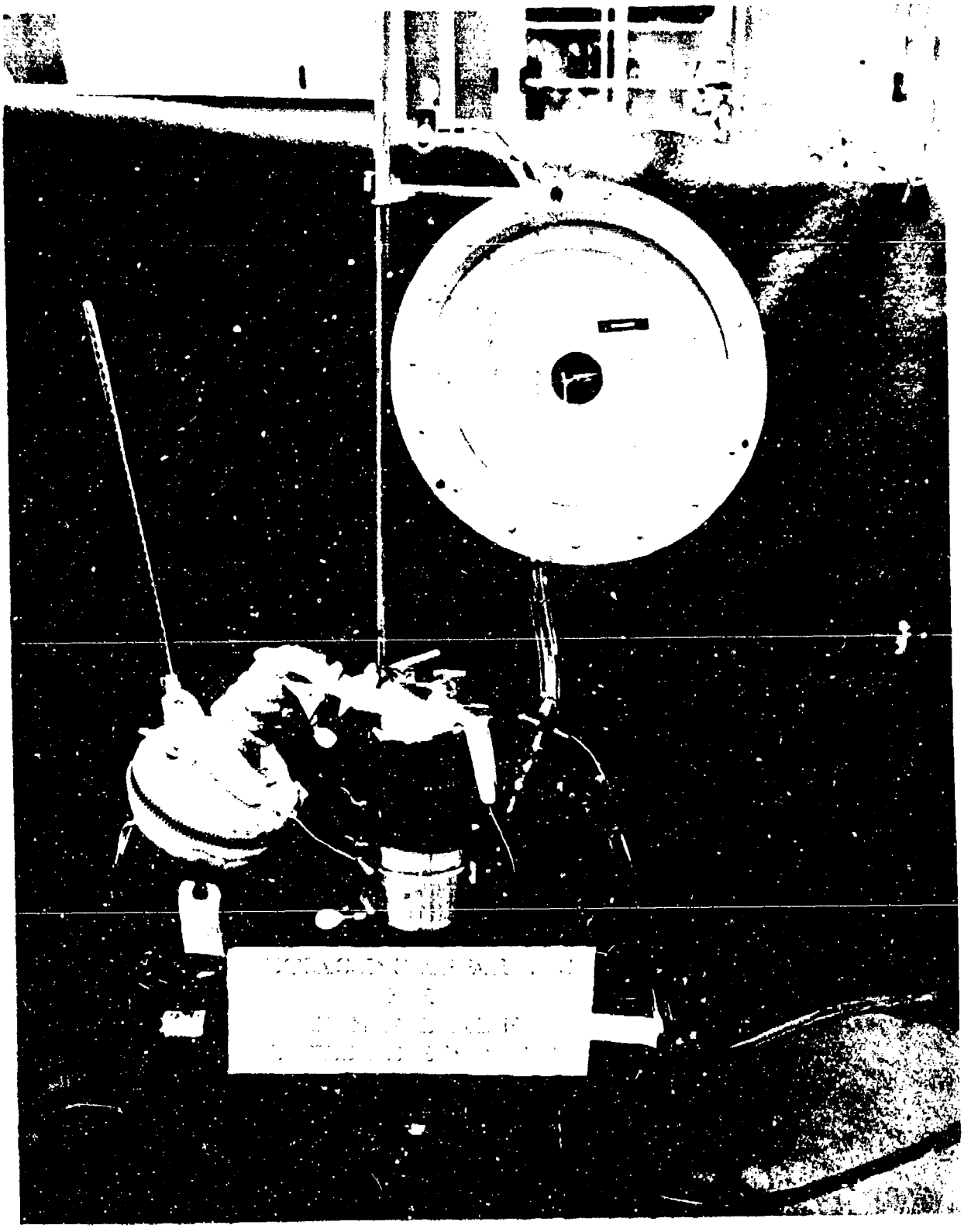

FIGURE 10
PREPREG GAS VOLATILIZATION EQUIPMENT

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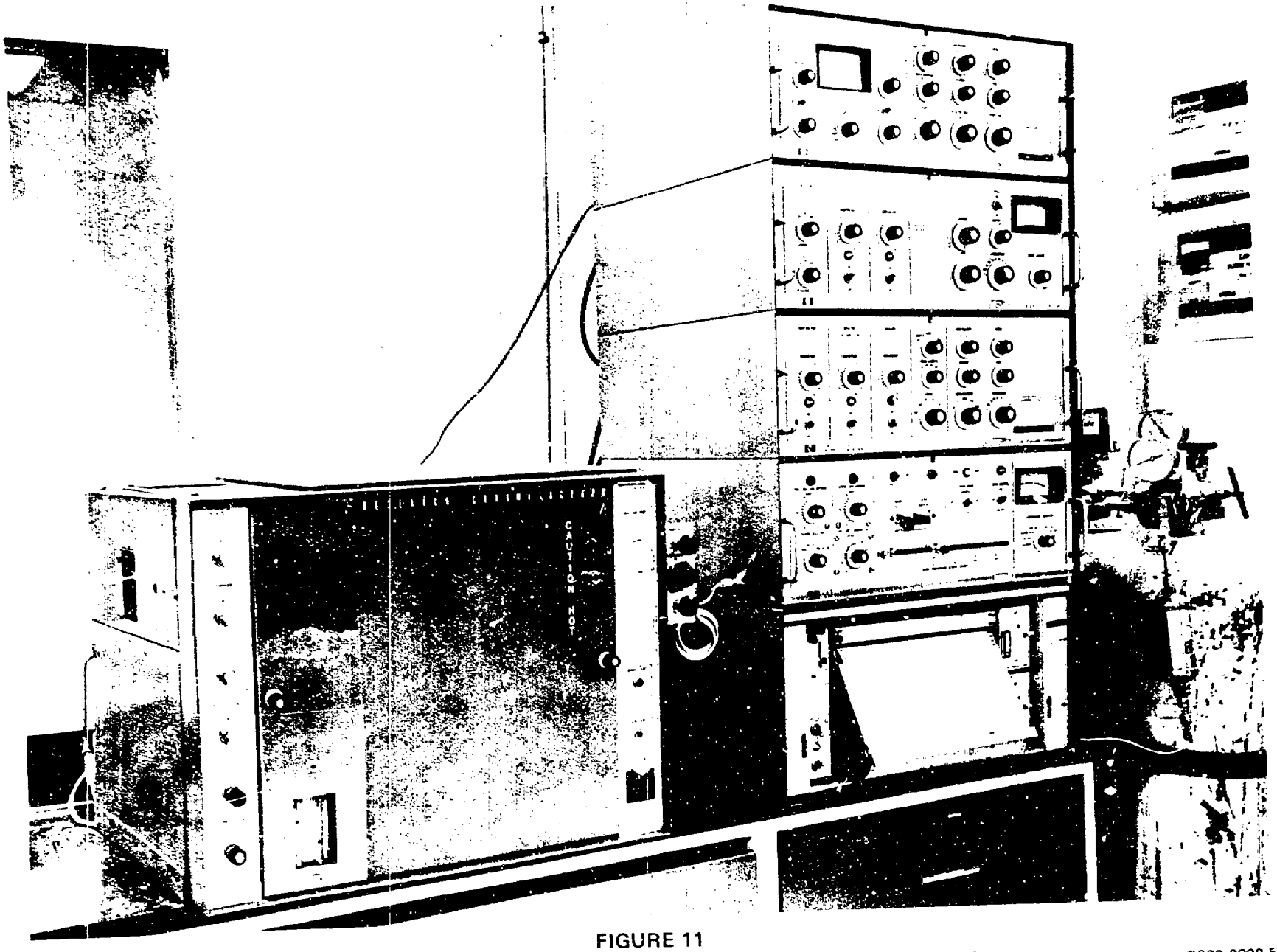

FIGURE 11
GAS CHROMATOGRAPH EQUIPMENT FOR VOLATILE ANALYSIS

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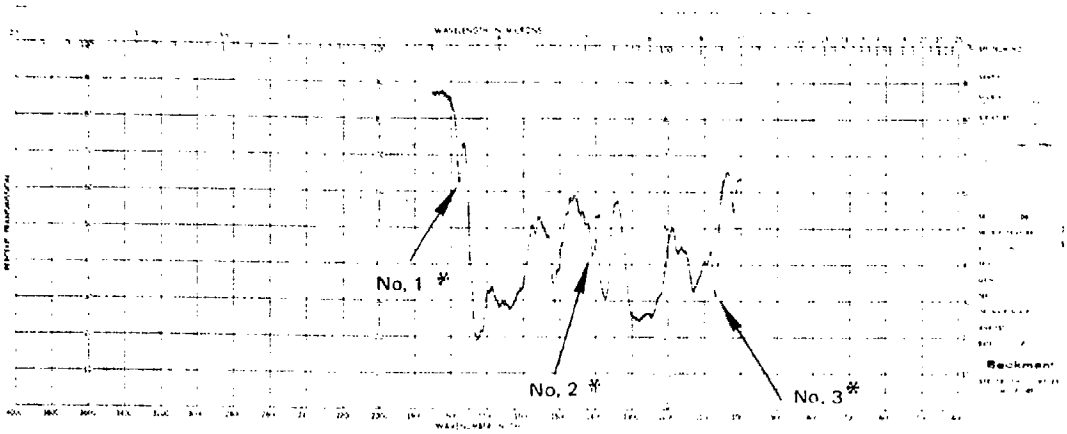
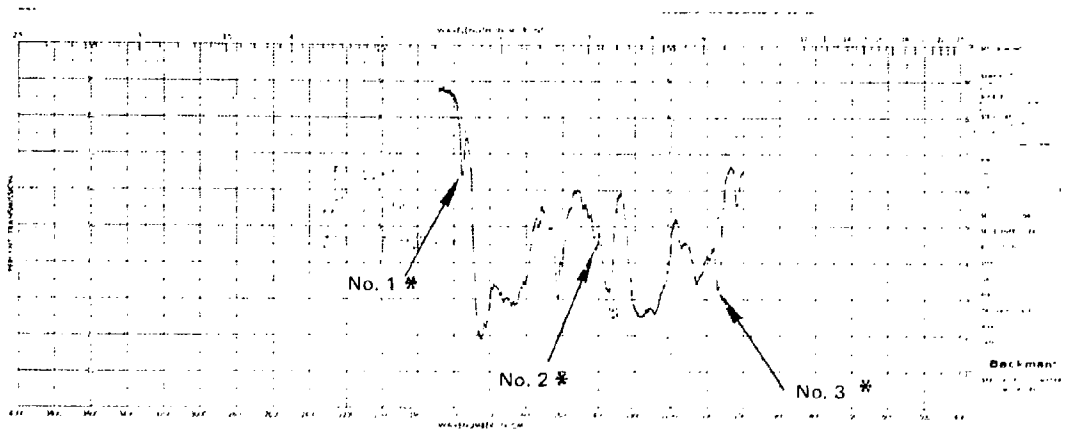
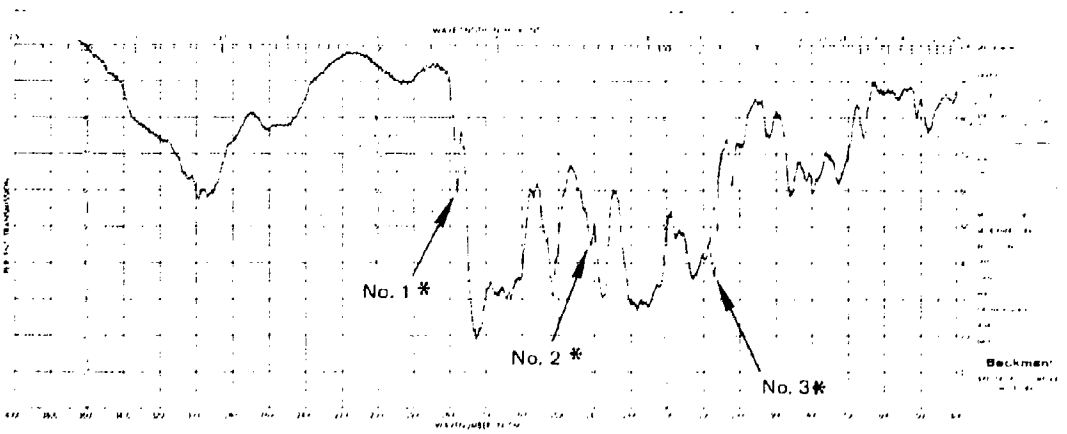


After 15 min at 200°F



After 1 hr at 200°F

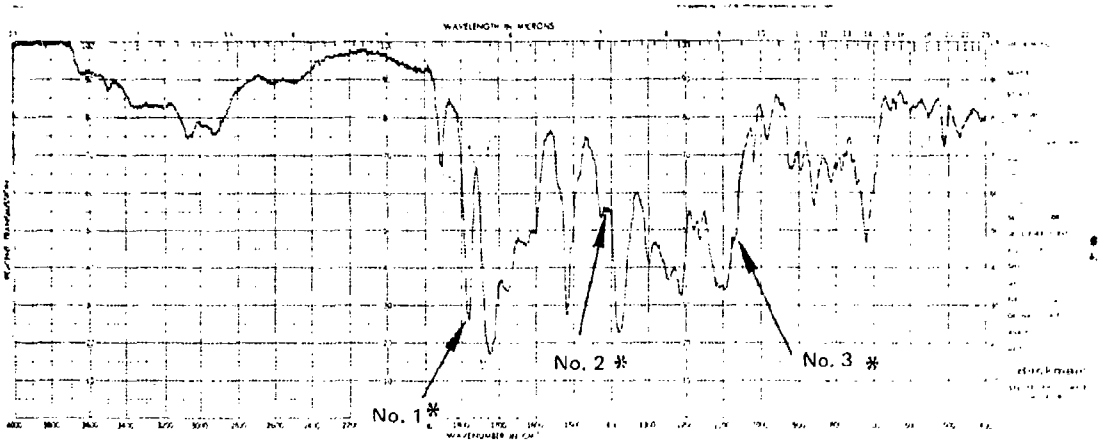


After 1 hr at 275°F

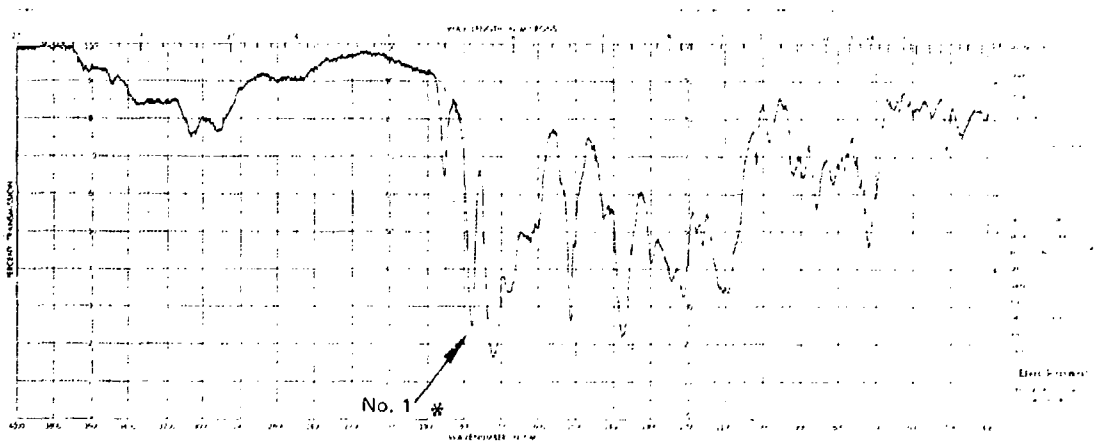
* No. 1 (imide), No. 2 (n-methyl pyrrolidone), No. 3 (ethanol)

FIGURE 12
INFRARED SPECTROGRAMS FOR SB 703

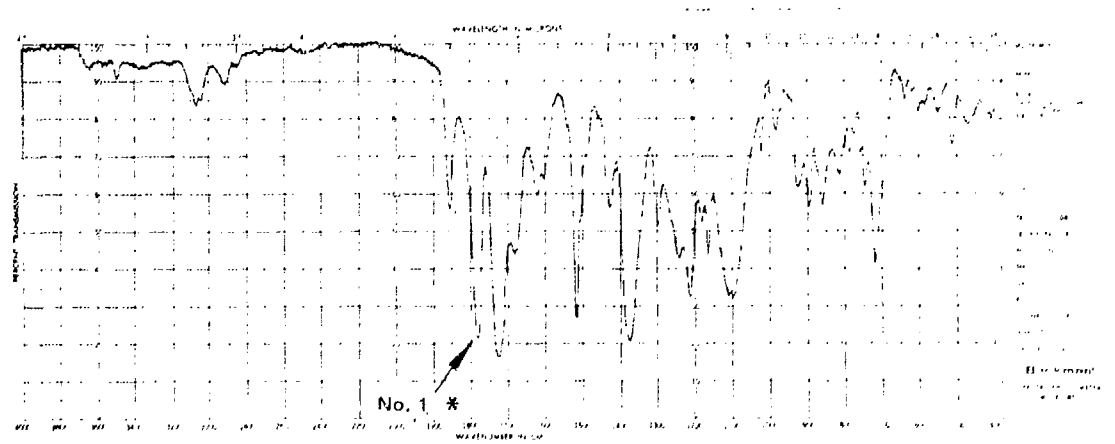
GP72-0328-72



After 2-1/2 hr at 340°F



After 2 hr of Post Cure at 400°F



After 2 hr of Post Cure at 600°F

* No. 1 (imide), No. 2 (n-methyl pyrrolidone), No. 3 (ethanol)

FIGURE 12
INFRARED SPECTROGRAMS FOR SB 703 (Continued)

GP72-0328 73

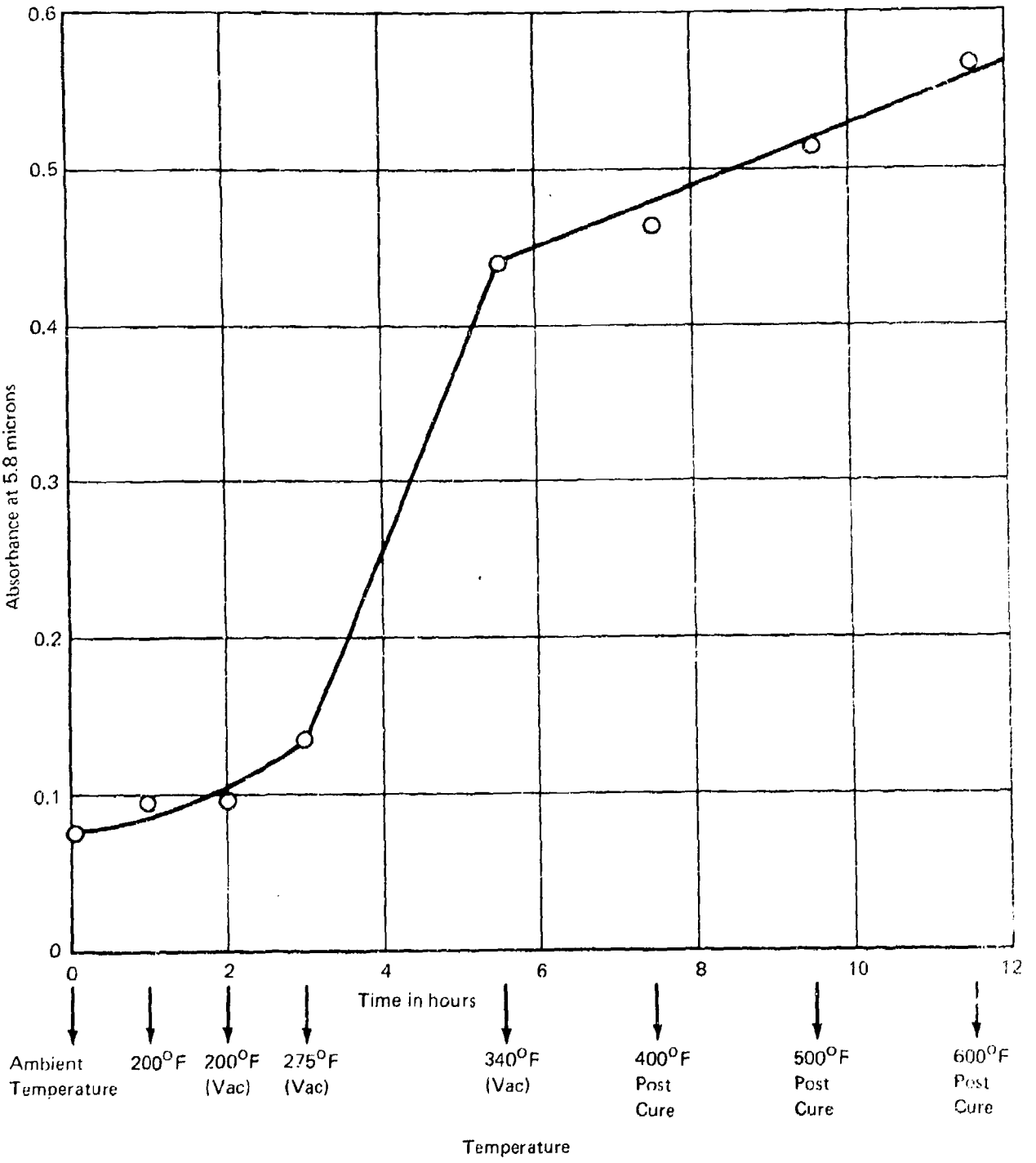


FIGURE 13
DEGREE OF SB 703 POLYMERIZATION

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Solvent retention characteristics were monitored by measurement of absorption bands assignable to the solvents in the resin system. The intensity of the 9.4 micron band was used to measure the ethyl alcohol content, and the intensity of the 7.1 micron band was used to measure the n-methyl pyrolidone (NMP) content. Table XV shows the absorbance values of the 9.4 and 7.1 micron bands as a function of cure time. All traces of detectable solvents were not removed until after the 500°F interval in the post cure cycle. The most rapid rate of solvent removal occurred between the 275°F and 340°F portion of the cure cycle. Figure 14 shows the infrared spectroscopy equipment used for this study.

Thermal-Gravimetric Analysis (TGA) with Mass Spectrometry - The gas chromatography and infrared spectroscopy are excellent instruments for detecting the condition of a material at a steady-state condition. However, they are not readily adaptable to material analysis is a transient system. The TGA can be used to plot weight loss at any time during the cure cycle while the mass spectrometer identifies materials being released.

Figure 15 is a plot of volatile constituents (ethanol, water, and NMP) release vs. temperature for a constant heat-up rate of 1-3°F/min. The TGA with mass spectrometry and infrared spectroscopy (I.R.) agree quite well as to the maximum volatile release temperature range (270 - 310°F). The TGA with mass spectrometry is,of course, used as a transient analysis system while the I.R. analyzed the steady state conditions at various points in the cure cycle. It should be pointed out that Figure 15 does not represent quantity ratios for various volatiles, but rather only a relative rate of volatilization over the temperature range shown. In summary, the Figure 15 data confirms the volatile release information developed by I.R. and it also indicates that ethanol is a more critical product of reaction than is water.

3.2.2.2 Optimum Prepreg Resin Content - The 28.0% resin solids content prepreg resulted in laminate thickness of 0.0057 - 0.0070 in/ply after cure, so it was decided to prepreg material with 22.0%, 24.0%, and 26.0% resin solids to get the desired 0.0050 - 0.0056 in/ply thickness. Laminates fabricated with various resin contents were then evaluated for thickness/ply, delaminations and interlaminar shear strength. Table XVI summarizes the results of 22 - 28% resin solids content prepreg evaluation. The 22% and 24% resin solids prepreg consistently resulted in 15 and 40 ply laminates that were too thin, approximately 0.0044 - 0.0049 in/ply. Although the 15 ply laminates

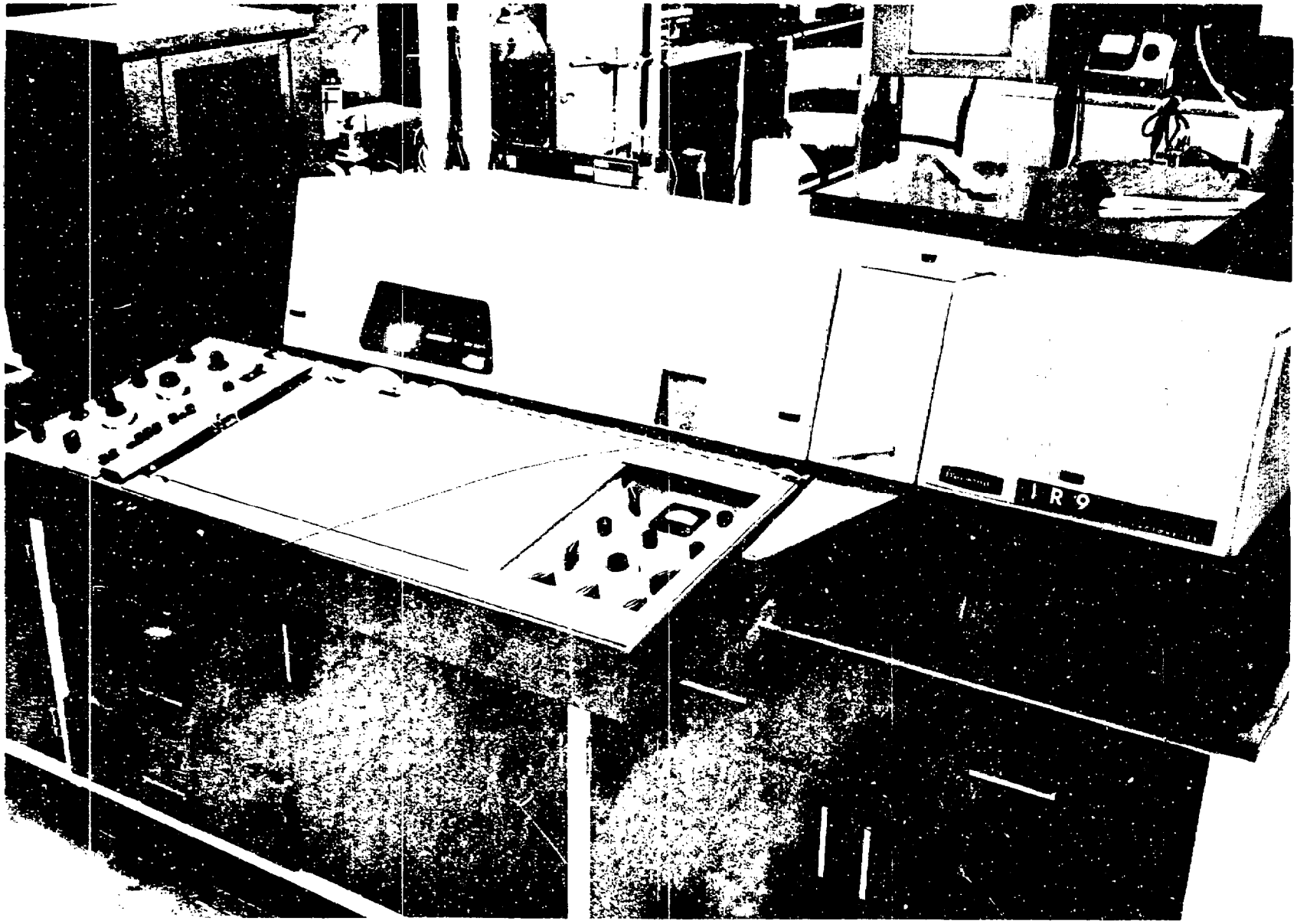

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FIGURE 14
INFRARED SPECTROSCOPY EQUIPMENT

TABLE XV
SB 703 SOLVENT RETENTION DATA

Cure Schedule (Cumulative)	Absorbance at 7.1 Microns (N-Methyl Pyrrolidone)	Absorbance at 9.4 Microns (Ethyl Alcohol)
As Received	0.095	0.225
1 hr at 200°F (No Vacuum - "B" Stage)	0.065	0.218
1 hr at 200°F (Full Vacuum)	0.065	0.218
1 hr at 275°F (Full Vacuum)	0.067	0.170
2-1/2 hr at 340°F (Full Vacuum)	Trace	Trace
2 hr of Post Cure at 400°F	Trace	Trace
2 hr of Post Cure at 500°F	Not Detected	Not Detected

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TABLE XVI
RESIN SOLIDS CONTENT EFFECT ON LAMINATE PROPERTIES

Laminate	Resin Solids Content (%)	"B" Stage Cycle	Thickness Ply (mils)	Shear Strength (ksi)		Remarks
				RT	550°F	
011971	28	2 hr @ 200°F	5.9	–	–	Delaminated After Post Cure
012971	24	2 hr @ 213 ± 10°F	5.5	–	–	Delaminated After Cure
	24	2 hr @ 200°F	5.5	–	–	Delaminated After Cure
020271	22	1 hr @ 200°F	4.7	–	–	Some Voids Apparent
		1.5 hr @ 200°F	4.7	8.3	8.4	
		2.0 hr @ 200°F	5.3	–	–	
021171	24	1.75 hr @ 200°F	4.5	8.5	7.9	Laminates All Too Thin (15 and 40 (Ply)
	28	2 hr @ 200°F	4.5	8.5	8.5	
	26	2 hr @ 200°F	4.8	11.4	9.8	15 Ply Data
			4.8	9.5	4.0	40 Ply Data
021871	26	2 hr @ 200°F	5.4	–	–	Splice Plate Good Appearance
	28	2.5 hr @ 200°F	6.0	–	–	Precipated w/Some Voids
	28	3 hr @ 200°F	6.0	–	–	Precipated w/Some Voids
022571	26	2 hr @ 200°F	4.7	5.8	7.0	Resin Poor w/Polymer
			4.6	6.2	3.7	
	28	2 hr @ 200°F	4.7	7.5	8.1	Precipitation
			4.6	7.4	4.3	
022871	26	2.5 hr @ 200°F	4.8	–	–	
	28	2.5 hr @ 200°F	4.7	–	–	

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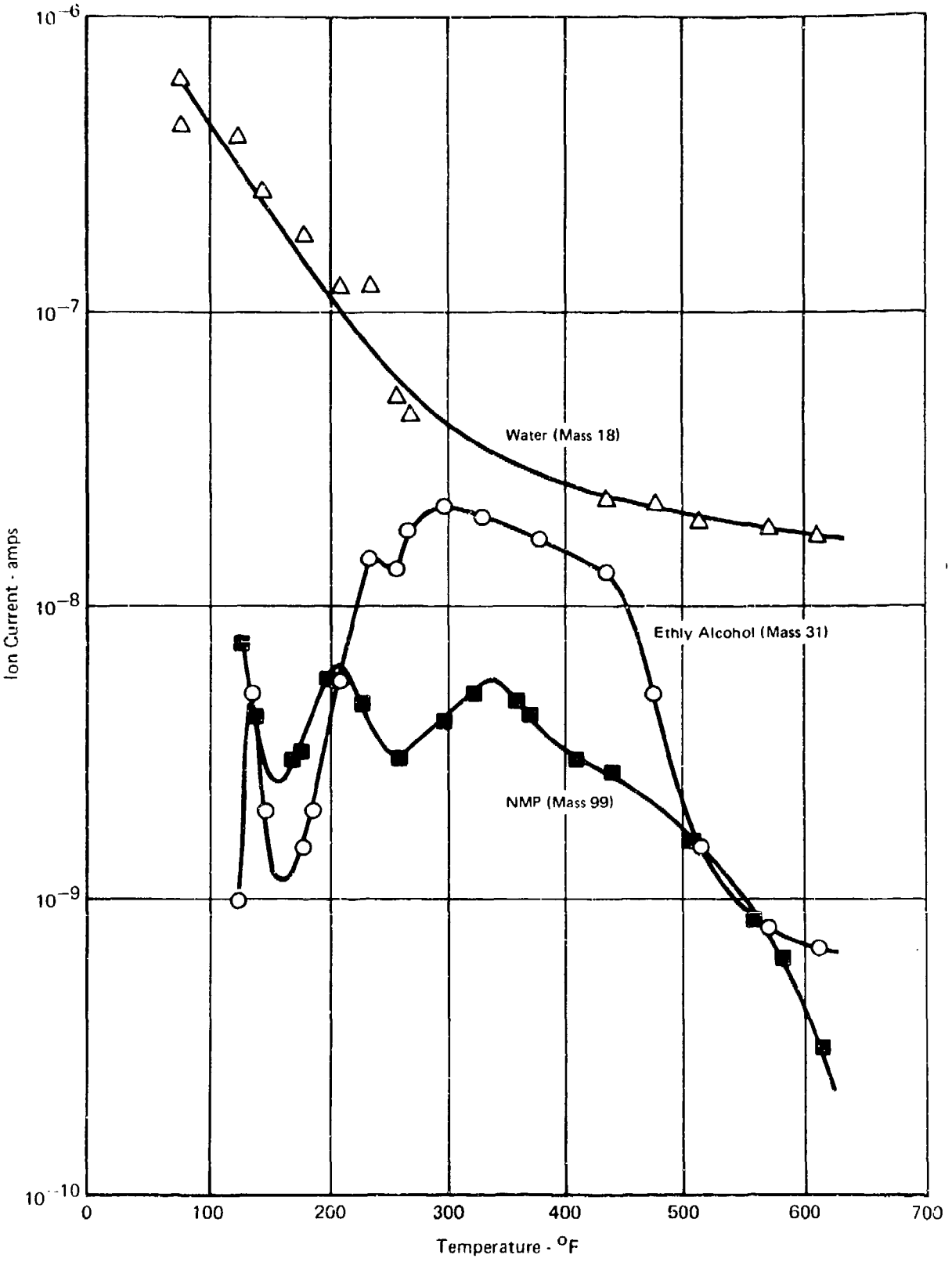

FIGURE 15
OUTGASSING CHARACTERISTICS OF "A" STAGED SB 703

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appeared satisfactory, the resultant interlaminar shear strengths were quite low, particularly at room temperature. The 26% and 28% resin solids prepreg gave more encouraging results. The thicknesses varied from 0.0050 - 0.0062 in/ply, which was an improvement over previous measurements.

In spite of the thickness/ply improvement, there was no marked improvement in delamination resistance for 40 ply laminates. The resin content variation effort was conducted concurrently with the prepreg analysis and there were indications of prepreg volatile content variability. This was demonstrated most conclusively by 28% resin solids prepreg 40 ply laminates 021171 and 021871B. Laminate 021171 was 0.175-0.187 in. thick, while laminate 021871B was 0.228 - 0.248 inch thick.

The 26% resin solids prepreg was selected for the remainder of the program based on experience with it during both this study and the process cycle alteration study. It has generally exhibited thickness/ply in the range of 0.0050 - 0.0056 inches.

3.2.2.3 Process Cycle Modifications - Laminates made during the prepreg analysis and variable resin content studies were fabricated with essentially the same processing cycle. Slight changes of "B" stage time at 200°F occurred (2 to 3 hours), and post cure cycles varied between a stepped cycle (2 hours each at 400, 450, 500, 550, and 600°F) and continuous, slow heat-up to 600°F and hold for 6 hours. However, these changes did not make significant differences in 40 ply thick laminate acceptability and indications were that processing cycles changes were necessary.

Initial effort altered the "B" stage temperature and increased the autoclave pressure from 100 to 200 psi. "B" stage temperatures of 215°F, 235°F and 300°F in air and 200°F under vacuum were evaluated. A combination of 3 hours at 215°F "B" stage and 200 psi autoclave pressure gave the best results, but did not completely eliminate the delamination of 40 ply specimens. These processing changes did result in improved thickness/ply control as shown in Table XVII. The initial cycle refers to a "B" stage of 2 hours at 200°F; cure cycle at 100 psi with temperature stops at 200°F, 235°F, and 350°F; and a post cure of 2 hours each at 400°F, 450°F, 500°F and 550°F plus 6 hours at 600°F. The altered cycle changed the "B" stage to 3 hours at 215°F and cure cycle pressure to 200 psi. Other factors remained constant.

TABLE XVII
COMPARISON OF REGULAR AND ALTERED CURE CYCLE
(Prepreg Batch 021171)

	Regular Cycle		Altered Cycle	
	24%	28%	24%	28%
Thickness/Ply — 40 Ply Laminate	0.0045	0.0060	0.0054	0.0053

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"B" stage temperatures of 235°F and 300°f in an air circulating overn, and 205°F at 26 - 27 in. Hg. vacuum were not as successful. The 235°F and 300°F "B" stage cycles imidized the SB703 polyimide resin too far for adequate interply bonding during the cure cycle. Both laminates had per ply thicknesses of 0.0065 - 0.0066 inches, which was unacceptable. The laminate fabricated with the vacuum (26 - 27 in. Hg. vacuum) "B" staged prepreg flowed excessively, was full of voids and thickness/ply was less than 0.005 in.

Parallel studies were initiated to explore better methods of removing the volatiles during the processing cycle. One approach was to add a cure cycle hold at 300°F since the volatile load was maximum at that temperature. A second approach was to fabricate a 20 ply laminate and then bond 10 plies on either side in a separate fabrication cycle. The third approach was to complete the entire cure/post cure cycle in the autoclave under 200 psi. The fourth approach was to lengthen the post cure cycle to 26 hours and evaluate the influence of vacuum during post cure. None of these approaches solved the delamination of 40 ply laminates problem. Table XVIII summarizes the results.

TABLE XVIII
PROCESS VARIATION EVALUATION

Approach	Shear Strength (ksi)		Remarks
	RT	550°F	
1. Cure Cycle Hold at 300°F	10.4	3.9	Slight delamination
2. Two Step Fabrication Cycle	–	–	Delaminated
3. Cure and Post Cure in Autoclave at 200 psi	4.8–6.4	2.6–3.2	Slight delamination
4. 26 Hour Post Cure Cycle	7.4–10.3	5.1	a) 1/2 delaminated b) 1/2 did not delaminate

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The longer post cure cycle offered some encouragement, and, after discussions with WRD and Monsanto the following new approaches were tried:

- Cure cycle that eliminates all steps below 350°F with pressure application at approximately 250°F.
- Post cure cycle from 350°F to conclusion of 600°F exposure in 5 to 6 days.
- Determine the maximum thickness laminate that can be fabricated without delamination and with acceptable shear properties.

Direct Heat-Up Cure Cycle - Two laminates were fabricated using the direct heat-up to 350°F at 3 - 4°F/min. with pressure application at 240 - 250°F. The first laminate was made with full vacuum (29 in. Hg.) applied at room temperature and maintained throughout the cycle. A second laminate was processed with 5 in. Hg applied from room temperature to 230°F and full vacuum applied from 230°F to cure cycle completion. In both cases, the resin flow during cure was excessive and the thickness/ply was less than 0.005 inch.

Extended Post Cure - A longer post cure cycle was evaluated with 15 and 40 ply laminates using the regular cure cycle with 200 psi autoclave pressure. It consisted of 24 hour holds at each of 400, 500, and 600°F. Both laminates were satisfactory as evidenced by the Table XIX summary. The 5 day post cure

cycle gave 40 ply laminates with no delaminations. This complete process cycle is given in Paragraph 3.2.3 below.

3.2.3 Fabrication Scale-Up - The maximum thickness laminate that (a) could be reliably fabricated with no delamination, and (b) exhibit shear strength at room temperature and 550°F comparable to 15 ply data was demonstrated. The reliability aspect would be established by making 4 consecutive autoclave runs with 15 through 40 ply panels using the same process cycle from "B" stage through post cure. Table XX shows the resultant data.

Figure 16 is a visual comparison of a series of 15 through 40 ply laminates fabricated with the extended post cure cycle. The excellent quality of laminates up to 40 plies thick is visually demonstrated. There is no significant difference between 15 and 40 ply laminates, except for the one instance of a 30 ply and one instance of a 40 ply panel that delaminated.

The scale-up of boron/SB703 prepreg involved more effort than originally anticipated, and the delay in obtaining satisfactory 40 ply laminates necessitated a redirection of Structural Component Fabrication and Test Task. A thinner skinned wing compression panel was chosen to demonstrate the technology developed. The detailed analytical tests did not assist directly in resolving delamination problems, but did provide information about characteristics of the SB703 cure cycle. The eventual almost completely successful fabrication and test of 40 ply laminates indicates that the potential exists to fabricate thicker boron/SB703 structural laminates and components. The autoclave cure cycle finally selected met the original objective of a production type cycle. The complete process cycle selected for the F-4 Rudder skin fabrication, design allowables generation, and the F-15 Wing Compression Panel skin fabrication is as follows:

"B" Stage	3 hours at 215°F
Cure Cycle	Apply full vacuum Heat to 215°F and hold 1 hour Heat to 235°F and hold 1 hour Apply 200 psi during hold Heat to 350°F and hold 2 hours
Post Cure Cycle	Hold 24 hours each at 400°F, 450°F, 500°F, 550°F and 600°F

TABLE XIX
EFFECT OF 5 DAY POST CURE CYCLE ON 15 AND 40 PLY LAMINATES

Property	15 Ply	40 Ply
0° Flexure Strength (ksi)	249.0 @ RT 163.0 @ 550°F	– –
0° Flexure Modulus (msi)	26.1 @ RT	–
Shear Strength (ski)	14.7 @ RT 8.2 @ 550°F	13.5 @ RT 7.5 @ 550°F
Thickness/Ply	5.6	5.4
Delaminate	No	No

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TABLE XX
RESULTS OF MAXIMUM PANEL THICKNESS STUDY

Property	Set No	Panel Thickness (Plies)					
		15	20	25	30	35	40
Delamination	1	No	No	No	Yes	No	No
	2	No	No	No	No	No	Yes
	3	No	No	No	No	No	No
	4	No	No	No	No	No	No
Thickness/Ply (mils)	1	5.7	5.4	5.5	–	5.4	5.3
	2	6.0	5.6	5.5	5.5	5.5	–
	3	5.7	5.5	5.5	5.7	5.4	5.5
	4	5.5	5.7	5.7	5.5	5.7	5.5
Shear Strength at RT (ksi)	1	13.8	14.0	13.9	–	13.7	12.3
	2	14.7	14.4	14.4	12.6	14.9	–
	3	12.8	14.0	15.2	14.6	13.3	12.2
	4	12.9	13.3	13.2	12.9	12.0	12.2
Shear Strength at 550°F (ksi)	1	8.5	8.6	6.6	–	4.4	4.0
	2	8.6	7.6	7.7	6.9	7.3	–
	3	8.5	8.4	7.8	7.4	7.1	6.5
	4	8.3	7.9	8.2	7.8	7.0	7.0

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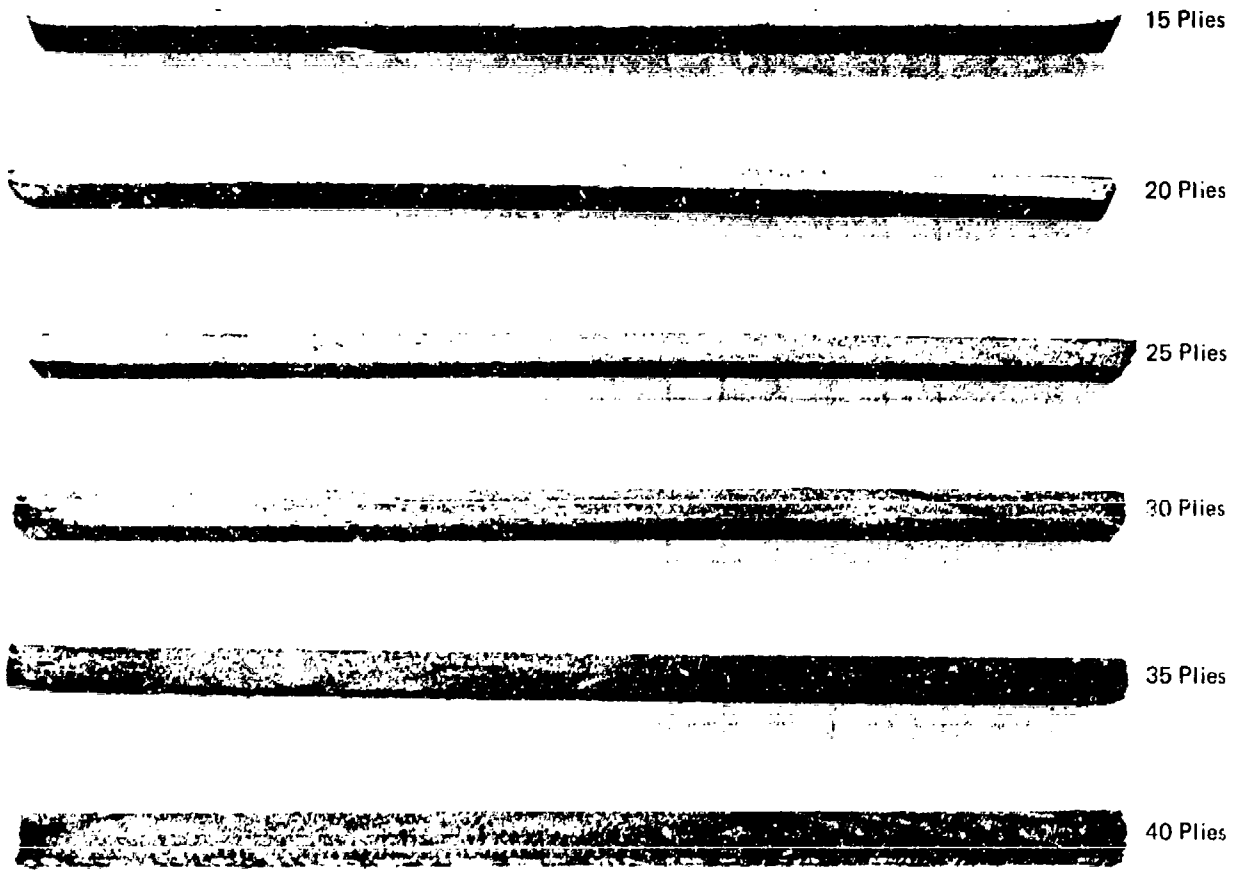

FIGURE 16
MAXIMUM PLY THICKNESS STUDY (15-40 PLIES)

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3.3 Graphite/SB703 Scale-Up

Graphite/SB703 was considered for spar/rib and stiffener type structure. Initially only spar/rib structure with a "C" channel configuration was planned, and, as a result, fabrication scale-up was performed with that configuration only. Both male and female tooling concepts were evaluated as the F-4 Rudder spar/rib required male tooling (prepreg laid over outside contour of tool) and the F-15 Stabilator spar required female tooling (prepreg laid inside tool cavity) for proper dimensional control. The male tooled parts did not present any problems whether they were unidirectional, 0° ±45°, or all ±45° orientation. Female tooled parts evidenced unacceptable voids in the radii.

The graphite/SB703 prepreg was not sufficiently tacky for easy fabrication of "C" channels. The typical "C" channel had flanges 1.5 in. wide and a web 1.5 in. high with .06 in. radii. Prepreg formability was then aided by spraying with a light, mist coat of solvent n-methyl pyrrolidone and sandwiching it between two sheets of Mylar for 6 hours minimum. This made the prepreg very pliable, but the cure cycle had to be modified for removal of the solvent. The addition of a 4 hour hold at 175°F and 2 hours at 200°F with 3 - 5 in. Hg. vacuum to the regular cure cycle resulted in low void, 31.2% by-weight resin content laminates.

"C" channel sections (both 0° and ±45°) designed to simulate the F-4 rudder spar, were made with the modified cure cycle on male tooling. Tensile specimens were cut from the 0° channel cap sections and tested at room temperature. The values of 210-236,000 psi for one channel and 190-208,000 psi for another channel indicated the acceptability of the graphite/epoxy spar fabrication procedures. Similar efforts with "C" channels made on female tooling were not as successful because of voids in the radii resulting from lack of pressure. When the Advanced Fighter Wing Compression Panel was substituted for the stabilator, the type of graphite/SB703 structure changed from female tool fabricated spars/rib to male tool produced hot stiffeners. Therefore, the scale-up efforts for the F-4 Rudder spar was directly applicable to the Wing Compression Panel stiffeners. The hat stiffeners thickness of seventeen 0°, ±45° plies had already been demonstrated with a "C" channel.

The successful fabrication and test of a male tooled "C" channel plus the satisfactory properties generated from flat laminates demonstrated the acceptability of graphite/SB703 for use on the F-4 Rudder, Wing Compression Panel, and for design allowables testing. Results of this effort are summarized in Table XXI with most of the data presented relevant to cure cycle development using the mist coat of solvent addition. Two separate cure cycles were developed for graphite/polyimide laminates. The first covers laminates made without the addition of NMP for handleability:

- o Apply full vacuum and heat to 200°F
- o Hold at 200°F for 1 hour
- o Heat to 350°F; however, when the temperature reaches 250°F apply 100 psi
- o Hold at 350°F for 2 hours
- o Cool down under pressure.

The second covers laminates fabricated with the NMP solvent coat:

- o Apply 1 - 3 in. Hg. vacuum
- o Heat to 175°F and hold 4 hours
- o Heat to 200°F and hold 2 hours
- o Apply full vacuum and heat to 350°F; however, when the temperature reaches 250°F apply 100 psi
- o Hold at 350°F for 2 hours
- o Cool down under pressure.

The post cure cycle for all graphite/polyimide parts consisted of heating from 350°F to 600°F at less than 1°F/min. and holding for 6 hours. It should be pointed out that this post cure cycle covers parts only up to .125 in. thick. Thicker parts would probably require a slower post cure, such as that used with boron/polyimide.

3.4 Honeycomb Sandwich Panel Scale-Up

A two foot by three foot polyimide sandwich panel was fabricated to determine suitability of polyimide bonding operations. Panel fabrication consisted of bonding post cured 6 ply (+45, -45, 0, 0, -45, +45) boron/SB703 polyimide skins to HRH 327 fiberglass/polyimide core using FM-34 polyimide adhesive. The post cured sandwich panel, Figure 17, was NDT inspected for void areas and none were found. Then flatwise tension and edgewise compression specimens were machined from the panel, Figure 18. During the

fabrication of the 2' x 3' boron/polyimide skins, vacuum was lost temporarily when the cure temperature reached 240°F. The vacuum system was repaired and then the skins continued their cure cycle. The skins appeared to have a high void content. However, since the intent of fabricating the large sandwich panel was to determine suitability of the bonding operation on large sandwich panels, it was decided that the skins were satisfactory for that purpose. Subsequent mechanical property tests did not confirm the bonding operation since premature failures initiated in the skins for both flatwise tension and edgewise compression at R.T. and 550°F.

Dissection and visual inspection of the panel bond lines showed good filleting and low void content of the FM-34/BR-34 adhesive system. This inspection correlated quite well with the NDT. In addition, the skin flatwise tension values, although much lower than anticipated (100 psi at R.T.), were still higher than the flatwise tension loads required for the F-4 Rudder. The primary requirement for high flatwise tension loads had been the Stabilator Torque Box where values to 200 psi at R.T. were anticipated. Therefore, the two foot by three foot panel was not repeated with more acceptable boron/SB703 skins.

TABLE XXI
GRAPHITE/SB 703 SCALE-UP LAMINATES/SHAPES

Laminate Configuration	Cure Cycle	Remarks
1.5" x 1.5" x 15" x 7 ply "C" channel - male mold	Heat to 200°F under full vacuum and hold. Heat to 250°F and apply 100 psig. Heat to 350°F.	0.059-0.063 in. thick, very little resin bleed but cover plates slipped causing poor pressure distribution.
3" x 3" x 11 ply laminate	Same as above.	0.075-0.077 in. thick, heavy resin bleed but good appearance.
1.5" x 1.5" x 15" x 7 ply	Same as above.	0.048-0.051 in. thick, resin content: 20.5% Specific gravity: 1.26 . Results of beam tension specimens machined from channel are shown in Table .
3" x 3" x 12 ply laminate	Same as above.	0.080-0.082 in. thick. Heavy bleed with appearance good. Prepreg was sprayed with solvent prior to lay-up. From this date all graphite/PI prepreg sprayed with solvent.
1.5" x 1.5" x 15" x 17 to 10 (tapered) ply "C" channel - female mold	Same as above.	Prepreg was sprayed with solvent prior to lay-up. Nylon vacuum bag broke when cure cycle reached 300°F; solvent degraded nylon bag.
Same as above except light coat solvent added	Same as above except added 1 hour hold at 125°F.	Heavy resin bleed, delaminated in radius during post-cure.
3" x 3" x 10 ply laminate (solvent added)	Same as above except heat to 200°F under full vacuum.	Laminate started bleeding at 140°F; resin bleed by 200°F was excessive.
3" x 3" x 10 ply laminate (solvent added)	Same as above except heat to to 200°F under 2-3" Hg vacuum.	Resin bleed started when temperature reached 125°F.
3" x 3" x 12 ply laminate	Same as above except heat under 2" Hg vacuum to 175°F and hold, then heat to 200°F and hold. Apply full vacuum and heat to 250°F, then apply 100 psig. Heat to 350°F.	0.085-0.090 in. thick. Resin content: 31.2%
1.5" x 1.5" x 15" x 17 to 10 (tapered) ply "C" channel - female mold (solvent added)	Same as previous laminate	Resin bleed fair. Cross section of channel showed voids and deliminations due to poor pressure distribution in female tool.
5" x 4" x 8 ply laminate (solvent added)	Heat to 200°F under 5" Hg and hold. Heat to 250°F at 5" Hg vacuum, apply full vacuum and 100 psig. Then continue heat to 350°F.	Resin bleed fair. Resin content (Postcured): 27.9%. Mechanical properties acceptable.
2" x 4" x 21" x 8 ply "C" channel - male mold (solvent added)	Same as above.	Resin bleed fair. Cross section of channel good. Beam tension specimens from cap: (average 3 specimens) 202,000 psi.

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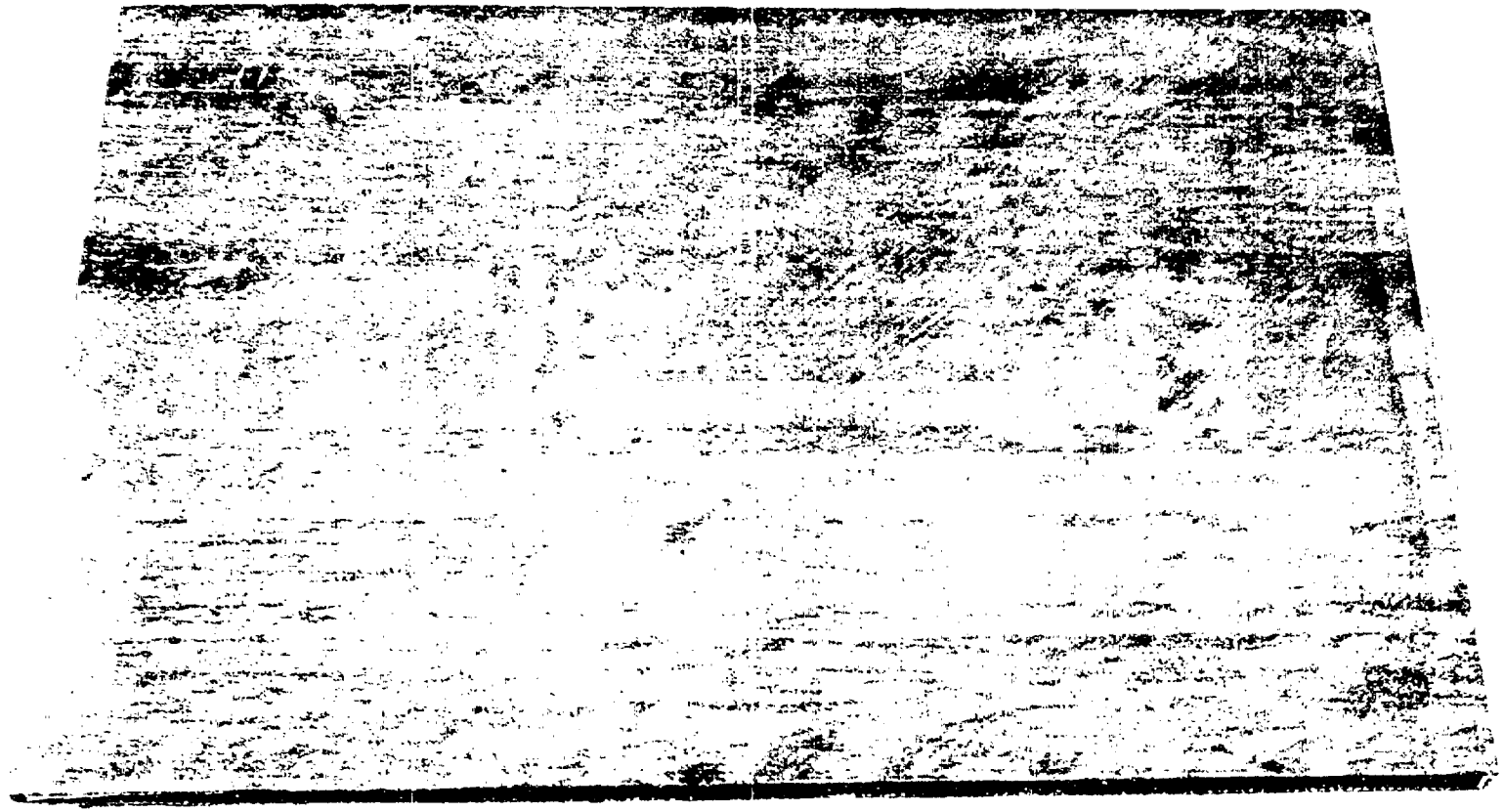


FIGURE 17
POST CURED 2 FT x 3 FT POLYIMIDE SANDWICH PANEL

GP72-0328-55

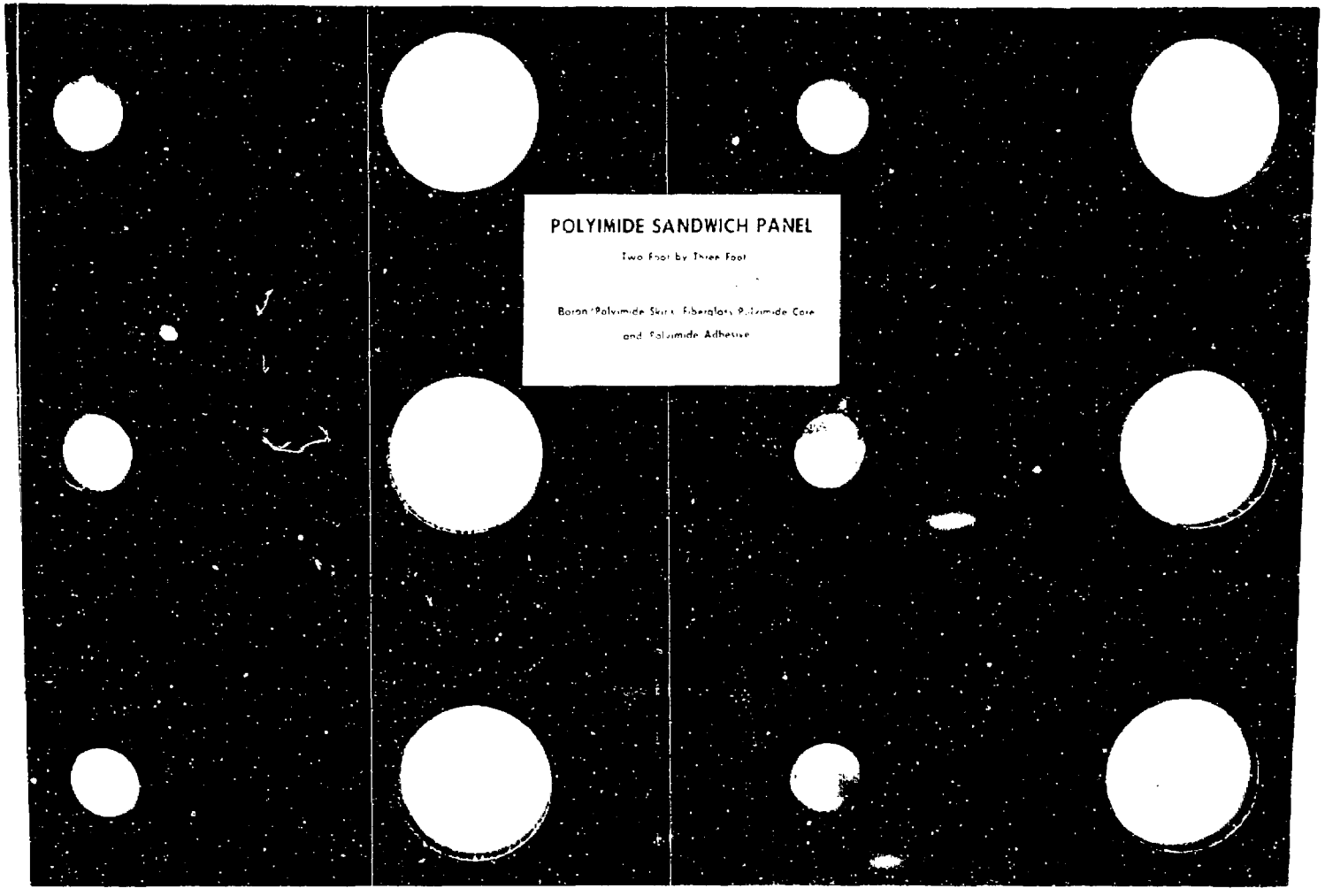


FIGURE 18
TEST SPECIMENS MACHINED OUT OF POST CURED 2 FT x 3 FT
POLYIMIDE SANDWICH PANEL

GP72-0328-54

4. STRUCTURAL ANALYSIS/DESIGN

4.1 Introduction

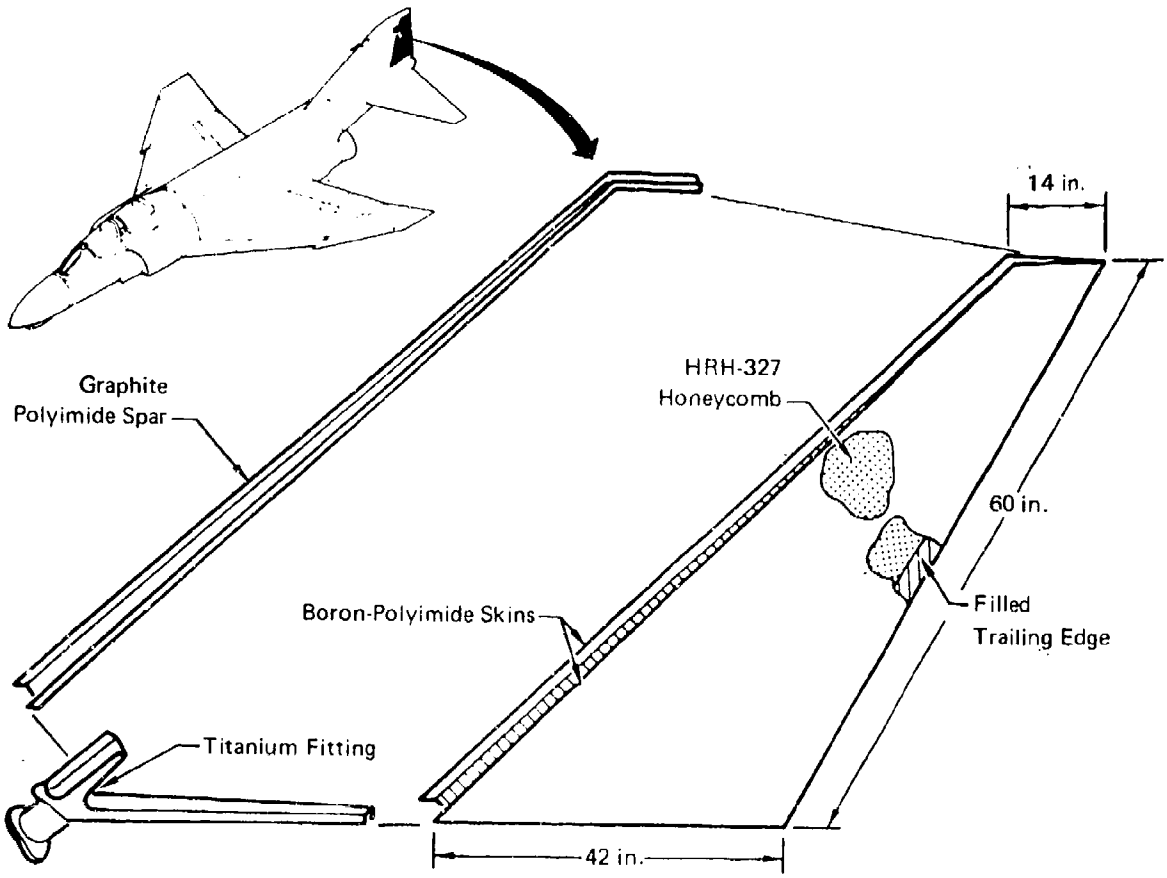
The purpose of this task was to design and analyze an F-4 rudder, an F-15 wing compression panel, and an F-15 horizontal stabilator. All of the above items had already been designed, analyzed, and tested utilizing epoxy type materials. The analysis/design effort on these structural items utilizing polyimide materials is presented in the following paragraphs.

4.2 F-4 Rudder

The F-4 rudder geometry and skin lay-up are shown in Figure 19. The rudder was designed with the same skin lay-up as used in the previous F-4 boron epoxy rudder. As in the boron/epoxy rudder, a structural analysis was performed to determine the boron/polyimide skin thickness required to satisfy the strength and stiffness requirements. In order to determine the effects of rudder stiffness on the load distribution the rudder was analyzed for the combined rudder-fin loading condition using the stiffness for four plies at ± 45 degrees. Similar to the boron epoxy rudder, the polyimide rudder is approximately two times as stiff in torsion as the conventional aluminum rudder. As a result of the structural analysis a single ply was added along the spar with the fibers perpendicular to the spar and two plies were added along the drive rib. The single ply along the spar added to react the hinge and balance weight loads, and to reduce the transverse strains along the spar. The two plies along the drive rib were added to provide strain compatibility between the skin and rib. The leading edge spar is graphite polyimide with a ± 45 degree lay-up. This lay-up was chosen to provide adequate strength combined with relatively low bending stiffness so that fin bending will not induce excessive rudder hinge loads. Spar size was established primarily by stiffness and geometrical compatibility considerations and, consequently, the strength margins of safety are generally high.

4.3 F-15 Wing Compression Panel

The polyimide compression panel is similar to an F-15 composite wing panel. The size of this panel is 14.6 x 24 inches. The panel consists of two 9-ply boron/polyimide skins $(\pm 45°, 0_2°, 90°, 0_2°, \pm 45°)_T$ separated by an 0.127 inch thick polyimide honeycomb core. The two skins in Figure 21 are labeled "-2001" and "-2003". The loaded edge of the panel is reinforced by gradually increasing the face sheet plies from 9 to 13 starting at 5.50 in. from the end of the panel as shown in Section C-C of Figure 21. The HRH 327



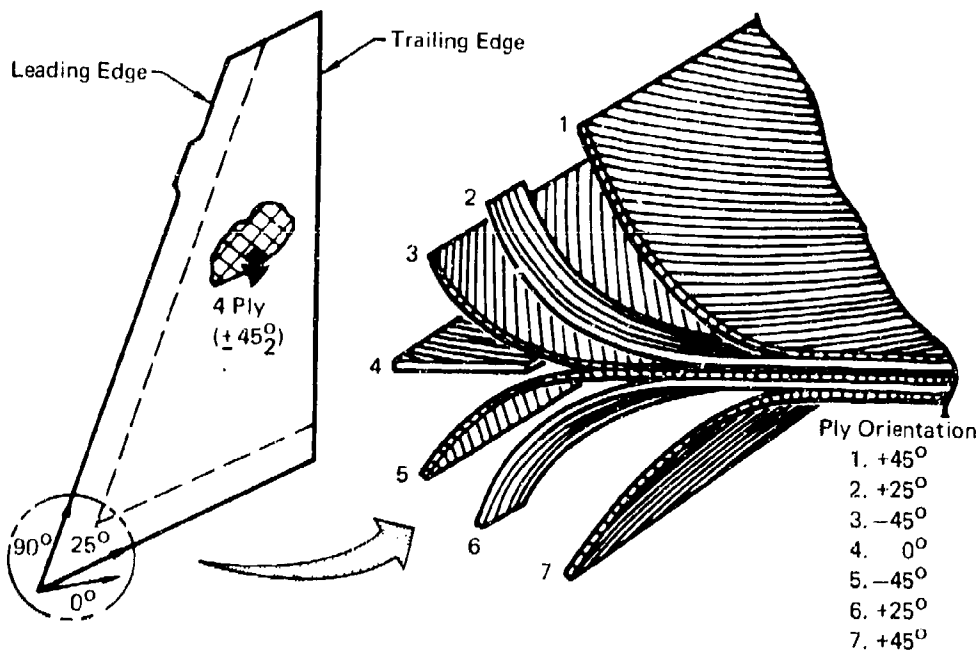
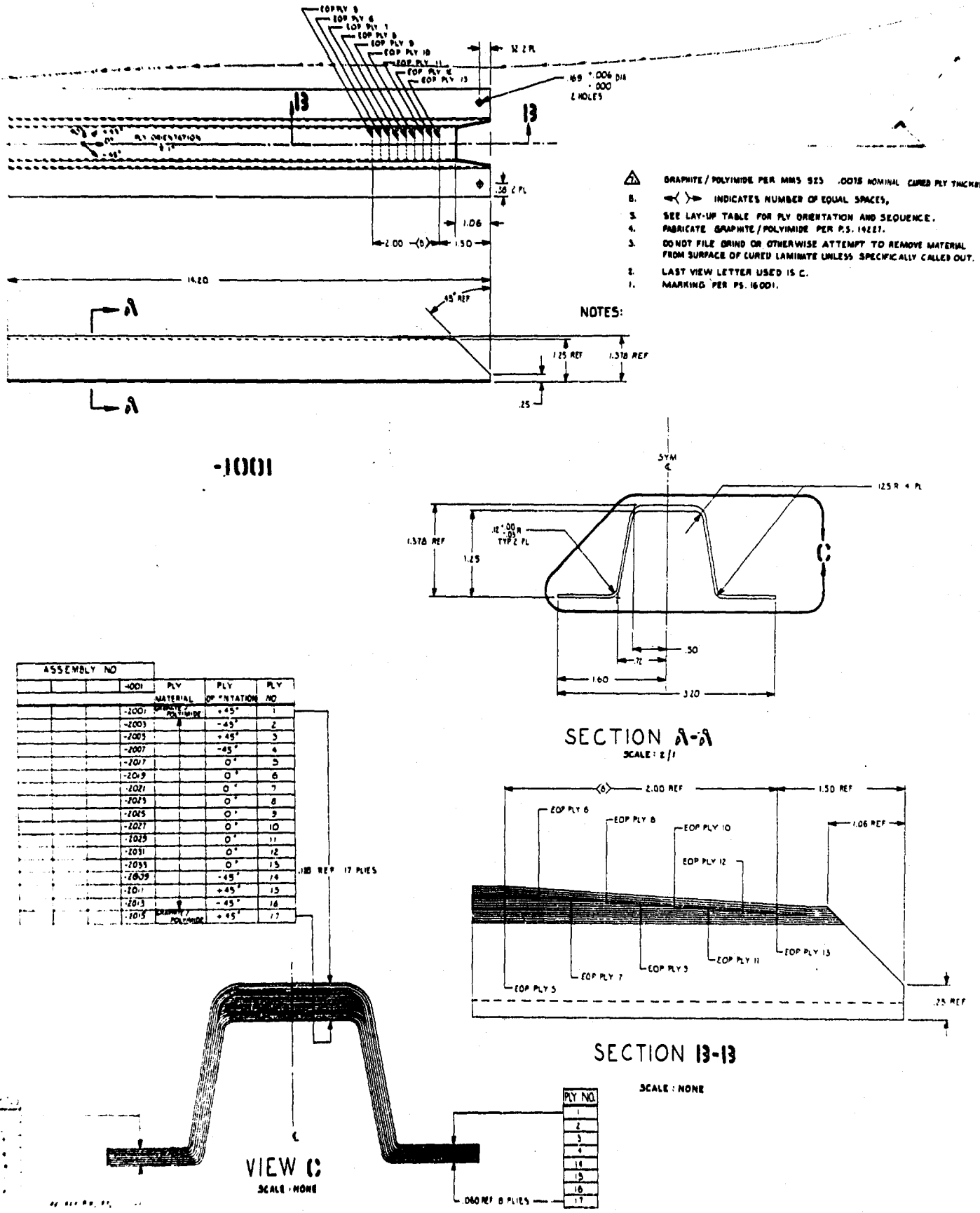


FIGURE 19
F-4 POLYIMIDE RUDDER SCALE-UP COMPONENT

GP72-0593-1



ASSEMBLY NO -1001	PLY MATERIAL	PLY ORIENTATION	PLY NO
-2001	GRAPHITE / POLYIMIDE	+45°	1
-2003		-45°	2
-2005		+45°	3
-2007		-45°	4
-2017		0°	5
-2019		0°	6
-2021		0°	7
-2023		0°	8
-2025		0°	9
-2027		0°	10
-2029		0°	11
-2031		0°	12
-2033		0°	13
-2039		-45°	14
-2011		+45°	15
-2013		-45°	16
-2015	GRAPHITE / POLYIMIDE	+45°	17

FIGURE 20
GRAPHITE POLYIMIDE COMPOSITE HAT STIFFENER

GP72-0893-18

honeycomb core is replaced by 10 plies ($\pm 45°$, $0_2°$, $90°_2$, $0_2°$, $\pm 45°$) of graphite/polyimide on the loading edge of the panel as shown in Section C-C of Figure 21 labeled "-2005". The hat-section stiffeners are scarfed at 45 degrees and are bonded and bolted at the corners with radius blocks under single fasteners. A design drawing for the three stiffeners is shown in Figure 20. The hat stiffeners consist of a basic 8 ply $\pm 45°$ lay-up that extends across the entire cross-section. In addition, the cap area contains 9 plies of 0° graphite/SB703 as shown in view C of Figure 20. The stiffeners are constructed entirely of graphite/polyimide; whereas, the composite wing design used a hybrid lay-up with a graphite/epoxy as the basic material and additional boron/epoxy plies in the cap of the stiffener. The stiffness of the graphite/polyimide stiffener is comparable to the stiffness of the hybrid stiffener. Since the post cure cycles were different, the technology is not advanced to the state of hybrid lay-ups of boron and graphite/polyimide. Hybrid lay-ups of polyimide materials will require and additional development effort which is beyond the scope of this program.

Referring to Section C-C of the design drawing of the F-15 wing compression panel shown in Figure 21, eighteen (18) plies of boron/polyimide (two 9-ply skins) are used in the skin panel instead of sixteen (16) plies of boron/epoxy used in the composite wing panel. The composite wing panel lay-up is slightly unsymmetrical (+45°, -45°, 0°, 90°, 0°, 0°, -45°, +45°). Because of the long post cure times for boron/polyimide it was felt advisable to use a symmetrical lay-up $(\pm 45°, 0_2°, 90°, 0_2°, \pm 45°)_T$. A structural analysis was performed on the wing compression panel indicating an overall increase of 16 percent in bending stiffness over that of the previous epoxy panel. Adequate strength levels were established with margins of safety higher than the epoxy panel. These extra margins of safety were primarily due to the symmetrical lay-up (one extra 0° ply in each skin) as opposed to the unsymmetrical lay-up of the epoxy panel.

4.4 F-15 Stabilator Torque Box

The purpose of this task was to:

(a) Establish design criteria, load environment, and structural arrangement for the F-15 stabilator torque box.

(b) Assess and modify, as required, analytical procedures, computer programs, and failure criteria to account for difference encountered through the use of the polyimide resin system.

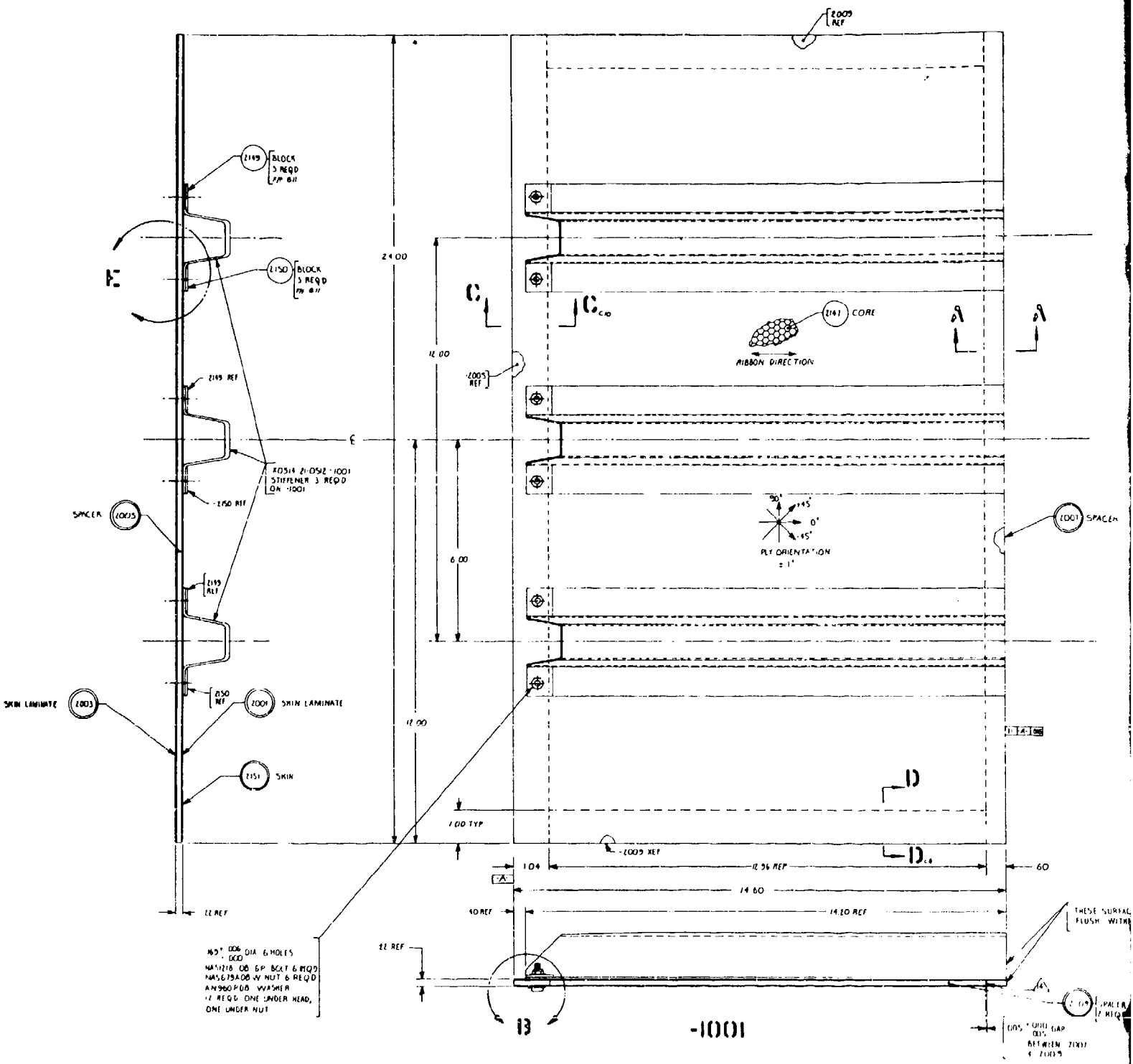


FIGURE 21

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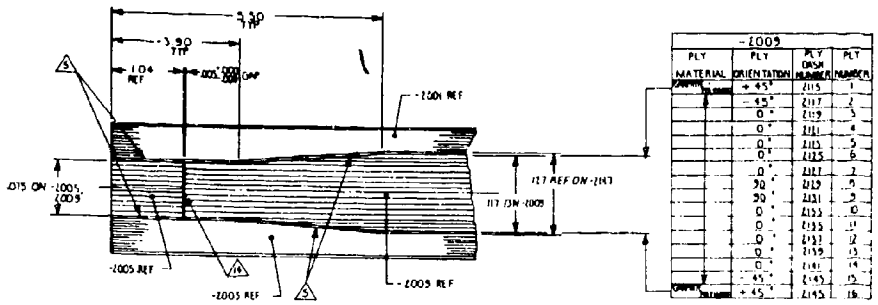
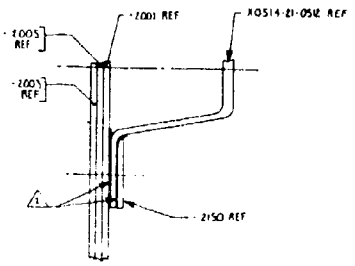
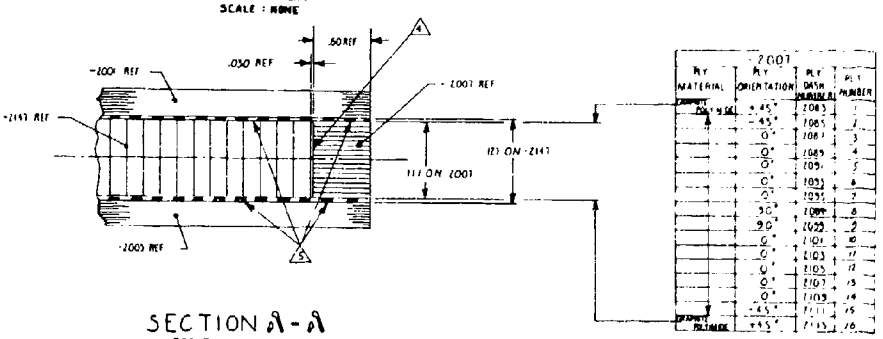
POLYIMIDE WING COMPRESSION TEST PANEL



VIEW 13
TYP L CORNERS
SCALE : NONE



VIEW E
TYP SKIN, STIFFENER, PLATE INTERFACE
SCALE 2/1

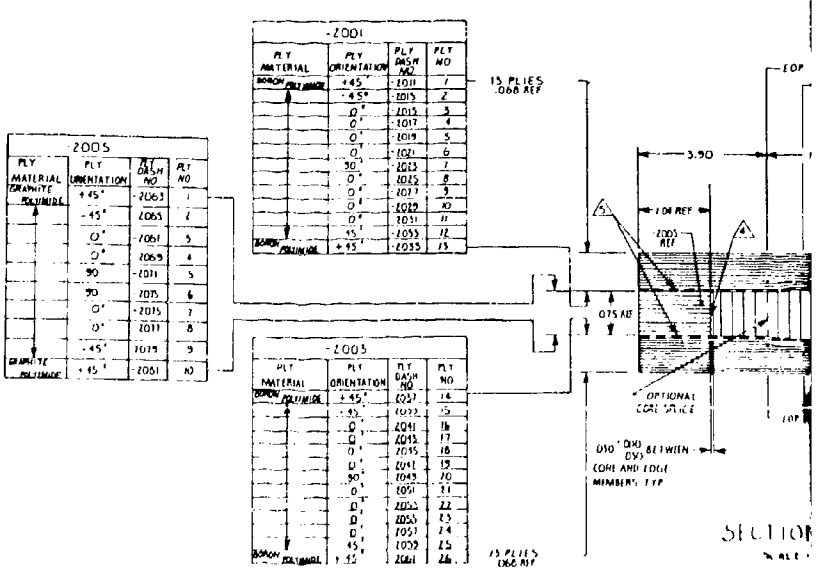


SECTION A-A
SCALE : NONE

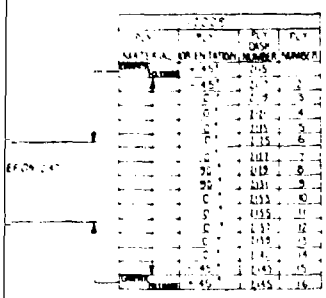
(ECC) SPACER

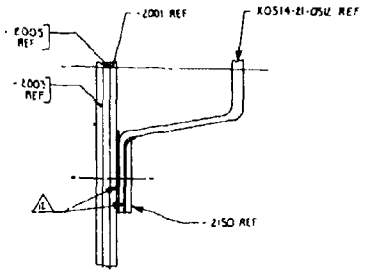
16. DIRECT CONTACT OF CADMIUM OR SILVER PLATE WITH TITANIUM IS PROHIBITED.
15. ODD DASH NUMBERS SHOWN, EVEN DASH NUMBERS OPPOSITE.
14. BOND -2005 SPACER TO -2003, 2007 SPACER WITH FM 34 ADHESIVE.
13. FABRICATE -2151 PER P.S. 14118.
12. METLBOND 840 ADHESIVE.
11. FABRICATE PER P.S. 14117.
10. FABRICATE PER P.S. 14114.
9. INSTALL FASTENERS PER P.S. 18000.
8. GRAPHITE POLYIMIDE PER MMS 515 .0075 NOMINAL PLY THICKNESS.
7. BORON POLYIMIDE PER MMS 511 .0052 NOMINAL PLY THICKNESS.
6. SEE PLY LAYUP TABLES FOR PLY ORIENTATION AND SEQUENCE.
5. BOND -2147 CORE TO SKIN, SKIN TO SPACERS, WITH FM 34 ADHESIVE
4. BOND -2147 CORE TO SPACER WITH FM 69 ADHESIVE
3. LAST VIEW LETTER USED IS E.
2. DO NOT FILE GRIND OR OTHERWISE ATTEMPT TO REMOVE MATERIAL FROM SURFACE OF CURED LAMINATE UNLESS SPECIFICALLY CALLED OUT.
1. MARKING PER P.S. 18001.

NOTES:

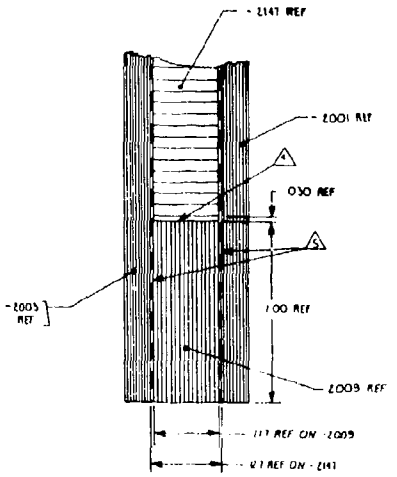


SECTION

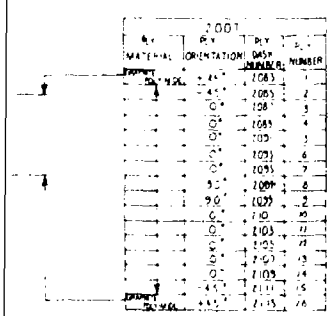


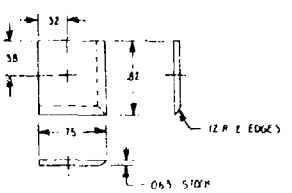


VIEW E
TYP SKIN, STIFFENER, PLATE INTERFACE
SCALE 2/1

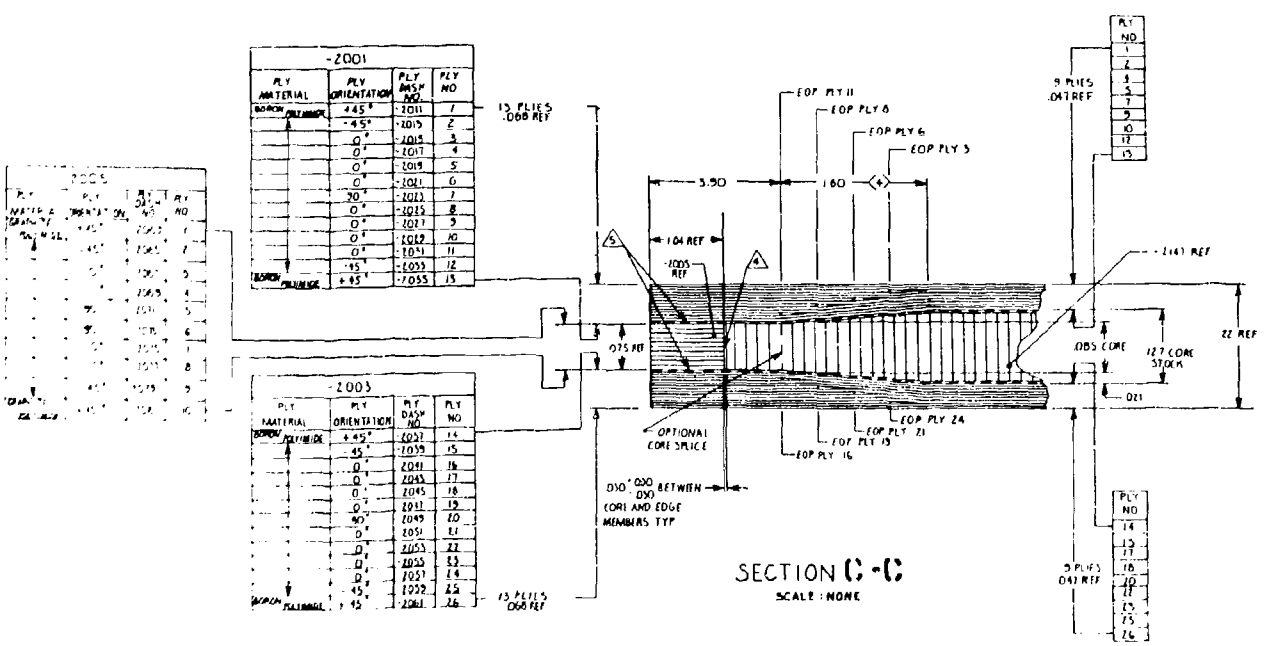


SECTION D-D





-2149 SHN
2150 OPP
SCALE 2/1



SECTION C-C
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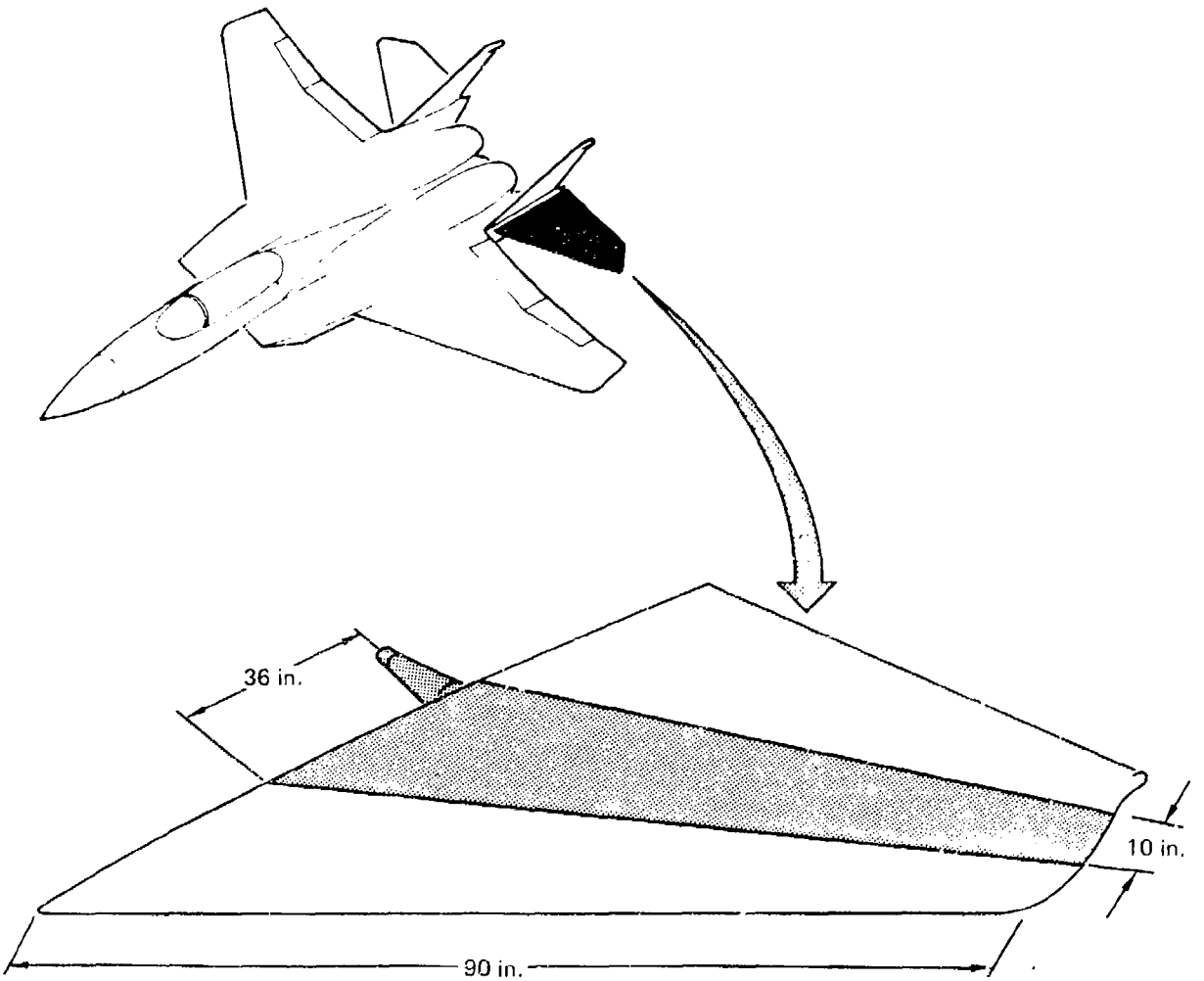

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FIGURE 22
F-15 STABILATOR TORQUE BOX

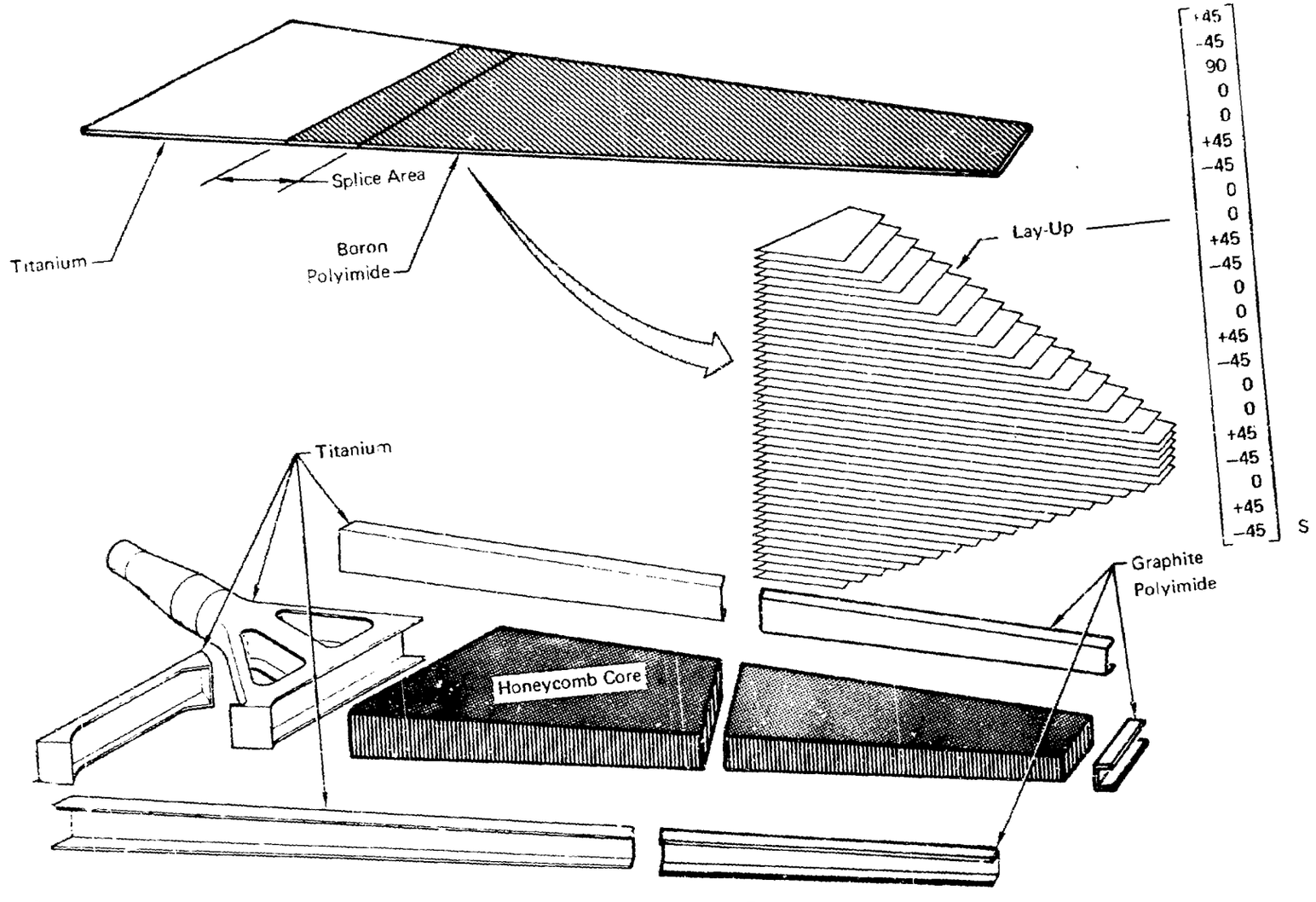

FIGURE 23
POLYIMIDE F-15 STABILATOR TORQUE BOX ASSEMBLY

(c) Perform a detail design strength and deflection analysis of the stabilator torque box. Prepare drawings suitable for release to manufacturing.

The F-15 stabilator torque box geometry is shown in Figure 22. The polyimide stabilator torque box assembly is shown in Figure 23. This figure indicates areas where polyimide materials were to replace epoxy materials. Basically, the epoxy stabilator titanium splice plate remained the same with boron/polyimide replacing boron/epoxy in the skins and graphite/polyimide replacing titanium in the outboard spars and closure member. Polyimide honeycomb core also replaced the aluminum core.

A finite element internal loads and deflection analysis was conducted to determine the effect of using HRH 327 polyimide honeycomb core in place of the aluminum core used in the production torque box. Results of this analysis showed that, although the shear stiffness of the polyimide core was less than that of the aluminum core, load distributions and deflections of the polyimide torque box were within 3% of production torque box analytical results.

Design drawings for the torque box were initiated but due to the laminate fabrication problems, inability to fabricate the necessary thick section laminates, continued work on the F-15 stabilator was terminated. Consideration was given to alternate structural components to fulfill the objectives of the HITAC program. An F-15 composite wing compression panel was ultimately selected. The fabrication and test effort on this panel is discussed in Section 6.0 of this report. The F-4 boron and graphite/polyimide rudder in fact was maintained and was successfully fabricated and tested, as discussed in detail in Section 6.0 of this report.

5. DESIGN MECHANICAL PROPERTIES

5.1 Introduction

The purpose of this task was to establish design allowable strengths and elastic constants for design and analysis of polyimide structures by means of room temperature and 550°F tests of boron/polyimide (Boron/Skybond 703 Prepreg) and graphite/polyimide (Modmor II Graphite/Skybond 703 Prepreg) specimens in tension, compression, and in-plane shear. A summary of this data is shown in Tables XXXV through XXXVIII for boron/polyimide and Tables LI through LIV for graphite/polyimide. "B" basis strength allowables, as reported in these tables, were calculated on the basis of ten test points. Elastic moduli and Poisson's ratio data are mean values. Cure and post cure cycles for boron and graphite/polyimide were performed as discussed in Section 3.2.3 and 3.3, respectively, and represent a best effort for this program. It is realized that the long post cure cycle for boron/polyimide adversely affects, to a certain extent, transverse properties; however, in order to fabricate acceptable thick laminates the long post cure cycle was necessary. Under this program it was not required to fabricate graphite/polyimide laminates thicker than 0.125 in.

5.2 Test Methods

5.2.1 Tension - Tension properties have been evaluated by two methods. Sandwich beam specimens were used to establish room temperature properties since this method gives high properties with less data scatter than the coupon method. For 550°F testing tension coupons were used. Tension design allowable data was also developed with tension coupons at room temperature to give a correlation between the sandwich beam and coupon test methods. The number of specimens and configurations tested are presented in Figure 24. Ten specimens were used to establish the 0 - 90 degree type properties because of statistical requirements in establishing "B" basis allowables. Approximately one third of the specimens were instrumented so that representative stress-strain curves could be established.

5.2.2 Compression - Compression properties were also evaluated by two methods. The sandwich beam method was used to establish room temperature properties. The honeycomb stabilized edgewise compression method was selected to establish 550°F data. Edgewise compression tests were also conducted at room temperature to give a correlation between this method and the sandwich

beam test method. The number of specimens and configurations tested are presented in Figure 25. The same rationale holds for the number of specimens and instrumentation as for the tension specimens, as explained in Paragraph 5.2.1.

5.2.3 In-Plane Shear - Inplane shear properties were evaluated by both the rail shear and picture frame test methods. Shear properties have been successfully evaluated for both $\pm 45°$ and $(0°, 90°)$ laminates by the rail shear test method, but this method gives low results for $\pm 45°$ laminates. For this reason, evaluation of shear properties for a $\pm 45°$ lay-up was attempted by the picture frame shear test. Two laminates were fabricated and tested by this method. The shear failure stresses were very low due to stress concentrations in the corners. As a result, $\pm 45°$ shear testing was accomplished by the rail shear test method. The number of picture frame specimens and configurations for the program are presented in Figure 26. Rail shear specimens tested are presented in Figure 27.

Room Temperature Tests

Specimen Configuration	Filament Material	Fiber Pattern	No. of Specimens	No. of Instrumented Specimens	Instrumentation*	Channels Per Specimen	Measured Data
Coupon	Boron	A B	10 10	3 3	St rain Gages Double Axis	2	E, μ, F_{tu}, ϵ
Coupon	Graphite	A B	10 10	3 3	Strain Gages Double Axis	2	E, μ F_{tu}, ϵ
Sandwich Beam	Boron	A B C	10 10 3	3 3 1	Strain Gage Single Axis	1	E, ϵ F_{tu}
Sandwich Beam	Graphite	A B C	10 10 3	3 3 1	Strain Gage Single Axis	1	E, ϵ F_{tu}

550°F Tests

Specimen Configuration	Aging Time (hrs)	Filament Material	Fiber Pattern	No. of Specimens	No. of Instrumented Specimens	Instrumentation*	Channels Per Specimen	Measured Data
Coupon	1/2	Boron	A B	10 10	3 3	Strain Gages Double Axis	2	E, μ, E_{tu}, ϵ
Coupon	1/2	Graphite	A B	10 10	3 3	Strain Gages Double Axis	2	E, μ, F_{tu}, ϵ
Coupon	100	Boron	A B	10 10	3 3	Strain Gages Double Axis	2	E, μ, F_{tu}, ϵ
Coupon	100	Graphite	A B	10 10	3 3	Strain Gages Double Axis	2	E, μ, F_{tu}, ϵ

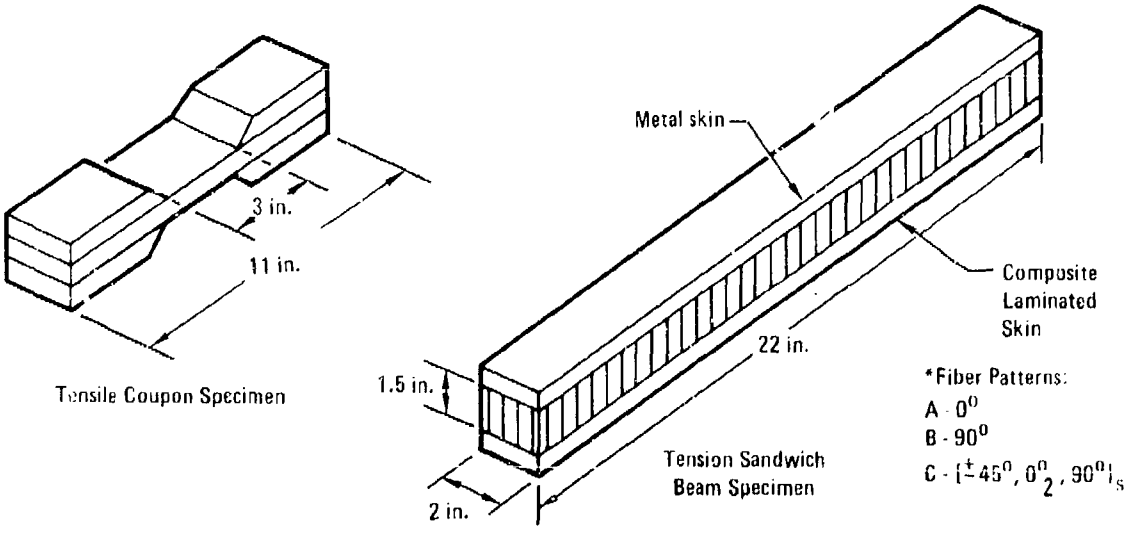

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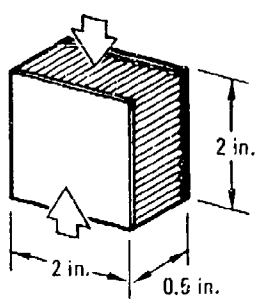
FIGURE 24
TESTS FOR DETERMINATION OF TENSION DESIGN ALLOWABLES

Room Temperature Tests

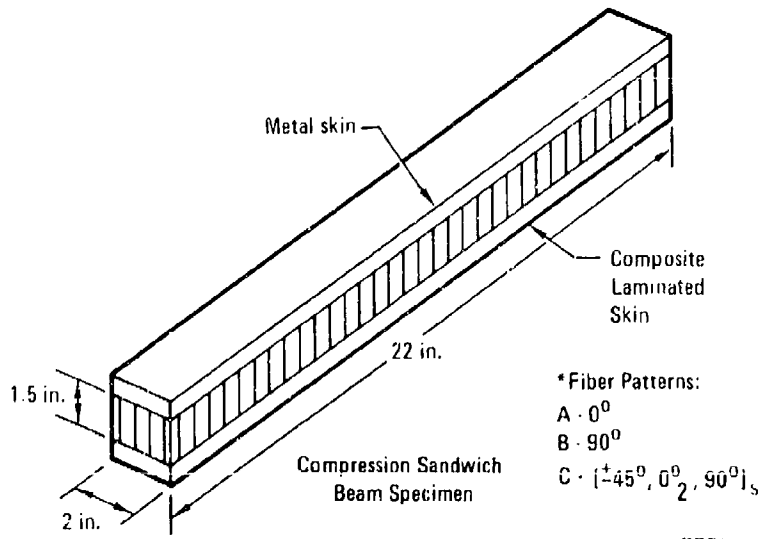
Specimen Configuration	Filament Material	Fiber* Pattern	No. of Specimens	No. of Instrumented Specimens	Instrumentation	Channels Per Specimen	Measured Data
Sandwich Beam 	Boron	A B C	10 10 3	3 3 1	Strain Gages Single Axis	1	E, ϵ F_{cu}
Sandwich Beam	Graphite	A B C	10 10 3	3 3 1	Strain Gages Single Axis	1	E, ϵ F_{cu}
Edgewise 	Boron	A B	10 10	3 3	Strain Gage Single Axis	1	E, ϵ F_{cu}
Edgewise	Graphite	A B	10 10	3 3	Strain Gage Single Axis	1	E, ϵ F_{cu}

550°F Tests

Specimen Configuration	Aging Time (hrs)	Filament Material	Fiber* Pattern	No. of Specimens	No. of Instrumented Specimens	Instrumentation	Channels Per Specimen	Measured Data
Edgewise	1/2	Boron	A B	10 10	3 3	Strain Gage Single Axis	1	E, ϵ F_{cu}
Edgewise	1/2	Graphite	A B	10 10	3 3	Strain Gage Single Axis	1	E, ϵ F_{cu}
Edgewise	100	Boron	A B	10 10	3 3	Strain Gage Single Axis	1	E, ϵ F_{cu}
Edgewise	100	Graphite	A B	10 10	3 3	Strain Gages Single Axis	1	E, ϵ F_{cu}



Edgewise Compression Specimen



Compression Sandwich Beam Specimen

*Fiber Patterns:
A - 0^0
B - 90^0
C - $[\pm 45^0, 0^0_2, 90^0]_s$

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FIGURE 25
TESTS FOR DETERMINATION OF COMPRESSION DESIGN ALLOWABLES

Room Temperature Tests

Specimen Configuration	Filament Material	Fiber* Pattern	No. of Specimens	No. of Instrumented Specimens	Instrumentation	Channels Per Specimen	Measured Data
Picture Frame	Boron	D	3	1	Strain Gages Rosette	3	G F_{su}
Picture Frame	Graphite	D	3	1	Strain Gages Rosette	3	G F_{su}

550°F Tests

Specimen Configuration	Soak Time (hrs)	Filament Material	Fiber* Pattern	No. of Specimens	No. of Instrumented Specimens	Instrumentation	Channels Per Specimen	Measured Data
Picture Frame		Boron	D	3	1	Strain Gages Rosette	3	G F_{su}
Picture Frame		Graphite	D	3	1	Strain Gages Rosette	3	G F_{su}

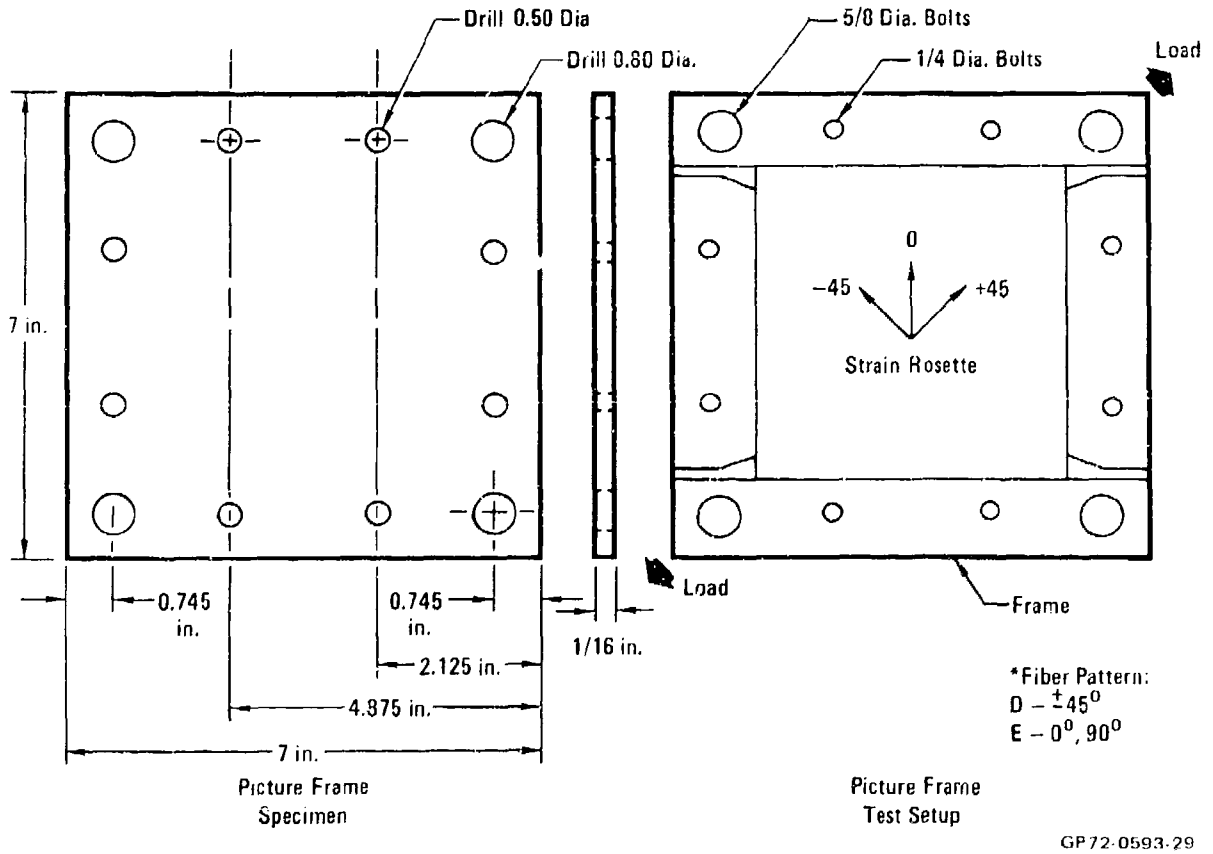

FIGURE 26
PICTURE FRAME TEST FOR DETERMINATION OF SHEAR DESIGN ALLOWABLES

Room Temperature Tests

Specimen Configuration	Filament Material	Fiber* Pattern	No. of Specimens	No. of Instrumented Specimens	Instrumentation	Channels Per Specimen	Measured Data
Rail Shear	Boron	D F	10 10	10 10	Strain Gages Rosette	3	G, F_{su}
Rail Shear	Graphite	D E	10 10	10 10	Strain Gages Rosette	3	G F_{su}

550°F Tests

Specimen Configuration	Soak Time (hrs)	Filament Material	Fiber* Pattern	No. of Specimens	No. of Instrumented Specimens	Instrumentation	Channels Per Specimen	Measured Data
Rail Shear		Boron	D E	10 10	10 10	Strain Gage Rosette	3 3	G F_{su}
Rail Shear		Graphite	D E	10 10	10 10	Strain Gage Rosette	3 3	G F_{su}

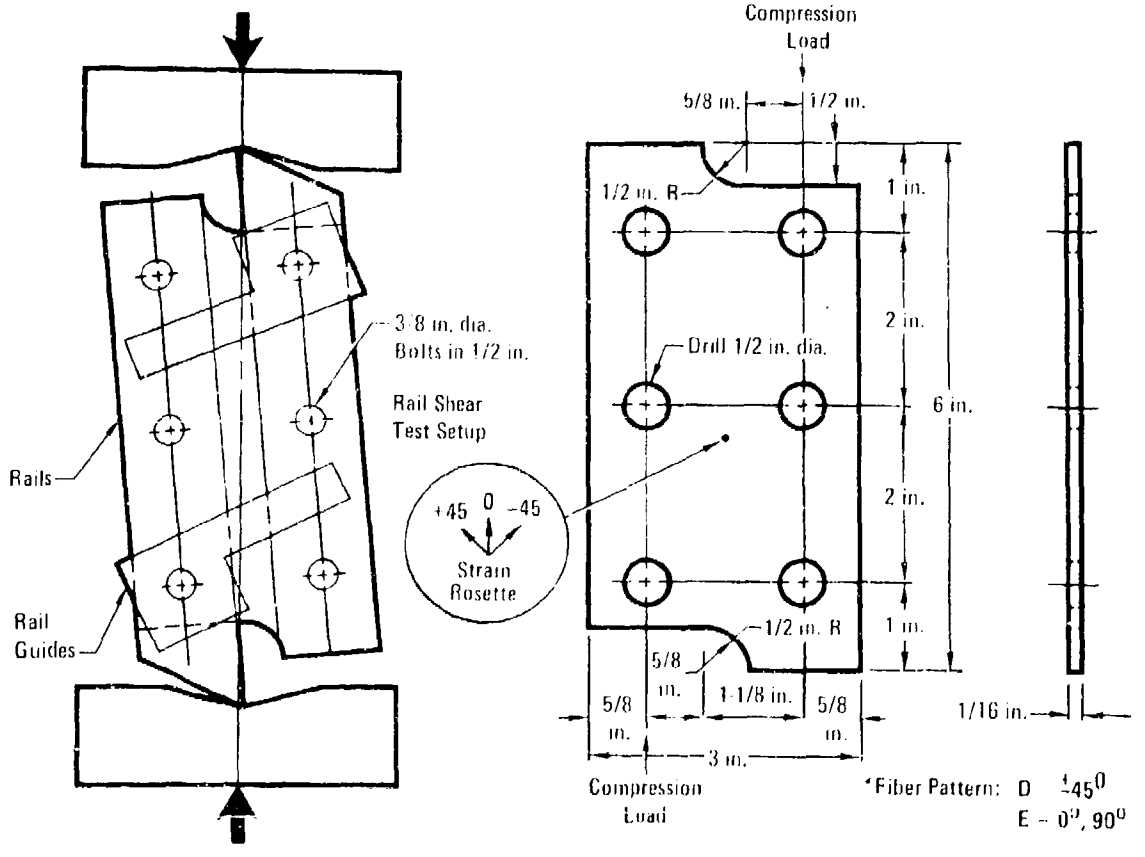

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FIGURE 27
RAIL SHEAR TEST FOR DETERMINATION OF SHEAR DESIGN ALLOWABLES

5.3 Boron/Polyimide Design Allowables (Boron/Skybond 703)

Test results for tension, compression and in-plane shear tests for both room temperature and 550°F are presented in Tables XXII through XXXIV. Typical stress-strain curves for all tests are presented in Figures 28 through 42. A summary of this data is shown in Tables XXXV through XXXVIII. "B" basis strength allowables, as reported in these tables, were calculated on the basis of ten test points. Elastic moduli and Poisson's ratio data are mean values.

5.3.1 Tension Design Allowables - Boron/polyimide longitudinal and transverse tension data are presented in Tables XXII through XXXIV. Representative stress-strain curves are given in Figures 28 through 42. Excellent longitudinal tension properties were obtained for this material at room temperature, with approximately 50 percent retention of strength properties at 550°F. However, the transverse tension properties were not as impressive with strength values of 5000 psi, and approximately 3000 microinches of strain. These lower transverse properties are attributed to the long post cure times for boron/polyimide; however in order to fabricate thick laminates the long post cure cycle was necessary.

5.3.2 Compression Design Allowables - Boron/polyimide longitudinal and transverse compression data is presented in Tables XXVIII through XXXII. Typical stress-strain curves are given in Figures 34 through 38. Room temperature mean longitudinal compression data obtained by the sandwich beam method, Table XXVII, is similar to boron/epoxy data. However, because of data scatter the "B" basis data is somewhat lower than boron/epoxy. This data scatter is attributed to variations in interlaminar shear properties that were obtained for the two batches of material used to fabricate these specimens. Test data was also obtained by the edgewise compression method at both room temperature and 550°F. This data is presented in Tables XXIX, XXX, and XXXII. Inspection of these test specimens indicated that there were mixed mode failures of shear and compression. As a result of the evaluation, it is felt that edgewise compression testing is not sufficient for establishing longitudinal compression design allowables. To obtain longitudinal compression data at 550°F three sandwich beam specimens were fabricated using high temperature honeycomb core.

These test results are presented in Table XXXI and Figure 36. The specimens exhibited good compression failures with approximately 30 percent retention of properties at 550°F. This is similar to boron/epoxy data at 350°F.

5.3.3 In-Plane Shear Design Allowables - The results of the (0°, 90°) and (±45°) rail shear tests for both room temperature and 550°F are presented in Tables XXXIII and XXXIV, respectively. Typical stress-strain diagrams are given in Figures 39 through 42. "B" basis allowables were established for (0°, 90°) and mean data for the (±45°) lay-ups.

TABLE XXII
BORON POLYIMIDE LONGITUDINAL AND TRANSVERSE SANDWICH BEAM TENSION PROPERTIES AT ROOM TEMPERATURE

Specimen Number	Laminate Orientation (degrees) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
7-138-1	0°	215000		
-2		223000		
-3		214000	7140	32.0
-4		212000	7210	30.8
-5		219000	7175	32.0
9-158-6	0°	233000	7560	32.2
-7		234000	7390	33.2
-8		229000	7740	32.5
-9		220000	7500	32.7
-10		229000	7630	32.6
F_{tu} Mean		222600	7420	32.2
F_{tu} "B"		203900		
7-138-1	90°	5120	2370	2.48
-2		5120	2240	2.02
-3		5160	2165	2.27
-4		5115		
-5		5075		
9-158-1	90°	5375	4050	2.06
-2		5536	4900	2.30
-3		5185	2025	3.32
-4		5271	3350	2.70
-5		5261		
F_{tu} Mean		5221	3014	2.45
F_{tu} "B"		4884		

Notes: (1) Type material – Boron/Skybond 703
(2) See Figures 28 and 29 for Stress-Strain Diagrams

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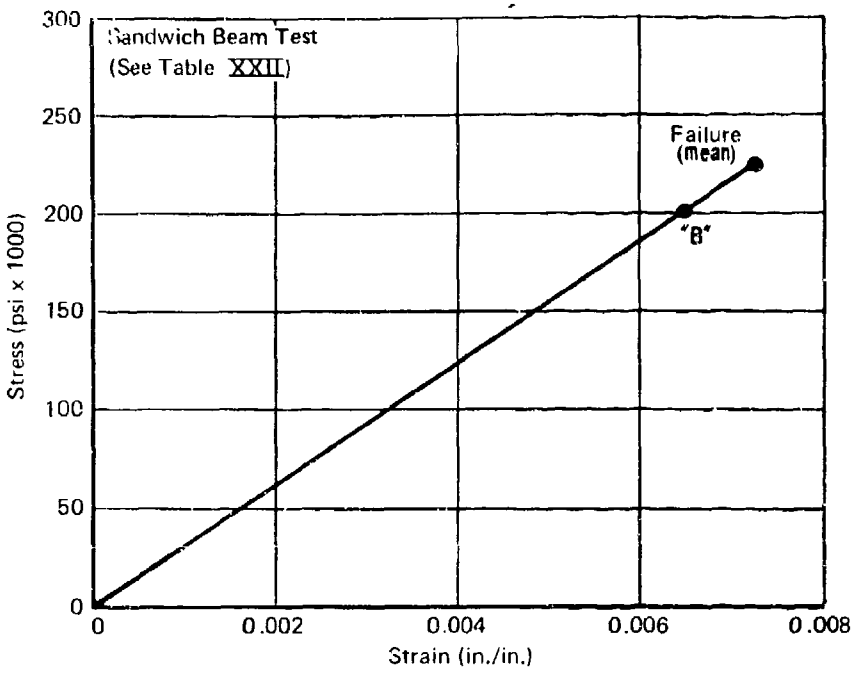

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FIGURE 28
TYPICAL TENSION STRESS - STRAIN DIAGRAM FOR BORON POLYIMIDE 0° LAMINATE AT ROOM TEMPERATURE
(Boron/Skybond 703)

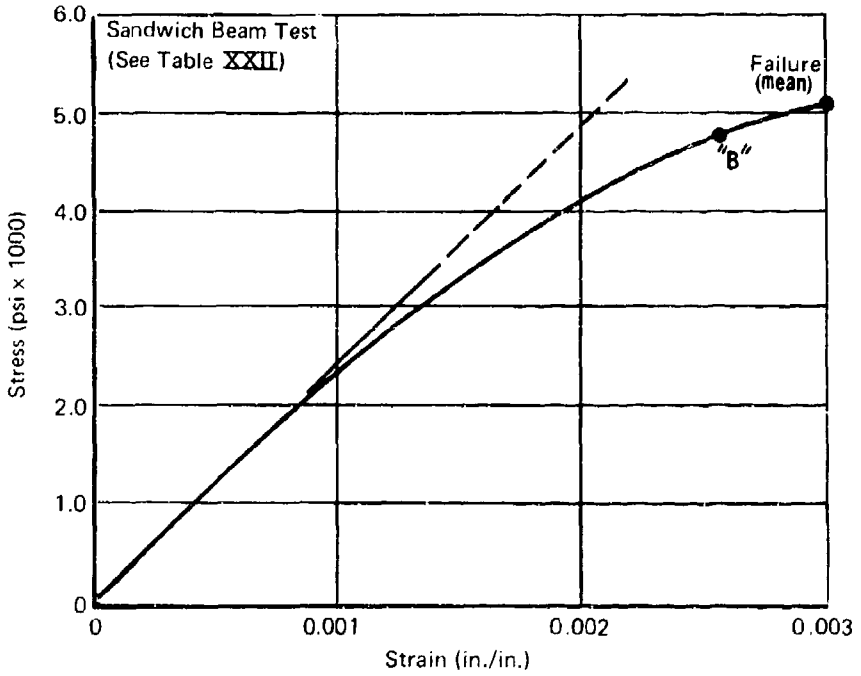

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FIGURE 29
TYPICAL TENSION STRESS - STRAIN DIAGRAM FOR BORON-POLYIMIDE 90° LAMINATE AT ROOM TEMPERATURE
(Boron/Skybond 703)

TABLE XXIII
BORON POLYIMIDE LONGITUDINAL COUPON TENSION MECHANICAL PROPERTY DATA AT ROOM TEMPERATURE

Specimen Number	Laminate Orientation (degrees) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
7-138-1	0°	167700		
-2		174300		
-3		172400		
-4		166100		
-5		171300		
7-158-6	0°	196670	6650	30.7
-7		195150		33.1
-8		180480		
-9		171440	5460	32.4
-10		178890		
F_{tu} Mean		177440	6055	32.0
F_{tu} "B"		152336		

Notes: (1) Type material – Boron/Skybond 703
(2) See Figures 30 and 31 for Stress-Strain Diagrams

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TABLE XXIV

BORON POLYIMIDE LONGITUDINAL AND TRANSVERSE TENSION MECHANICAL PROPERTY DATA AT 550°F 30 MINUTE SOAK

Specimen Number	Laminate Orientation (degrees) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
7-138-21	0°	138300		
-22		141800		
-23		122000		
-24		120700		
-25		127300		
7-158-26	0°	120400		
-27		125000		
-28		122200	4810	27.8
-29		118700	4810	27.2
-30		124000	4450	29.1
F_{tu} Mean		126000	4690	28.0
F_{tu} "B"		107600		
9-158-1	90°	5600		
-2		6030		
-3		5545		
-4		5680		
-5		5410		
9-158-6	90°	5440		
-7		5460		
9-163-8		5430	5425	1.90
-9		5790	8500	1.50
-10		5860	8275	1.63
F_{tu} Mean		5620	7400	1.68
F_{tu} "B"		5130		

Notes:

(1) Type material - Boron/Skybond 703

(2) See Figures 30 and 31 for Stress - Strain Diagrams

(3) 0° - Coupon Test, 90° - Sandwich Beam Test

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TABLE XXV
BORON POLYIMIDE LONGITUDINAL AND TRANSVERSE TENSION MECHANICAL PROPERTY DATA AT 550°F 100 HOUR SOAK

Specimen Number	Laminate Orientation (degrees) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
7-138-11	0°	117800		
-12		120800		
-13		122800		
-14		123200		
-15		123100		
7-158-16	0°	127700		
-17		134900		
-18		124200		
-19		136700	5000	32.7
-20		143500		
F_{tu} Mean		127500	5000	32.7
"B"		108200		
9-158-11	90°	4950		
-12		4990		
-13		5140		
-14		5276		
-15		4872		
9-163-16	90°	5330		
-17		5410		
-18		5760	8080	1.55
-19		5750	7800	1.65
-20		5720	7950	1.52
F_{tu} Mean		5320	7940	1.56
"B"		4520		

Notes: (1) Type material – Boron/Skybond 703
(2) See Figures 30 and 31 for Stress-Strain Diagrams
(3) 0° – Coupon Test, 90° – Sandwich Beam Test

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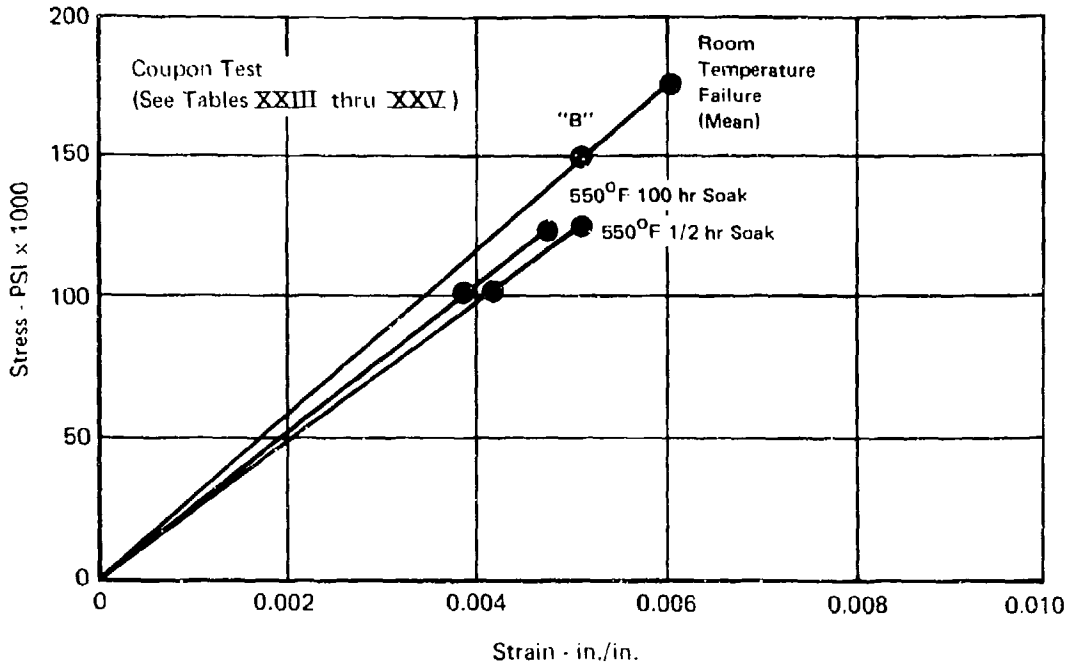

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FIGURE 30
TYPICAL TENSION STRESS-STRAIN DIAGRAMS FOR BORON POLYIMIDE 0° LAMINATE
(Boron/Skybond 703)

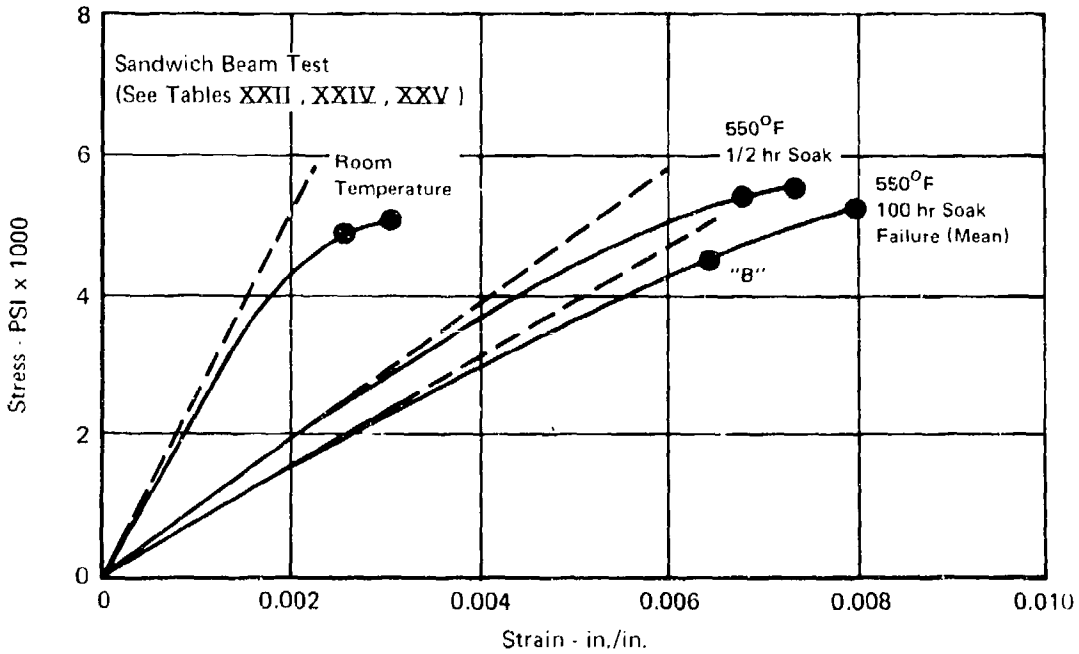

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FIGURE 31
TYPICAL TENSION STRESS-STRAIN DIAGRAMS FOR BORON POLYIMIDE 90° LAMINATE
(Boron/Skybond 703)

TABLE XXVI
BORON POLYIMIDE SANDWICH BEAM TENSION PROPERTIES AT ROOM TEMPERATURE

Specimen Number	Laminate Orientation (degrees) (Loading at 0^o)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
9-158-1	$[\pm 45^o, 0_2^o, 90^o]_S$	129000	8470	17.0
-2		113000	7820	15.9
-3		116000	7980	15.3
F_{tu} Mean		119000	8090	16.0

Notes: (1) Type material – Boron/Skybond 703
(2) Resin content of incoming material
(3) See Figures 32 and 33 for Stress-Strain Diagrams

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TABLE XXVII
BORON POLYIMIDE SANDWICH BEAM COMPRESSION PROPERTIES AT ROOM TEMPERATURE

Specimen Number	Laminate Orientation (degrees) (Loading at 0^o)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
9-158-4	$[\pm 45^o, 0_2^o, 90^o]_S$	194000	13030	16.0
-5		176000	10650	17.5
-6		191000	12550	16.2
F_{cu} Mean		187000	12080	16.5

Notes: (1) Type material – Boron/Skybond 703
(2) See Figures 32 and 33 for Stress-Strain Diagrams

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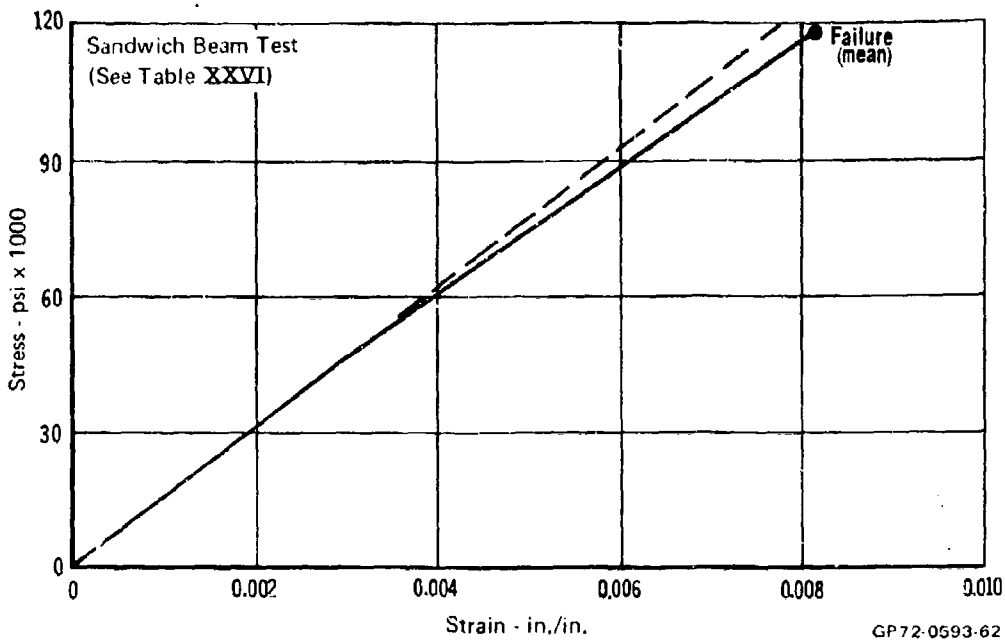

FIGURE 32
TYPICAL TENSION STRESS-STRAIN DIAGRAM FOR BORON POLYIMIDE
$[\pm 45^{o}, 0_{2}^{o}, 90^{o}]_{S}$ LAMINATE AT ROOM TEMPERATURE
(Boron/Skybond 703)

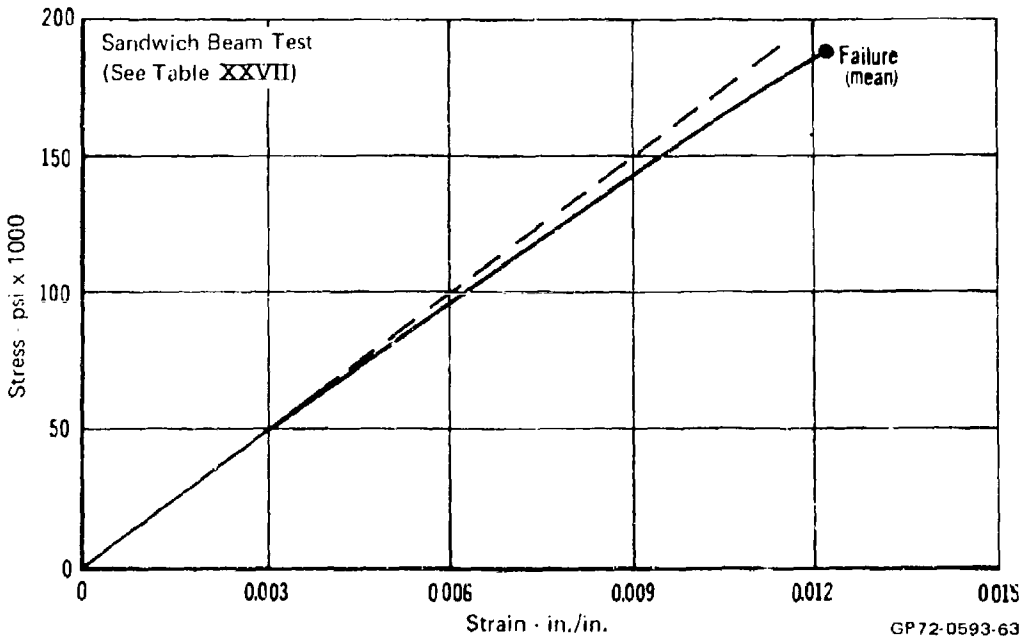

FIGURE 33
TYPICAL COMPRESSION STRESS-STRAIN DIAGRAM FOR BORON POLYIMIDE
$[\pm 45^{o}, 0_{2}^{o}, 90^{o}]_{S}$ LAMINATE AT ROOM TEMPERATURE
(Boron/Skybond 703)

TABLE XXVIII
BORON POLYIMIDE LONGITUDINAL AND TRANSVERSE SANDWICH BEAM COMPRESSION PROPERTIES AT ROOM TEMPERATURE

Specimen Number	Laminate Orientation (degrees) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
7-138-11	0°	334000	10220	32.7
-12		313000		
-13		306000	9250	32.0
-14		304000	9450	32.7
-15		310000		
9-158-14	0°	422000	13000	33.0
-15		435000	14250	33.0
-16		455000	14100	32.7
-17		404000	12600	32.5
-18		374000	11625	33.0
F_{cu} Mean		365700	11845	32.7
F_{cu} "B"		226200		
7-138-6	90°	26200	11536	2.22
-7		26250	13580	3.06
-8		24000	14420	2.16
-9		23700		
-10		22400		
9-158-9	90°	15140		
-10		21650		
-11		17450	11270	2.53
-12		15290	7300	2.07
-13		15820	11125	2.61
F_{cu} Mean		20790	11539	2.44
F_{cu} "B"		10280		

Notes: (1) Type material -- Boron/Skybond 703
(2) See Figures 34 and 35 for Stress-Strain Diagrams

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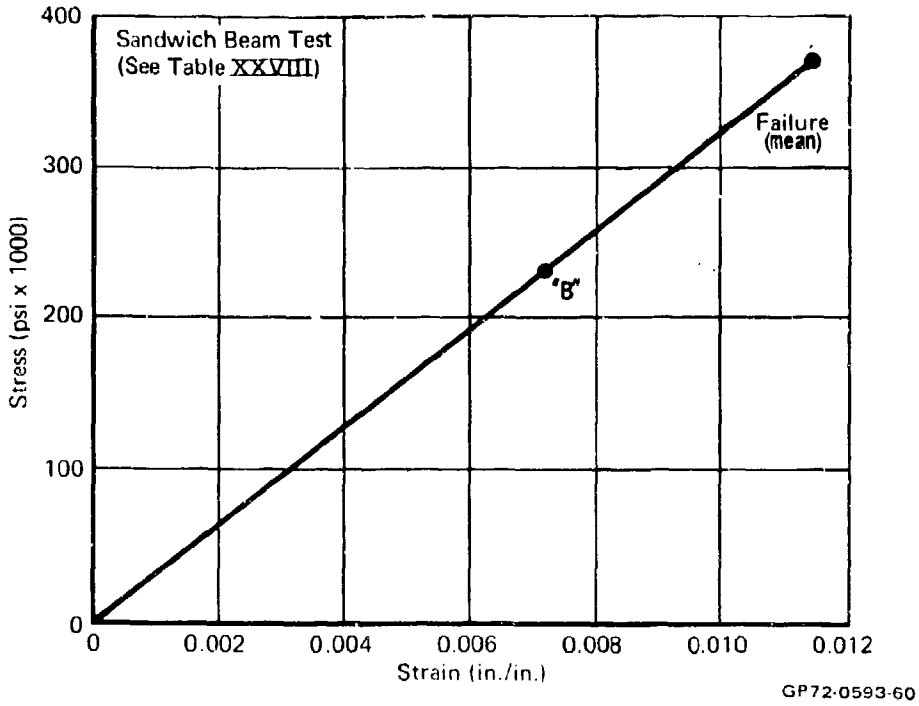

FIGURE 34
TYPICAL COMPRESSION STRESS - STRAIN DIAGRAM FOR BORON POLYIMIDE 0° LAMINATE AT ROOM TEMPERATURE (BORON/SKYBOND 703)

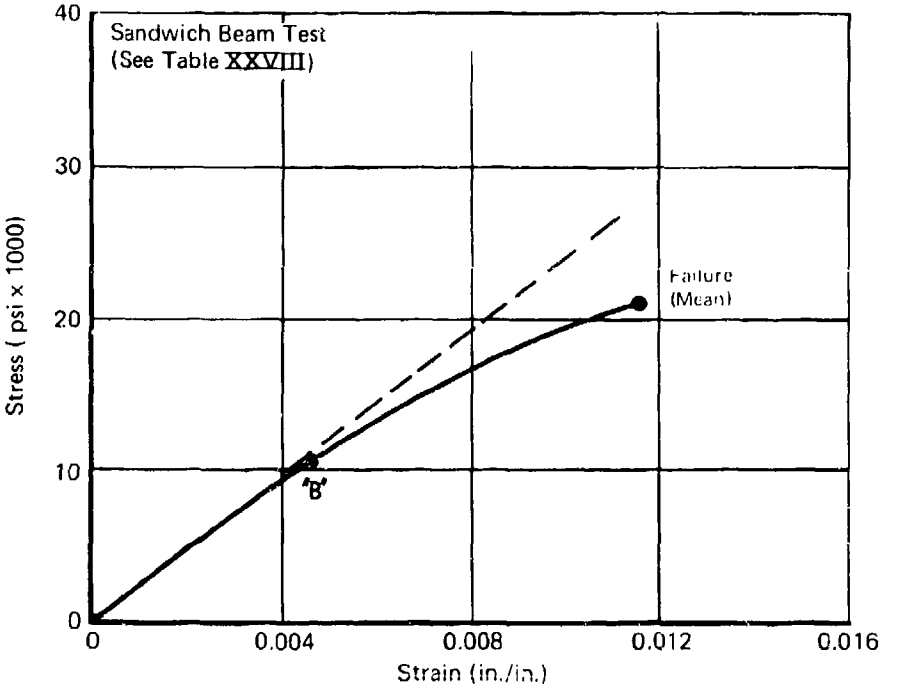

FIGURE 35
TYPICAL COMPRESSION STRESS-STRAIN DIAGRAM FOR BORON POLYIMIDE 90° LAMINATE AT ROOM TEMPERATURE (BORON/SKYBOND 703)

TABLE XXIX

BORON POLYIMIDE LONGITUDINAL AND TRANSVERSE EDGEWISE COMPRESSION MECHANICAL PROPERTY DATA AT ROOM TEMPERATURE

Specimen Number	Laminate Orientation (degrees) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
9-162-1	0°	134677		
-2		134409		
-3		133333		
-4		132796		
-5		132258		
9-162-6	0°	138172		
-7		123656		
-8		116398	1950	37.8
-9		127554	3085	37.0
-10		96774	2700	41.5
F_{cu} Mean		127000	2575	38.8
F_{cu} "B"		97930		
9-158-1	90°	32640		
-2		32060		
-3		34850		
-4		31180		
-5		33090		
9-158-6	90°	33680		
-7		33820		
-8		34560	9216	4.73
-9		33820	9258	4.50
-10		38090	8470	5.37
F_{cu} Mean		33780	8981	4.86
F_{cu} "B"		29350		

Notes: (1) Type material – Boron/Skybond 703
(2) See Figures 37 and 38 for Stress - Strain Diagrams

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TABLE XXX
BORON POLYIMIDE LONGITUDINAL AND TRANSVERSE EDGEWISE COMPRESSION MECHANICAL PROPERTY DATA AT 550°F 30 MINUTE SOAK

Specimen Number	Laminate Orientation (degrees) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
9-162-11	0°			
-12		48790		
-13		63978		
-14		48062		
-15		48450		
9-162-16	0°	39478		
-17		46124		
-18		53100	680	43.2
-19		55600	1650	41.5
-20		52000	920	28.5
F_{cu} Mean		50620	1083	37.7
F_{cu} "B"		33930		
7-158-11	90°	16350		
-12		17320		
-13		16800		
-14		16060		
-15		16000		
7-158-16	90°	15880		
-17		16650		
-18		15500	10360	2.24
-19		12059	12740	0.946
-20		13295	13160	1.26
F_{cu} Mean		15590	12087	1.50
F_{cu} "B"		11724		

(1) Type material - Boron/Skybond 703

(2) See Figures 37 and 38 for Stress - Strain Diagrams

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TABLE XXXI
BORON POLYIMIDE LONGITUDINAL SANDWICH BEAM COMPRESSION MECHANICAL PROPERTY DATA AT 550°F 30 MINUTE SOAK

Specimen Number	Laminate Orientation (Deg) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (msi)
8-163-1	0°	110,100	3350	30.0
-2		118,900	4130	30.0
-3		91,800	4000	28.0
F_{cu} Mean		107,000	3830	29.3

Note: (1) Type Material - Boron/Skybond 703

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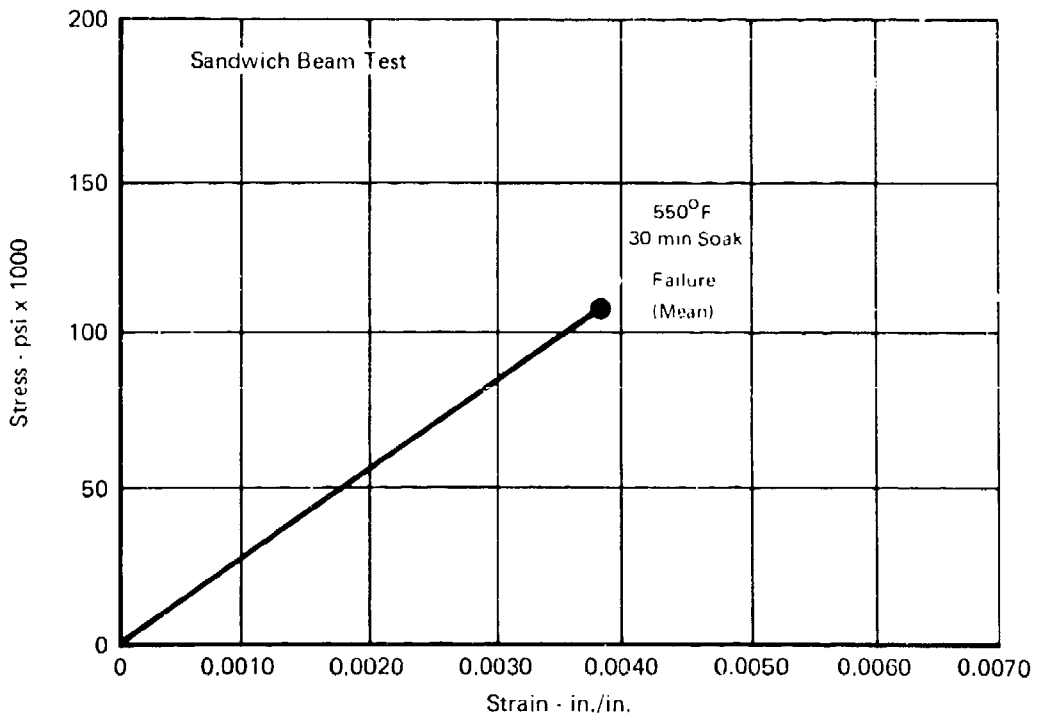

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FIGURE 36
TYPICAL COMPRESSION STRESS-STRAIN DIAGRAM FOR BORON POLYIMIDE 0° LAMINATE
(Boron/Skybond 703)

TABLE XXXII
BORON POLYIMIDE LONGITUDINAL AND TRANSVERSE EDGEWISE COMPRESSION MECHANICAL PROPERTY DATA 550°F 100 HOUR SOAK

Specimen Number	Laminate Orientation (degrees) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
9-162-21	0°	53495		
-22		47984		
-23		40457		
-24		44355		
-25		56586		
9-162-26	0°	54167		
-27		–		
-28		(3)		19.6
-29		(3)		18.1
-30		(3)		18.9
F_{cu} Mean		49510	(3)	18.9
F_{cu} "B"		30570		
7-158-21	90°	15910		
-22		16740		
-23		15530		
-24		15940		
-25		16820		
7-158-26	90°	15180		
-27		14880		
-28		13941	8120	1.35
-29		12235	9870	0.932
-30		14235	10290	0.913
F_{cu} Mean		15140	9427	1.07
F_{cu} "B"		11860		

(1) Type material - Boron/Skybond 703
(2) See Figures 37 and 38 for Stress - Strain Diagrams
(3) Honeycomb core buckled invalidating failure points

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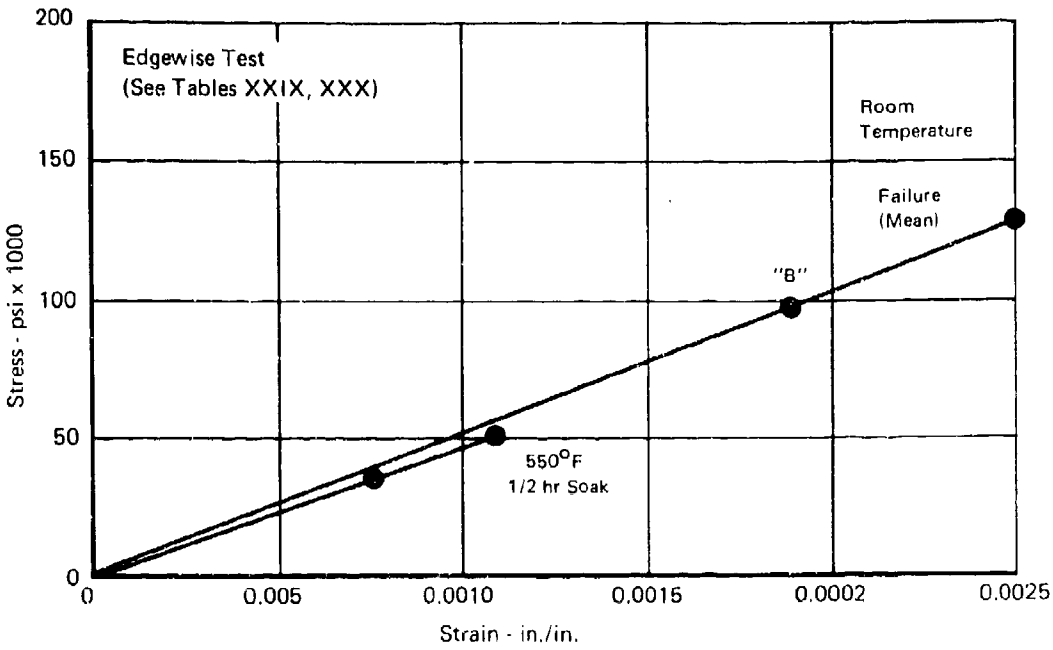

FIGURE 37
TYPICAL COMPRESSION STRESS-STRAIN DIAGRAMS FOR BORON POLYIMIDE 0° LAMINATE
(Boron/Skybond 703)

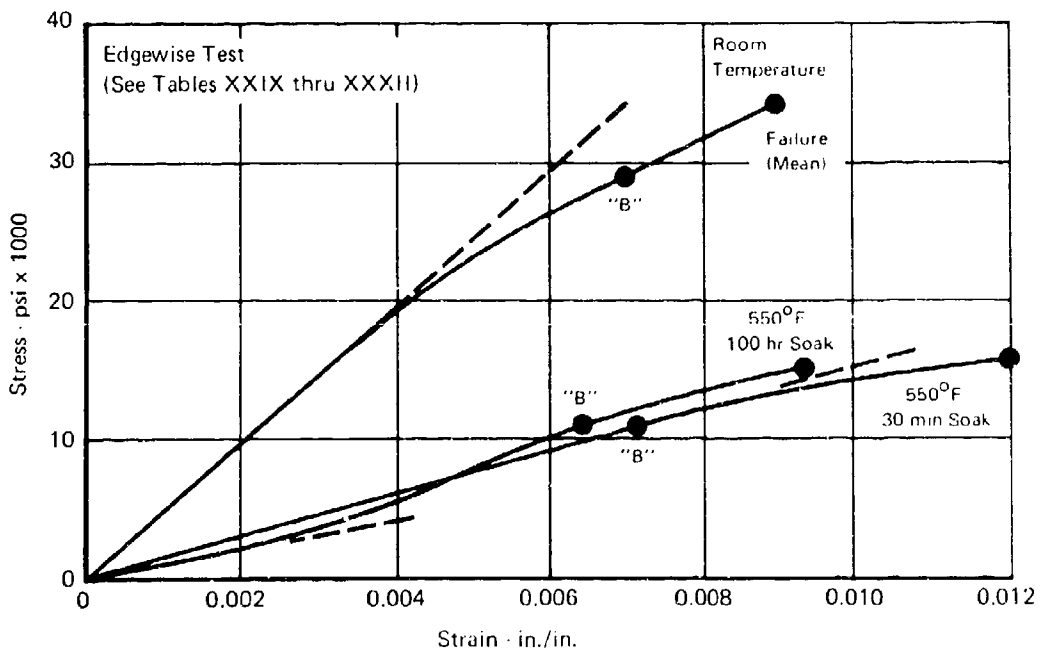

FIGURE 38
TYPICAL COMPRESSION STRESS-STRAIN DIAGRAMS FOR BORON POLYIMIDE 90° LAMINATE
(Boron/Skybond 703)

TABLE XXXIII
BORON POLYIMIDE RAIL SHEAR MECHANICAL PROPERTY DATA FOR $0^o - 90^o$ LAMINATE

Specimen Number	Test Temperature	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
9-158-1	Room Temperature	9972	54000	0.810
-2		10072	78000	0.700
-3		10530	62200	1.020
-4		10050	37300	0.850
-5		10169	47000	0.810
9-158-6	Room Temperature	10390	88000	0.892
-7		11036	57700	0.892
-8		10193	46700	0.810
-9		10072	41000	1.020
-10		10580	60000	1.140
F_{su} Mean		10310	57190	0.894
"B"		9346		
9-158-1	550°F	6490	41400	0.430
-2		7060	34800	0.285
-3		5180	24800	0.350
-4		5790	35200	0.350
-5		6310	40800	0.430
9-158-6	550°F	5220	35600	0.430
-7		6940	37500	0.430
-8		6120	33000	0.420
-9		5530	36200	0.340
-10		6000	(3)	(3)
F_{su} Mean		6060	247400	0.385
"B"		4520		

Notes: (1) Type material – Boron/Skybond 703

(2) See Figures 39 and 40 for Stress - Strain Diagrams

(3) Strain Gauge Failed in Run

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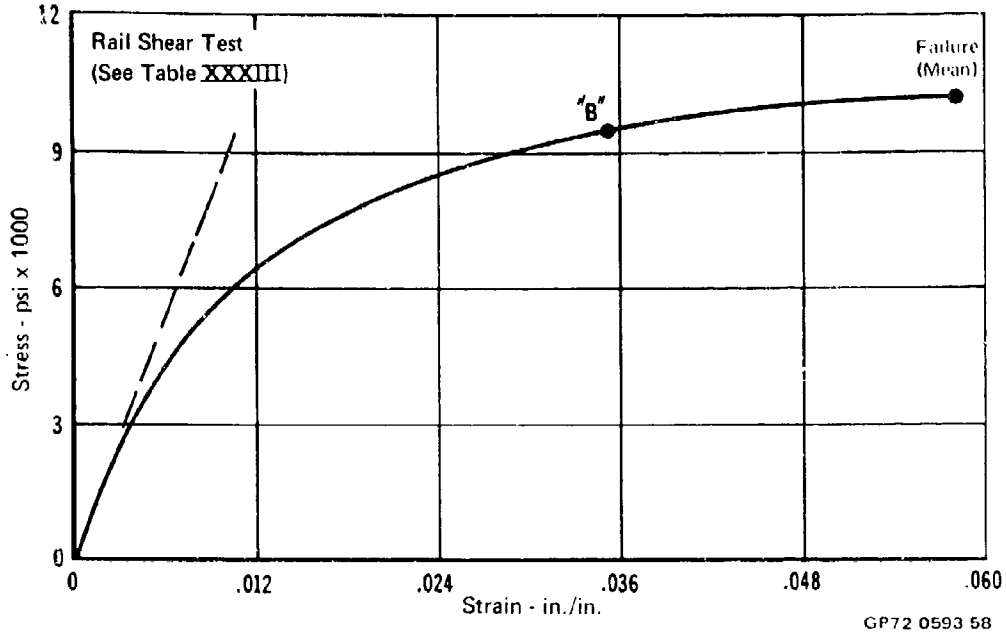

FIGURE 39
TYPICAL SHEAR STRESS-STRAIN DIAGRAM FOR BORON POLYIMIDE
$0^o - 90^o$ LAMINATE AT ROOM TEMPERATURE
(Boron/Skybond 703)

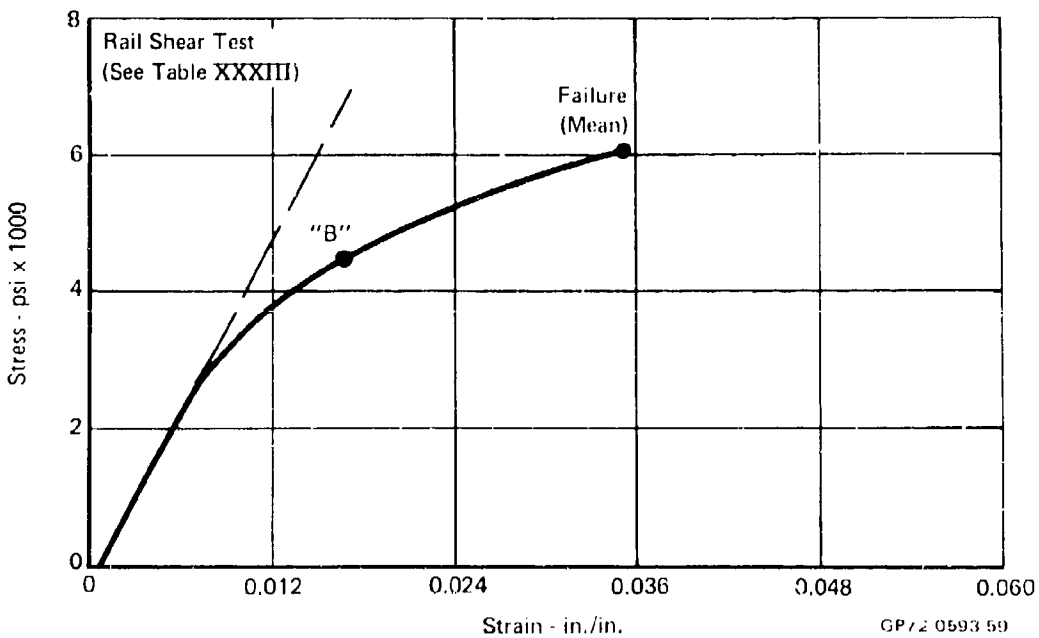

FIGURE 40
TYPICAL SHEAR STRESS-STRAIN DIAGRAM FOR BORON POLYIMIDE
$0^o - 90^o$ LAMINATE AT 550^oF
(Boron/Skybond 703)

TABLE XXXIV
BORON POLYIMIDE RAIL SHEAR MECHANICAL PROPERTY DATA FOR ±45° LAMINATE

Specimen Number	Test Temperature	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
9-163-1	Room Temperature	60,600	10,900	7.30
-2		57,900	10,600	8.60
-3		46,990*	7,980*	7.93
F_{su} Mean		59,250	10,750	7.94
9-163-4	550°F	44,420	(3)	(3)
-5		41,120	10,550	8.8
-6		43,260	9,600	6.2
F_{su} Mean		42,930	10,080	7.5

Notes: (1) Type material – Boron/Skybond 703
(2) See Figures 41 and 42 for Stress - Strain Diagrams
(3) Strain gauge failed at start of run

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*Specimen hole delaminations invalidated failure points.

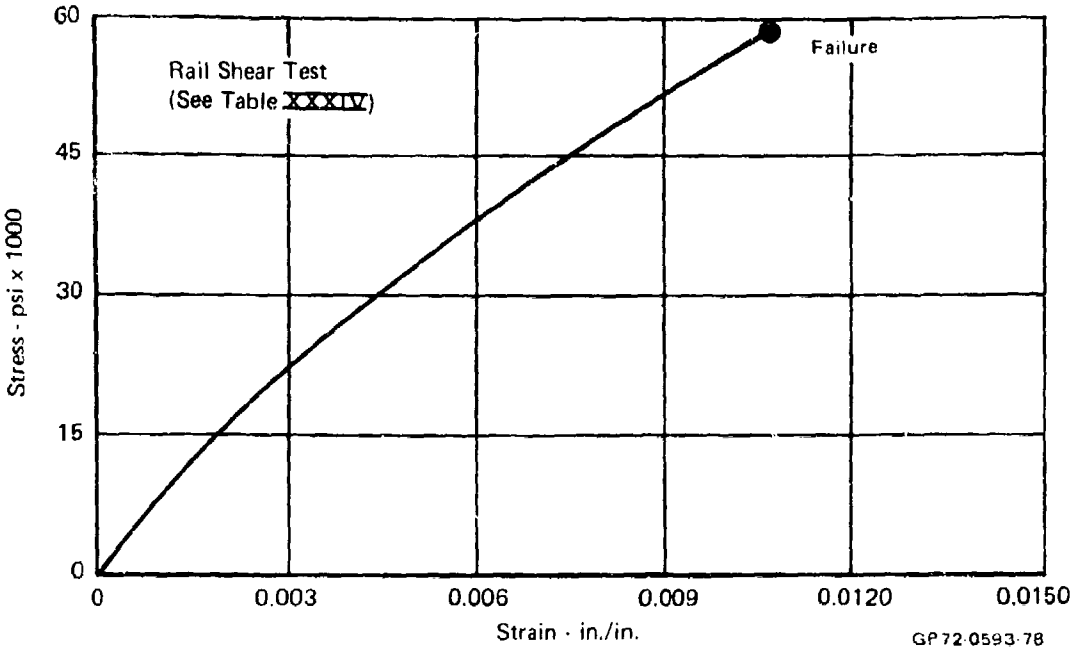

FIGURE 41
TYPICAL SHEAR STRESS-STRAIN DIAGRAM FOR BORON POLYIMIDE ±45° LAMINATE AT ROOM TEMPERATURE
(Boron/Skybond 703)

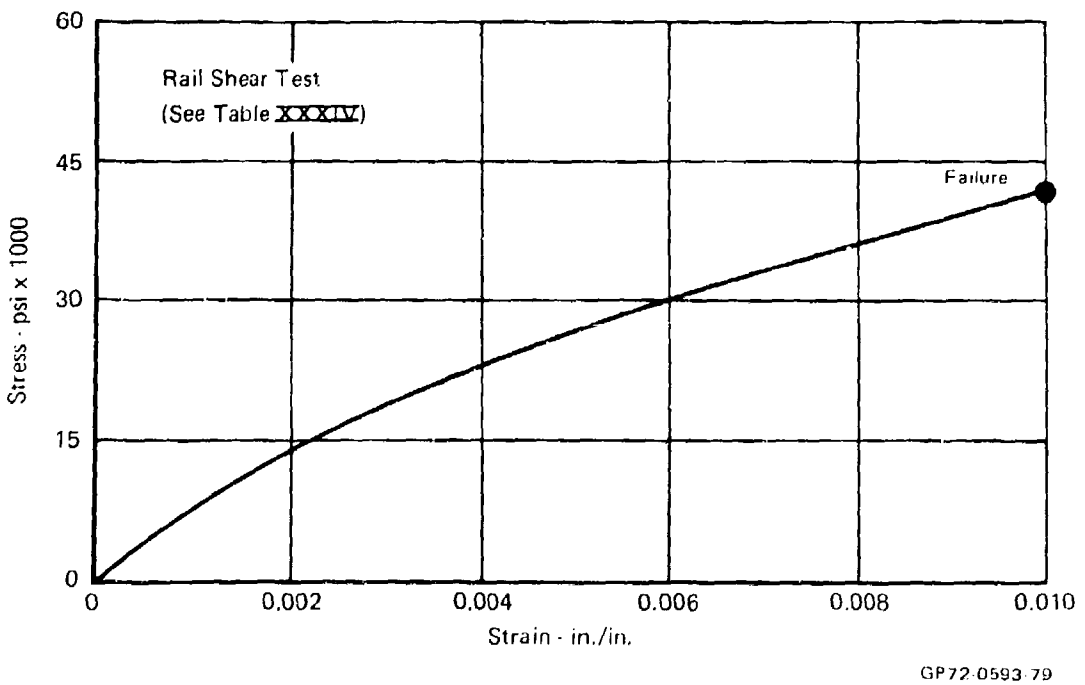

FIGURE 42
TYPICAL SHEAR STRESS-STRAIN DIAGRAM FOR BORON POLYIMIDE ±45° LAMINATE AT 550°F
(Boron/Skybond 703)

TABLE XXXV
BORON POLYIMIDE/SKYBOND 703
LONGITUDINAL "B" BASIS DESIGN ALLOWABLES

Property	Room Temperature	550°F 30 minutes SOAK	550°F 100 hours SOAK
F_{tu}	203900 psi	107600 psi	108200 psi
F_{cu}	226200 psi	33930* psi	30570 psi
E	32.3 msi	28.0 msi	32.7 msi
ν	0.31	0.30	0.19

Notes: (1) Elastic Constants are Mean Values

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*Mean value = 106950 from sandwich beam test (Ref. Table XXXI)

TABLE XXXVI
BORON POLYIMIDE/SKYBOND 703
TRANSVERSE "B" BASIS DESIGN ALLOWABLES

Property	Room Temperature	550°F 30 minutes SOAK	550°F 100 hours SOAK
F_{tu}	4880 psi	5130 psi	4520 psi
F_{cu}	29350 psi	11720 psi	11860 psi
E	2.45 msi	1.68 msi	1.56 msi

Notes: (1) Elastic Constant is Mean Value

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TABLE XXXVII
BORON POLYIMIDE/SKYBOND 703 0^{o}-90^{o} IN PLANE SHEAR "B" BASIS DESIGN ALLOWABLES

Property	Room Temperature	550°F
F_{su}	9346 psi	4520 psi
G	0.894 mis	0.385 msi

Notes: (1) Elastic Constant is Mean Value

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TABLE XXXVIII
BORON POLYIMIDE/SKYBOND 703 $\pm 45^{o}$ IN PLANE SHEAR MEAN TEST RESULTS

Property	Room Temperature	550°F
F_{su}	59250 psi	42190 psi
G	7.94 msi	7.5 msi

GP72-0593-44

5.4 Graphite/Polyimide Design Allowables (Modmor II Graphite/Skybond 703)

Tension, compression, and in-plane shear properties for both room temperature and 550°F are presented in Tables XXXIX through L. Stress-strain curves are also presented in Figures 43 through 55. A summary of this data is shown in Tables LI through LIV. "B" basis strength allowables, as reported in these tables, were calculated on the basis of ten test points. Elastic modulus and Poisson's ratio data are mean values.

5.4.1 Tension Design Allowables - Graphite/polyimide longitudinal and transverse tension data are presented in Tables XXXIX through XLII. Representative stress-strain curves are given in Figures 43 through 47. The sandwich beam average ultimate longitudinal tensile strength at room temperature, presented in Table XXXIX, is 21 percent greater than the corresponding coupon value, presented in Table XL. The sandwich beam "B" basis ultimate longitudinal tensile strength at room temperature is 32.5 percent greater than the corresponding coupon value as presented in Tables XXXIX and XL. The value calculated for coupon "B" basis ultimate longitudinal tensile strength at room temperature (see Table XL) is low because of the scatter present in the test data.

The sandwich beam average ultimate transverse tensile strength value, presented in Table XXXIX is 390 percent greater than the corresponding coupon value, presented in Table XL. These coupons did not exhibit good tension failures. For this reason all 550°F transverse tension data was developed by the sandwich beam method as presented in Tables XLI and XLII.

5.4.2 Compression Design Allowables - The sandwich beam average ultimate longitudinal compressive strength presented in Table XLV is 75 percent greater than the longitudinal edgewise average ultimate compressive strength, Table XLVI at room temperature. Test data obtained from longitudinal edgewise compression test presented in Tables XLVI through XLVIII were much lower than expected. Representative stress-strain curves are presented in Figures 51 and 52. Inspection of the test specimens indicated that there were mixed mode failures of shear and compression. As a result of this evaluation, it is felt that edgewise compression testing is not sufficient for establishing longitudinal compression design allowables. The room temperature longitudinal sandwich beam "B" basis ultimate compressive strength is 65 percent greater than the longitudinal edgewise "B" basis ultimate compressive strength (Table XLV and Table XLVI. Typical stress-strain curves are shown in Figures 49 and 50.

The sandwich beam average ultimate transverse compressive strength is 6 percent less thatn the edgewise average ultimate transverse compressive strength at

room temperature. The sandwich beam "B" basis ultimate transverse compressive strength is approximately the same as the edgewise "B" basis ultimate transverse compressive strength at room temperature, Table XLV and XLVI. Therefore, good agreement was obtained between the two test methods for transverse compression. Summary design allowables are presented in Table LI and typical stress-strain curves are shown in Figure 52.

5.4.3 In-Plane Shear Design Allowables - The results of the (0°, 90°) and (±45°) rail shear tests for both room temperature and 550°F are presented in Table XLIX and L, respectively. Representative stress-strain curves are given in Figures 53 through 55. These values for graphite/polyimide are approximately 30 percent lower than graphite/epoxy in plane shear values (15000 psi) at room temperature. In-plane shear allowables are presented in the summary tables (Table LIII and LIV).

TABLE XXXIX

GRAPHITE POLYIMIDE LONGITUDINAL AND TRANSVERSE SANDWICH BEAM TENSION PROPERTIES AT ROOM TEMPERATURE

Specimen Number	Laminate Orientation (degrees) (Loading at 0^o)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
6-131-1	0^o	216000	9466	21.4
-2		205000	8560	23.1
-3		201000		
-4		218000		
6-132-6	0^o	210000	8050	25.2
-7		218000		
-8		218000		
-9		204000		
-10		213000		
F_{tu} Mean		213000	8692	23.2
F_{tu} "B"		193800		
6-134-1	90^o	9250	4430	1.88
-2		9475	4655	2.22
-3		10350		
-4		9075		
-5		9350		
6-135-6	90^o	10148	4520	1.98
-7		10620		
-8		10500		
-9		10150		
-10		10000		
F_{tu} Mean		9890	4535	2.02
F_{tu} "B"		8580		

Notes: (1) Type material - MODMOR II Graphite/Skybond 703
(2) See Figures 43 and 44 for Stress - Strain Diagrams

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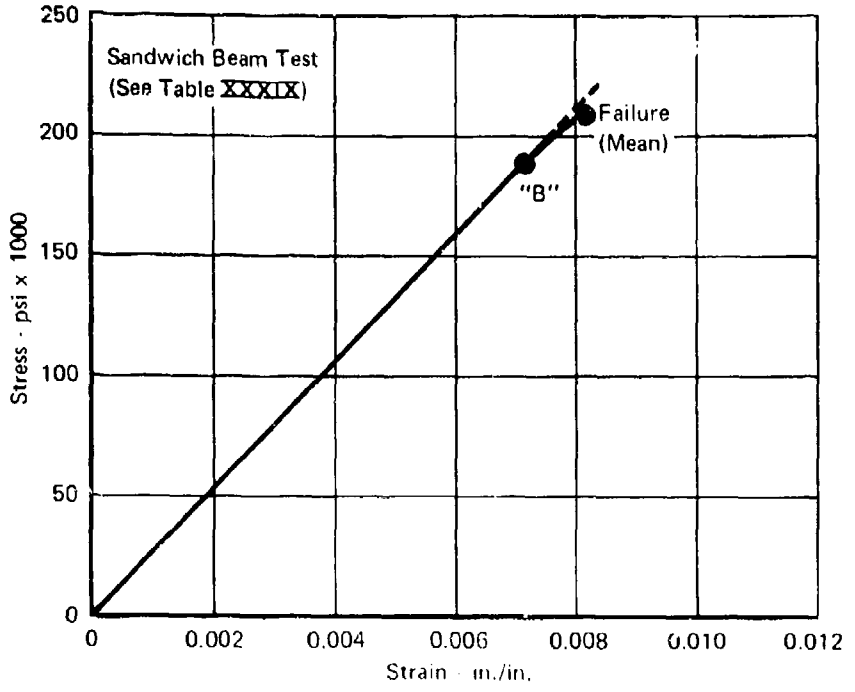

FIGURE 43
TYPICAL TENSION STRESS-STRAIN DIAGRAM FOR GRAPHITE POLYIMIDE 0° LAMINATE AT ROOM TEMPERATURE
(MODMOR II/Skybond 703)

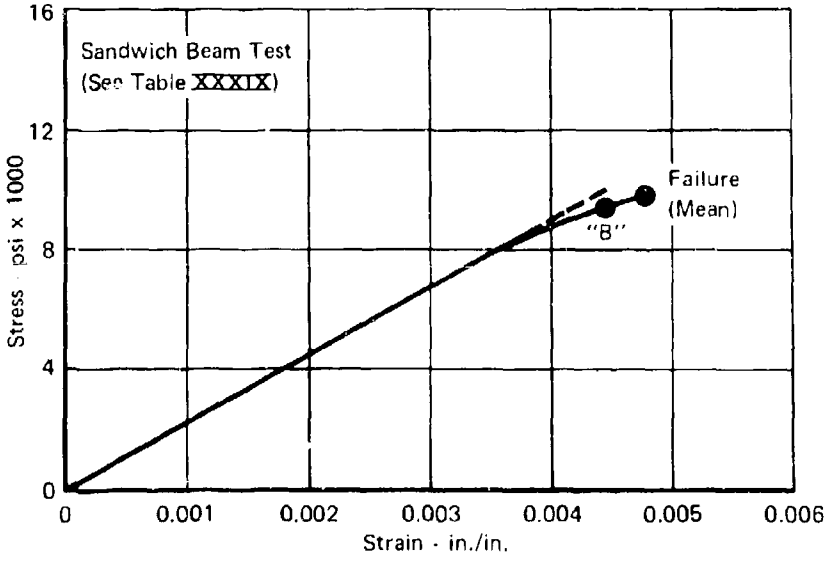

FIGURE 44
TYPICAL TENSION STRESS-STRAIN DIAGRAM FOR GRAPHITE POLYIMIDE 90° LAMINATE AT ROOM TEMPERATURE
(MODMOR II/Skybond 703)

TABLE XL
GRAPHITE POLYIMIDE LONGITUDINAL AND TRANSVERSE COUPON TENSION MECHANICAL PROPERTY DATA AT ROOM TEMPERATURE

Specimen Number	Laminate Orientation (degrees) (Loading at 0^o)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
6-133-1	0^o	176000		
-2		186000		
-3		186000		
-4		185000		
-5		180000		
6-134-6	0^o	151000	9240	17.3
-7		190000		
-8		160000	9170	17.15
-9		179000	9700	18.7
-10		169000		
F_{tu} Mean		176200	9370	17.7
F_{tu} "B"		146600		
6-131-1	90^o	4570		
2		4080		
6-133-3		2530		
4		2060		
5		1660		
6-133-6	90^o	1960	1950	1.05
7		2100	1980	1.14
8		3040	2890	0.98
9		820	1210	0.58
-10		2480	1370	1.20
F_{tu} Mean		2530	1880	0.99
F_{tu} "B"				

Notes: (1) Type material - MODMOR II Graphite/Skybond 703
(2) See Figures 45 and 46 for Stress - Strain Diagrams

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TABLE XLI
GRAPHITE POLYIMIDE LONGITUDINAL AND TRANSVERSE TENSION MECHANICAL PROPERTY DATA AT 550°F 30 MINUTE SOAK

Specimen Number	Laminate Orientation (degrees) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
6-133-11	0°	167000		
-12		180000		
-13		166000		
-14		164000		
-15		167000		
6-134-16	0°	167000	7950	20.1
-17		176000	8400	22.8
-18		163000	5936	28.0
-19		174000		
-20		167000		
F_{tu} Mean		169100	7429	23.6
F_{tu} "B"		156000		
6-131-1	90°	4190		
-2		4570		
-3		4280		
-4		4500		
-5		4140		
6-131-6	90°	4240		
-7		4560		
-8		4400	4800	1.16
-9		4280	5500	1.07
-10		4280	4700	1.23
F_{tu} Mean		4340	5000	1.15
F_{tu} "B"		3980		

Notes: (1) Type material - MODMOR II Graphite/Skybond 703
(2) See Figures 45 and 46 Stress-Strain Diagrams
(3) 0° – Coupon Test, 90° – Sandwich Beam Test

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TABLE XLII
GRAPHITE POLYIMIDE LONGITUDINAL AND TRANSVERSE TENSION MECHANICAL PROPERTY DATA AT 550°F 100 HR SOAK

Specimen Number	Laminate Orientation (degrees) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
6-133-21	0°	157000	8470	20.4
-22		156000	8240	20.5
-23		153000	6890	21.2
-24		171000		
-25		159000		
6-134-26	0°	166000		
-27		170000		
-28		152000		
-29		167000		
-30		169000		
F_{tu} Mean		162000	7867	20.7
F_{tu} "B"		144700		
6-131-11	90°	4360		
-12		4380		
-13		4770		
-14		4680		
-15		4280		
6-131-16	90°	3940		
-17		4110		
-18		4270	5800	1.13
-19		4170	3825	1.27
-20		4230	4500	1.20
F_{tu} Mean		4340	4708	1.20
F_{tu} "B"		3981		

Notes: (1) Type material - MODMOR II Graphite/Skybond 703
(2) See Figures 45 and 46 Stress-Strain Diagrams
(3) 0° – Coupon Test, 90° – Sandwich Beam Test

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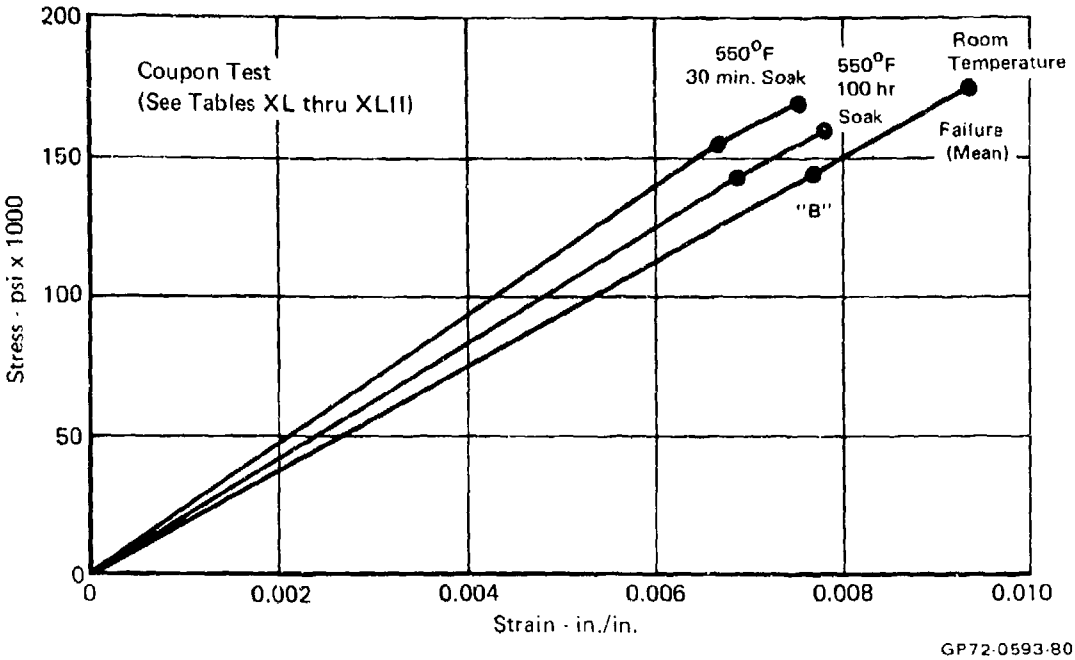

FIGURE 45
TYPICAL TENSION STRESS-STRAIN DIAGRAMS FOR GRAPHITE POLYIMIDE 0° LAMINATE
(MODMOR II/Skybond 703)

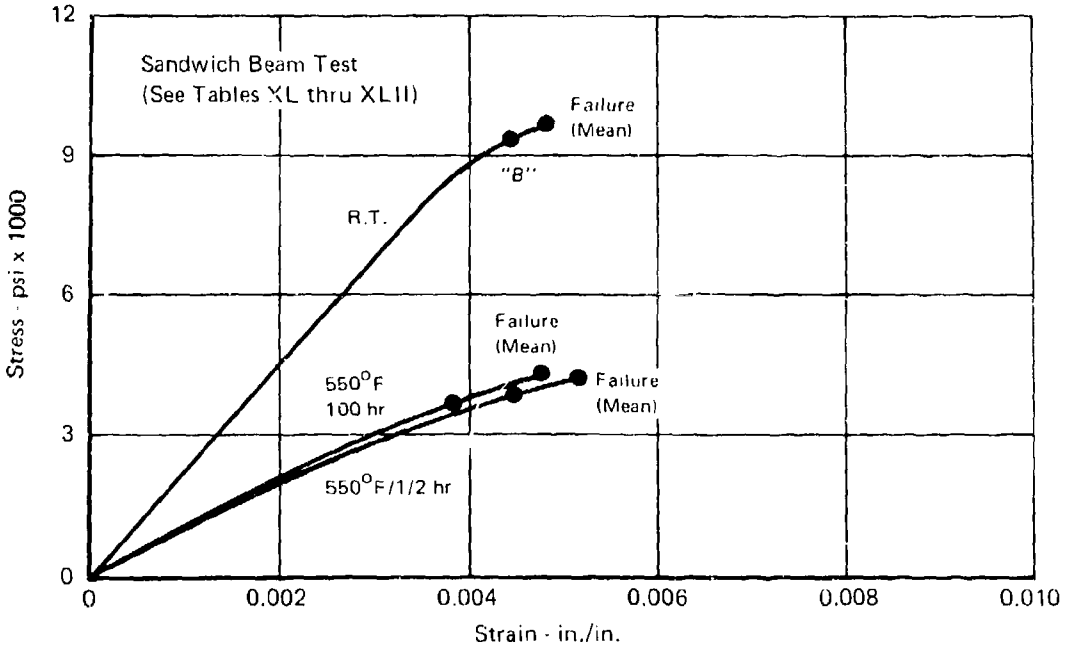

FIGURE 46
TYPICAL TENSION STRESS-STRAIN DIAGRAMS FOR GRAPHITE POLYIMIDE 90° LAMINATE
(MODMOR II/Skybond 703)

TABLE XLIII
GRAPHITE POLYIMIDE SANDWICH BEAM TENSION PROPERTIES AT ROOM TEMPERATURE

Specimen Number	Laminate Orientation (degrees) (Loading at 0^{o})	Failure Stress (psi)	Failure Strain (μ in./i	Elastic Modulus (MSI)
6-131-1	$[\pm 45^{o}, 0_2^{o}, 90^{o}]_S$	92500	8995	10.2
-2		101000		
-3		99000		
F_{tu} Mean		97500		

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TABLE XLIV
GRAPHITE POLYIMIDE SANDWICH BEAM COMPRESSION PROPERTIES AT ROOM TEMPERATURE

Specimen Number	Laminate Orientation (degrees) (Loading at 0^{o})	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
6-131-4	$[\pm 45^{o}, 0_2^{o}, 90^{o}]_S$	72500	8750	8.45
-5		68200		
-6		68400		
F_{cu} Mean		69700		

Notes: (1) Type material - MODMOR II Graphite/Skybond 703
(2) See Figures 47 and 48 for Stress-Strain Diagrams

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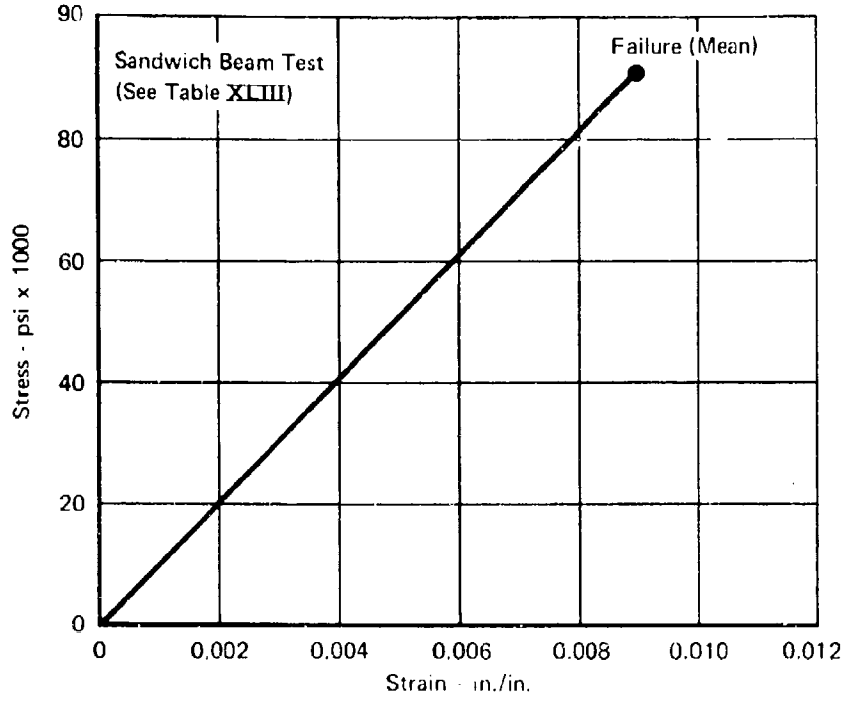

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FIGURE 47

TYPICAL TENSION STRESS-STRAIN DIAGRAM FOR GRAPHITE POLYIMIDE $[\pm 45^{o}, 0_{2}^{o}, 90^{o}]_{S}$ LAMINATE AT ROOM TEMPERATURE

(MODMOR II/Skybond 703)

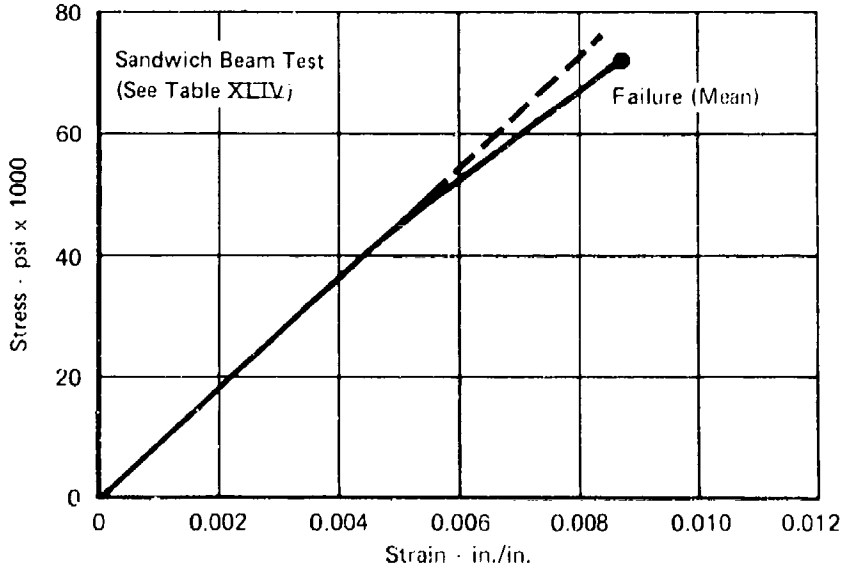

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FIGURE 48

TYPICAL COMPRESSION STRESS-STRAIN DIAGRAM FOR GRAPHITE POLYIMIDE $[\pm 45^{o}, 0_{2}^{o}, 90^{o}]_{S}$ LAMINATE AT ROOM TEMPERATURE

(MODMOR II/Skybond 703)

TABLE XLV
GRAPHITE POLIMIDE LONGITUDINAL AND TRANSVERSE SANDWICH BEAM COMPRESSION PROPERTIES AT ROOM TEMPERTURE

Specimen Number	Laminate Orientation (degrees) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
6-131-11	0°	178000	9015	21.6
-12		163000	7797	22.6
-13		166000		
-14		177000		
-15		164000		
6-132-1	0°	203000	10150	21.8
-17		199000		
-18		173000		
-19		173000		
-20		188000		
F_{cu} Mean		178300	9034	22.0
F_{cu} "B"		144900		
6-134-11	90°	19900	11608	1.13
-12		20200	11485	1.17
-13		18000		
-14		19300		
-15		18700		
6-135-16	90°	19450	11515	1.18
-17		17510		
-18		18250		
-19		19880		
-20		18750		
F_{cu} Mean		18990	11536	1.16
F_{cu} "B"		16880		

Notes: (1) Type material - MODMOR II Graphite/Skybond 703
(2) See Figures 49 and 50 for Stress-Strain Diagrams

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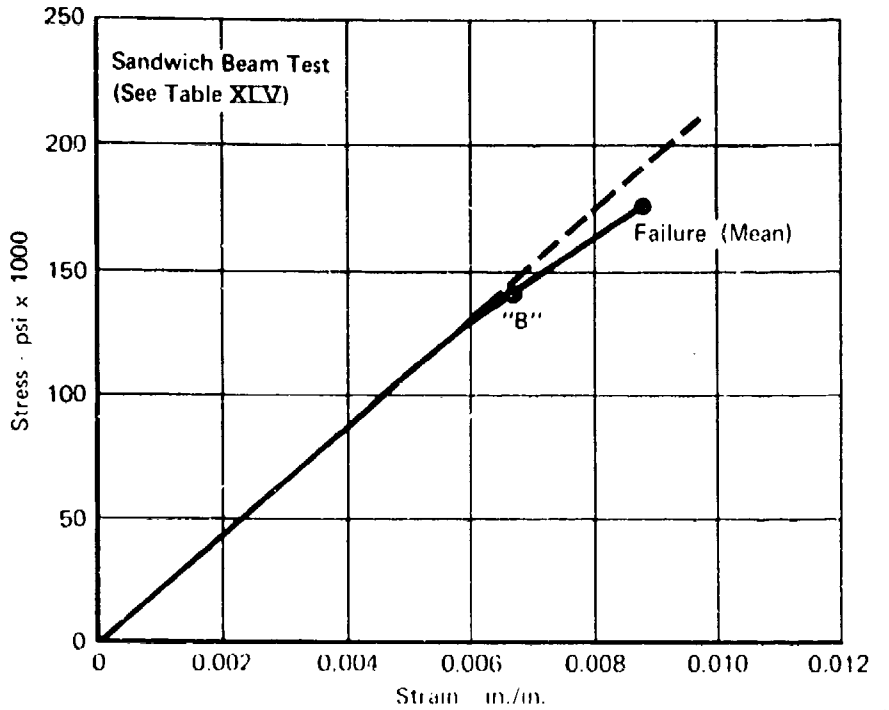

FIGURE 49

TYPICAL COMPRESSION STRESS-STRAIN DIAGRAM FOR GRAPHITE POLYIMIDE 0° LAMINATE AT ROOM TEMPERATURE

(MODMOR II/Skybond 703)

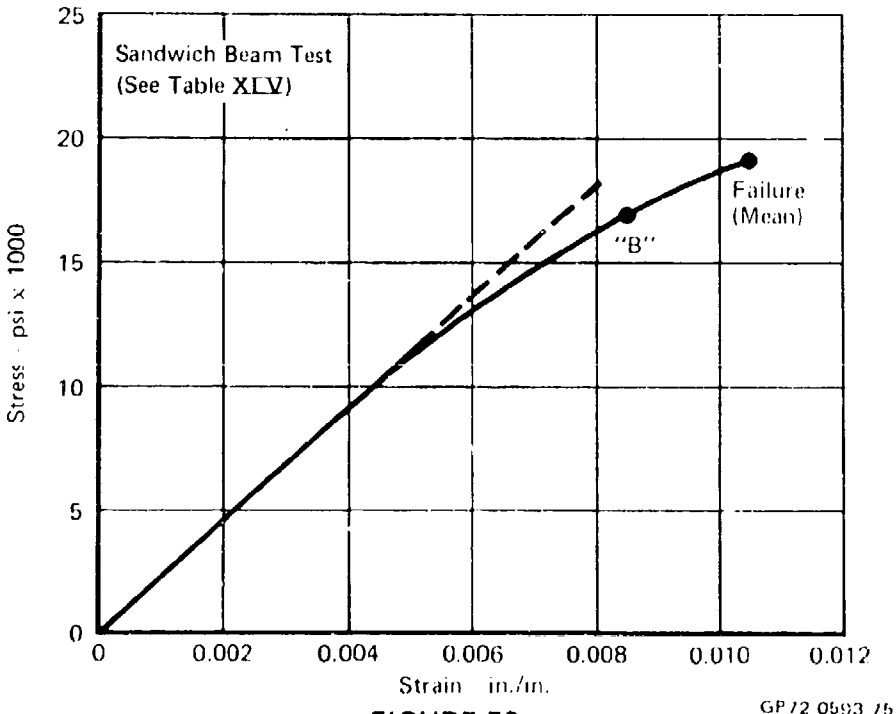

FIGURE 50

TYPICAL COMPRESSION STRESS-STRAIN DIAGRAM FOR GRAPHITE POLYIMIDE 90° LAMINATE AT ROOM TEMPERATURE

(MODMOR II/Skybond 703)

TABLE XLVI
GRAPHITE POLYIMIDE LONGITUDINAL AND TRANSVERSE EDGEWISE COMPRESSION MECHANICAL PROPERTY DATA AT ROOM TEMPERATURE

Specimen Number	Laminate Orientation (degrees) (Loading at 0^{o})	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
6-133-1	0^{o}	101500		
-2		97300		
-3		97300		
-4		114000	6030	18.7
-5		112000	7000	19.2
6-135-6	0^{o}	101100		
-7		99500	5680	19.4
-8		100000		
-9		97400		
-10		98500		
F_{cu} Mean		101800	6237	19.1
F_{cu} "B"		87500		
6-134-1	90^{o}	21000	16500	0.70
-2		21600	19040	0.650
-3		22300	21000	0.695
-4		20600		
-5		21000		
6-135-6	90^{o}	18300		
-7		24700		
-8		22800		
-9		21100		
-10		19000		
F_{cu} Mean		21240	18850	0.681
F_{cu} "B"		17000		

Notes: (1) Type material - MODMOR II Graphite/Skybond 703
(2) See Figures 51 and 52 for Stress - Strain Diagrams

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TABLE XLVII
GRAPHITE POLYIMIDE LONGITUDINAL AND TRANSVERSE EDGEWISE COMPRESSION MECHANICAL PROPERTY DATA AT 550°F 30-MINUTE SOAK

Specimen Number	Laminate Orientation (degrees) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
6-133-11	0°	61600		
-12		65300		
-13		64500		
-14		68500	2870	25.8
-15		65000	2855	21.4
6-135-16	0°	59600		
-17		61400		
-18		70600		
-19		60300		
-20		62500	2790	20.4
F_{cu} Mean		63880	2838	22.5
"B"		55500		
6-134-11	90°	11000		
-12		10600		
-13		10400		
-14		9600	11300	0.875
-15		8800	16950	0.810
6-135-16	90°	10600		
-17		10500		
-18		10500		
-19		10300		
-20		9500	15630	0.820
F_{cu} Mean		10180	14630	0.835
"B"		8610		

Notes: (1) Type material - MODMOR II Graphite/Skybond 703
(2) See Figures 51 and 52 for Stress - Strain Diagrams

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TABLE XLVIII
GRAPHITE POLIMIDE LONGITUDINAL AND TRANSVERSE EDGEWISE COMPRESSION MECHANICAL PROPERTY DATA AT 550°F 100-HOUR SOAK

Specimen Number	Laminate Orientation (degrees) (Loading at 0°)	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
6-133-21	0°	44400		
-22		48600		
-23		41700		
-24		57200	2850	21.4
25		55500	2625	19.8
6-135-26	0°	47300		
-27		50600		
-28		51500		
-29		50000		
-30		51500	2500	18.7
F_{cu} Mean		49800	2658	20.0
"B"		38900		
6-134-21	90°	10000		
-22		10250		
-23		9750		
-24		9450	16600	0.835
-25		9650	14450	0.810
6-135-26	90°	10350		
-27		9350		
-28		10800		
-29		9250	22500	
-30		9500		
F_{cu} Mean		9835	17810	0.822
"B"		8700		

Notes: (1) Type material - MODMOR II Graphite/Skybond 703
(2) See Figures 51 and 52 for Stress - Strain Diagrams

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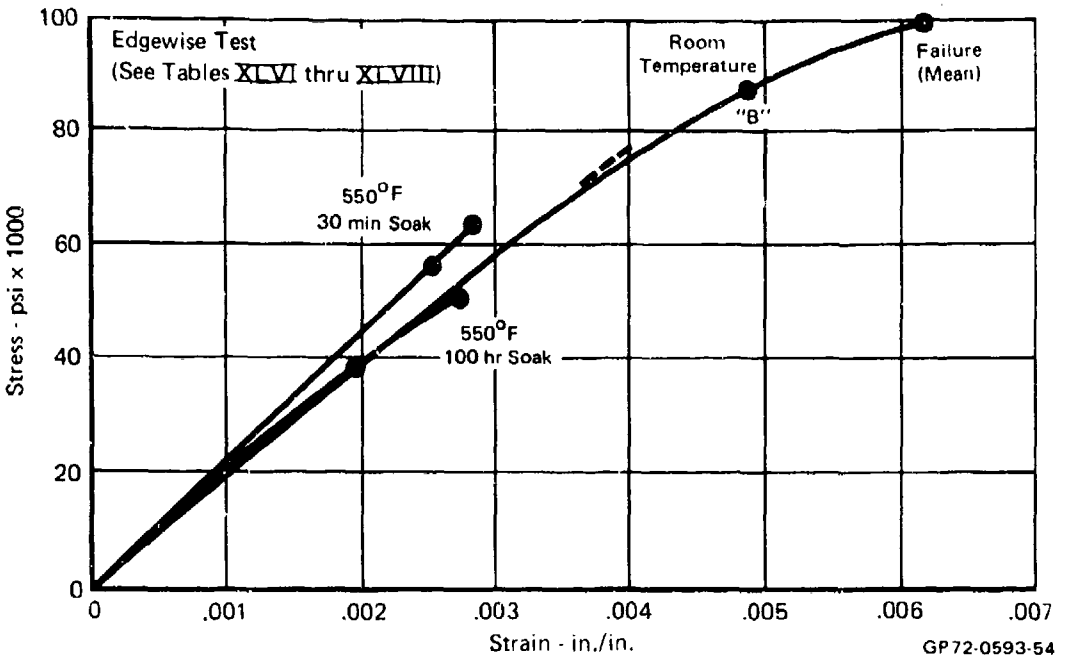

FIGURE 51
TYPICAL COMPRESSION STRESS-STRAIN DIAGRAMS FOR GRAPHITE POLYIMIDE 0° LAMINATE
(MODMOR II/Skybond 703)

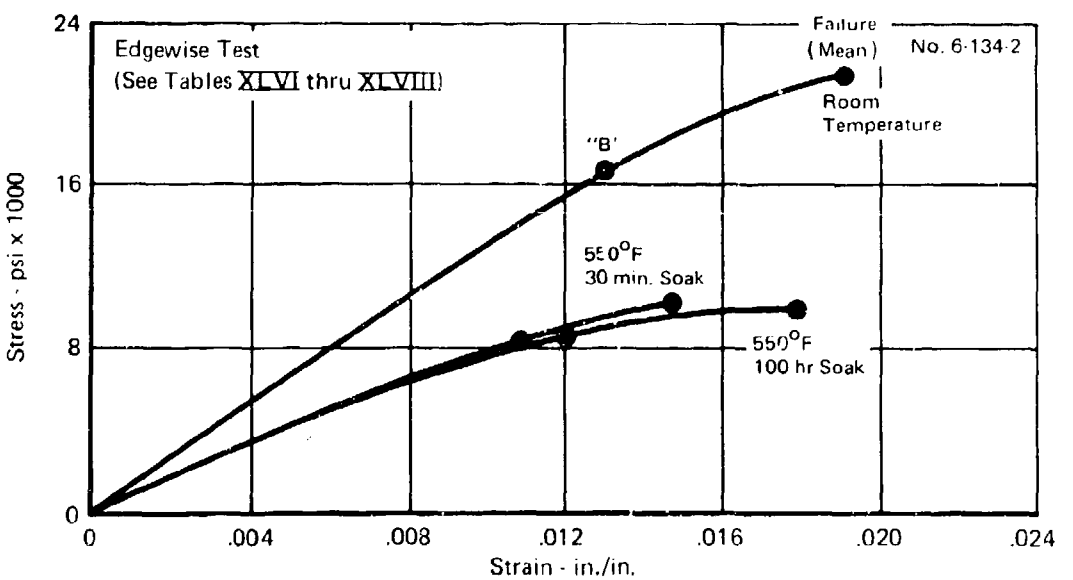

FIGURE 52
TYPICAL COMPRESSION STRESS-STRAIN DIAGRAMS FOR GRAPHITE POLYIMIDE 90° LAMINATE
(MODMOR II/Skybord 703)

TABLE XLIX
GRAPHITE POLYIMIDE RAIL SHEAR MECHANICAL PROPERTY DATA FOR $0^{o} - 90^{o}$ LAMINATE

Specimen Number	Test Temperature	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
6-131-A1	Room Temperature	10780	43000	0.629
-A2		10780	46500	0.629
-A3		8430	20250	0.700
-A4		11020	45600	0.700
-A5		11130	32000	0.723
6-134-A6		10060	40800	0.795
-A7		10850	31000	0.795
-A8		10790	36400	0.795
-A9		8300	22600	0.733
-A10		10320	35800	0.636
F_{su} Mean		10250	35400	0.713
F_{su} "B"		7800		
6-131-1	$550^{o}F$	6283	29570	0.354
-2		7094	34800	0.418
-3		5717	35600	0.357
-4		5680	27600	0.284
-5		6433	43200	0.284
6-134-6	$550^{o}F$	5570	35400*	0.568
-7		7320	44000*	0.568
-8		6510	46000*	0.460
-9		6320	40000*	0.350
-10		5960	39000*	0.720
F_{su} Mean		6290	37500	0.436
F_{su} "B"		4910		

Notes: (1) Type material - MODMOR II Graphite/Skybond 703
(2) See Figures 53 and 54 for Stress - Strain Diagrams
*Values are extrapolated due to instrumentation failure.

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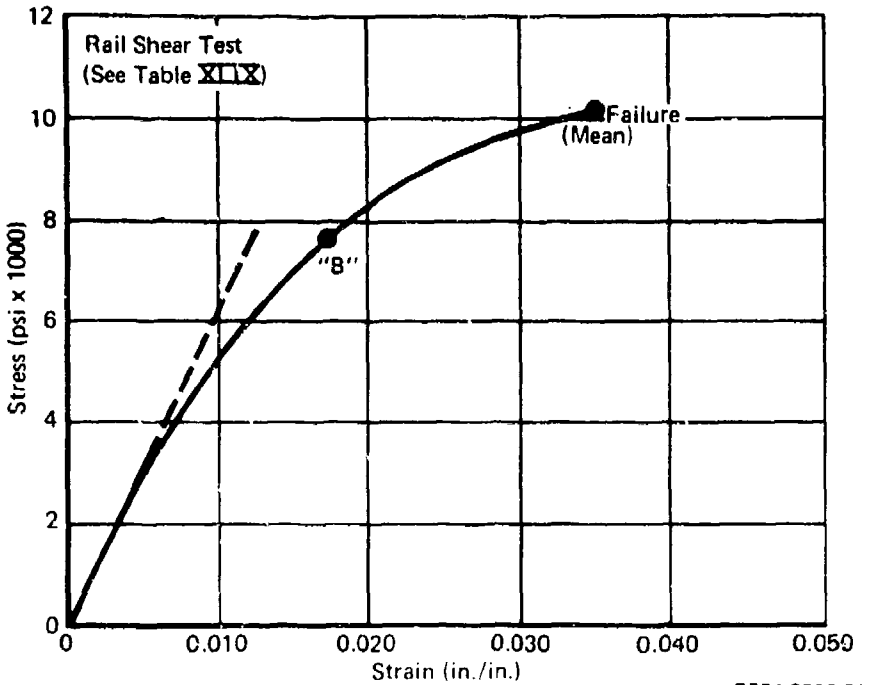

FIGURE 53

TYPICAL SHEAR STRESS - STRAIN DIAGRAM FOR GRAPHITE POLYIMIDE 0^{o} - 90^{o} LAMINATE AT ROOM TEMPERATURE
(MODMOR II/SKYBOND 703)

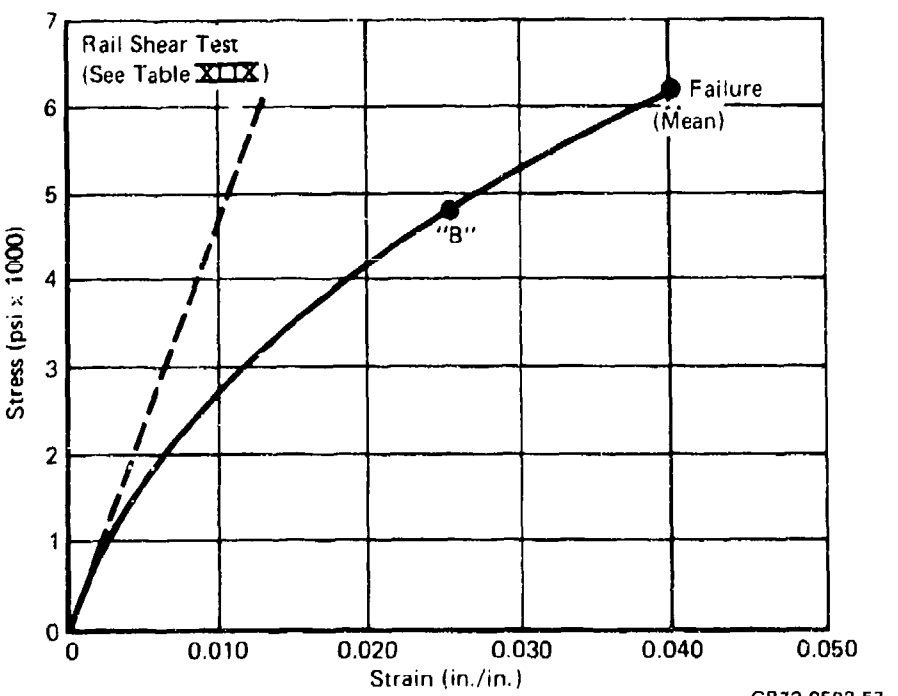

FIGURE 54

TYPICAL SHEAR STRESS - STRAIN DIAGRAM FOR GRAPHITE POLYIMIDE 0^{o} - 90^{o} LAMINATE AT 550^{o}F
(MODMOR II/SKYBOND 703)

TABLE L
GRAPHITE POLYIMIDE RAIL SHEAR MECHANICAL PROPERTY DATA FOR $\pm 45^{o}$ LAMINATE

Specimen Number	Test Temperature	Failure Stress (psi)	Failure Strain (μ in./in.)	Elastic Modulus (MSI)
6-131-1 -2 -3	Room Temperature	40,200 41,800 33,600	10,300 12,900	7.80 5.00
F_{su} Mean		38,500	11,600	6.40
6.131-4 -5 -6	$550^{o}F$	21,500 (3) (3)		
F_{su} Mean		21,500	(3)	(3)

Notes: (1) Type material - MODMOR II Graphite/Skybond 703
(2) See Figure 55 for Stress - Strain Diagram
(3) Rails Detached During Test Invalidating Instrumented Specimens

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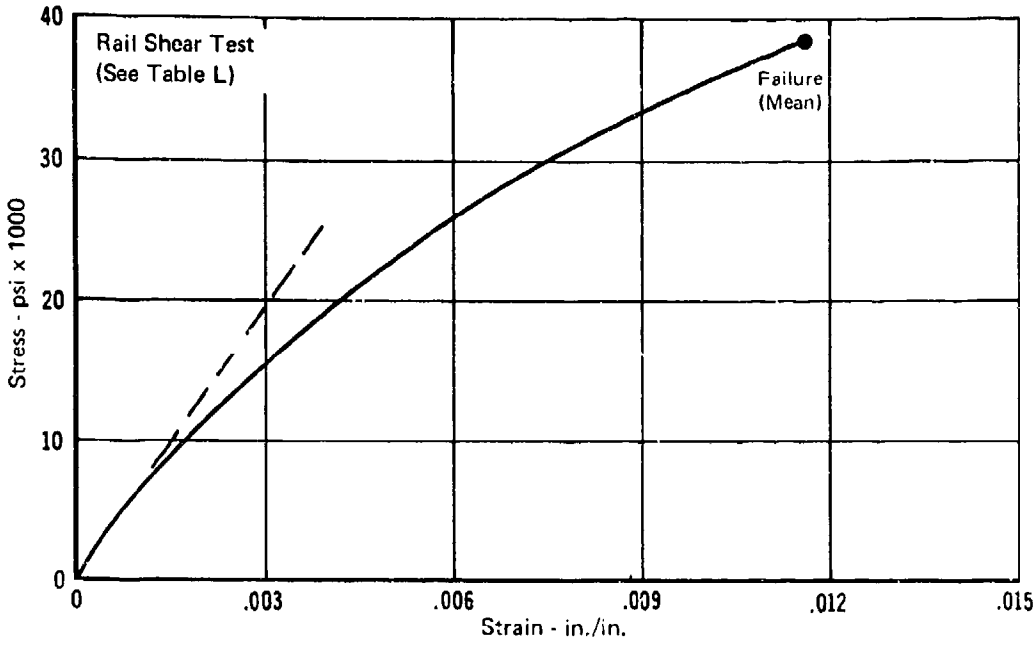

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FIGURE 55
TYPICAL SHEAR STRESS-STRAIN DIAGRAM FOR GRAPHITE POLYIMIDE ±45° LAMINATE AT ROOM TEMPERATURE
(MODMOR II/Skybond 703)

TABLE LI
GRAPHITE POLYIMIDE MODMOR II/SKYBOND 703
LONGITUDINAL "B" BASIS DESIGN ALLOWABLES

Property	Room Temperature	550°F 30 Minutes SOAK	550°F 100 Hour SOAK
F_{tu}	193,800 psi	156,000 psi	144,700 psi
F_{cu}	145,000 psi	55,500 psi	38,900 psi
E	23.2 msi	23.6 msi	20.7 msi
ν	0.33	0.21	0.20

Notes: (1) Elastic Constants are Mean Values

GP72-0593-49

TABLE LII
GRAPHITE POLYIMIDE MODMOR II/SKYBOND 703
TRANSVERSE "B" BASIS DESIGN ALLOWABLES

Property	Room Temperature	550°F 30 minutes SOAK	550°F 100 hour SOAK
F_{tu}	8,600 psi	4000 psi	4000 psi
F_{cu}	17,000 psi	8600 psi	8700 psi
E	2.02 msi	1.15 msi	1.20 msi
E_c	0.681 msi	0.835 msi	0.822 msi

Notes: (1) Elastic Constants are Mean Values

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TABLE LIII
GRAPHITE POLYIMIDE MODMOR II/SKYBOND 703 0°-90°
IN PLANE SHEAR "B" BASIS DESIGN ALLOWABLES

Property	Room Temperature	550°F
F_{su}	7800 psi	4900 psi
G	0.713 msi	0.436 msi

Notes: (1) Elastic Constant is Mean Value

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TABLE LIV
GRAPHITE POLYIMIDE MODMOR II/SKYBOND 703 ±45°
IN PLANE SHEAR MEAN TEST RESULTS

Property	Room Temperature	550°F
F_{su}	38,500 psi	21,500 psi
G	6.4 msi	* msi

*Rails Detached During Rail Shear Test Invalidating Instrumented Specimens

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6. COMPONENT FABRICATION/TEST

6.1 Introduction

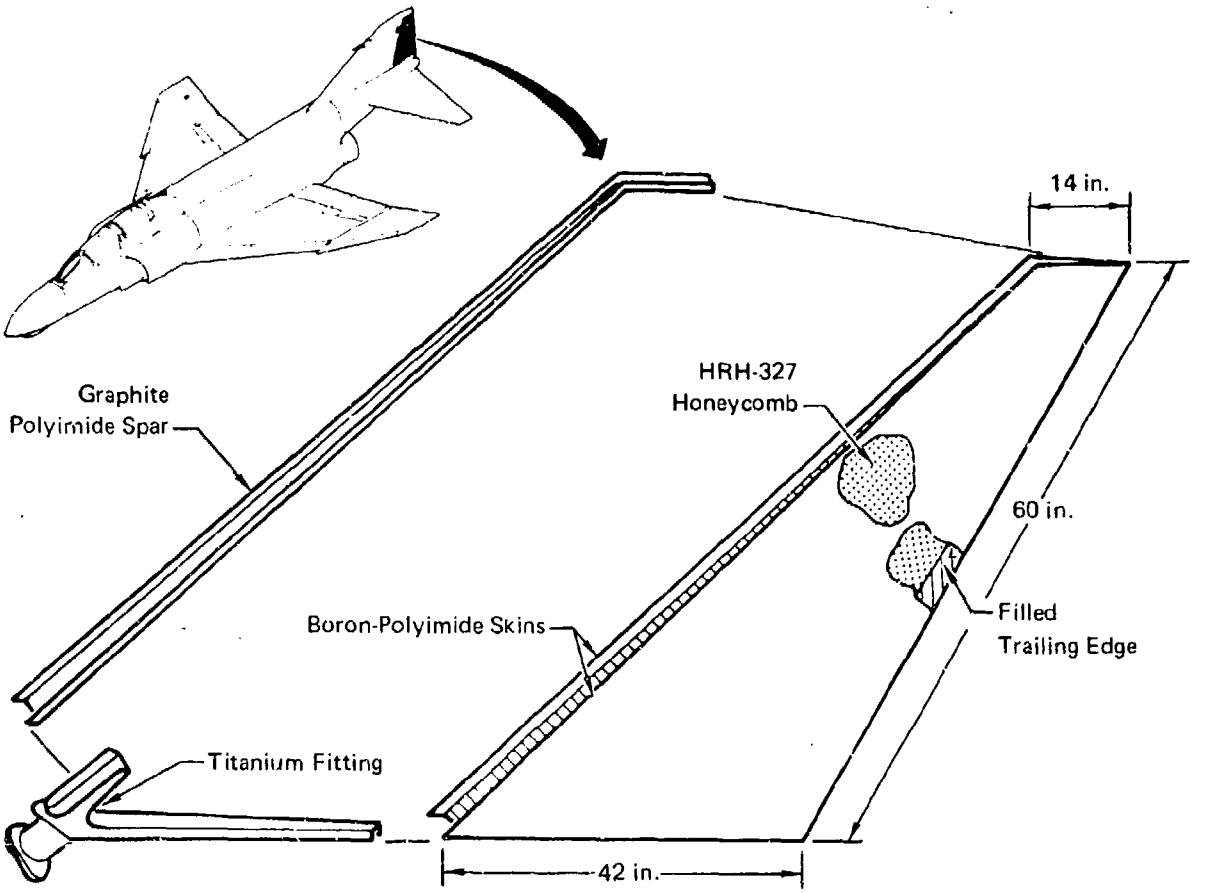
The purpose of the component fabrication/test task was to demonstrate the adequacy of polyimide materials for use in aircraft structures. An F-4 polyimide rudder was successfully fabricated and tested to failure. The fabrication of the F-15 horizontal stabilator torque box had to be discontinued because of difficulties encountered in fabricating thick boron/polyimide laminates. In its place, an F-15 composite wing compression panel was fabricated and static tested to failure.

The F-4 polyimide rudder is shown in Figure 56 indicating dimensions, and material utilization. NDT techniques (ultrasonic "C" scan and radiography) were very helpful in defining acceptable rudder component/assemblies. The rationale for selecting the F-4 rudder was based on potential for scale-up to primary assembly structures, and the attractive opportunity for comparison between the polyimide rudder and the earlier F-4 metal and boron/epoxy rudders.

6.2 F-4 Rudder Fabrication/Test

The F-4 polyimide composite rudder was fabricated with boron/SB703 skins, graphite/SB703 spar/rib, and HRH-327 fiberglass polyimide honeycomb core. Polyimide adhesives were used for 550°F capability. Variability of boron/SB703 prepreg required several skin fabrication cycles to obtain two acceptable skins. NDT (ultrasonic "C" scan and radiography) was very helpful in defining acceptable components/assemblies. The graphite/SB703 spar fabrication and rudder bonding operations were performed without difficulties.

6.2.1 Graphite/SB703 Spar Assembly - The graphite/SB703 spar (see Figure 52) was successfully cured and post cured in the first attempt using the processing cycle as outlined in Section 3. The prepreg lay-up used the low boiling point pyrolidone solvent to improve handleability and tack for the "C" channel spar shape. Figures 57 and 58 show the spar/rib ready for cure and after cure respectively. The spar passed all quality control tests, NDT and destructive testing of control specimens. Destructive test results of control specimens are given in Table LV.



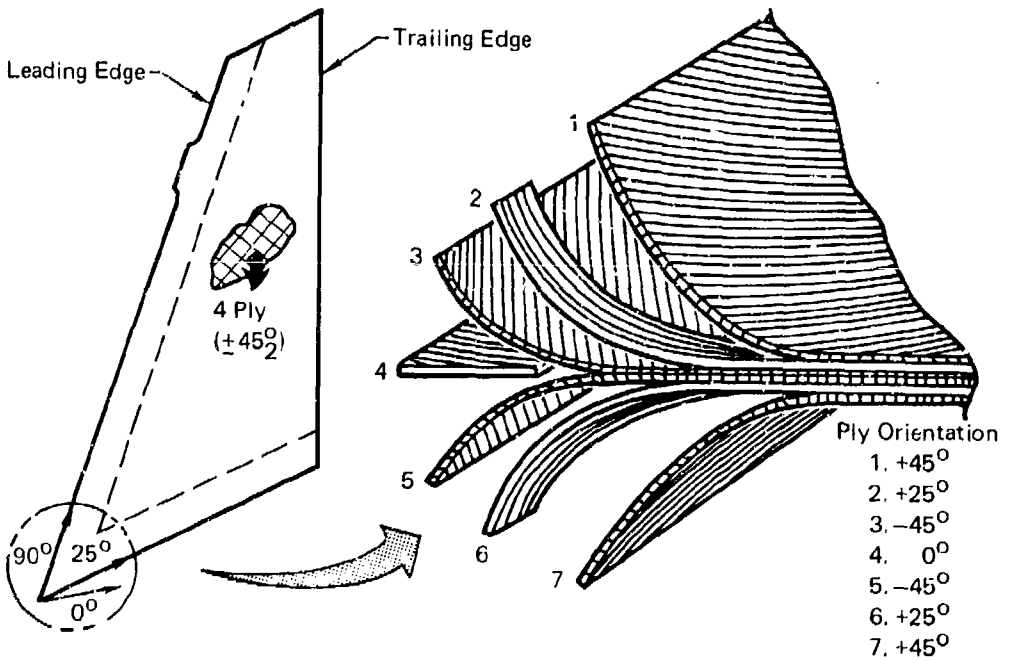


FIGURE 56
F-4 POLYIMIDE RUDDER SCALE-UP COMPONENT

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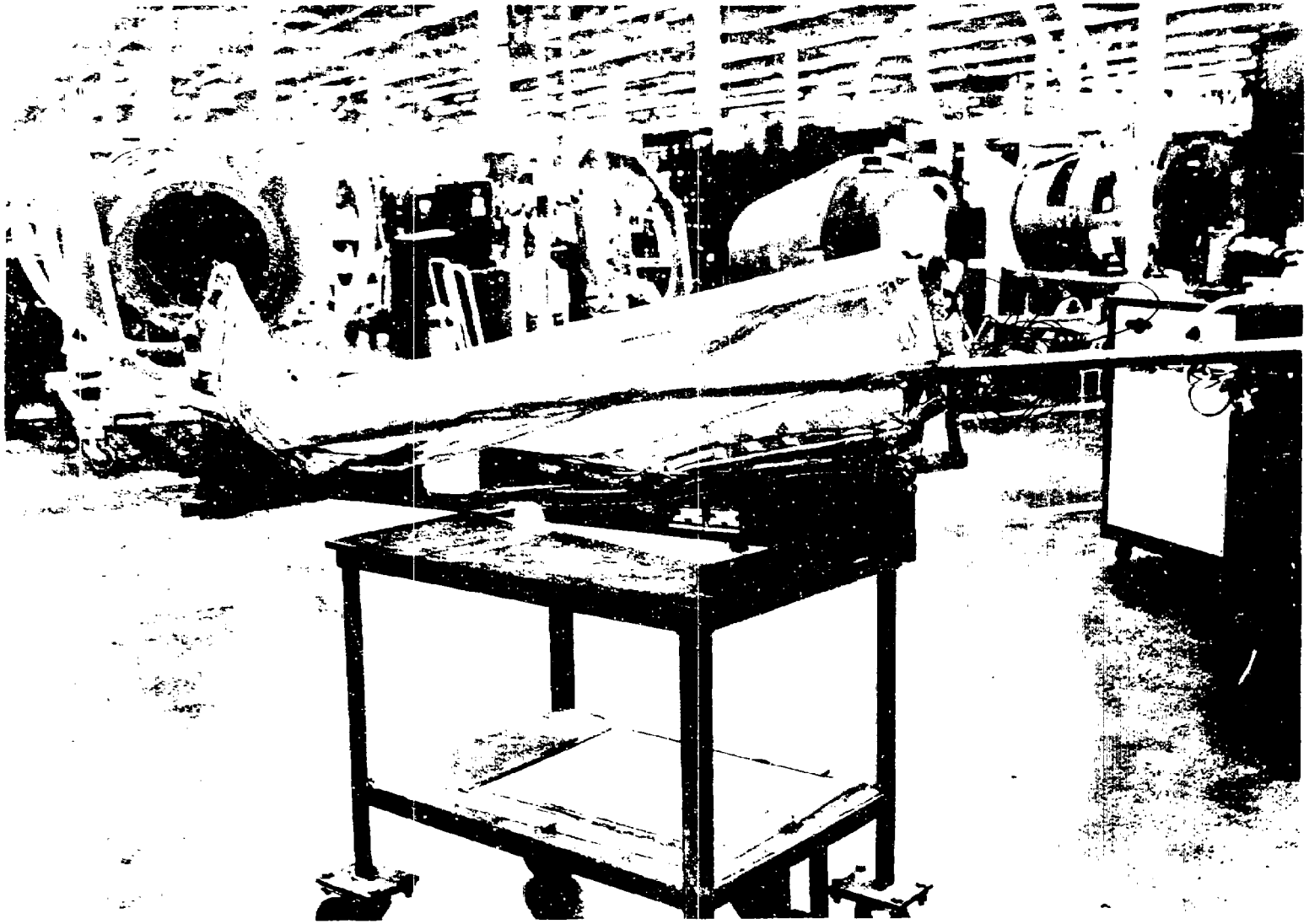

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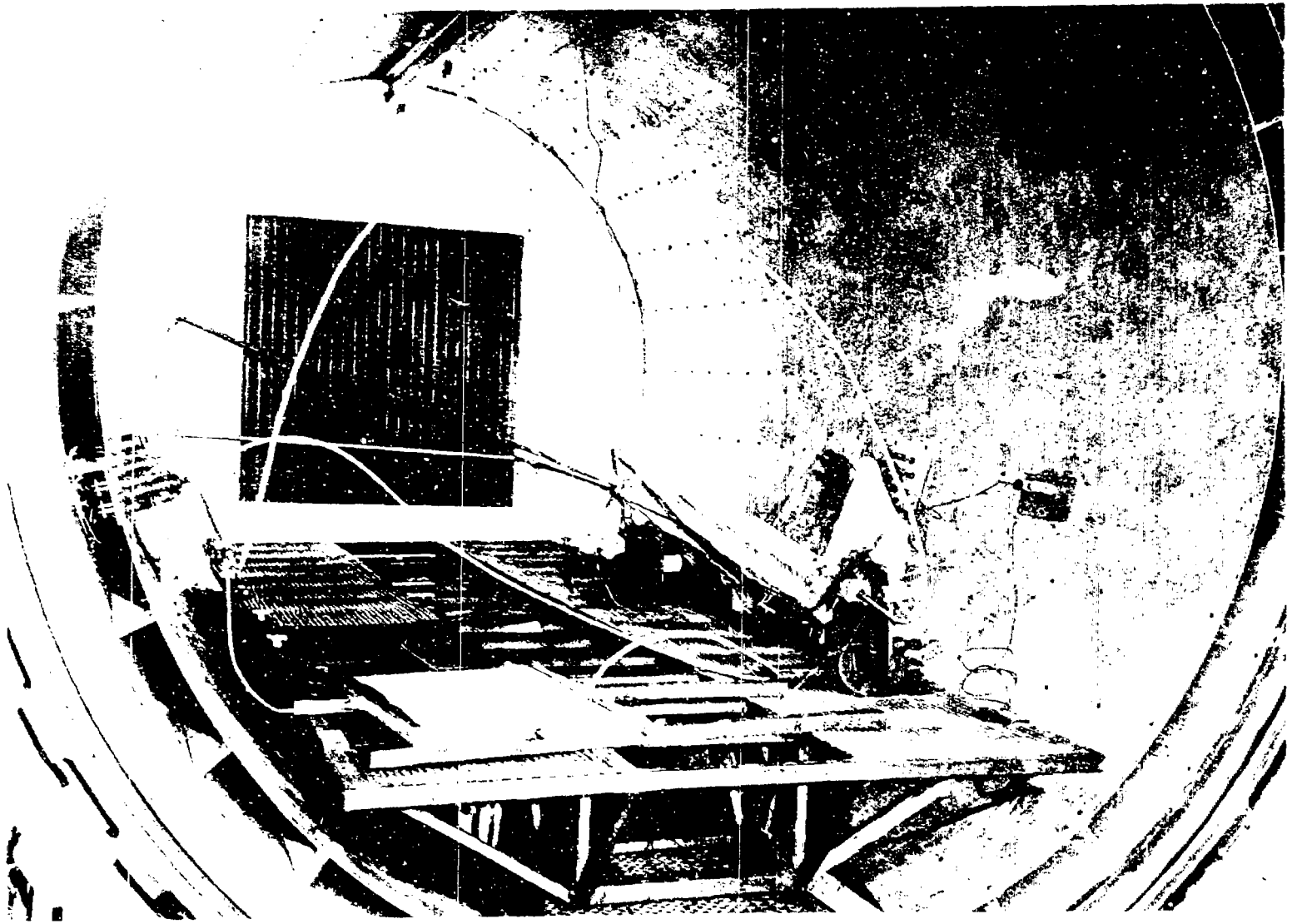
FIGURE 57
GRAPHITE/SB703 SPAR IN BAG



GP72 0593-103

FIGURE 58
BAGGED SPAR IN AUTOCLAVE

TABLE LV
GRAPHITE POLYIMIDE SPAR QUALITY ASSURANCE MECHANICAL PROPERTIES

Process Control Test	Temperature	Required Minimum	Test Result
Interlaminar Shear	R.T.	8,000 psi	12,950 psi
Interlaminar Shear	550°F	5,000 psi	7,670 psi
0° Flexure	R.T.	170,000 psi	199,000 psi
0° Flexure	550°F	110,000 psi	130,000 psi

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Following spar cure and post cure as established in Section 3, secondary bonds of the titanium fittings and titanium hinge fittings to the spar were performed, followed by the bond of the titanium fairing to the spar (see Figure 56). Metlbond 840 adhesive was used in all bonds. Quality assurance control specimen bonds, performed with the spar bond, were acceptable per Table LVI. A photograph of the completed spar assembly is shown in Figure 59. The completed spar assembly was ultrasonically C-scan inspected and x-ray inspected and no defects were found.

TABLE LVI
QUALITY ASSURANCE RESULTS OF GRAPHITE POLYIMIDE SPAR ASSEMBLY BONDS

Condition	Test	Temperature	Specification Minimum	Test Result
Ti Cleaning Coupon	Single Lap Shear	R.T.	2250 psi	2640 psi
		550°F	1000 psi	1840 psi
Ti Hinge & Rib Bond	Single Lap Shear	R.T.	2250 psi	3150 psi
		550°F	1000 psi	2173 psi
Ti Fairing Bond	Single Lap Shear	R.T.	2250 psi	3005 psi
		550°F	1000 psi	2025 psi

Note: Metlbond 840 adhesive used in all bonds.

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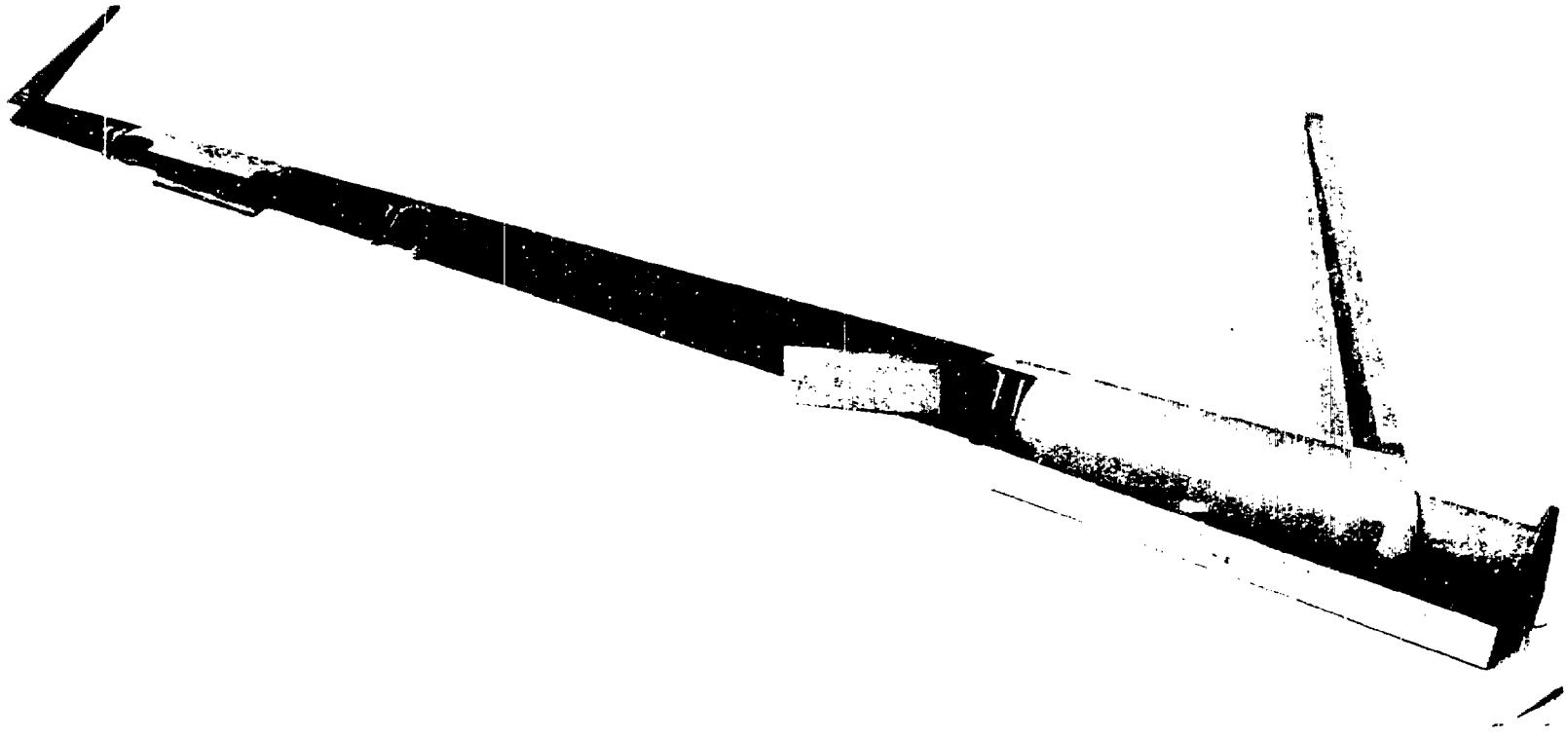

FIGURE 59
GRAPHITE POLYIMIDE SPAR ASSEMBLY

GP72-0593-104

6.2.2 Boron Polyimide Skins - The first set of boron polyimide skins was cured and found to be excessively porous. Following tests of the process control panel (see Table LVII) fabricated with the skins, the skins were rejected because of failure to meet minimum specifications. The principal cause was excessive "A" stage advancement of the prepreg used to fabricate these skins. As a result of this experience, further controls were established for the "A" stage operation such as time, temperature, vacuum and agitation speed.

The fabrication of a second set of rudder skins appeared at first to be successful. The completed skins (Figure 60) had acceptable resin distribution and no porosity was visible. Fifteen ply quality assurance specimens, fabricated with the 4-to-7-ply skins, yielded acceptable test results per Table LVIII. Both sets of skins were fabricated per P.S. 14224 (Appendix).

TABLE LVII

BORON POLYIMIDE SKIN (FIRST SET)

QUALITY ASSURANCE PANEL TEST RESULTS

Process Control Test	Temperature*	Specificiation Minimum	Individual Test Results
0° Flexure	All at R.T.	190,000 psi	68,000 psi
			97,600 psi
			202,000 psi
Interlaminar Shear		11,000 psi	7,800 psi
			6,400 psi
			7,100 psi

*550°F tests not performed because of poor R.T. results.

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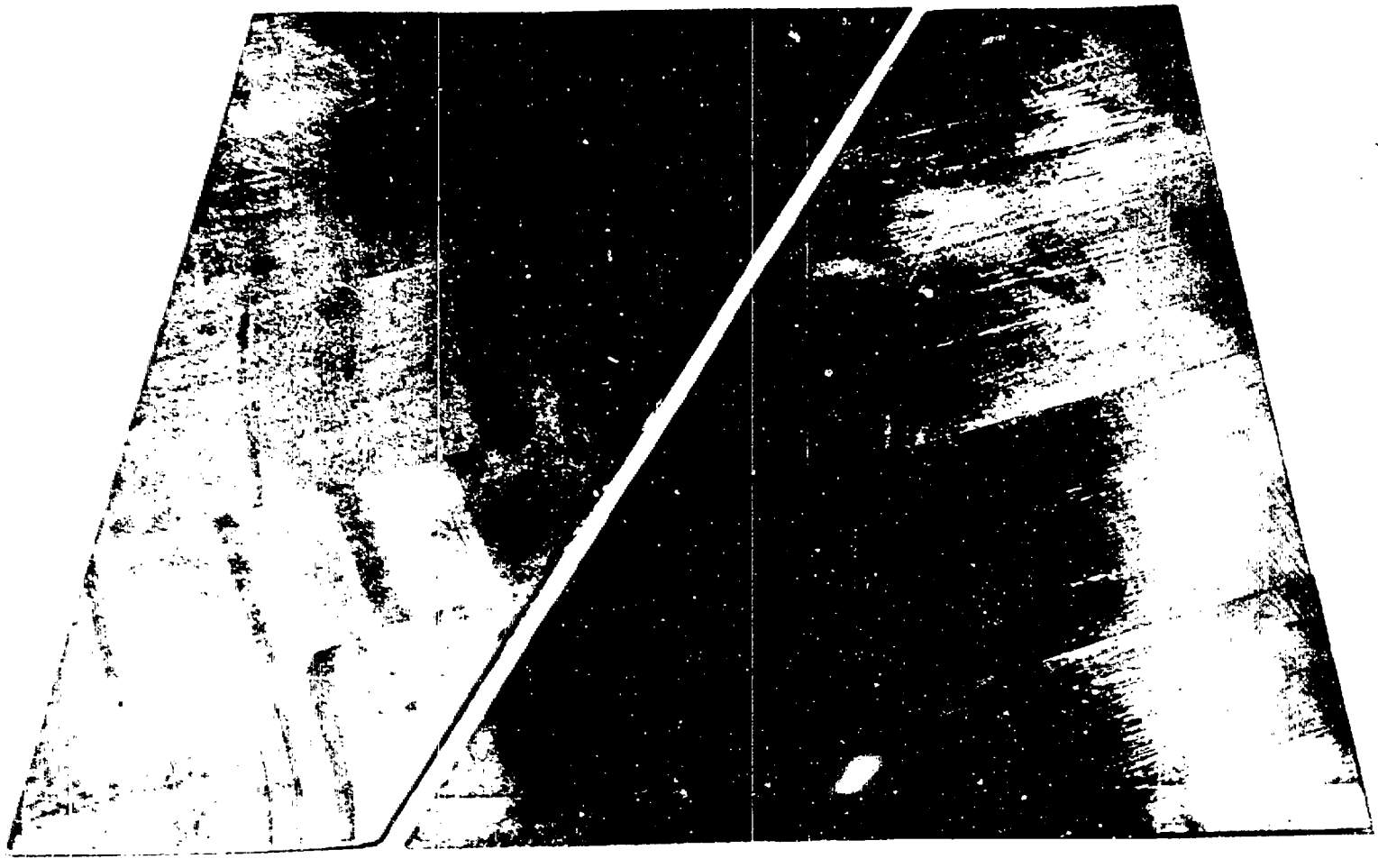

FIGURE 60
BORON POLYIMIDE RUDDER SKINS (SET NO. 2)

GP72-0593-105

TABLE LVIII
BORON POLYIMIDE SKIN (SECOND SET)
QUALITY ASSURANCE PANEL TEST RESULTS

Property	Test Temperature °F	Spec Minimum psi	Average Test Results psi
0° Flexural Strength	R.T.	190,000	226,000
	550°F	155,000	200,000
Interlaminar Shear Strength	R.T.	11,000	11,800
	550°F	6,000	8,080

GP72-0328-108

The skins were machined, Figure 61, and then ultrasonically C-scan inspected for delaminations. C-scan of one of the skins, Figure 62, showed it to be acceptable, while C-scan of the second skin, Figure 63 showed that the skin was delaminated. Plugs were machined out of the skin to confirm the delaminations. Visual examination of the plugs showed that plugs taken from areas which the C-scan showed delaminations were indeed delaminated, and the plug taken from an area which C-scan showed no delaminations was not delaminated. Figure 64 shows photomicrographs of two of the delaminated plugs and of the undelaminated plug. Additional plugs were machined from delaminated and undelaminated areas of the skins according to the C-scan. Correlation between the flatwise tension results with the plugs and C-scan results was excellent, as shown in Table LIX.

TABLE LIX
FLATWISE TENSION RESULTS OF PLUGGED
BORON POLYIMIDE RUDDER SKIN

Percent Delaminated	Flatwise Tension Strength, psi
66	395
33	685
0	1385
0	1420
0	1040
0	1205
0	1460
0	1470

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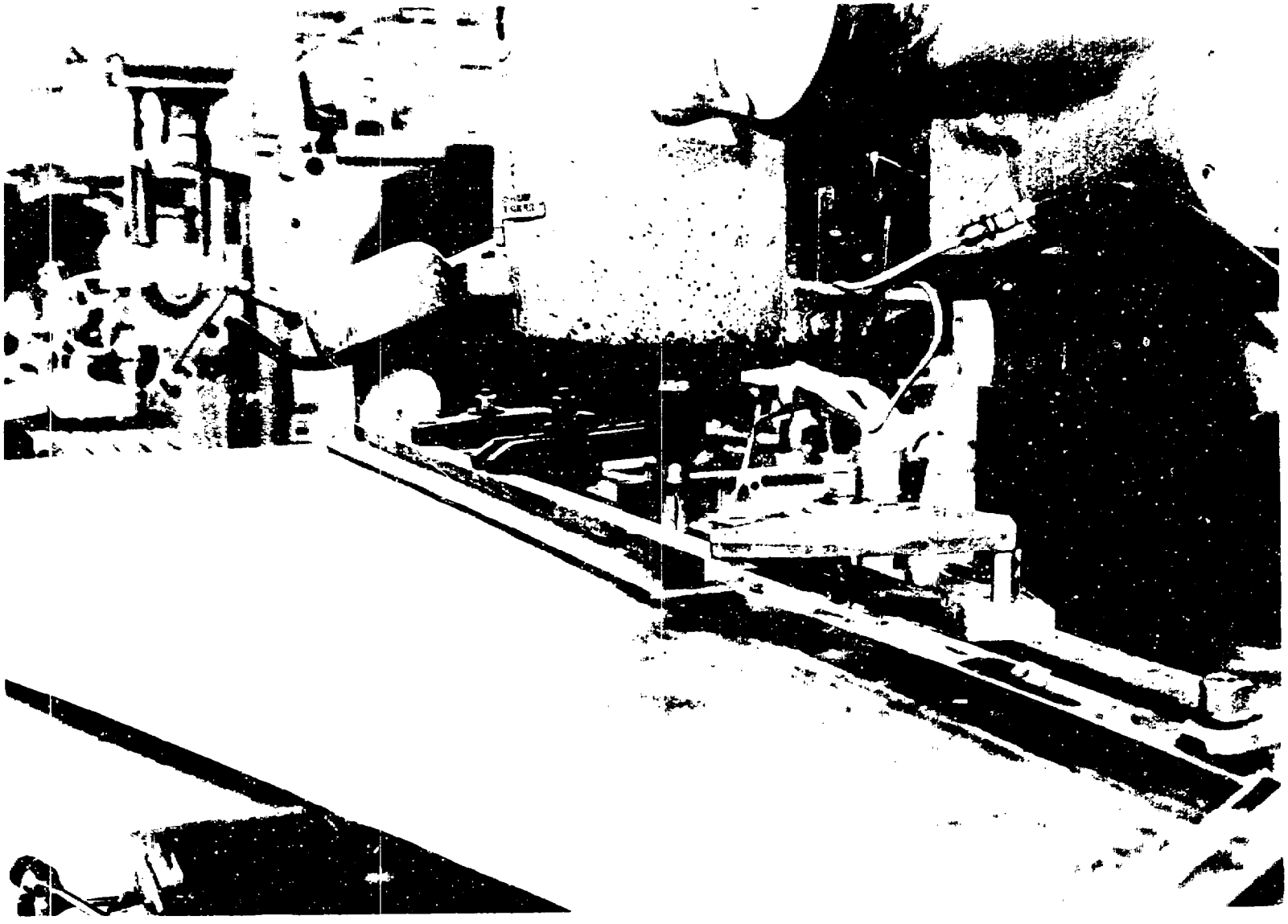

FIGURE 61
MACHINING OF BORON POLYIMIDE RUDDER SKIN

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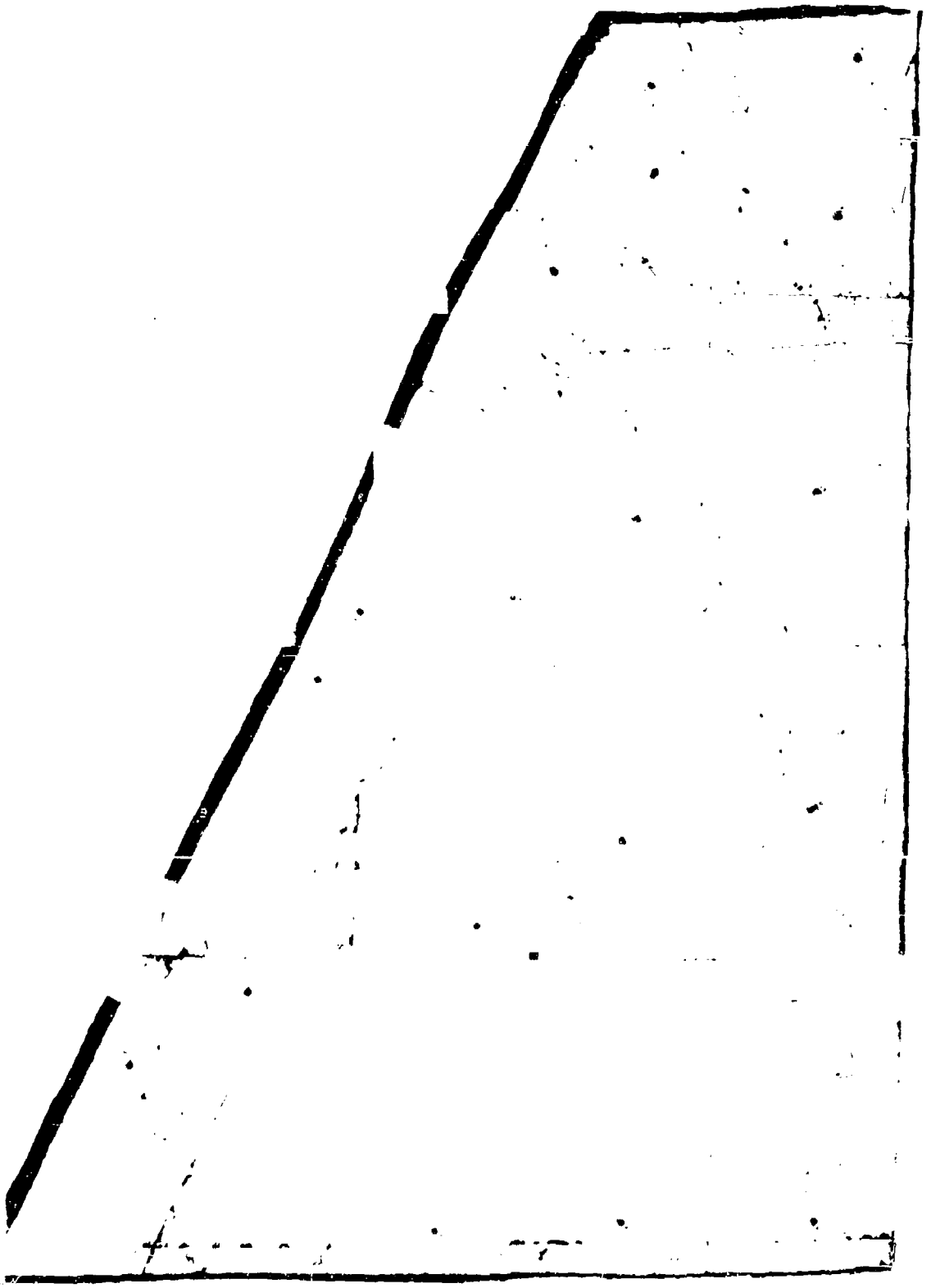


FIGURE 62 GP72-0593-116
C-SCAN OF UNDELAMINATED BORON POLYIMIDE RUDDER SKIN

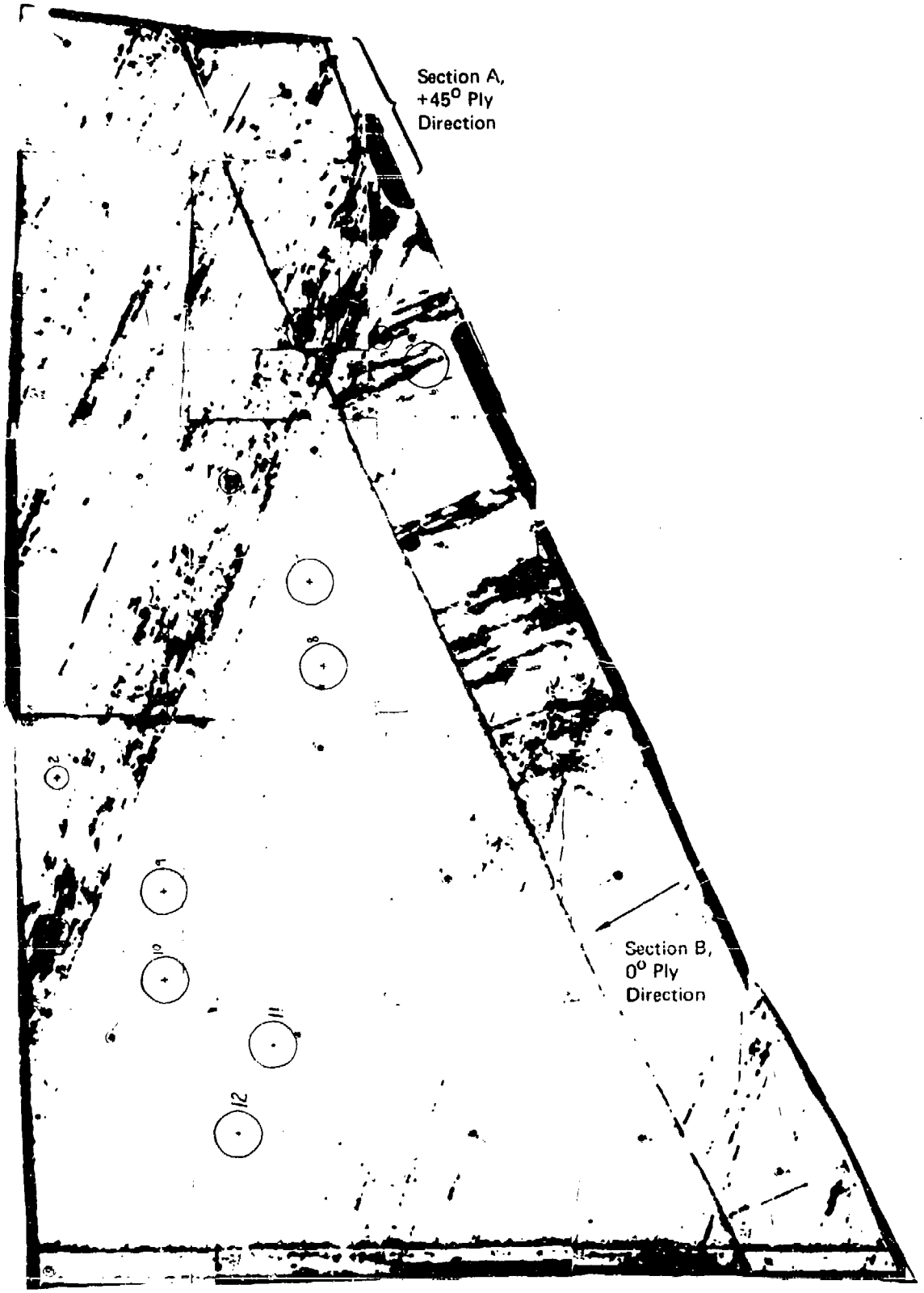
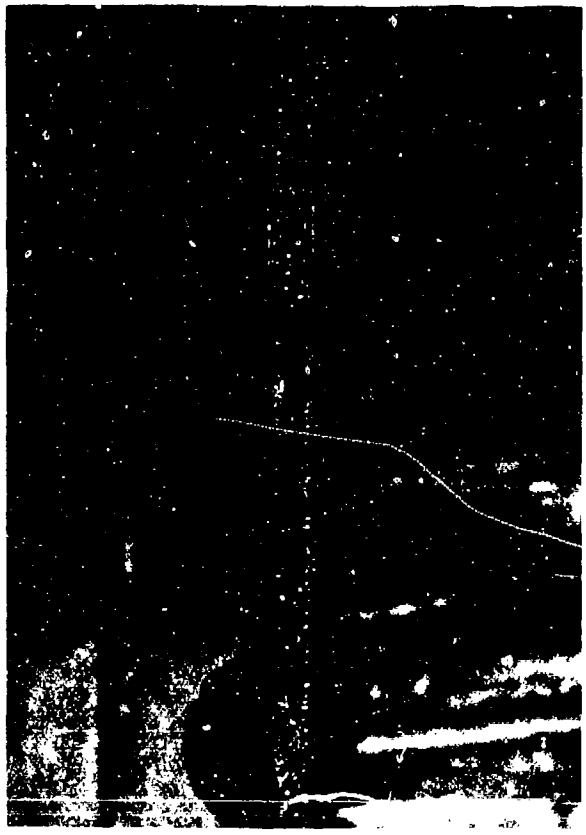
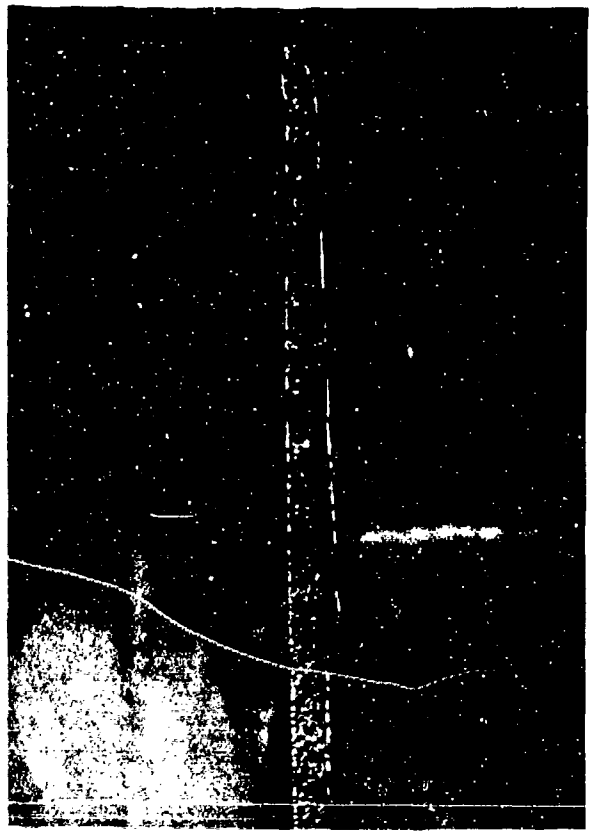


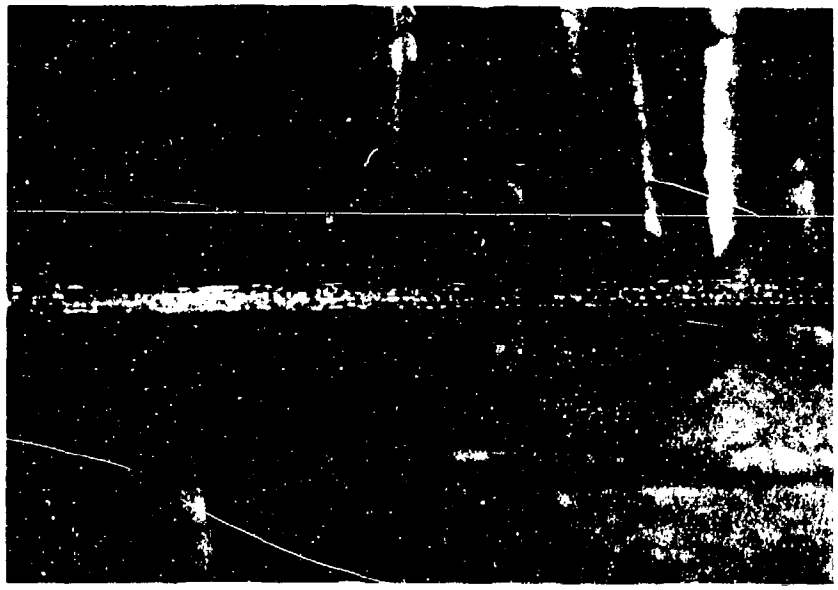
FIGURE 63
C-SCAN OF DELAMINATED BORON POLYIMIDE RUDDER SKIN



Plug No. 3 Delamination is Between -45^{o} Ply and 0^{o} Ply.



Plug No. 1 Delamination is Between $+45^{o}$ Ply and -45^{o} Ply.



Plug No. 2 No Delamination

FIGURE 64
PHOTOMICROGRAPHS OF BORON POLYIMIDE PLUGS MACHINED FROM DELAMINATED RUDDER SKIN

GP72-0593-107

A third skin (needed to complete the set of two required for a rudder) was fabricated and it successfully passed NDT. Fifteen ply quality assurance specimens fabricated with the skin yielded acceptable test results, Table LX.

TABLE LX
BORON POLYIMIDE SKIN (THIRD SET)
QUALITY ASSURANCE PANEL TEST RESULTS

Property	Test Temperature °F	Specification Minimum psi	Average Test Results psi
0° Flexural Strength	R.T.	190,000	270,000
	550°F	155,000	224,000
Interlaminar Shear Strength	R.T.	11,000	11,800
	550°F	6,000	7,380

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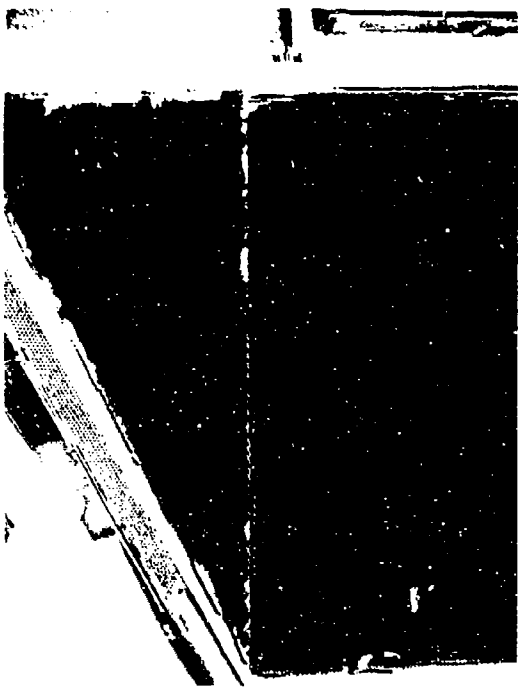
6.2.3 Bonding of Rudder Assembly - The HRH-327 fiberglass polyimide core was cut into five pieces for bonding into the rudder assembly, and Verifilm check was performed to determine uniform bonding pressure between spar assembly, core and bottom skin bonding surfaces. The same processing was utilized as explained in Section 3 of this report. The fiberglass polyimide core was then primed with BR-34 primer and dried at 400°F. Placement of this core into the rudder assembly showed that the core had shrunk 2 inches in the spanwise direction, during the 400°F drying operation. It was suspected that this honeycomb core had not been post cured to 600°F by the supplier. Another possible explanation for the core shrinkage was the low density and relaxation characteristics of the core material. The shrunken fiberglass/polyimide core, Figure 65, was replaced with a larger piece of core which was post cured, primed, dried, and cut to size.



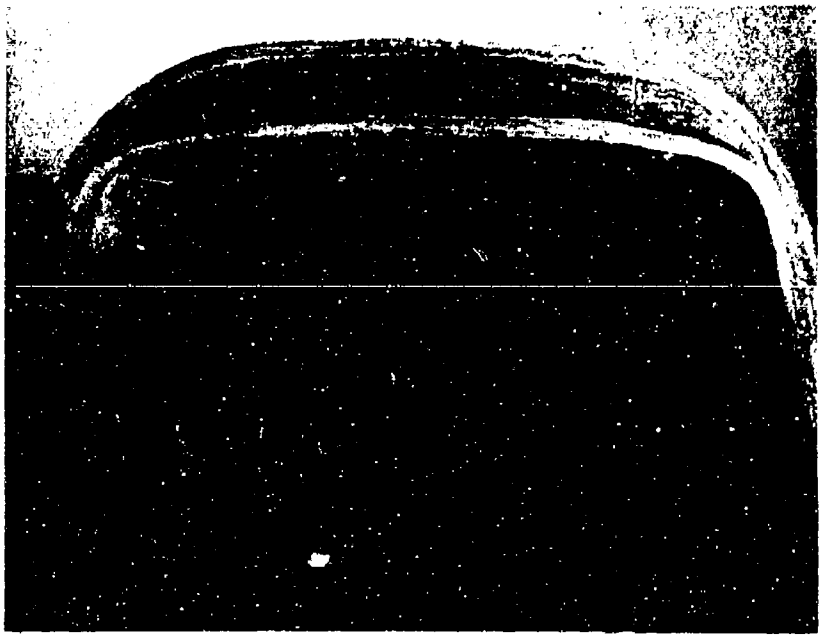


FIGURE 65
FIBER GLASS POLYIMIDE CORE ON RUDDER SUBASSEMBLY SHOWING AMOUNT OF SHRINKAGE OF CORE DURING 400°F CORE PRIMER DRY OPERATION

GP72-0593-108

The bottom boron polyimide skin bonding surface was lightly grit blasted, and then the bottom skin, core and spare assembly were bonded in an autoclave. Quality assurance bonds, performed with the rudder assembly bond, yielded results shown in Table LXI.

TABLE LXI
QUALITY ASSURANCE RESULTS OF BOTTOM SKIN-CORE-SPAR ASSEMBLY BOND

Process Control Test	Temperature	Specification Minimum (psi)	Test Result
Lap Shear, Ti-Ti	R.T.	2250	2090
	550°F	1000	2145
Flatwise Tension, Skin-Core	R.T.	400	345
	550°F	75	165

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Metlbond 840 and FM-34 adhesives were used for skin to spar (lap shear) and skin to core (flatwise tension), respectively. All three of the room temperature flatwise tension specimens failed in the skin to flatwise tension block bond line, and not in core to skin bond line. Of the three 550°F flatwise tension specimens, two failed in the skin to flatwise tension block bond line and one specimen failed in core to skin bond line. The lap shear specimens, as shown in Table LXI, were machined into dog bones inadvertently and failures occurred in metal and not in the adhesive.

The trailing edge of the bonded subassembly was then filled with FM-29 foam adhesive and cured. After post cure (8 hours total, including 2 hours cycle at 600°F) of the subassembly, the fiberglass polyimide core on the bonded subassembly was machined to the correct height as shown in Figure 66.

A Verifilm check of the top skin to rudder assembly was then accomplished. After the top boron/polyimide skin was lightly grit blasted and the top of the core on the rudder subassembly was primed and dried, the top skin was bonded to the rudder subassembly in an autoclave. The bonded rudder assembly after post cure is shown in Figure 67.

6.2.4 NDT Inspection of F-4 Rudder Assembly - The F-4 rudder assembly was ultrasonically "C" scan inspected, and radiographically examined. The NDT detected several small unbonds in skin to edgemember areas and a few apparent voids in the core to edgemember bonds. In

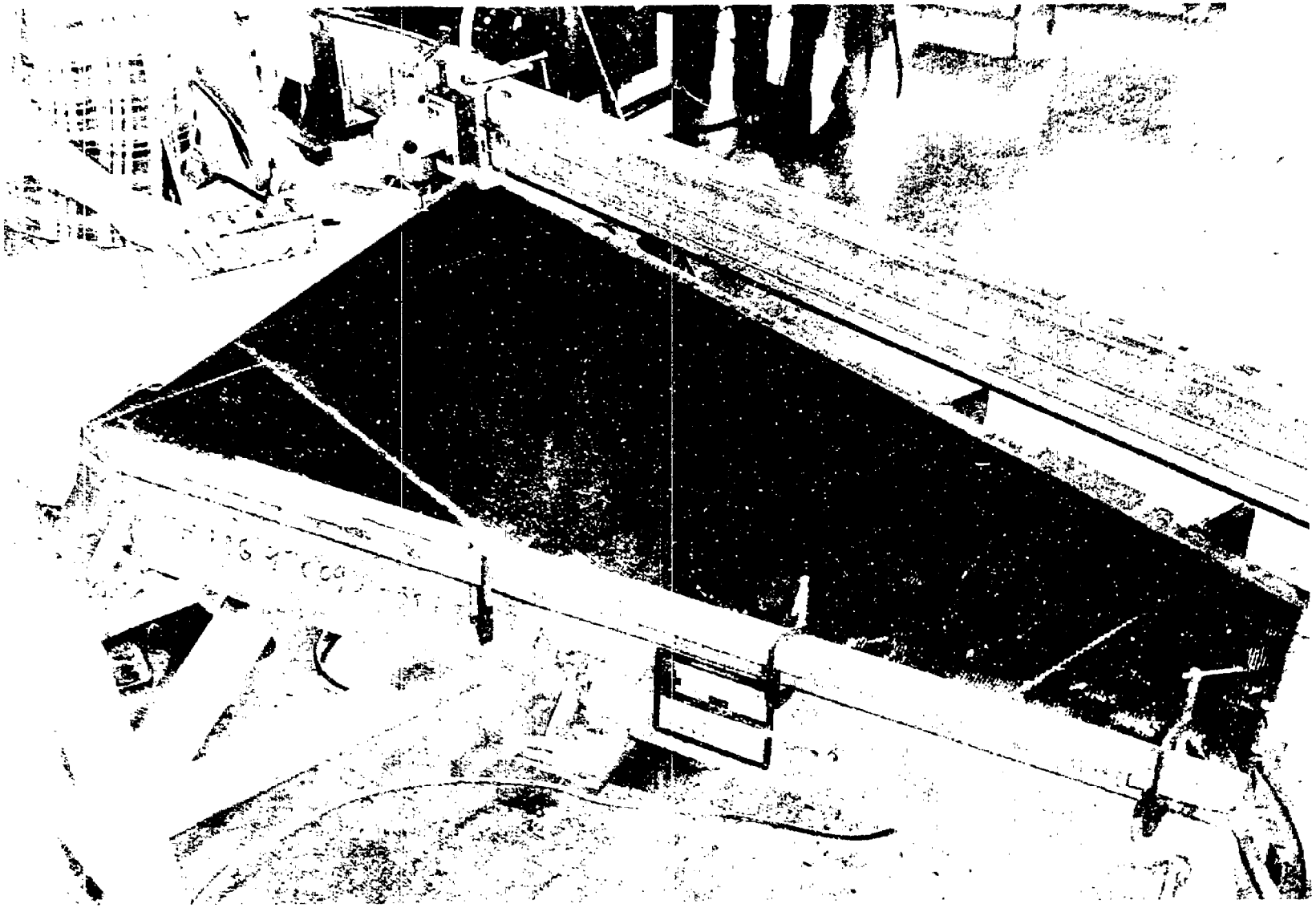

FIGURE 66 MACHINING OF FIBER GLASS POLYIMIDE CORE ON RUDDER SUBASSEMBLY

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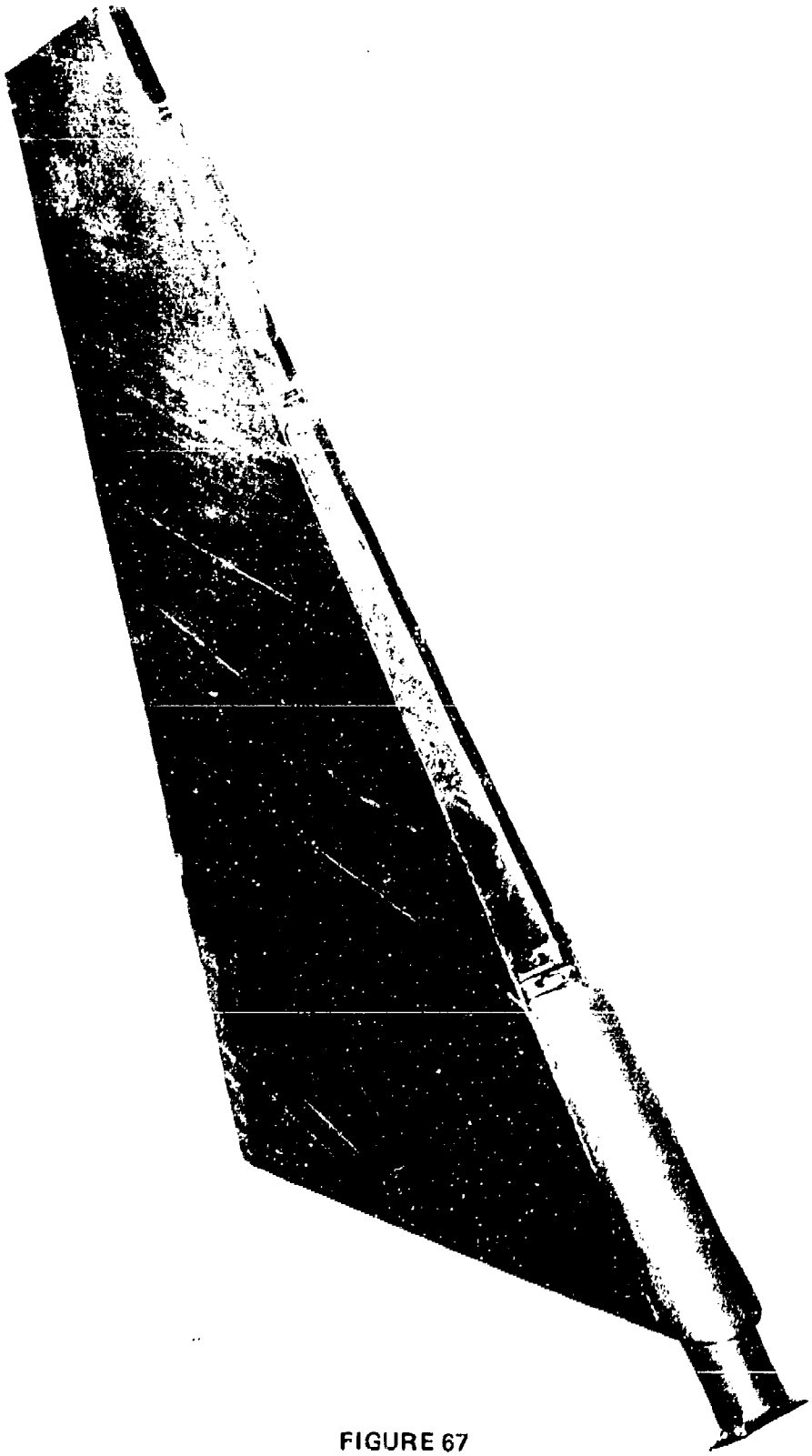

FIGURE 67
F-4 POLYIMIDE RUDDER

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addition, areas of either bondline voids or thick primer and/or adhesive in the core to skin areas were detected. Three coupons were cut from selected areas of the rudder to determine what the NDT was indicating. Shown in Figure 68 are the three areas where three coupons were cut to evaluate core to skin bonds. The selection of areas from which these specimens were taken was based on NDT results and locations which would have a minimum effect on the rudder load carrying capability. The specimens were visually inspected and then tested in flatwise tension. Visual inspection of these specimens showed a thick adhesive build-up. Table LXII shows the results of the flatwise tension tests. All specimens failed in areas other than in the skin to core bond. Therefore, the skin to core bonds were concluded to be adequate and NDT results were taken to be indications of thick primer and/or adhesive rather than of unbonded areas.

TABLE LXII
POLYIMIDE RUDDER FLATWISE TENSION TESTS

Specimen No.	NDT Results (Ranking) [1]	Failure Load (lb)	Flatwise Tension Stress (psi)	Failure Remarks
1	1	280	–	Core Split; No Bond Failure
2	2	1450	462	Skin Delaminated; No Bond Failure; Core Was Starting to Split
3	3	1180	376	Skin Delaminated; No Bond Failure

[1] 1 best to 3 worst.

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Repair of F-4 Rudder Assembly - The skin to edgemember unbonded areas as indicated in Figure 62 were repaired using a hypodermic needle to apply the adhesive. Areas of the rudder from which the flatwise tension specimens were taken were also repaired using boron/polyimide patches. These repair procedures were similar to those used to repair boron/epoxy rudders.

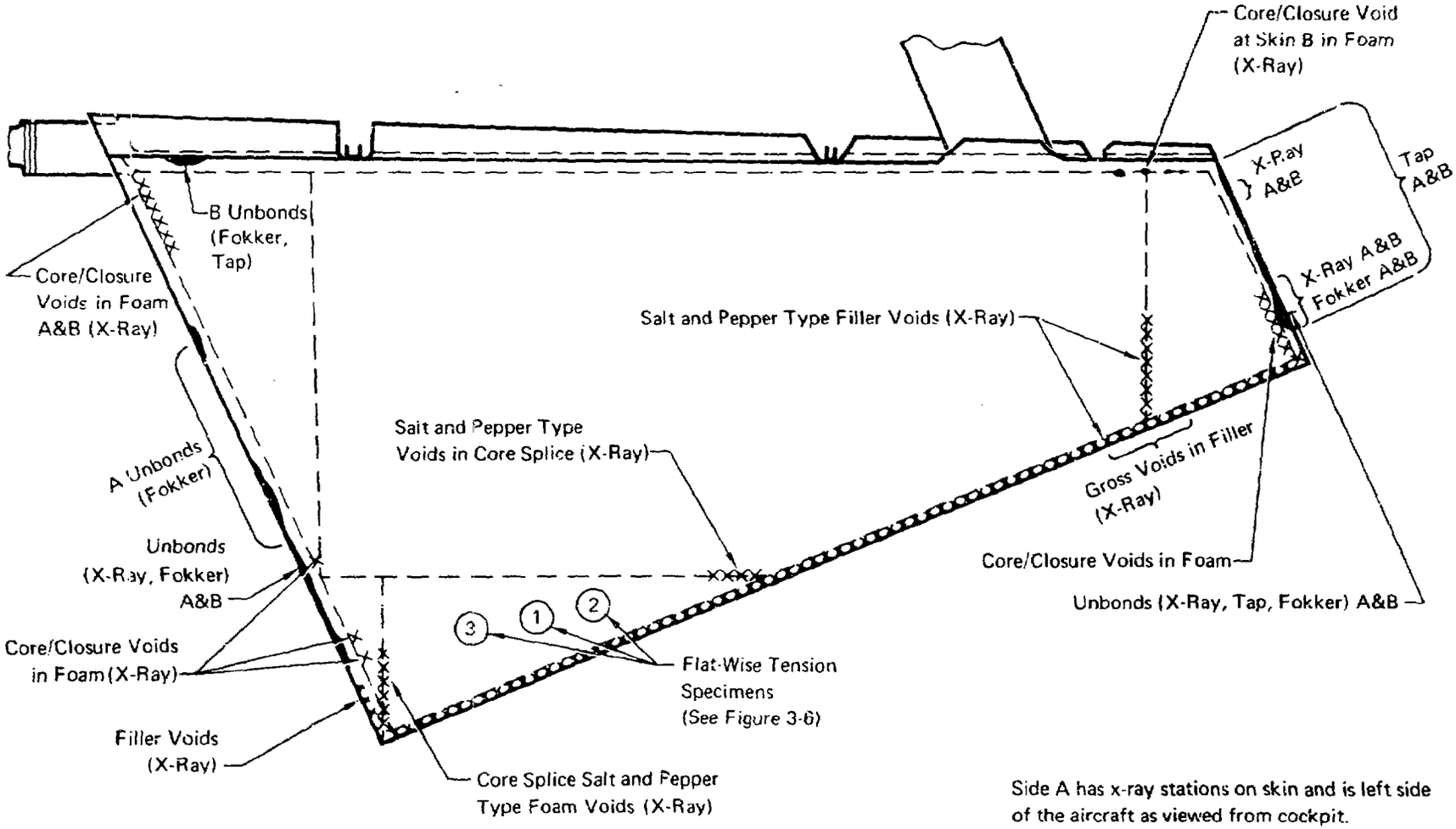


FIGURE 68
NDT INSPECTION OF F-4 POLYIMIDE RUDDER

GP72-0593-114

6.2.5 F-4 Rudder Test - The polyimide rudder was subjected to 400% design limit load (4640 pounds) without catastrophic failure. An inspection of the rudder at the end of the test indicated that a small area of the skin was delaminated at the lower-forward corner of the rudder. Strain gage data indicates that this delamination occurred at approximately 240% design limit load. The F-4 rudder is a torsional stiffness (GJ) critical structure. Therefore, the skin gage required for stiffness considerations results in an over strength structure. Consistently with these results, the boron/epoxy rudder met stiffness requirements and was capable of sustaining loads of 380 - 410 percent of design limit load.

(a) Rudder Test Setup - An F-4 static article aft-fuselage, including the vertical fin, was mounted on a rigid backup structure at the fuselage station 515.0 splice (Figure 69). The polyimide rudder was installed on the vertical fin using production fittings as in a normal service installation. A fixed length simulated actuator was installed between the rudder and the empennage assembly to hold the rudder in the neutral position (aligned with the aircraft centerline). In addition, a linkage was installed in the location of the rudder damper. This linkage was adjusted so that it would react approximately one half of the rudder hinge moment and the fixed length simulated actuator would react the other half.

Neoprene rubber tension pads were bonded with EC1300 adhesive on the left hand surface of the rudder as shown in Figure 70. The pads were linked through a whiffletree loading system to a hydraulic cylinder. Pressure was applied to the cylinder with a hydraulic hand pump. Loads were measured using a calibrated strain link. Strain gages were bonded to both the left and right-hand surfaces of the rudder at the locations shown in Figure 71. Tension pads in the area of strain gages were trimmed to provide a minimum of 1/4 inch clearance with the strain gages. Deflection transducers were attached to the right-hand surfaces of the fin and the rudder at the locations shown in Figure 72.

(b) Test Procedure - The rudder was loaded to 400% design limit load (4640 pounds). The loading was identical to that of the boron/epoxy rudders. No loads were applied to the fin. Loads were applied in increments of 20% design limit load up to 100% and in increments of 10 and 30% above 100%. Loads were applied at a rate not in excess of 20% design limit load per 30 seconds. The outputs from the deflection transducers and strain gages were recorded continuously throughout the test to

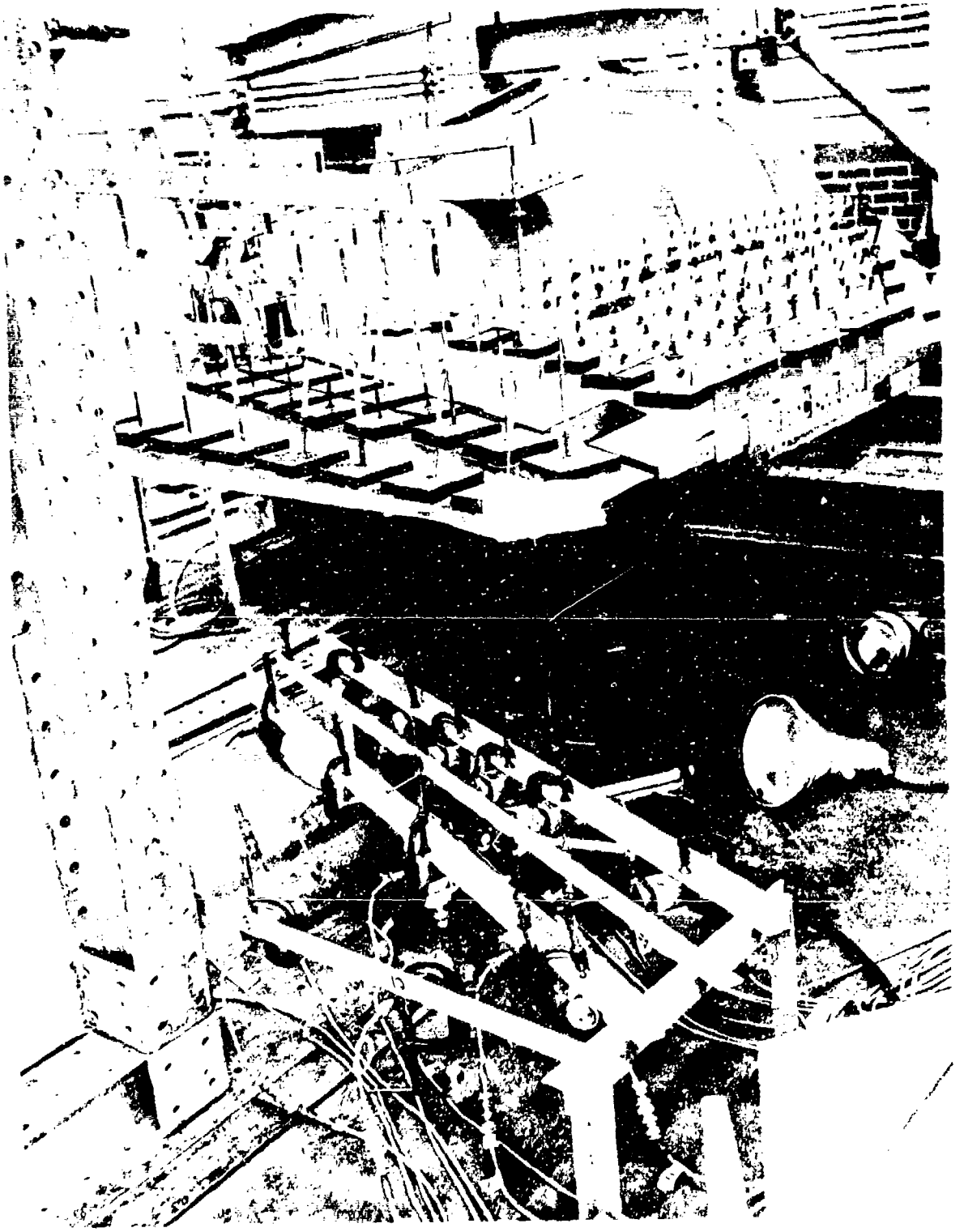
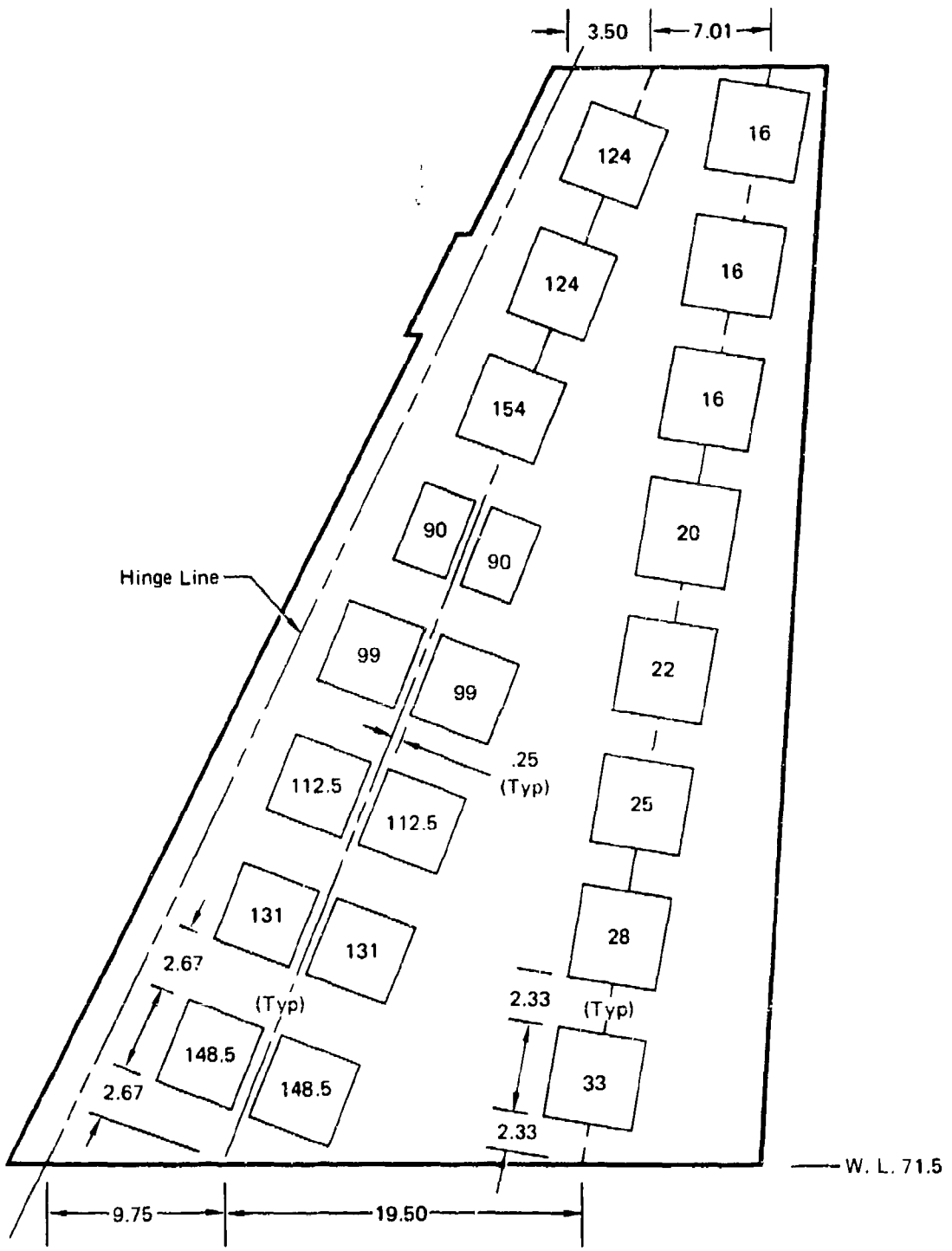


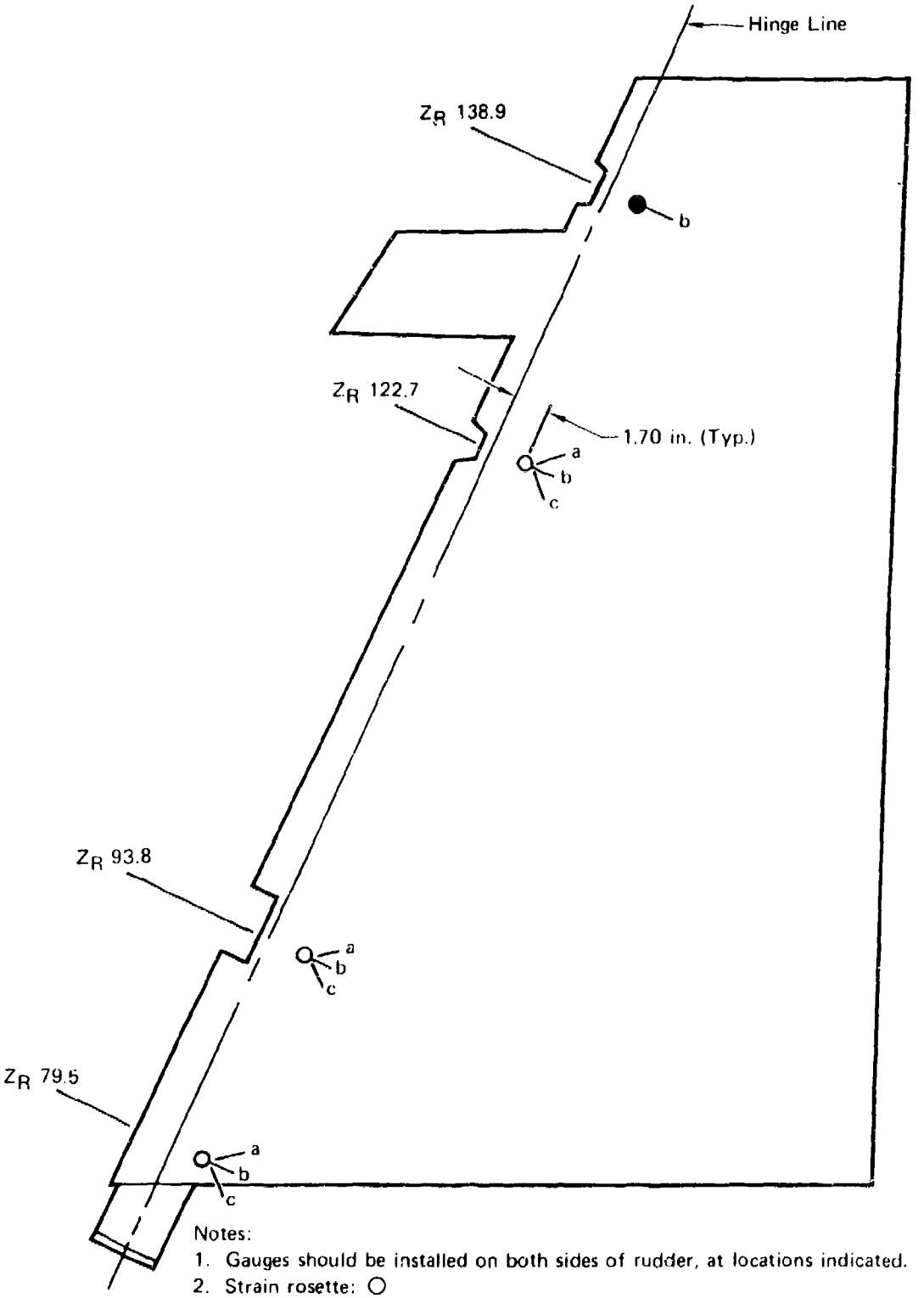
FIGURE 69
F-4 POLYIMIDE RUDDER STATIC TEST SET-UP



Notes:
1. XX indicates tension pad and its loading in lb @ 150%
2. All tension pads to be 5 inch x 5 inch except two pads with 90 lb loads. These pads to be 4 inch x 5 inch.
3. Ultimate load = 1740 lb ultimate hinge moment = 13,500 in. lb center of pressure at 30% chord. Ultimate load = 150% design limit load.

GP72-0593-117

FIGURE 70
RUDDER TENSION PAD LAYOUT AND ULTIMATE LOADS



Notes:

1. Gauges should be installed on both sides of rudder, at locations indicated.
2. Strain rosette: ○
3. Strain gauge: ●

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FIGURE 71
STRAIN GAUGE LOCATIONS FOR RUDDER TEST

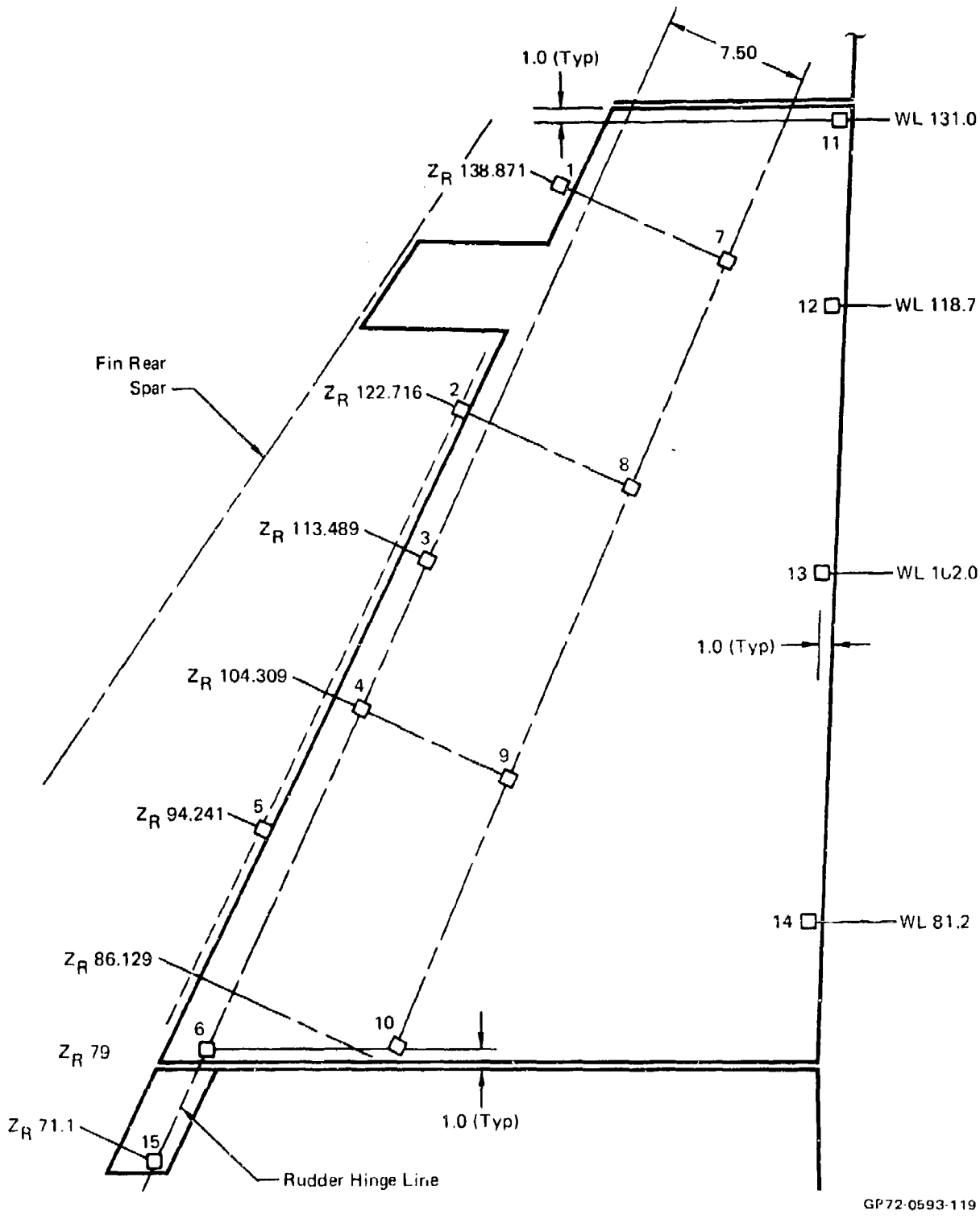

FIGURE 72
RUDDER DEFLECTION POINT LOCATIONS

400% design limit load and back to zero load. Certain strain gages and deflection transducers were monitored at the various load increments.

(c) Test Results - The rudder sustained 400% design limit load (4640 pounds) without catastrophic failure. An inspection of the rudder at the end of the test indicated that the skin had a small delamination at the lower-forward corner of the rudder. Deflection versus load graphs are presented in Figure 73 through 75. Strain versus load plots are presented in Figures 76 through 79. The strain data presented in Figure 79 (strain gages 7A, 7B, and 7C) indicates that the initial skin delamination occurred at approximately 240% design limit load.

Photographs showing the rudder at no load, 150%, and 400% design limit load are presented in Figures 80 through 82.

(d) Conclusions - Based on the results of this test, it is concluded that the F-4 polyimide rudder can satisfactorily react loads to 400% design limit load without major failure, as a matter of fact, this test result is consistent with previous tests conducted on the MCAIR boron/epoxy rudders. For example, the A-32 boron/epoxy rudder had an initial failure at 246% design limit load. The rib adjacent to the lower hinge buckled and partially separated from the skin. This failure did not prevent successful continuation of the test to 400% design limit load.

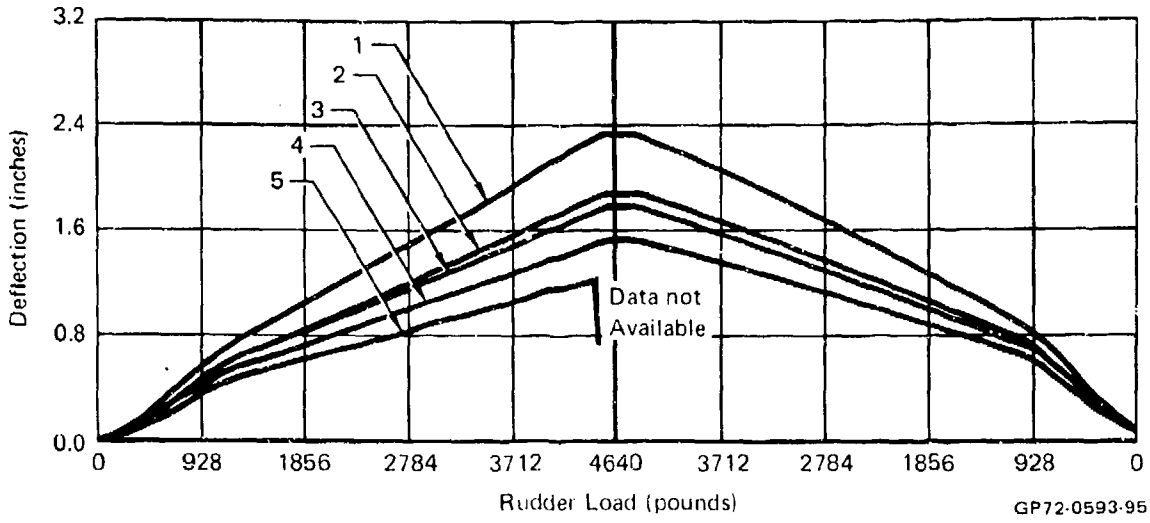

FIGURE 73
DEFLECTION vs RUDDER LOAD (TRANSDUCERS 1 THROUGH 5)

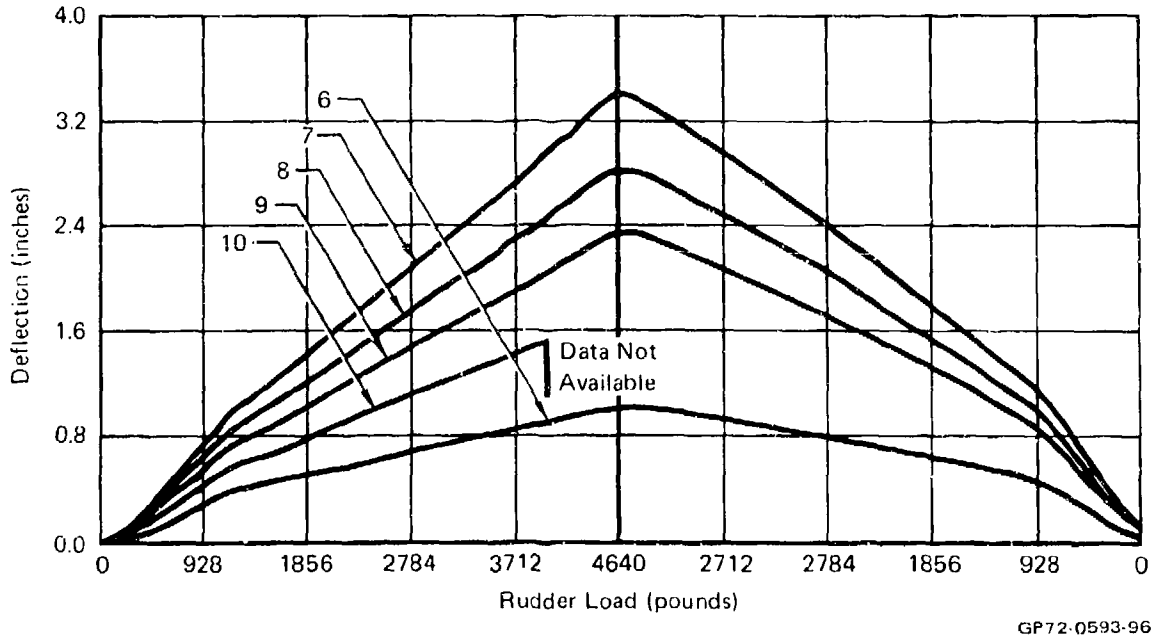

FIGURE 74
DEFLECTION vs RUDDER LOAD (TRANSDUCERS 6 THROUGH 10)

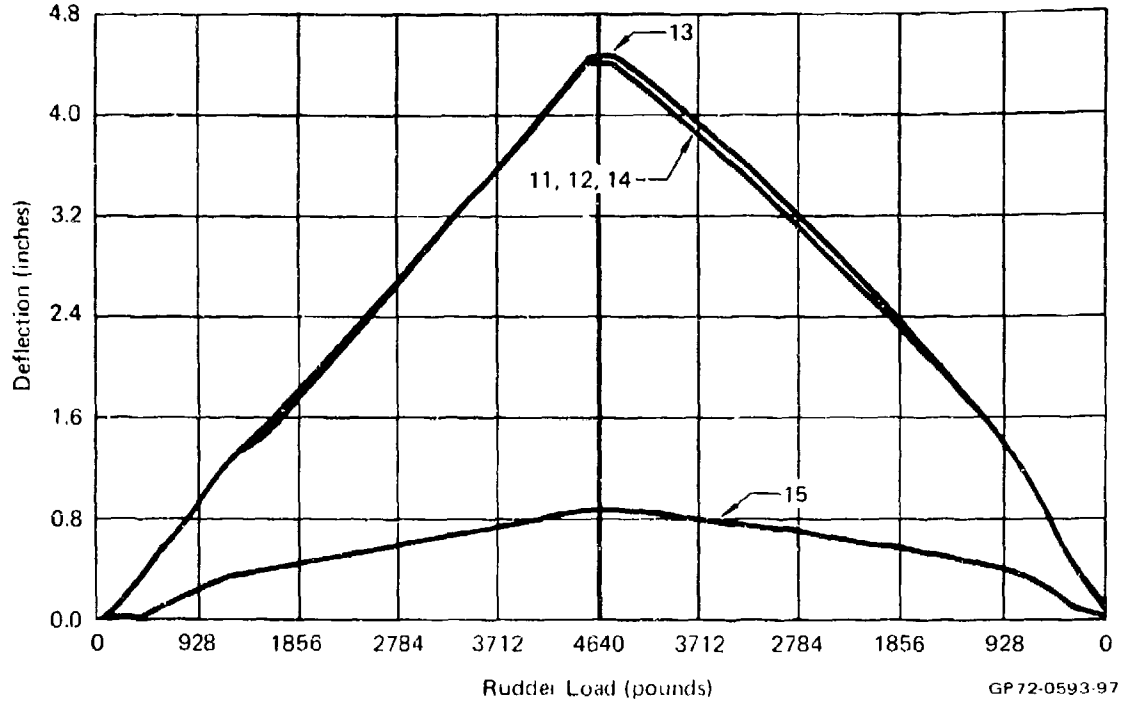

FIGURE 75
DEFLECTION vs RUDDER LOAD (TRANSDUCERS 11 THROUGH 15)

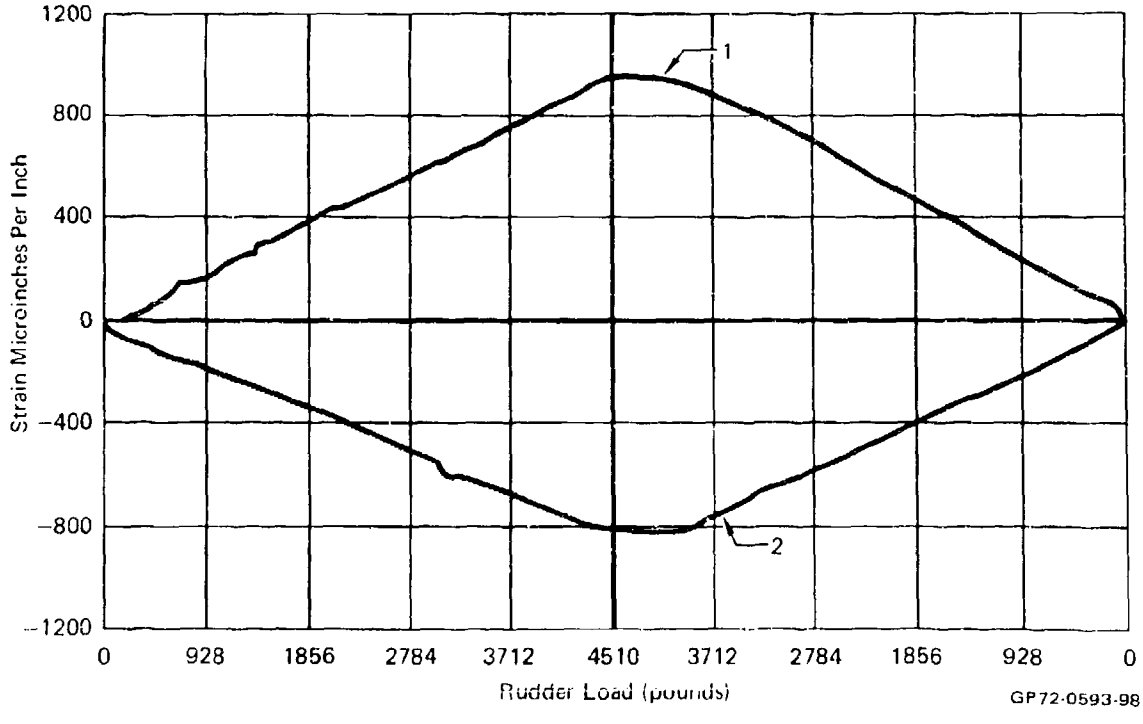

FIGURE 76
STRAIN vs RUDDER LOAD (STRAIN GAGES 1 AND 2)

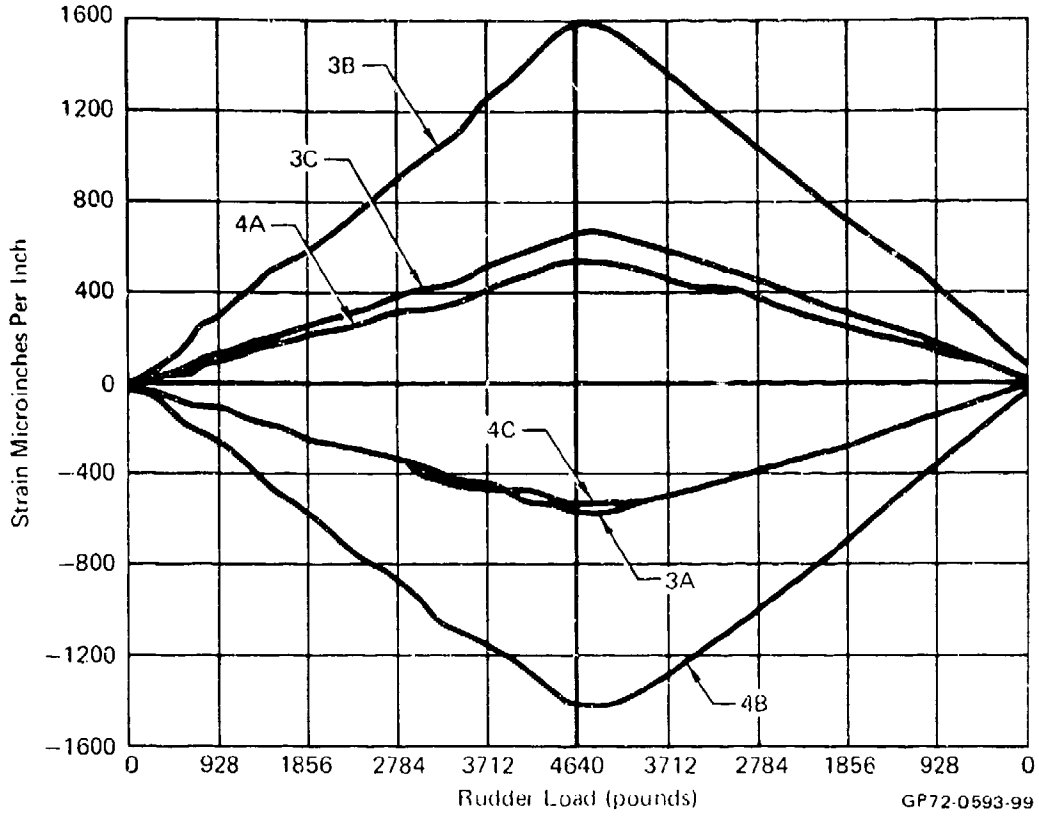

FIGURE 77
STRAIN vs RUDDER LOAD (STRAIN GAGES 3 AND 4)

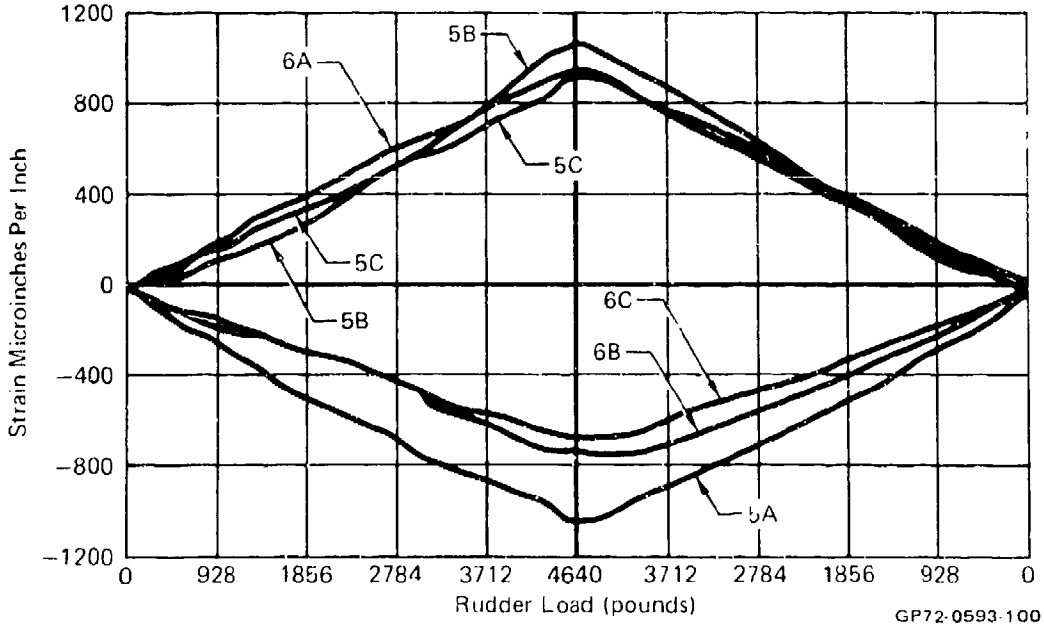

FIGURE 78
STRAIN vs RUDDER LOAD (STRAIN GAGES 5 AND 6)

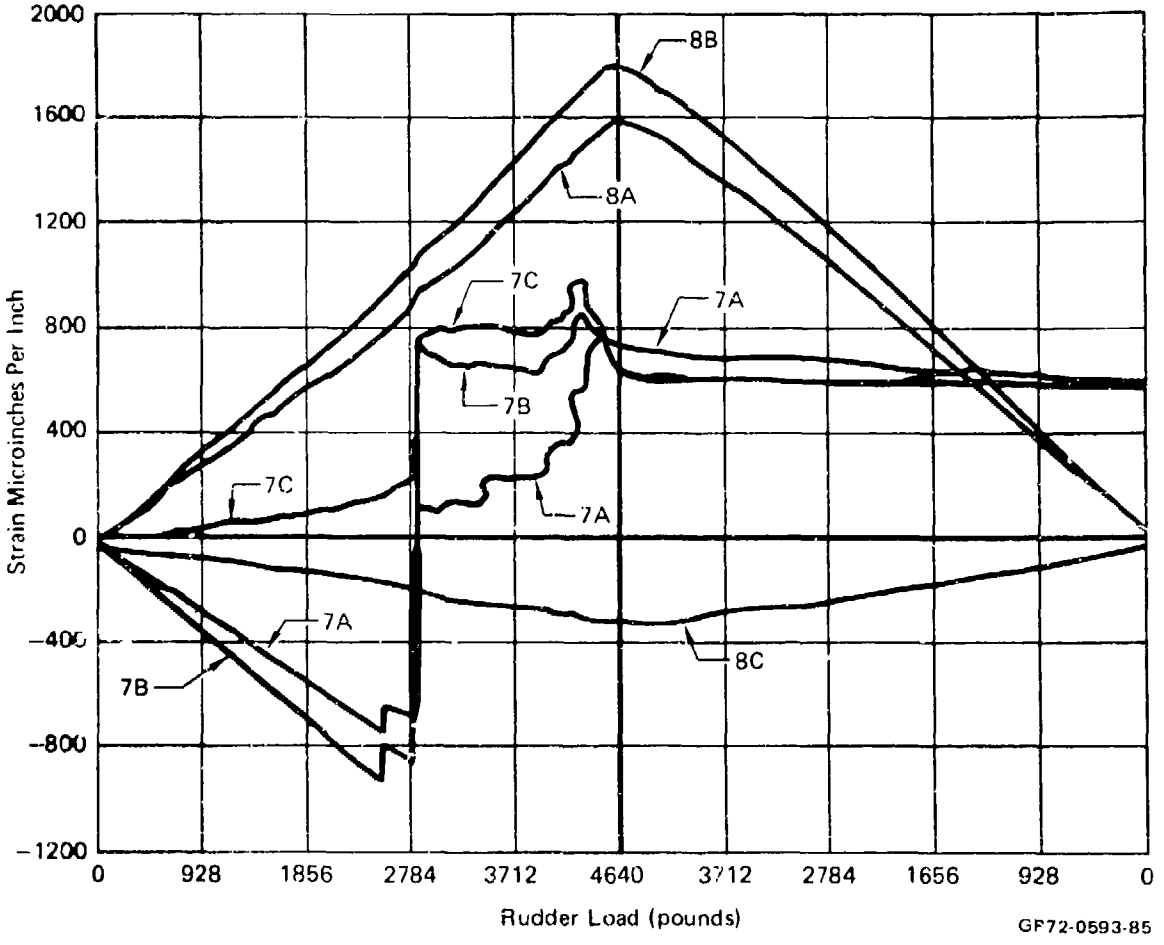

FIGURE 79
STRAIN vs RUDDER LOAD (STRAIN GAGES 7 AND 8)

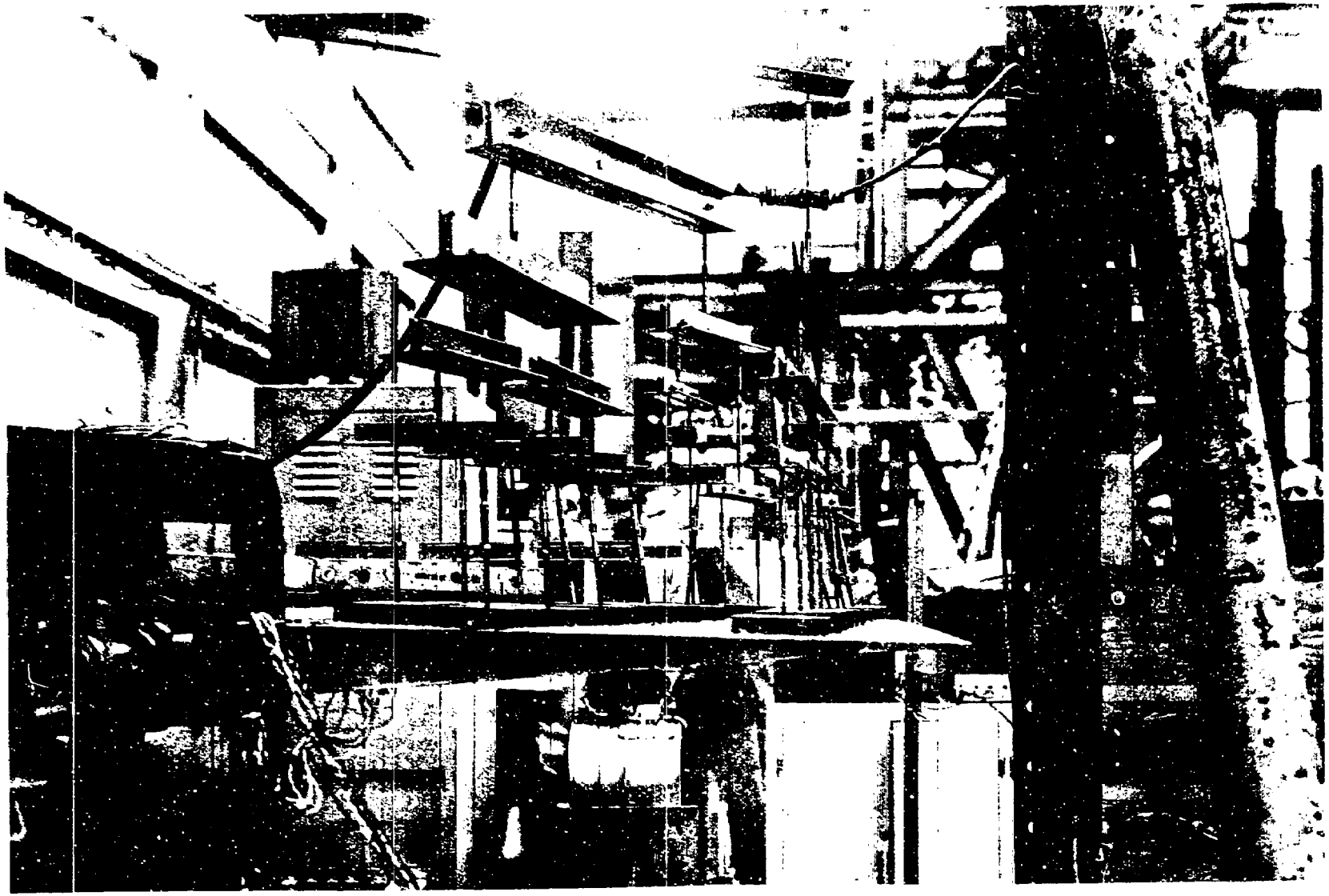

FIGURE 80
RUDDER WITH NO LOAD APPLIED

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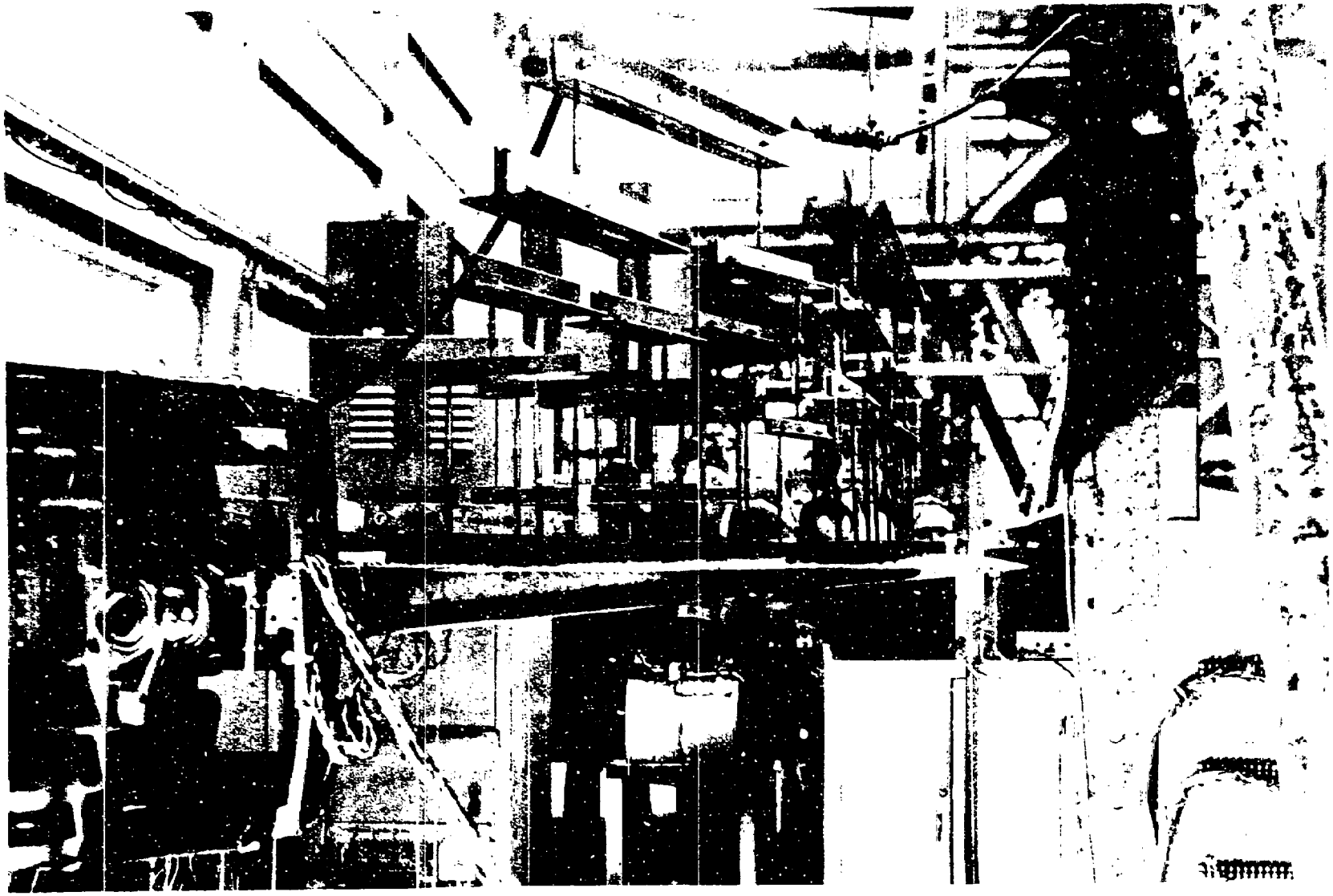

FIGURE 81
RUDDER DEFLECTION AT 150% LOAD

GP72-0593-113

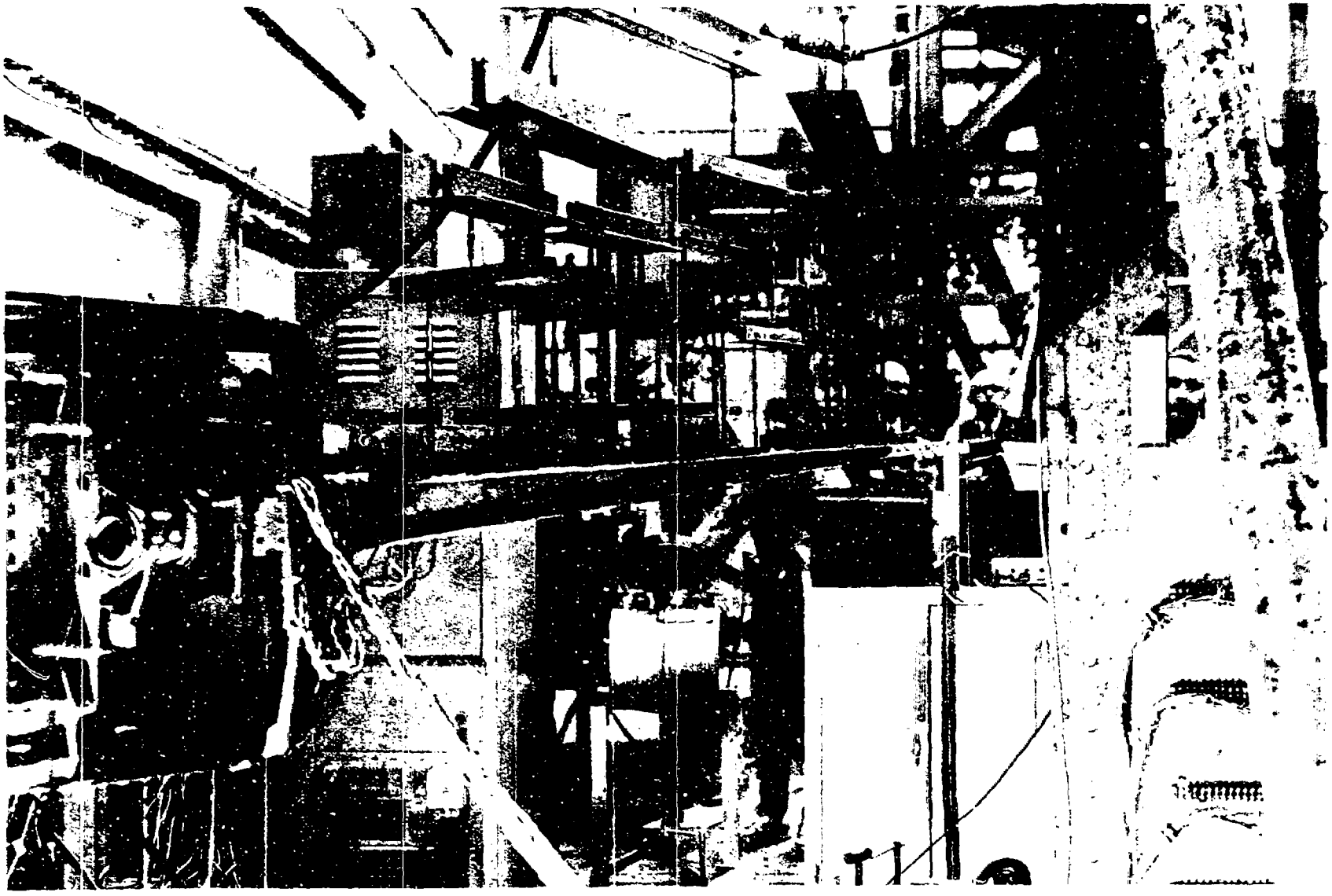

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FIGURE 82
RUDDER DEFLECTION AT 400% LOAD

6.3 F-15 Polyimide Wing Compression Panel Fabrication/Test

6.3.1 Introduction - The F-15 polyimide compression panel was fabricated with boron/SB703 skins, graphite/SB703 hat stiffeners and edge members, and HRH-327 fiberglass polyimide honeycomb core. Polyimide adhesives were used for 550°F capability.

The fabrication sequence for the compression panel was as follows:

(a) Fabricate two boron/SB703 skins

(b) Machine the HRH-327 honeycomb core to size

(c) Fabricate the graphite/SB703 edgemembers and hat stiffeners

(d) Bond the HRH-327 honeycomb core to the graphite/SB703 edgemembers

(e) Bond the boron/SB703 skins to the edgemember-honeycomb core subassembly

(f) Bond the graphite/SB703 hat stiffeners to the sandwich panel.

For each of these steps appropriate process control tests and NDT were performed to verify process/component acceptability.

Two of the fabrication operations required several attempts before parts acceptable for the compression panel could be obtained. Two sets of boron/SB703 skins and four sets of graphite/SB703 hats were fabricated. NDT revealed flaws (Section 6.3.4) which were confirmed by photomicrographic inspection.

6.3.2 Boron/SB703 Skins - Two 14.6 in. (0°) x 24 in. x 9 to 13 ply skins were fabricated with the process described in P.S. 14224 (Appendix), the same as was used for the F-4 Polyimide Rudder skins. NDT of the first set showed one of the skins to be unacceptable. NDT (ultrasonic "C" scan through transmission) results are shown in Figure 83. The principal causes for the unacceptable skin were considered to be (1) slower cool-down rate than required for the "B" stage cycle, and (2) slower heat-up rate than required for the cure cycle. These factors could have reduced the resin flow sufficiently to create the voids shown in Figure 83.

Another skin was fabricated with proper heat-up and cool down rates using the same cure/post cure cycle described above. It was satisfactory as evidenced by the ultrasonic "C" scan shown in Figure 84. Process control tests for the two sets of boron/SB703 skins are shown in Table LXIII.

FIGURE 83
NDT OF REJECTED B/SB703 SKIN

GP72-0593-6

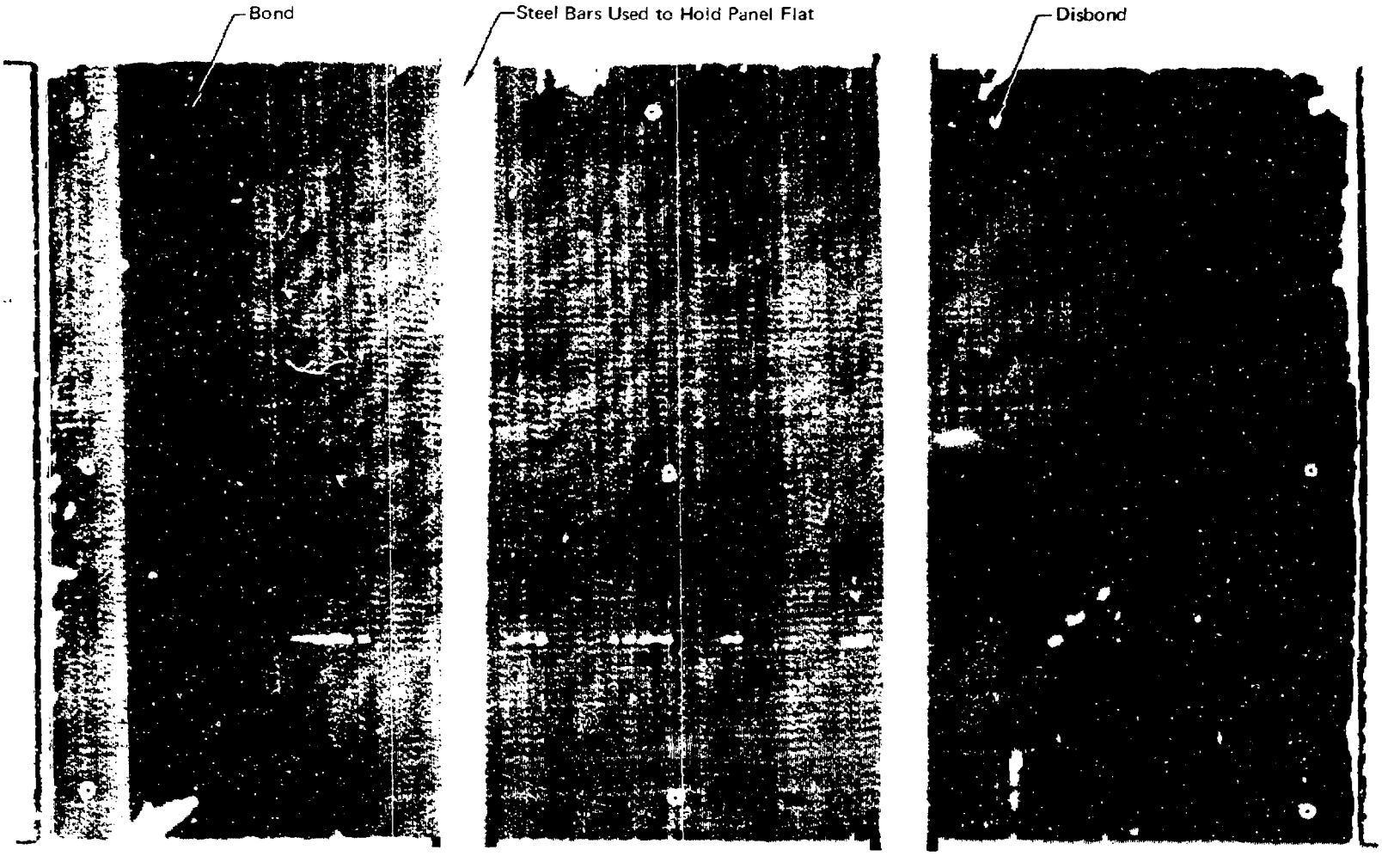

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FIGURE 84
NDT OF ACCEPTABLE B/SB703 SKIN

TABLE LXIII
BORON/SB703 SKIN PROCESS CONTROL TESTS

Mechanical Property	Test Temperature (°F)	Minimum Required (psi)	Actual Value	
			1st Set	2nd Set
0° Flexural Strength	RT	190,000	202000	241500
	550	155,000	167000	185600
Interlaminar Shear Strength	RT	11,000	16300	16 00
	550	6,000	7100	7000

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6.3.3 HRH-327 Honeycomb Core Machining - The HRH-327 honeycomb core was machined using a valve stem cutter in a manner similar to the machining of the F-4 rudder honeycomb core. The size was 4 in. (ribbon direction) x 24 in. x 0.117 in. high and no difficulties were encountered.

6.3.4 Graphite/SB703 Components - The compression panel requires 3 graphite/SB703 hat stiffeners and 4 graphite/SB703 edgemembers. The hat stiffeners consist of a basic 8 ply ±45° lay up that extends across the entire cross-section. In addition, the cap area contains 9 plies of 0° graphite/SB703. This lay-up differs from that used for the graphite/epoxy hats of the F-15 Composite Wing Program where the 0° plies are boron/epoxy. However, because of a lack of development effort with hybrid laminates and the difference in cure cycles between graphite/polyimide and boron/polyimide it was decided to maintain an all graphite/SB703 stiffener. The edgemembers consisted of a 16 ply $[\pm 45, 0_5, 90]_S$ lay-up. Fabrication of both hat stiffeners and edgemembers required the use of Mylar templates for flat pattern lay-out prior to collation on the tool. Hat stiffener fabrication also required the addition of a mist coat of MMP solvent on the prepreg just prior to collation on the tool to aid formability. Steel caul plates were used over

the lay-up to assist in obtaining more uniform pressure. Perforations (.06 in. dia.) on 0.5 in. centers permitted volatile removal. The fabrication was performed per P.S. 14227 (Appendix) and was similar to the process used for the F-4 rudder spar fabrication.

Visual appearance of the first set of hat stiffeners was not totally satisfactory. The caul plates left mark-off ridges in the cap and radii. Figure 85 shows the stiffeners. Subsequent NDT (see Figure 86) indicated the edgemembers had some voids. NDT also indicated some voids in the hat stiffeners (see Figure 87).

A second set of hat stiffeners was then fabricated. The caul plates were discarded to eliminate the markoff ridges in the radii and cap (both critical areas). Caul plates had been successfully eliminated for graphite/epoxy hats on the F-15 Composite Wing Program. A second set was laid up without caul plates, but during the cure cycle the bag broke at a part temperature of approximately 250°F. The part was under full vacuum and 100 psi pressure was being applied at the time of the break. This point of the cure cycle is very critical as it is the point where resin flow starts and the rate of imidization increases. The tool was quickly removed from the autoclave, a new bag installed and an attempt was made to complete the cure of the parts. However, sufficient resin flow could not be generated for adequate lamina bonding. NDT revealed the parts to have excessive voids and disbonds, which was not unexpected.

A third set of hat stiffeners were then fabricated using the same procedures as for the second set except that the tool was modified to eliminate the bag bridging problem. The cure cycle was successful with good vacuum (29+ in. Hg) being held throughout the critical parts of the cure cycle. All heat up and cool down rates were within specification limits. Removal of the hat stiffeners from the tool revealed 3 apparently acceptable parts. NDT of the hat stiffeners prior to post cure revealed excellent bonds everywhere, except in the cap area. Figure 88 shows the ultrasonic

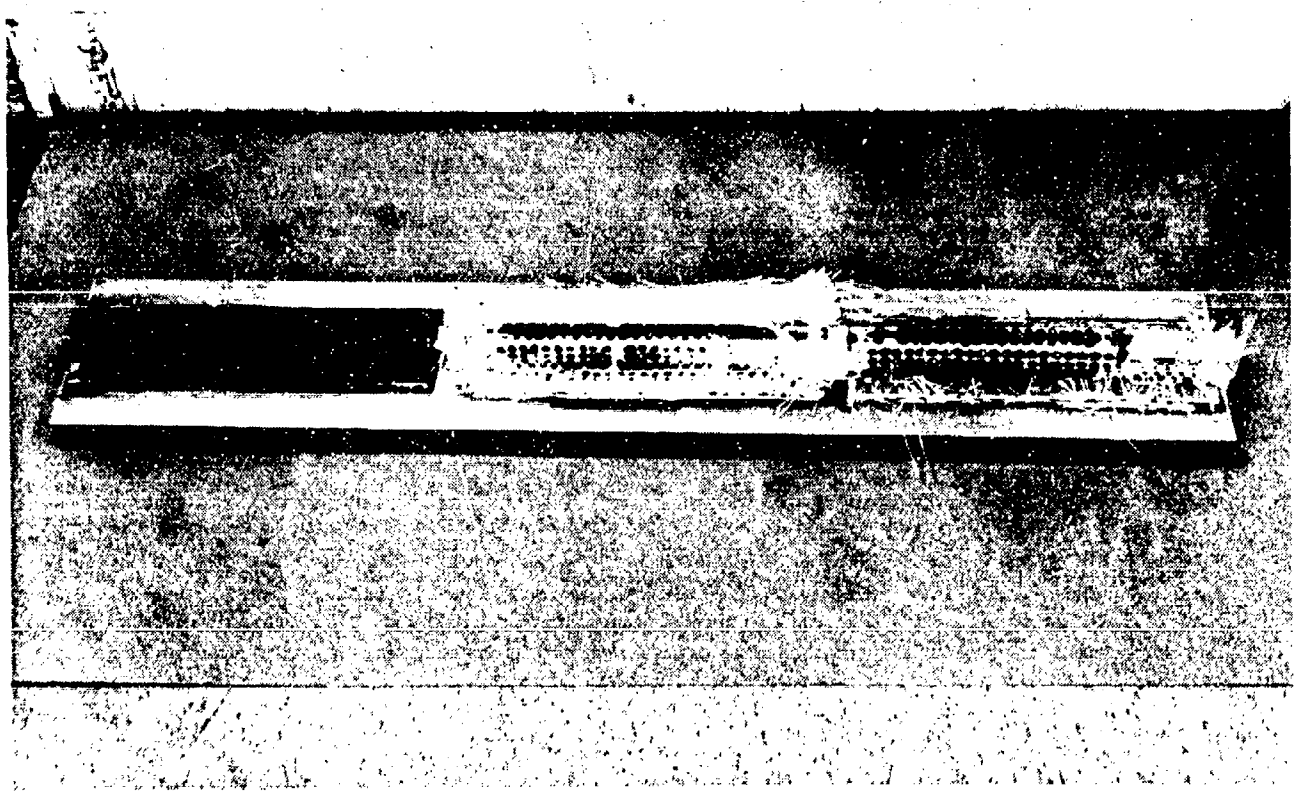

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FIGURE 85
G/SB703 HAT STIFFENER AFTER CURE

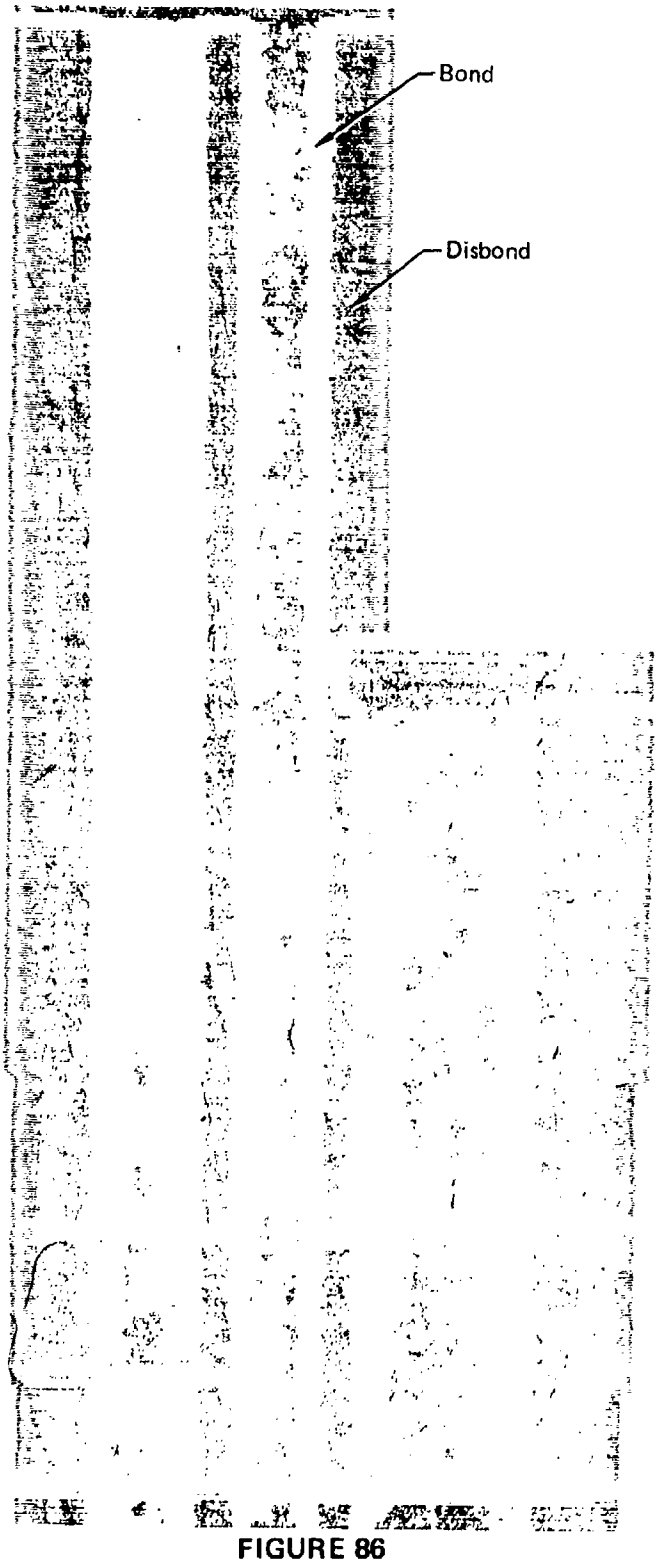
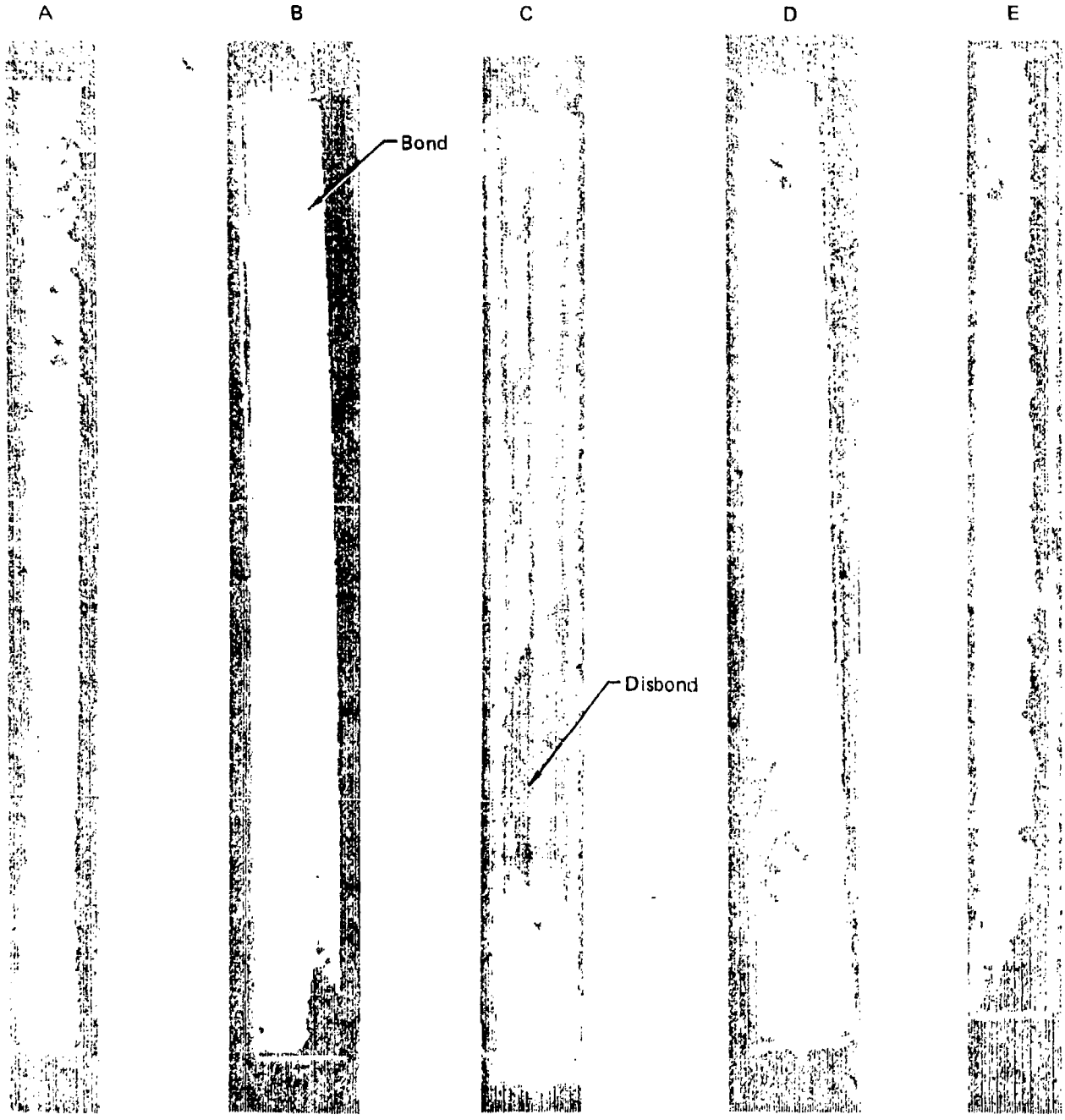


FIGURE 86
NDT OF G/SB703 EDGE MEMBERS

GP72-0593-8



A, E - Flanges
B, D - Webs.
C - Cap

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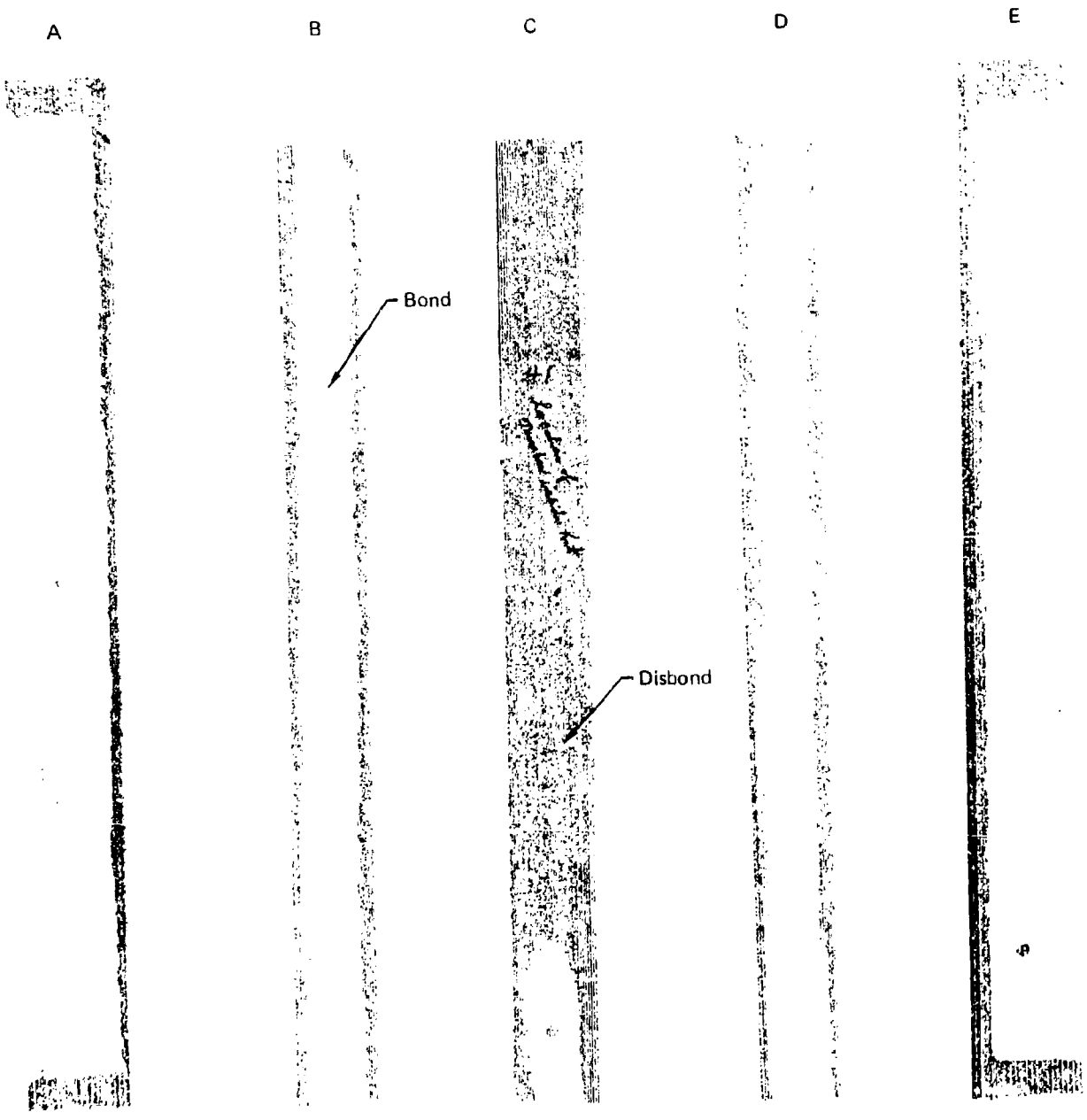
FIGURE 87
NDT OF G/SB703 HAT STIFFENER



A, E - Flanges
B, D - Webs
C - Cap

GP72-0593-10

FIGURE 88
NDT OF G/SB703 HAT STIFFENER (3RD RUN)

"C" scans for the hat stiffeners and Figure 89 shows a photomicrograph of a cross-section through the hat where NDT indicated a disbond. It was conjectured that the problem could have been one of thermal or mechanical strain where the nine 0° plies are sandwiched between four ±45° plies on both top and bottom.

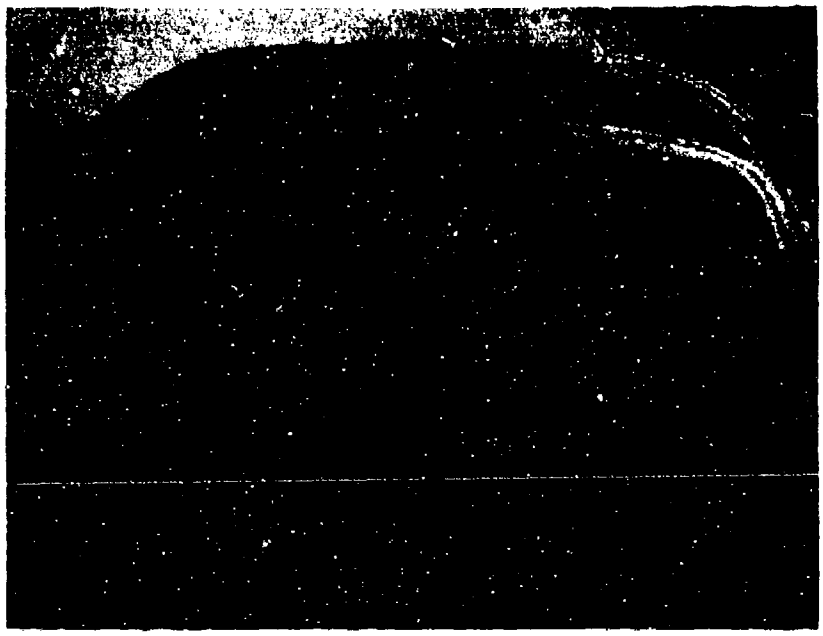
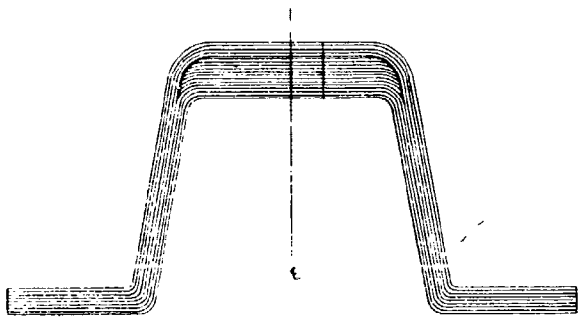


FIGURE 89 GP72-0593-11

HAT STIFFENER CROSS-SECTION (3RD RUN)

Sufficient graphite/SB703 prepreg remained for a fourth set of hat stiffeners. The approach for this set was to maintain the same fabrication procedures, but to redesign the cap area of the hat stiffener. The original and revised lay-up cross sections for the cap are shown in Figure 90. It was felt that dispersing the 0° plies among the 45° plies would reduce the strain concentration where the nine 0° plies had interface with the 45° plies.

The fabrication cycle was within specification requirements, but again NDT (after cure but prior to post cure) revealed disbonds in the cap area. Figure 91 shows a cross-section of one of these hat stiffeners.



Original

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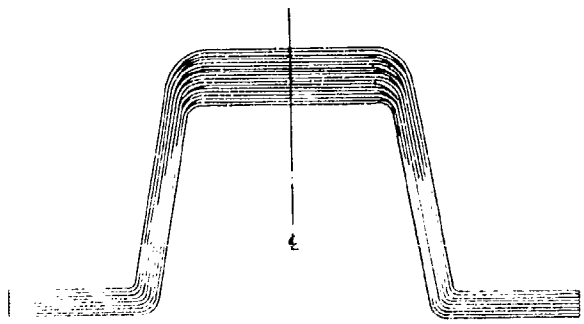

Revised

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FIGURE 90
G/SB 703 HAT STIFFENER CROSS-SECTIONS

GP72-0593-17

FIGURE 91 GP72-0593-12
CROSS-SECTION OF HAT STIFFENER USING REVISED LAY-UP (4TH RUN)
SANDWICH PANEL NDT

After fabricating 4 sets of graphite/SB703 hat stiffeners, sufficient time or material did not remain for a fifth attempt. Therefore, the first set was selected as the best all-around hat stiffeners available for the compression panel. Process control tests for fabrication of this set of hat stiffeners plus the graphite/SB703 edgemembers gave the results shown in Table LXIV. These results compare very favorably with those generated for the F-4 Polyimide Rudder Spar and are well above the minimum requirements.

TABLE LXIV
PROCESS CONTROL TEST RESULTS FOR G/SB703 COMPONENTS

Process Control Test	Temperature (°F)	Specification (psi)	Test Result (psi)
Interlaminar Shear Strength	RT	8,000	13,400
	550	5,000	6,700
0° Flexure Strength	RT	170,000	192,300
	550	110,000	113,100

GP72-0593-14

6.3.5 Edgemember to Honeycomb Core Bond - The graphite/SB703 16 ply thick edgemembers were bonded to the premachined HRH327 honeycomb core with the FM-29 Foaming Adhesive per P.S. 14225 (Appendix). Steel shims were used to underneath the edgemembers so that the honeycomb core would be .004-.005 in. higher on both top and bottom than the edgemembers. The FM-20 adhesive was cured by heating to 350°F in an air circulating oven and then holding for one hour. The post cure cycle was then completed by heating this subassembly to 600°F slowly in a nitrogen atmosphere. There was no danger of honeycomb core shrinkage, such as experienced with the F-4 Polyimide Rudder, because the honeycomb core had been post cured to 600°F before machining started. Visual inspection of the part after post cure revealed a satisfactorily bonded subassembly. The FM-29 adhesive process control test gave a core shear strength of 404 psi at room temperature compared to the 200 psi required by P.S. 14225.

6.3.6 Skin to Honeycomb Core Subassembly Bond - After selection of two satisfactory boron/SB703 skins and completion of the edgemember to honeycomb core bond, the next step was to bond the skins to the honeycomb core subassembly with FM-34 adhesive. Preparations prior to bonding consisted of (a) lightly grit blasting the boron/SB703 skin bonding surfaces, (b) apply BR-34 polyimide adhesive primer to the honeycomb core and drying at 400°F, and (c) lightly hand sanding the bonding surface of the graphite/SB703 edgemembers. None of these operations created any difficulties. The bond cycle itself was performed in autoclave at 50 psi with cure at 350°F per P.S. 14225 (Appendix). Post cure was delayed until after the graphite/SB703 hat stiffeners were bonded onto the panel so that two post cure cycles could be combined into one cycle. Panel appearance after bonding was satisfactory, as evidenced by sufficient adhesive filleting at the edge of the panel. NDT also revealed a satisfactory bond cycle as evidenced by the ultrasonic "C" scan of the panel shown in Figure 92. The flatwise tension and lap shear process control test values are shown in Table LXV. All of the flatwise tension specimens failed in the specimen to loading block bond line, and not in core to skin bond line. Because of past experience with this adhesive on the F-4 rudder (Table LXI) and the NDT results as mentioned above, it was felt that the bond between the core and skin was adequate.

TABLE LXV
PROCESS CONTROL TESTS FOR SANDWICH PANEL BONDING
(FM-34 ADHESIVE)

Mechanical Property	Test Temperature (°F)	Specification Minimum (psi)	Test Result (psi)
Flatwise Tension	RT	400	90*
	550	75	91
Single Lap Shear Strength	RT	2250	3610
	550	1500	1840

*Failures occured in the specimen to loading block bond line. No specimen failure

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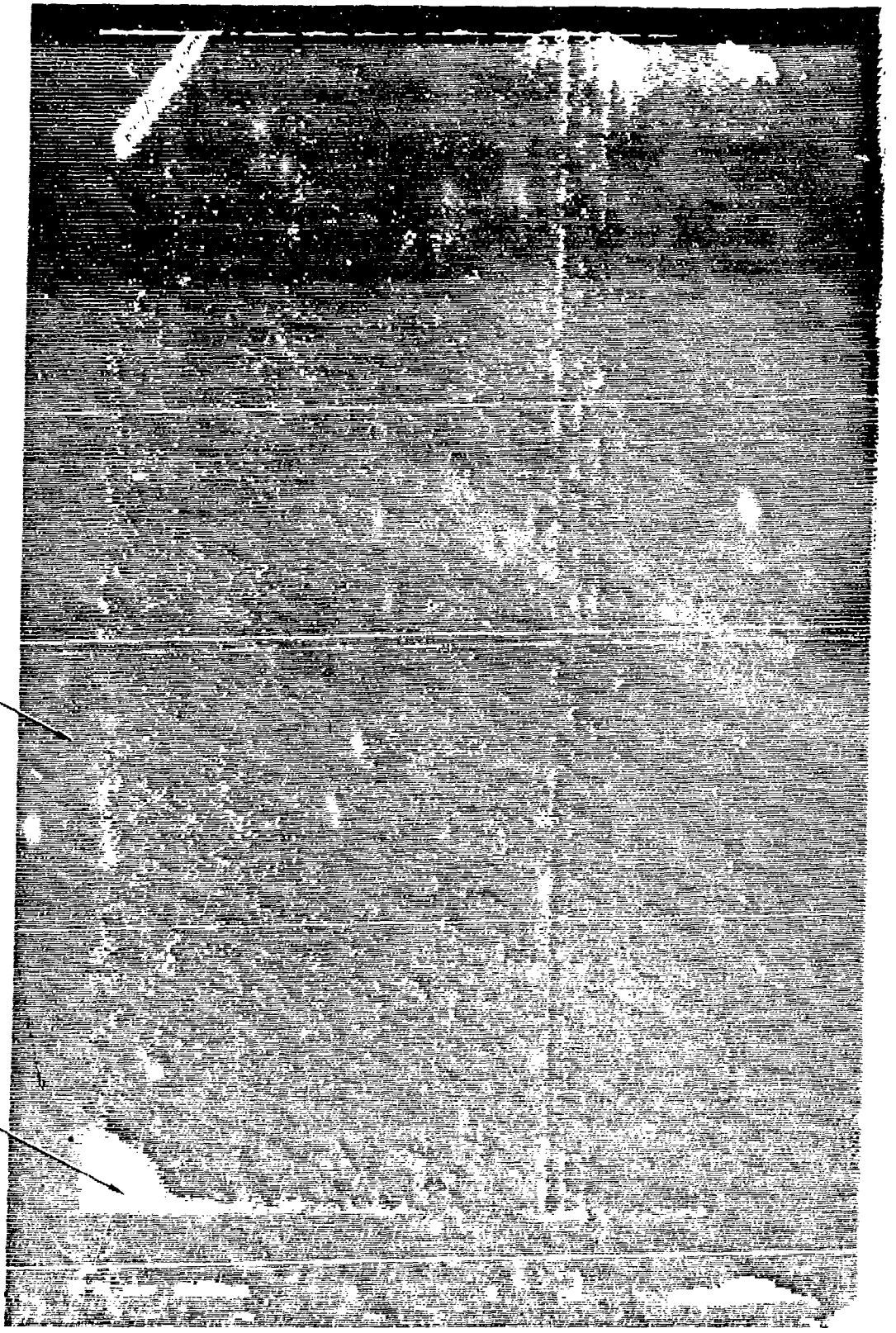

FIGURE 92

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6.3.7 Hat Stiffener to Sandwich Panel Bond - The final assembly operation involved bonding the three graphite/SB703 hat stiffeners onto the boron/SB703 skin sandwich panel with Metlbond 840 adhesive. Hat stiffener placement was dictated by the Engineering drawing, Figure 21. At the conclusion of the adhesive cure cycle, the entire sandwich panel was then post cured in a nitrogen filled retort.

The hat stiffeners were prepared for bonding by lightly sanding the bottom of the flanges with 200 grit sandpaper to remove resin gloss. The surfaces of the boron/SB703 skins were prepared for bonding by lightly grit blasting the areas where the stiffeners are bonded to the skins. Metlbond 840 was then placed between the skin and stiffener with an additional fillet of adhesive placed on the inside radii of the stiffeners-skin intersection. Peel fastener location holes were drilled through the hat stiffener flange and the sandwich panel skin/edgemember. The titanium plates were located for bonding. The panel assembly was then bagged, (see Figure 93) cured and post cured per P.S. 14225 (Appendix) requirements. The cure cycle basically consists of heating directly to 350°F under 50 psig and full vacuum. The post cure cycle is a stepped heat up from 350°F to 600°F in a nitrogen atmosphere. Then the peel fasteners were installed. The lap shear process control specimens were acceptable with the results shown in Table LXVI. These results are similar to those obtained for the F-4 Polyimide Rudder bond cycle. Adhesive filleting was satisfactory. The completed assembly is shown in Figure 94.

TABLE LXVI

METLBOND 840 PROCESS CONTROL TEST RESULTS

Property	Test Temp (°F)	Minimum Requirement	Test Value
Single Lap Shear Strength (psi)	RT	2250	2480
	550	1500	2100

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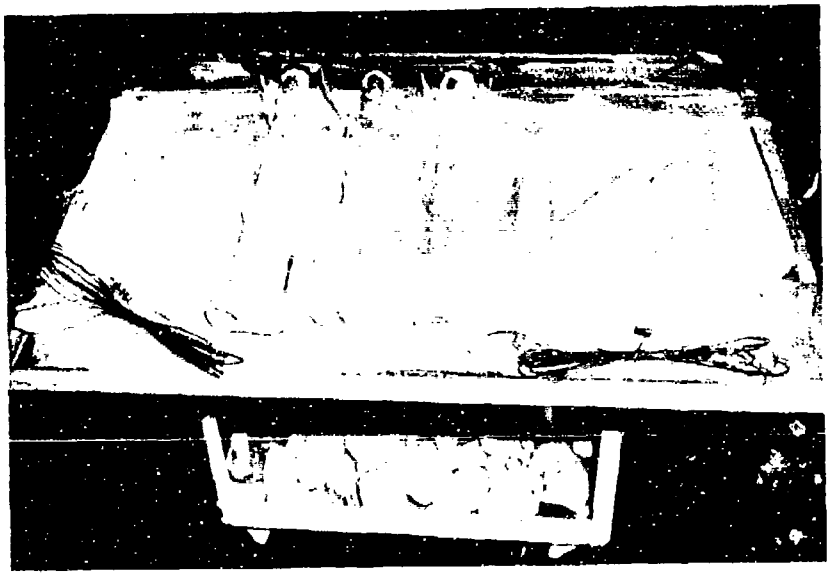


FIGURE 93
POLYIMIDE WING COMPRESSION PANEL PRIOR TO CURE

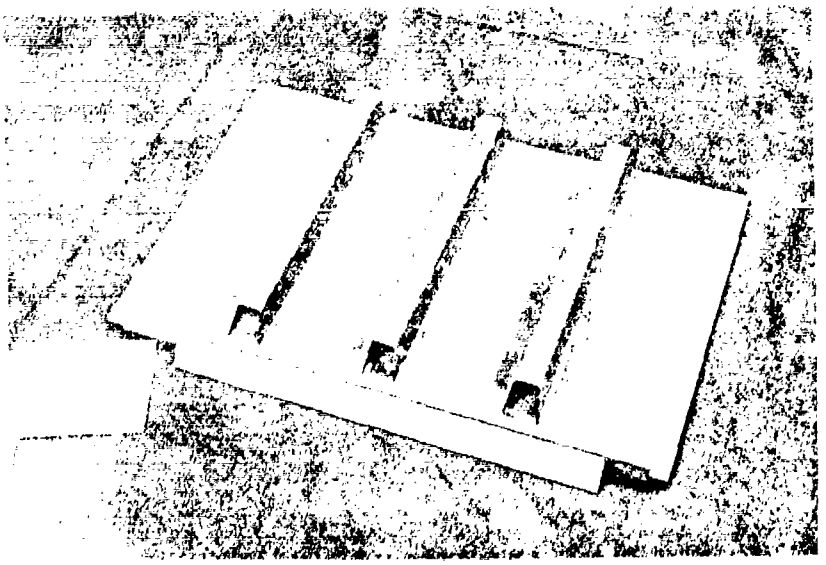


FIGURE 94
COMPLETED POLYIMIDE WING COMPRESSION PANEL

6.3.8 Compression Panel Test - The polyimide compression panel design drawings are shown in Section 4 of this report (Figures 20 and 21). Loading blocks were bonded to the panel for load introduction and slotted pipes were used on the edges to give a simple supported condition (Figure 95). The specimen was statically loaded in compression in 10% DLL increments. The panel failed prematurely at approximately 76% DLL with the hat stiffeners debonding from the panel.

(a) Panel Test Setup and Procedure - The test setup utilized a 400,000 lb. universal test machine. Machined knife edges were utilized to apply axial load to each panel. The test setup is shown in Figure 96. Strain gauges and linear motion transducers were bonded to each panel at the locations shown in Figure 97. Load was applied in 10% DLL increments until failure occurred. Strain and deflection data were recorded at each loading increment.

(b) Test Results - The panel was loaded in 10% DLL increments. While going from the 70 to 80 percent DLL increment, the panel failed with the stiffeners debonding from the panel. Figure 98 shows the compression panel after failure. At approximately 60 percent DLL visual inspection indicated that the middle stiffener was starting to debond and at 76 percent DLL all three stiffeners debonded. Strain gauges data (Figures 99 and 100) verify the visual observations. Specifically, strain gauge 17 on the stiffener cap indicated a loss in strain at 60 percent DLL as the load being applied to the panel was increasing. At the same time gauge 15 on the panel (stiffener side) indicated a loss in strain while gauge 16 on the opposite side of the panel (moldline side) showed an increase in strain. This indicates that the stiffeners were starting to unbond (the stiffener side of the panel was loosing stiffness), resulting in load being transfered to the moldline side of the panel. Deflection data is presented in Figure 101.

(c) Conclusions - Until the stiffeners started to debond the polyimide compression panel load distribution was similar to epoxy compression panels tested under the Composite Wing Program. Although NDT results showed debonds in the caps of the stiffeners, instrumentation and visual inspection indicated that the problem was in the polyimide adhesive bond line of the stiffener to panel.

Since NDT evaluation was not performed on the hat to panel bond there could have been debonds in this area. NDT standards had not been established previously for polyimide materials for this type bond. Another possible explanation for the adhesive failure is that because of the long post cure time the polyimide adhesives became brittle; therefore, there was not strain compatibility between the hat to adhesive to skin. Additional effort is needed to further develop the polyimide adhesives and fabrication techniques. Adhesive shear stress-strain curves are needed to obtain the adhesive characteristics as a function of post cure and appropriate fabrication techniques need to be established.

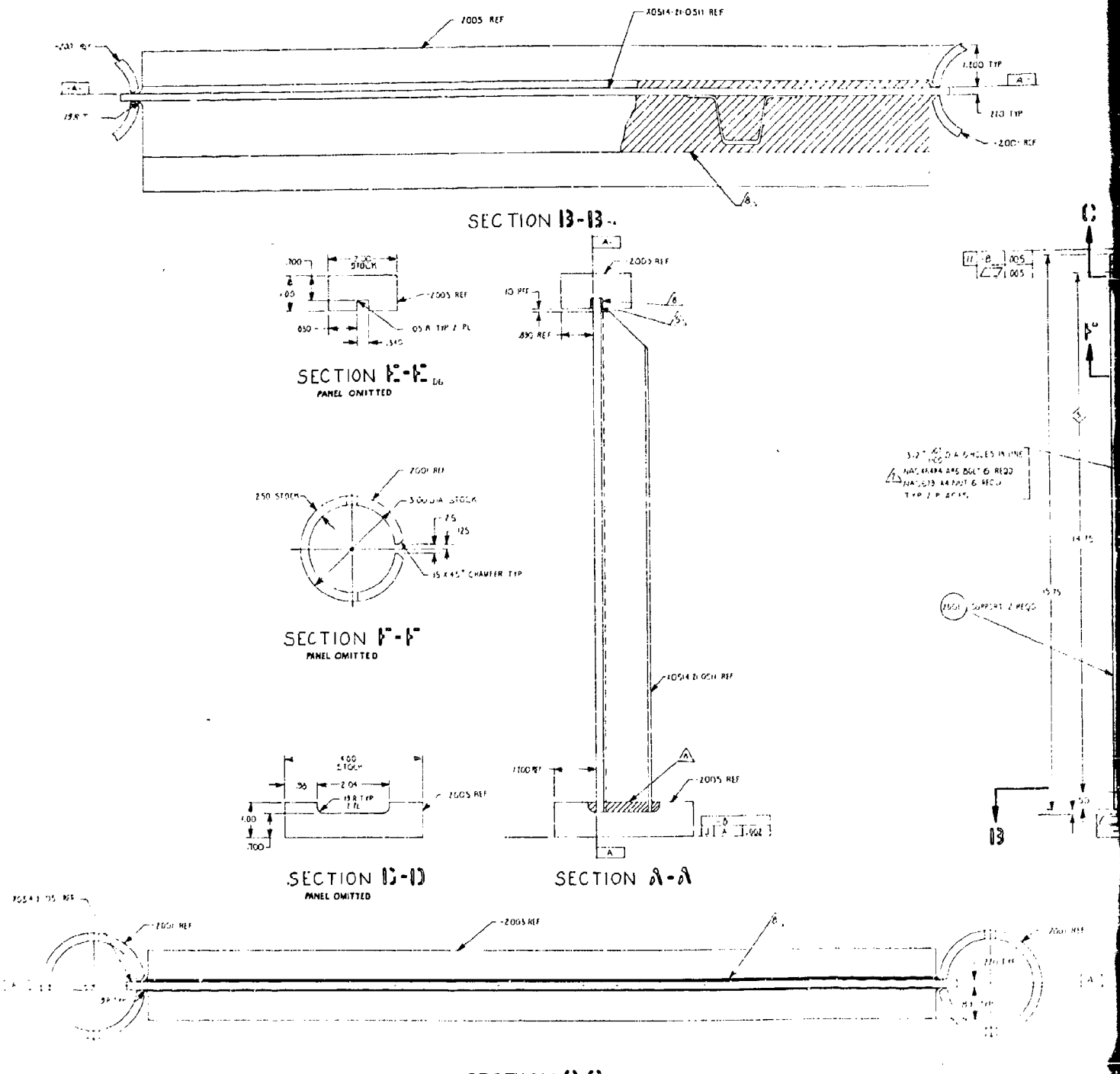
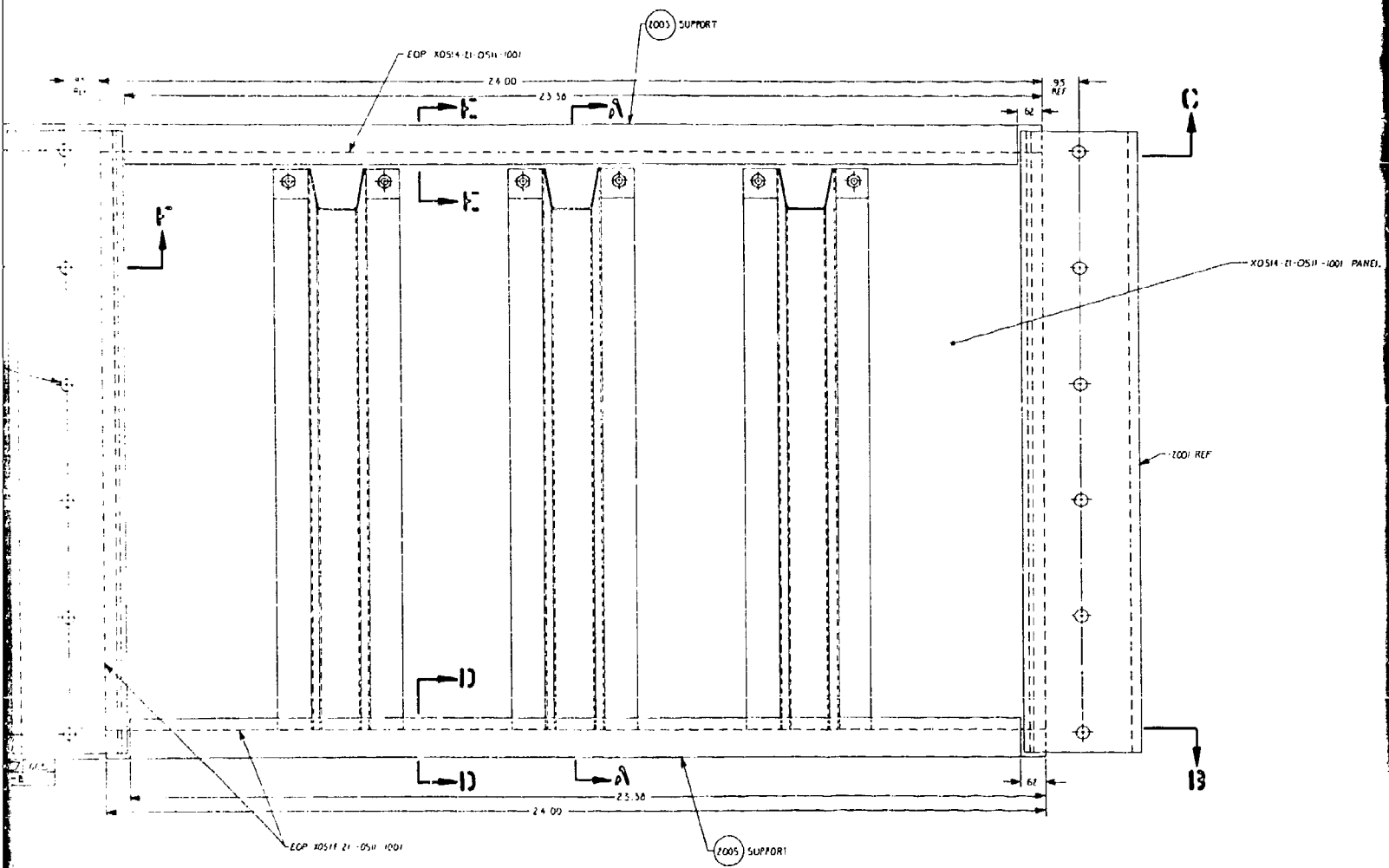


FIGURE 95

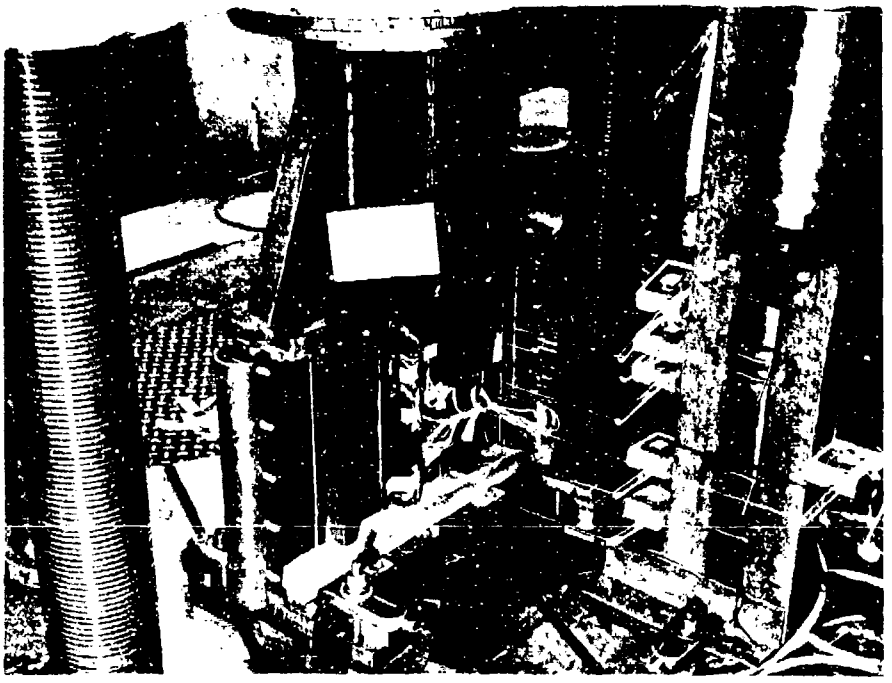
POLYIMIDE WING COMPRESSION TEST PANEL FIXTURE



-1001

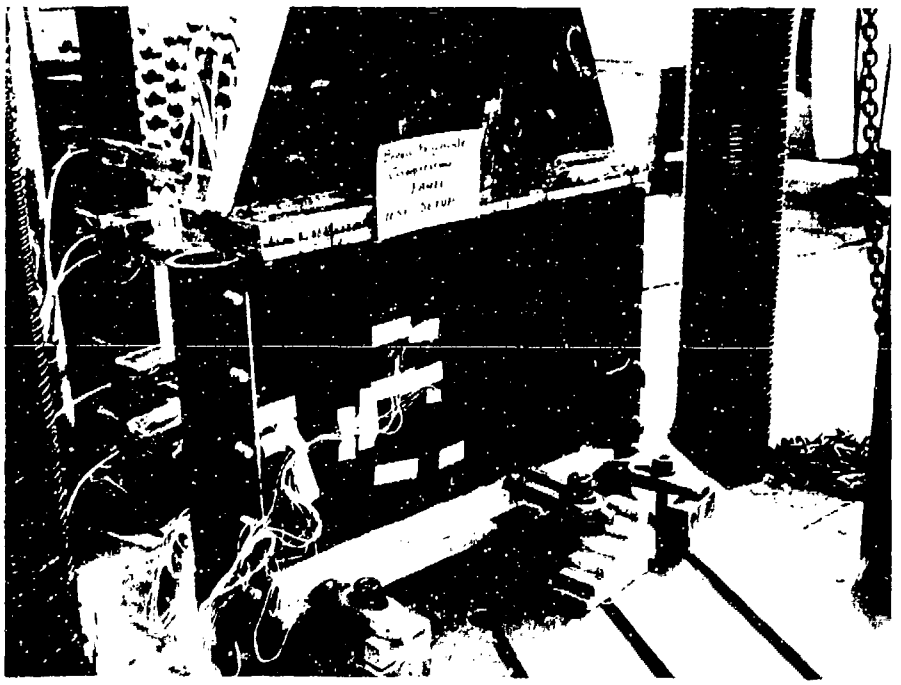
10	INSTALL FASTENERS PER PS 19000
9	MACHINE FINISH PER P.S 23041
8	POT WITH EPON 934 FILLED WITH ALUMINUM AND CURE PER P.S 11508 SHIMMING IS REQUIRED TO ASSURE THAT THE PANEL IS CENTERED IN THE MACHINED SLOT
7	INSTALLATION FASTENER
6	◄—◇—► INDICATES NUMBER OF EQUAL SPACES
5	REMOVE EXCESS POTTING FROM THIS AREA
4	LAST VIEW LETTER USED IS F
3	MARKING PER P.S 16001.
2	BREAK ALL SHARP EDGES PER P.S 23041
1	SURFACE ROUGHNESS 125 RHR OR BETTER.

NOTES :



Stiffened Side

GP72-0593-69



Moldline Surface Side

GP72-0593-70

FIGURE 96
POLYIMIDE WING COMPRESSION PANEL TEST SET-UP

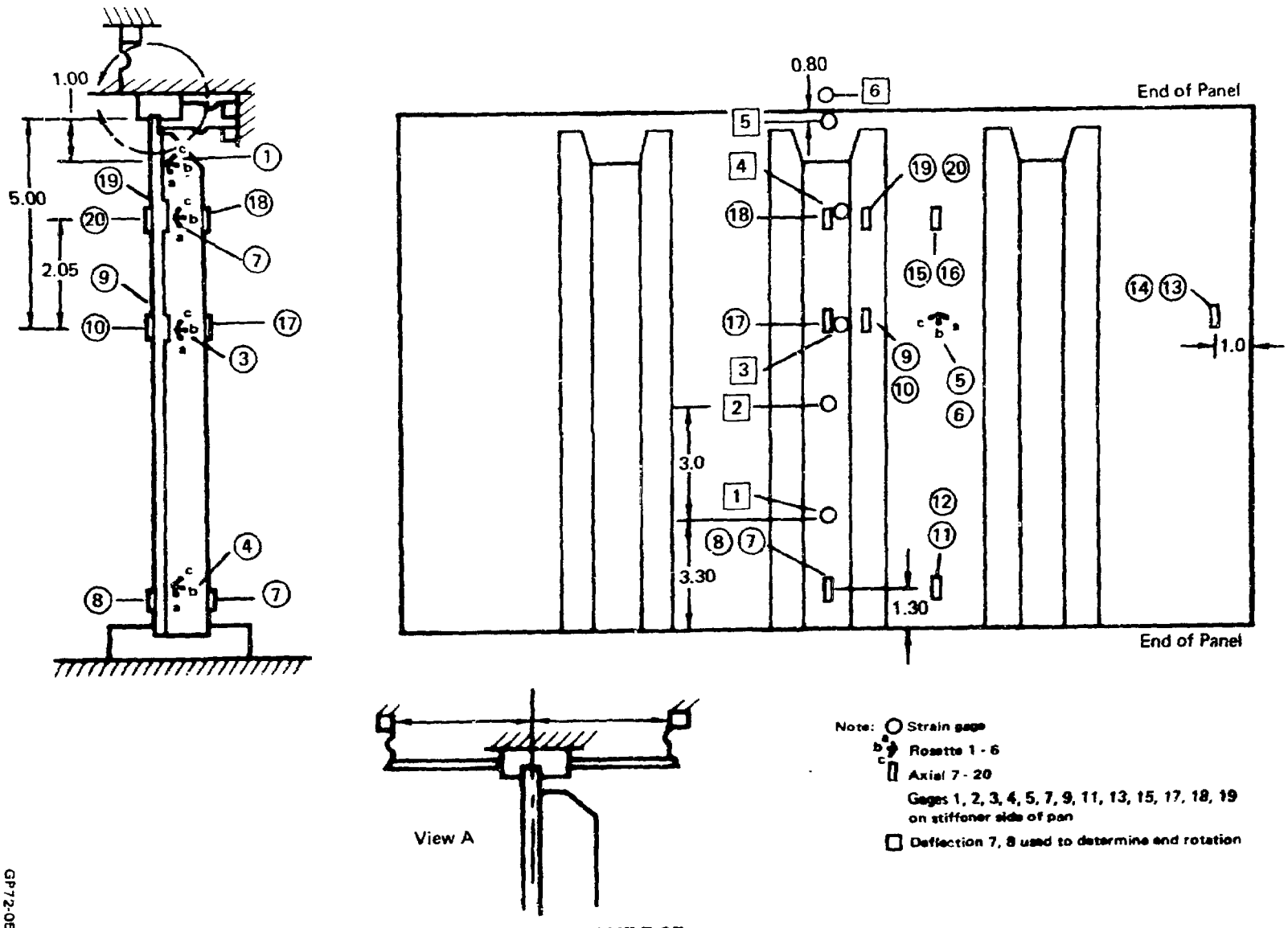
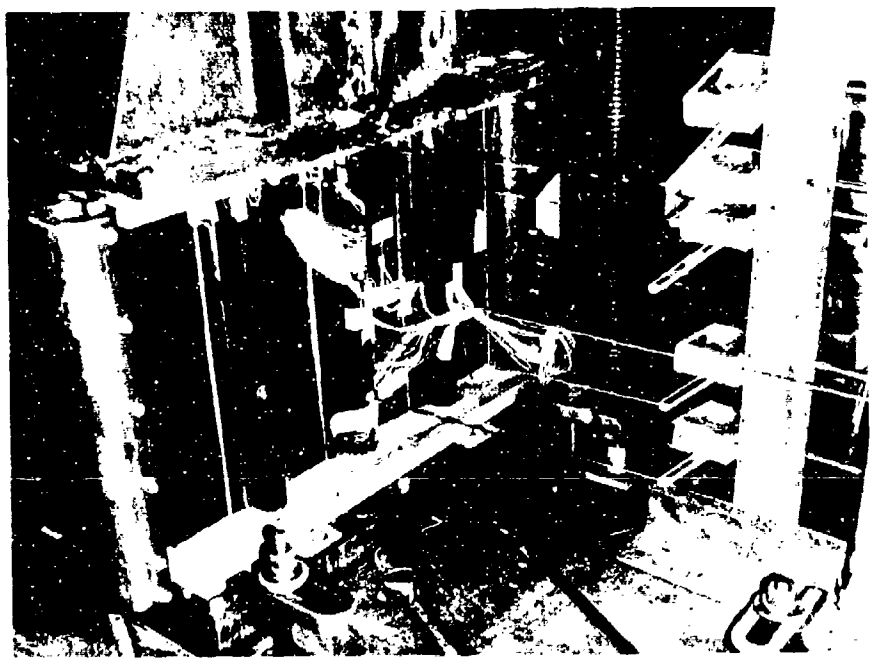


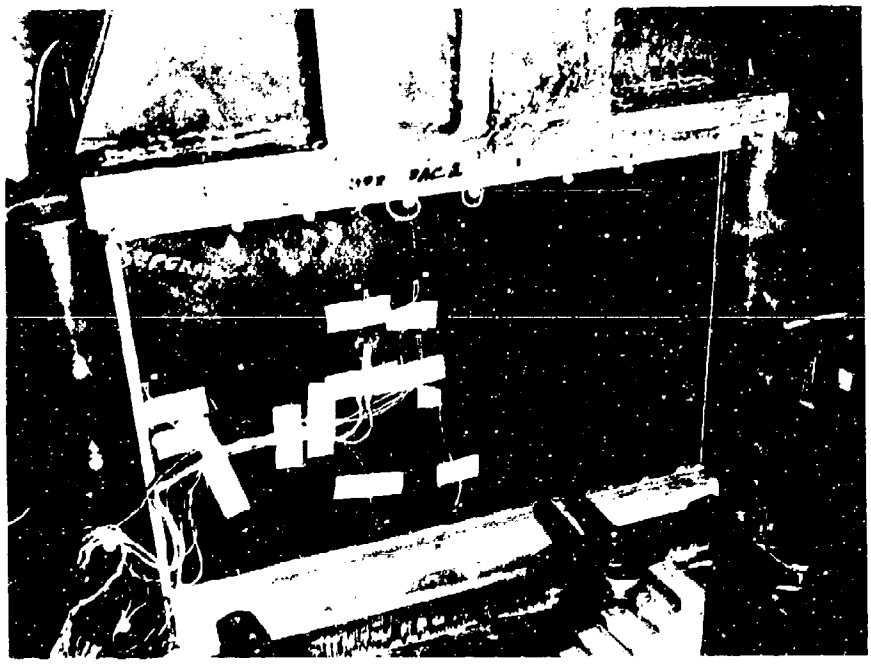
FIGURE 97
COMPRESSION PANEL DEFLECTION AND STRAIN GAGE LOCATION

GP72-0593-16



GP72-0593-67

Stiffened Side



Moldline Surface Side

GP72-0593-68

FIGURE 98
POLYIMIDE WING COMPRESSION PANEL AFTER FAILURE

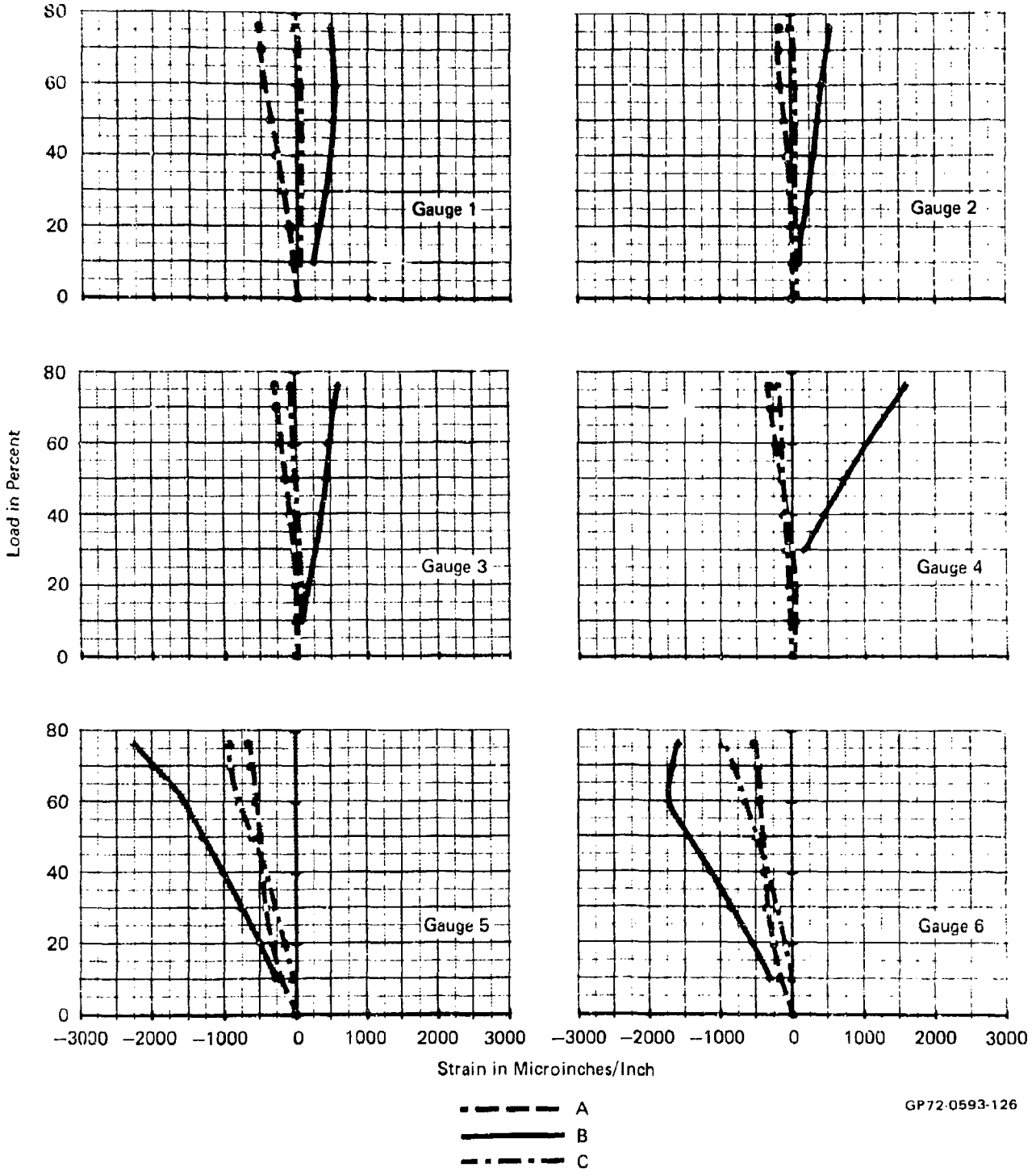


FIGURE 99
COMPRESSION PANEL STRAIN ROSETTES (GAUGES 1-6)

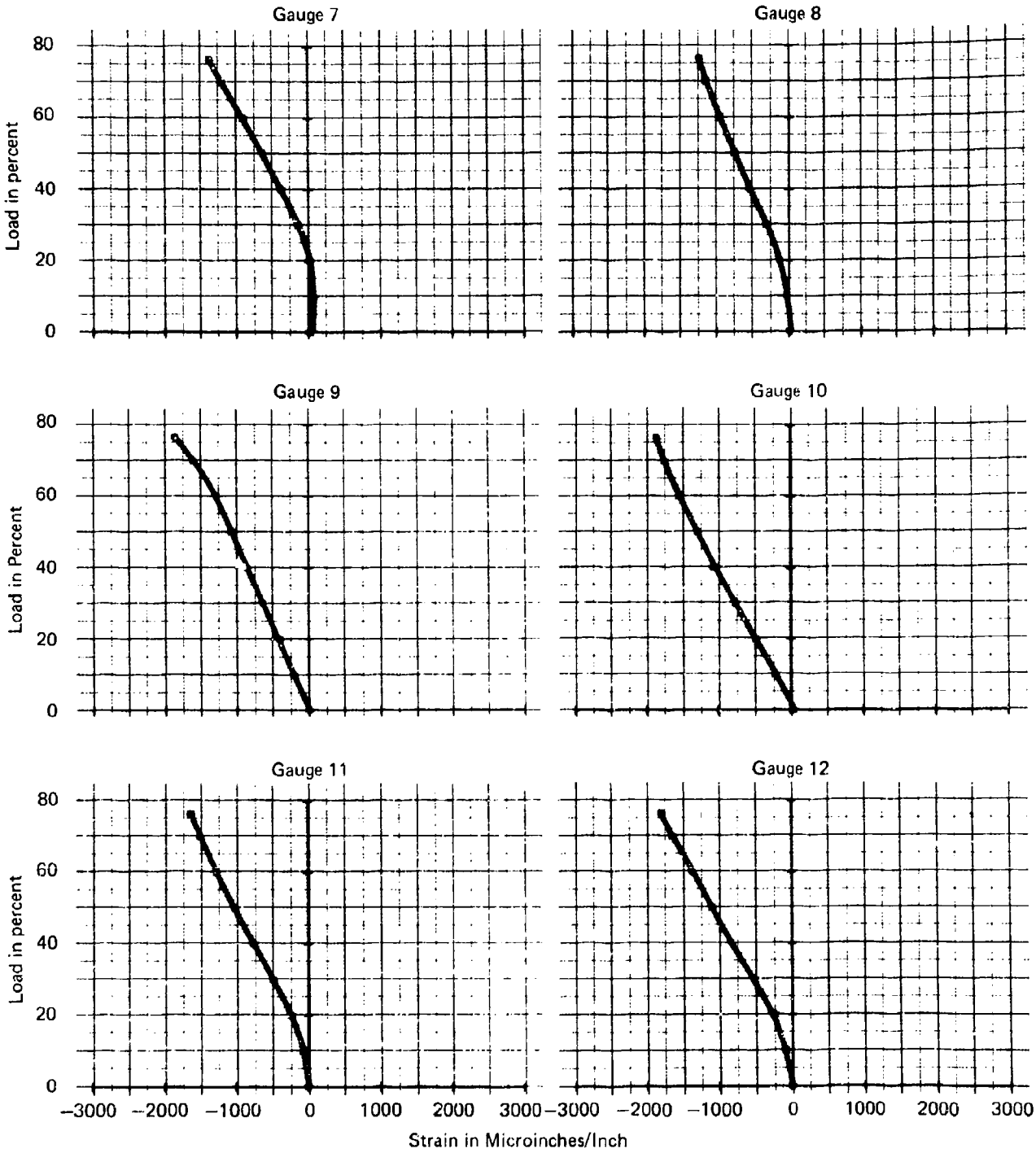


FIGURE 100
STRAIN DATA FOR POLYIMIDE COMPRESSION PANEL

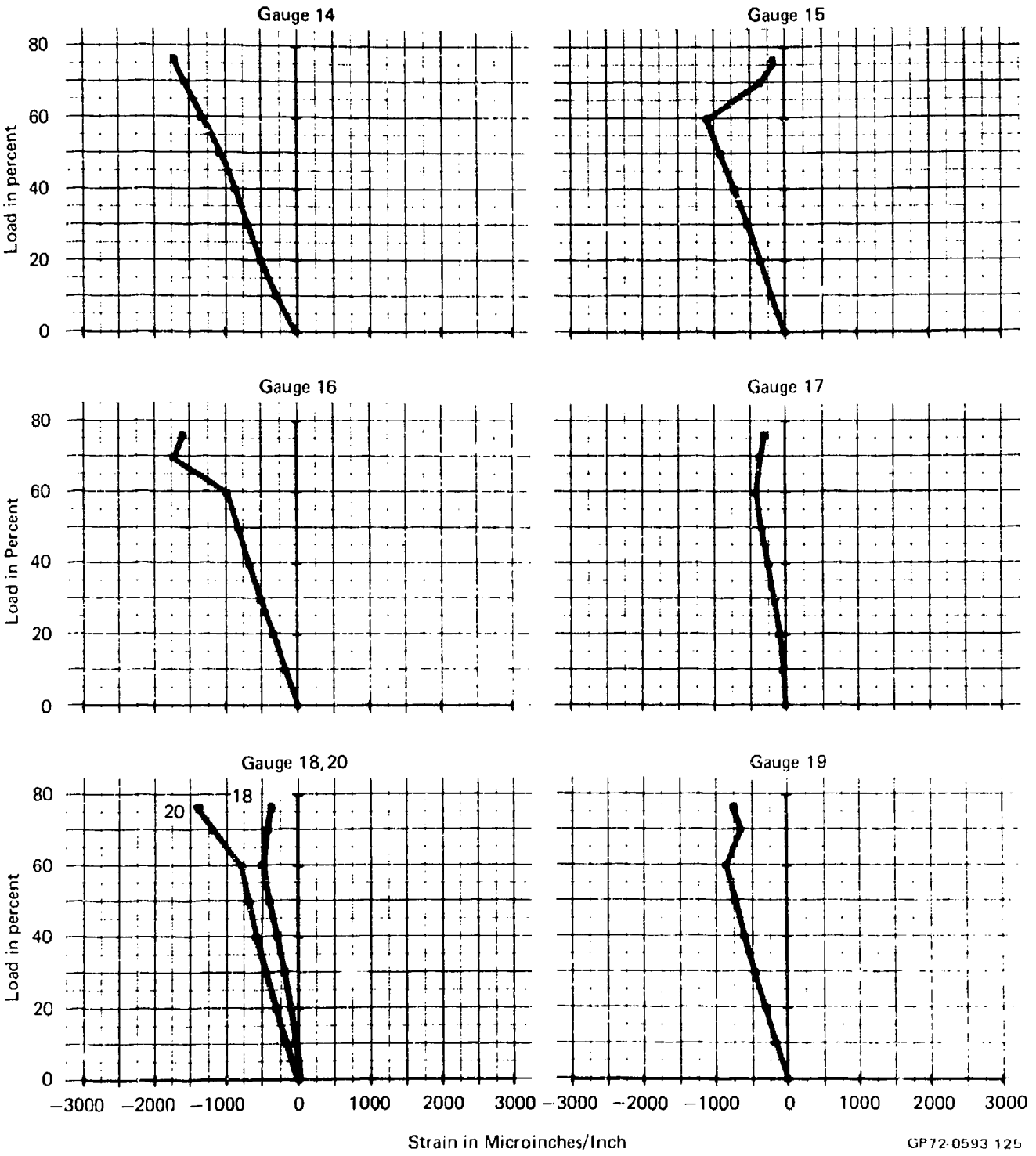
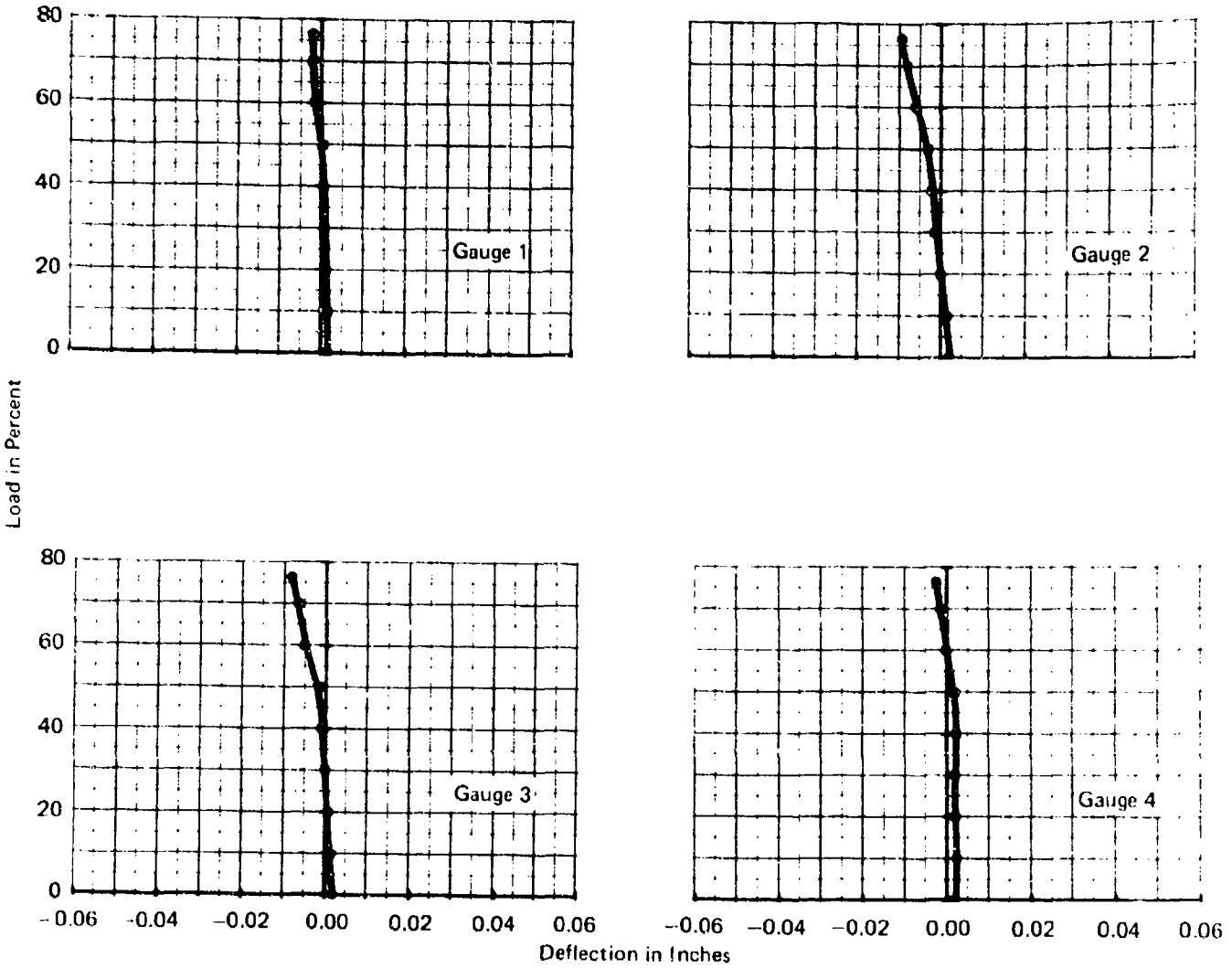


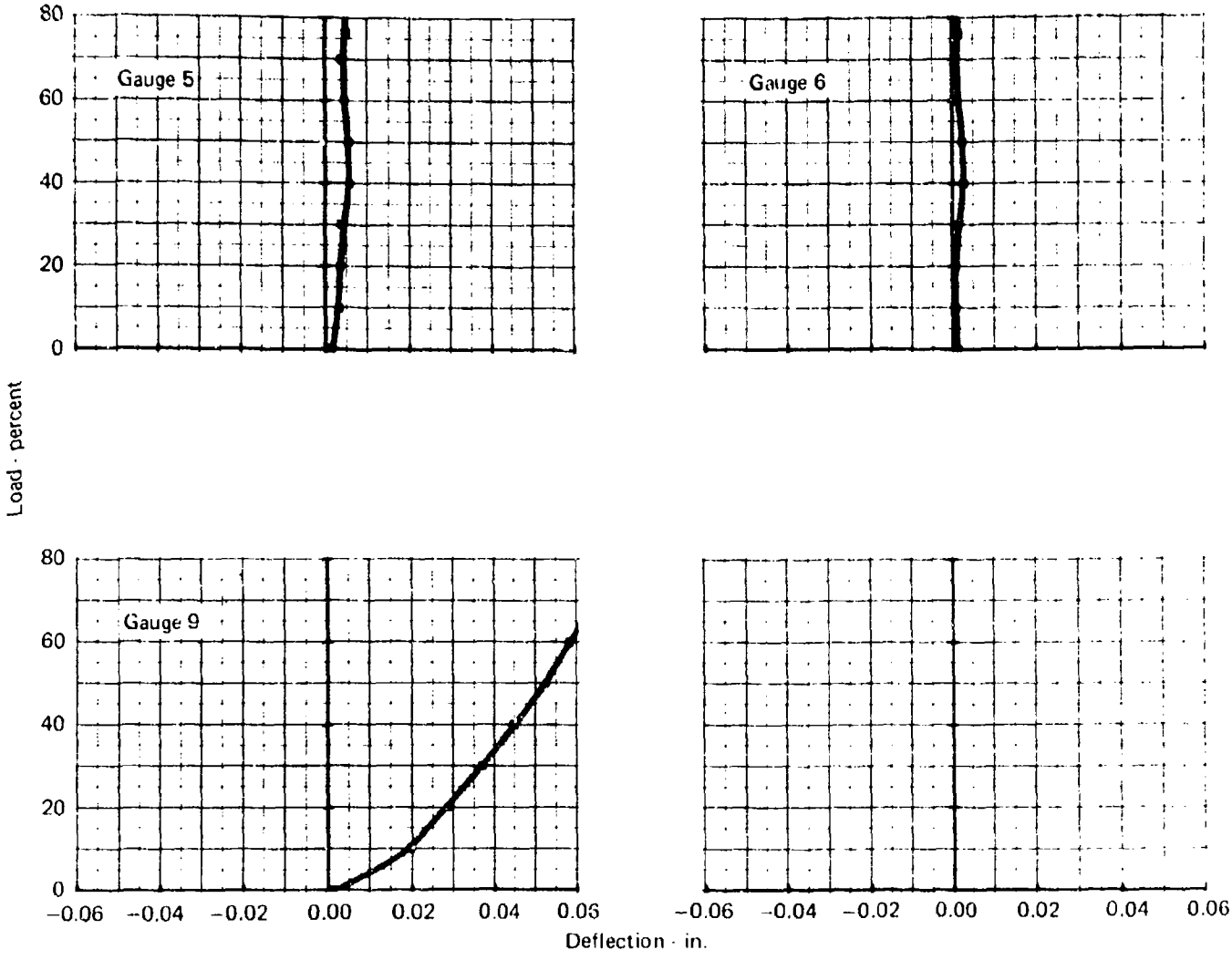
FIGURE 100 (Continued)
STRAIN DATA FOR POLYIMIDE COMPRESSION PANEL



Note: (1) Negative displacement is toward stiffener side of panel.

GP72 0593 124

FIGURE 101
COMPRESSION PANEL DEFLECTION GAUGES



Note: (1) Negative displacement is toward stiffener side of panel.
(2) Head Motion Defined Positive in Direction of Loading.

FIGURE 101 (Continued)
COMPRESSION PANEL DEFLECTION GAUGES

7. CONCLUSIONS AND RECOMMENDATIONS

The "High Temperature Advanced Composites" program has demonstrated that both boron/polyimide and graphite/polyimide structural parts can be successfully fabricated. The fabrication cycles for the graphite/polyimide matrix composites were shortened to near epoxy cycle times. The design allowables for tension, compression and shear properties are also very encouraging. NDT (ultransonics and radiography) proved useful in detecting defects and anomalies in polyimide structural elements.

The F-4 polyimide rudder test was successful as it attained loads of 400 percent design limit load without major failure. This test was similar to previous tests conducted on the MCAIR boron/epoxy rudders. For example, the A-32 boron/epoxy rudder had an initial failure at 246 percent design limit load. The rib adjacent to the lower hinge buckled and partially separated from the skin. This failure did not prevent successful continuation of the test to 400 percent design limit load. The successful fabrication and test of the polyimide rudder substantiated the structural integrity of boron and graphite polyimide composite materials for this type of structure.

However, problems still exist for fabricating complex highly loaded structures. The polyimide wing compression panel failed at 76 percent DLL with the hat stiffeners debonding from the panel. The reasons for the early failure were possible voids in the adhesive bond line, and strain incompatibility of the polyimide adhesive due to the post cure. Additional effort is needed to further develop the polyimide adhesives and fabrication techniques. Adhesive shear stress strain curves are needed to obtain the adhesive characteristics as a function of post cure and appropriate fabrication techniques need to be established. The material specification controls need to be further strengthened to insure the attainment of a reproducible product, batch after batch. Prepreg manufacturing variables and non-similar quality control tests are areas that must be closely controlled by the user. The technology must be extended to fabricate reproducible thick section (30 plies or greater) laminates and structure.

8. REFERENCES

1. Beeler, D. R. and Chase, V. A. "Advanced Polyimide Composites," Aeronautical and Space Engineering and Manufacturing Meeting, Society of Automotive Engineers, October 1968.

APPENDIX

PRELIMINARY MATERIALS AND PROCESS SPECIFICATIONS

The Appendix contains the material and process specifications required for the subject program. They consist of two MCAIR Material Specifications (MMS) and three MCAIR Process Specifications (P.S.) documents, and are listed below

- MMS-522, Boron/Polyimide Prepreg Material
- MMS-523, Graphite/Polyimide Prepreg Material
- P.S. 14224, Skins, Structural, Boron/Polyimide, Fabrication and Acceptance of
- P.S. 14225, Bonded Structure, Polyimide Matrix Composite, Fabrication and Inspection of
- P.S. 14227, Skins and Substructural Shapes, Structural, Graphite/Polyimide, Fabrication and Acceptance of

These are preliminary specifications and do not necessarily represent the final approved form of these documents. They have not been approved by any Government agency in their present form.

PRELIMINARY

MMS-522

BORON/POLYIMIDE PRE-PREG MATERIAL

1.0 APPLICATION

1.1 This specification establishes the requirements for "A" staged Polyimide resin impregnated Boron filament broadgood sheet containing a glass scrim cloth for ease of handling.

1.2 This pre-preg material is to be used for the fabrication of high performance composite structures over the approximate temperature range of -65°F to 550°F.

2.0 APPLICABLE DOCUMENTS

P.S. 14224 - Skins, Structural, Boron/Polyimide, Fabrication and Acceptance Of

MIL-B-83353, Boron Monofilament, Continuous, Vapor Deposited

3.0 REQUIREMENTS

3.1 CONSTITUENT MATERIALS

3.1.1 Boron Filament - The boron filament shall meet the requirements of MIL-B-83353 (USAF) Boron Monofilament, Continuous, Vapor Deposited.

3.1.2 Polyimide Resin - The polyimide resin shall be capable of structural application to 550°F and permit the pre-preg broadgoods and laminates produced from the broadgoods to meet the requirements of this specification.

3.1.3 Scrim Cloth - The glass scrim cloth shall consist of Type 104E and shall be continuous for the width of the pre-preg material.

3.1.4 Batch - A batch is defined as a quantity of boron polyimide broadgoods manufactured without interruption (normal work shift stoppages excepted) using polyimide resin from a single batch, boron filament from one or more lots, and without change of production equipment.

3.2 PHYSICAL PROPERTIES OF PRE-PREG MATERIAL - The "A" staged pre-preg material shall meet the physical property requirements of Table I.

3.3 PRE-PREG BROADGOODS - The length, width, and number of individual sheets of pre-preg shall be as specified on the purchase order.

3.4 FILAMENT SPACING - Spacing between adjacent filaments within the broadgoods sheet shall be 0.0002-0.015 inch, with the exception that two (2) gaps of 0.015-0.040 inch are permitted per 3.0 inch width provided all other provisions are met. The nominal filament spacing is 0.0008 inch.

3.5 BROKEN FILAMENTS - Broken filaments shall not be permitted within 1.0 inch of each other and no more than 5 per 10 feet of length.

3.6 CERTIFICATION - The vendor shall certify that his product meets the requirements of this specification or take specific exception in the purchase order. The vendor, by his certification, guarantees that the polyimide resin formulation is the same as that used in the initial qualification tests.

3.7 MECHANICAL PROPERTY OF CURED LAMINATES - The "A" staged pre-preg, when "B" staged and fabricated into a 6, 8, and 15 ply undirectional laminate per P.S. 14224, shall be capable of meeting the mechanical property requirements of Table II. Failure to meet any of the values of Table II permits retest for that property only. After the second test, the provisions of Paragraph 3.11 are in effect.

3.8 UNIFORMITY AND ORIENTATION - Filaments in the broadgoods sheet shall be oriented parallel to each other with the following exceptions. Single filament crossovers are acceptable. The width of broadgoods affected by one or more filament crossover groups (one group is two or more adjacent crossed filaments) shall be less than 0.40 inch. Single crossover groups between 0.20 and 0.40 inch wide shall not exceed 3.0 inches in length and are to be separated from each other by at least 5 feet.

3.9 PRE-PREG RELEASE SHEETS - The release sheet backing used on one side of the "A" staged boron/polyimide pre-preg material shall be Teflon coated glass fabric (CHR3TLL or equivalent) and on the other side the release material shall be a backing sheet which will easily separate from the pre-preg material without resin loss from the pre-preg.

3.10 DEFECTS DURING USAGE - Defects as defined by this specification found in the pre-preg material after acceptance has been completed are still cause for rejection of the unused portion of the batch. Defects caused by user mishandling, improper storage, or expiration of shelf life are not vendor responsibility.

3.11 PRE-PREG FLATNESS - Boron/polyimide broadgoods shall not: (1) curl over more than 5% of their width, (2) contain filament bundles, and (3) have separation of scrim cloth from the remainder of the pre-preg.

3.12 REJECTION - Boron/polyimide pre-preg broadgoods not conforming to the requirements of this specification shall be rejected.

4.0 QUALITY ASSURANCE

4.1 VENDOR BATCH CERTIFICATION TESTS - The vendor shall supply certified quantitative data for the following tests with each batch of broadgoods. In addition, the vendor shall supply data on the ultimate tensile strength, tensile modulus, and diameter of the boron filaments used in the manufacture of the broadgoods. The vendor shall send three (3) copies of the test data to the McDonnell Buyer. The Buyer in turn shall send two of these copies to Quality Assurance and the remaining copy to Material and Process Development Department. The test methods are described in Paragraph 4.3. The required tests are:

4.1.1 Resin Solids content of "A" staged resin prior to pre-pregging.

4.1.2 "A" stage pre-preg resin solids content.

4.1.3 Total volatile content of resin in "A" staged pre-preg.

4.1.4 Infrared spectrogram analysis of "A" staged resin.

4.2 MCDONNELL INCOMING INSPECTION TESTS - The following tests shall be performed at McDonnell for each incoming batch of broadgoods. The test methods are described in Paragraph 4.3. Perform these tests for each batch or 2500 ft² of prepreg, whichever is smaller.

4.2.1 "A" stage pre-preg resin solids content.

4.2.2 Total volatile content of resin in "A" staged pre-preg.

4.2.3 Infrared spectrogram analysis of "A" staged resin.

4.2.4 Horizontal shear strength at R.T. and 550°F.

4.2.5 Longitudinal Flexural Strength and Modulus at R.T. and 550°F.

4.2.6 Longitudinal Tensile Strength at R.T.

4.2.7 Transverse Tensile Strength at R.T.

4.3 TEST METHODS

4.3.1 Resin Solids Content of "A" Staged Resin (Prior to Pre-pregging)

4.3.1.1 Weigh a dried, tared (to nearest 0.0001 g) fritted crucible (W_1) and place the resin sample into it.

4.3.1.2 Weigh the crucible and resin to nearest 0.0001 g (W_2).

4.3.1.3 Heat the crucible and resin to 600°F at 1-3°F/min. and hold for 1 hour.

4.3.1.4 Allow the crucible and resin solids to cool to room temperature in a desiccator and weigh the crucible and resin solids to nearest 0.0001 g (W_3).

4.3.1.5 Calculate resin solids content as follows:

$$\% \text{ Resin solids content} = \frac{(W_3 - W_1)(100\%)}{W_2 - W_1}$$

4.3.1.6 Report the arithmetic mean of at least two specimens.

4.3.2 "A" Stage Prepreg Resin Solids and Resin Volatile Content

4.3.2.1 Take duplicate samples of boron/polyimide prepreg from the same sheet. Sample size is 2.0 in. x 2.0 in. Weigh one cleaned and dried 150 ml. Pyrex beaker to the nearest 0.0001 g and record weight as W_1.

4.3.2.2 Place a sample of prepreg in the beaker and weigh to the neatest 0.0001 g. Record as W_2.

4.3.2.3 Heat the sample to 600°F at a rate of 1-2°F/min. and hold for 1 hour. Cool to room temperature in a desiccator. Weigh the sample to the nearest 0.0001 g and record as W_3.

4.3.2.4 Weigh a cleaned and dried 150 ml. Pyrex beaker to the nearest 0.0001 g and record as W_4. Take the duplicate sample, place it in the beaker and weigh to the nearest 0.0001 g. Record as W_5.

4.3.2.5 Extract the polyimide resin from the prepreg with n, methyl-pyrrolidone (NMP) solvent at 100-130°F for 1 hour. Weigh another 150 ml. Pyrex beaker to the nearest 0.0001 g and record as W_6. Transfer the resin-NMP solution to this beaker, weight to the nearest 0.0001 g and record as W_7. Weigh the beaker with fibers remaining to the nearest 0.0001 g and record as W_8.

4.3.2.6 Boil the NMP off the resin-NMP solution by heating to 600°F at 1-2°F/min. and holding for 1 hour. Cool to room temperature in a desiccator. Weigh the remaining solids and record as W_9.

4.3.2.7 Calculate the prepreg resin solids as follows:

$$\text{Prepreg Resin Solids Content } (\%) = \frac{W_2 - W_4}{W_5 - W_4} (100)$$

$$\text{Resin Total Volatile Content } (\%) = \frac{W_2 - W_3 \quad (100)}{(W_2 - W_1) - (W_5 - W_5)}$$

Report the arithmetic mean of at least 3 specimens.

4.3.3 Filament Count - The filament count shall be established by photographing a 1.0-2.0 inch wide section of broadgoods, enlarging to 100X and counting the filaments/inch. The photograph method may involve radiography.

4.3.4 Infrared Analysis

4.3.4.1 Place the "A" staged pre-preg resin on a potassium bromide cell plate.

4.3.4.2 Place the potassium bromide plate in a Beckman IR9 Infrared Spectrophotometer or equivalent instrument and conduct the spectrum scan in direct transmission. Set the Beckman IR9 instrument as follows:

(a) Scanning Rate - 80 cm^{-1}/min.
(b) Fine gain control setting - 3
(c) Coarse gain control setting - 10
(d) Routine slit control
(e) Period setting - 2
(f) Resolution - 25 cm^{-1}

4.3.5 Horizontal Shear Strength

4.3.5.1 The specimen configuration and load support method shall be as specified in Figure I.

4.3.5.2 The specimen shall be loaded with 0.125 inch radius supports at 0.05 inch/min.

4.3.5.3 Calculate the horizontal shear strength as follows:

$$F_s = \frac{0.75P}{B(t)}$$, where F_s = Horizontal Shear Strength, lb/in^2
P = Load, lb.
B = Specimen Width, in.
t = Laminate thickness, in.

4.3.5.4 Report the arithmetic mean of at least three values.

4.3.6 Longitudinal Flexural Strength and Modulus

4.3.6.1 The specimen configuration and load support method shall be as shown in Figure II.

4.3.6.2 The specimen shall be loaded with 0.125 in. radius supports at a rate of 0.05 in./min.

4.3.6.3 Place a deflectrometer capable of measuring deflection to 0.001 inch under the span midpoint.

4.3.6.4 While the specimen is loaded record the deflection and load continuously. Remove the deflectometer prior to failure. Record the load at failure.

4.3.6.5 Calculate the longitudinal flexural strength as follows:

$$F_t = \frac{1.5\ PL}{B(t)^2}$$, where F_t = Flexural Strength, lb/in^2
P = Load, lb.
L = Test Span, in.
B = Specimen Width, in.
t = Specimen Thickness, in.

4.3.6.6 Calculate the longitudinal flexural modulus as follows:

$$E = \frac{(P_1 - P_2)(L)^3}{4B(t)^3(Y_1 - Y_2)}$$, where E = Modulus, lb/in^2
Y = Deflection, in.

4.3.6.7 Deflection data (Y_1, Y_2) are taken at the respective load (P_1, P_2) data points. Subscript 1 data is numerically larger than subscript 2 data. Do not take P or Y data near either end of the curve. Report the arithmetic mean of at least 3 values.

4.3.7 Longitudinal and Transverse Tension Properties

4.3.7.1 The specimen configuration shall be as shown in Figure III.

4.3.7.2 Filaments shall be parallel to the beam length for longitudinal tests and perpendicular to the beam length for transverse tests. The tolerance on alignment is ± 1°.

4.3.7.4 Calculate the tensile strength as follows:

$$F_t = \frac{PA}{2Bt\left[C + \frac{T + t}{2}\right]}$$, where F_t = Tensile Strength, lb/in^2
P = Load, lb.
B = Specimen Width, in.
T = Opposite Skin Thickness, in.
C = Honeycomb Core Thickness, in.
A = Moment Arm, in.

4.3.7.5 Report the arithmetic mean of at least 3 values.

5.0 PREPARATION FOR DELIVERY

5.1 MARKING OF CONTAINER - Each container shall be permanently marked with the following information:

5.1.1 Title, number and revision letter of this specification.

5.1.2 Vendor's name and address.

5.1.3 Date of impregnation.

5.1.4 Size and number of sheets of broadgoods material.

5.1.5 Resin manufacturer's batch number and trade name.

5.1.6 Vendor's pre-preg lot/batch number and trade name.

5.1.7 CAUTION: Ship and store at or below $0^{o}F$.

5.2 MARKING OF PLASTIC SHEET CONTAINERS - Each plastic bag shall be permanently marked with the vendor's lot/batch number, sheet number and trade name.

5.3 PACKAGING OF CONTAINER

5.3.1 Package each sheet of pre-preg material in individual sealed moisture proof plastic bags.

5.3.2 Place silica gel (or equivalent desiccant) inside each sealed plastic bag.

5.3.3 The sealed plastic bags are to be packed in an insulated shipping container that will be accepted for safe transportation by common carriers. Construct the containers so that sufficient room is available for the dry ice necessary to maintain the temperature inside the container at or below $0^{o}F$ for a minimum of three days.

5.5 SHIPPING - The container containing the pre-preg boron/polyimide broadgoods shall be shipped by air express from the vendor to McDonnell. Upon receipt of the container at McDonnell, it shall be opened to ascertain that solid dry ice remains in the container.

5.6 STORAGE AT MCDONNELL

5.6.1 Immediately upon receipt of the material at McDonnell, the pre-preg material shall be forwarded to the using department, removed from the shipping container, and placed in $0^{o}F$ storage.

5.6.2 The material information on the shipping container (see Paragraph 5.1) shall be recorded with the date of arrival included.

6.0 NOTES

Not applicable.

7.0 APPROVED PRODUCTS

Products which have been qualified under the requirements of this specification are listed below. Revision of the list will be made as necessary. The listing of a product does not release the supplier from compliance with the specification requirements. Reproduction or reference to the list for advertising purposes is expressly forbidden.

MCAIR DESIGNATION	SUPPLIERS DESIGNATION	TAPE	BROAD-GOODS	SUPPLIERS NAME & ADDRESS
MMS-522	Boron/Skybond 703 Polyimide Resin		X	Whittaker Research and Development 3540 Aero Court San Diego, California

NOTE: The accuracy of this method depends upon the care in handling the sample and the equipment. It is also suggested that the micro traps be connected with glass tubing and vinyl tubing prior to weighing in order to use them as a single unit. The traps should be connected with the first trap approximately 1 inch higher than the second.

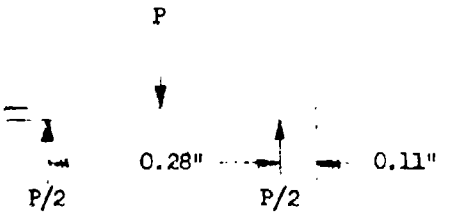


FIGURE I - HORIZONTAL SHEAR TEST

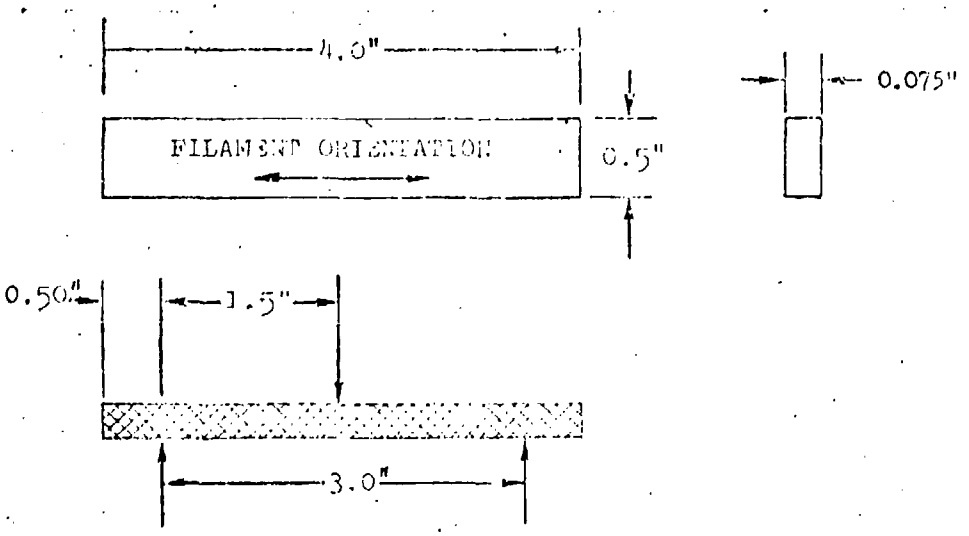
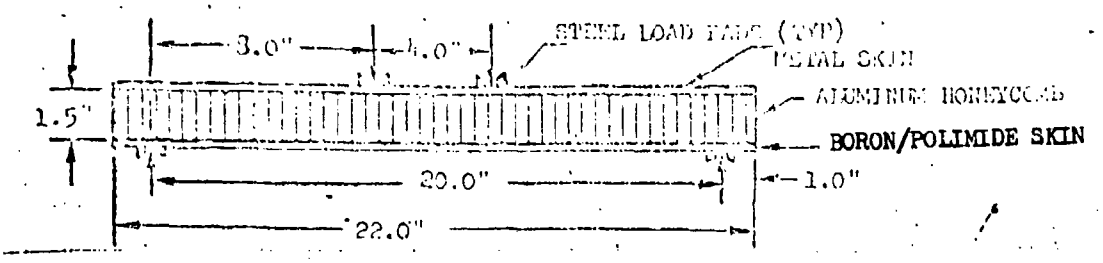


FIGURE II - LONGITUDINAL FLEXURE TEST



NOTES:

(1) For 0° tension and compression tests metal skin is 1 x 22 x 0.090 inch thick (minimum) 6Al-4V annealed titanium. Aluminum honeycomb is 1.5 inch thick 5052-H39 non-perforated, 1/8 inch cell - 0.003 inch foil gage (minimum).

(2) For 8 ply thick 90° tension tests: Metal skin is 1 x 22 x 0.063 inch thick 2024-T62 aluminum. Aluminum honeycomb is 1.5 inch thick 5052-H39 non-perforated, 3/16 inch cell - 0.0015 inch foil gage (minimum).

(3) Boron/Polyimide skin is approximately 1 inch wide x 22 inch x 6 ply thick for the 0° tension specimens and 1.00 inch wide x 22 inch x 8 ply thick for the 90° tension test specimens.

(4) The honeycomb core width shall be a minimum of one cell size (3/16" or 1/8" as applicable) wider on each side than the boron/polyimide skin and the ribbon direction parallel to the 22 inch direction.

(5) Bond specimens with FM-400 per P.S. 14081 except curing pressure may be 85 ± 5 psig.

(6) Measure and record the metal skin thickness, honeycomb core height, and the boron/polyimide skin width. Make measurements in the 4.0 inch center span.

(7) The steel load pads must have length sufficient to overhang the specimen edges, a minimum width of 0.75 inch, and a minimum radius of 0.125 inch at edges where they contact the specimen surface.

FIGURE III

LONGITUDINAL (0°) AND TRANSVERSE (90°) TENSION TESTS

PROPERTY	TEST METHOD PARAGRAPH	REQUIREMENT
Resin Solids Content of "A" Staged Resin	4.3.1	For Information Only
Pre-Preg Resin Solids Content	4.3.2	25-28% by weight
Total Volatile Content of Resin in "A" Staged Pre-Preg	4.3.2	40-43% by weight
Filament Count	4.3.3	208-216 filaments/inch
Infrared Analysis	4.3.4	For Information Only

TABLE I - PHYSICAL PROPERTIES OF

BORON/POLYIMIDE "A" STAGED PRE-PREG

MECHANICAL PROPERTY	TEMPERATURE (°F)	MINIMUM AVERAGE (PSI)	TEST METHOD PARAGRAPH
Longitudinal Flexural Strength, psi 15 Plies Thick	R.T. 550	190,000 155,000	4.3.6
Longitudinal Flexural Modulus, psi 15 Plies Thick	R.T. 550	26×10^6 21×10^6	4.3.6
Horizontal Shear Strength, psi 15 Plies Thick	R.T. 550	11,000 6,000	4.3.5
Longitudinal Tensile Strength, psi 6 Plies Thick	R.T.	*	4.5.7
Transverse Tensile Strength, psi 8 Plies Thick	R.T.	*	4.5.7

* Values to be negotiated

TABLE II - MECHANICAL PROPERTY REQUIREMENTS OF CURED LAMINATES

PREPARED Ron Kellenberger APPROVED _____
Engineering

APPROVED _____
AFPRO, Engineering

ISSUED 2 June 1971 REVISION A	BORON/POLYIMIDE PRE-PREG MATERIAL	MMS-522 PAGE 11 OF 11
	STANDARD MATERIAL SPECIFICATION	CODE IDENT.NO. 76301

MAC 2989C (2 JUL 70)

MCDONNELL DOUGLAS
ST. LOUIS

PRELIMINARY

MMS-523

GRAPHITE/POLYIMIDE PRE-PREG MATERIAL

1.0 APPLICATION

1.1 This specification establishes the requirements for "B" staged Polyimide resin impregnated Type II graphite fiber broadgood sheet.

1.2 This pre-preg material is to be used for the fabrication of high performance composite structures over the approximate temperature range of -65°F to 550°F.

2.0 APPLICABLE DOCUMENTS

P.S. 14227 - Skins and Substructural Shapes, Structural, Graphite/Polyimide, Fabrication and Acceptance of

3.0 REQUIREMENTS

3.1 CONSTITUENT MATERIALS

3.1.1 Graphite Fiber - The graphite fiber shall be a high strength, intermediate modulus type and shall have a minimum tensile strength of 350,000 psi, a minimum tensile modulus of 35×10^6 psi, no fiber twist, and a nominal fiber/tow count of 10,000. Tow splices are not permitted. Any sizing or surface finish used shall not be changed without written approval from MCAIR.

3.1.2 Polyimide Resin - The polyimide resin shall be capable of structural application to 550°F and permit the pre-preg broadgoods and laminates produced from the broadgoods to meet the requirements of this specification.

3.1.3 Batch - A batch is defined as a quantity of graphite/polyimide broadgoods manufactured without interruption (normal work shift stoppages excepted) using polyimide resin from a single batch, graphite fiber/tow from one or more lots, and without change of production equipment.

3.2 PHYSICAL PROPERTIES OF PRE-PREG MATERIAL - The uncured pre-preg material shall meet the physical property requirements of Table I.

3.3 BROADGOODS - The length, width, and number of individual pieces of pre-preg shall be as specified on the purchase order. Defect areas in any individual piece of broadgoods shall be identified by the vendor.

3.4 GAPS - The maximum allowable gap or distance between adjacent tows shall be 0.10 inches, as determined with a non-contact measurement apparatus. More than one gap of 0.075 - 0.10 inches per linear foot, in any one sheet, shall not be acceptable.

3.5 SPLICES - Tow splices are not permitted.

3.6 ALIGNMENT - The pre-preg shall meet both of the following alignment requirements:

3.6.1 The lay of the tow within the sheet shall not deviate from a straight line by more than 1/32 inch in a linear foot.

3.6.2 The edge of the sheet shall not deviate from a straight line by more than 0.010 inch per foot of length.

3.7 PRE-PREG RELEASE PAPER - The release paper backing used on the pre-preg broadgoods shall be made of a material that permits separation of the broadgoods from the paper without resin loss from the pre-preg, and shall not permit the broadgoods to shift or change position during shipment or storage.

3.8 PRE-PREG FLATNESS - The pre-preg broadgoods shall not curl over more than 5% of its width.

3.9 MECHANICAL PROPERTIES OF CURED LAMINATES - The mechanical property requirements of Table II shall be met when testing 4, 6 and 11 ply unidirectional laminates fabricated per F.S. 14227.

3.10 CERTIFICATION - The vendor shall certify that his product meets the requirements of this specification or take specific exception in the purchase order. The vendor, by his certification, guarantees that the polyimide resin formulation is the same as that used in the initial qualification tests.

3.11 DEFECTS DURING USAGE - Defects as defined by this specification found in the pre-preg material after acceptance has been completed are still cause for rejection of the unused portion of the batch. Defects caused by user mishandling, improper storage, or expiration of shelf life are not vendor responsibility.

3.12 WORKING LIFE - The prepreg broadgoods shall be capable of passing the resin tack test (Para. 4.4.6) and meeting the mechanical property requirements of Table II after 240 hours at 65 to 75°F.

3.13 REJECTION - Graphite/polyimide broadgoods not conforming to the requirements of this specification shall be rejected.

4.0 QUALITY ASSURANCE

4.1 VENDOR BATCH CERTIFICATION TESTS - The vendor shall supply certified quantitative data for the following tests with each batch of broadgoods. In addition, the vendor shall supply data on the ultimate tensile strength, tensile modulus, and diameter of the graphite filaments used in the manufacture of the broadgoods. The vendor shall send three (3) copies of the test data to the McDonnell Buyer. The Buyer in turn shall send two of these copies to Quality Assurance and the remaining copy to Material and Process Development Department. The test methods are described in Paragraph 4.4. The required tests are;

4.1.1 Longitudinal flexural strength and modulus at R.T. and 550°F.

4.1.2 Horizontal shear strength at R.T. and 550°F.

4.1.3 Pre-preg resin solids content.

4.1.4 Total volatile content of resin in pre-preg.

4.1.5 Infrared analysis.

4.1.6 Resin Tack

4.1.7 Drapability

4.2 MCDONNELL INCOMING INSPECTION TESTS - The following tests shall be performed at McDonnell for each incoming shipment of broadgoods. This includes different shipments of the same batch of broadgoods. The test methods are described in Paragraph 4.4. Perform these tests for each batch or 2500 ft^2 of prepreg, whichever is smaller.

4.2.1 Longitudinal flexural strength and modulus at R.T. and 550°F.

4.2.2 Horizontal shear strength at R.T. and 550°F.

4.2.3 Longitudinal tensile strength at R.T.

4.2.4 Transverse tensile strength at R.T.

4.2.5 Pre-preg resin solids content.

4.2.6 Total volatile content of resin in pre-preg.

4.2.7 Infrared analysis of "B" staged resin.

4.2.8 McDonnell reserves the right to perform any additional tests of Paragraph 3.0 as a further means of determining the acceptability of incoming material.

4.3 RETEST PROVISIONS

4.3.1 A batch of broadgoods which fails two (2) or more of the specified tests for vendor certification or McDonnell incoming inspection shall be rejected. The rejected batch of broadgoods, however, may be reworked, retested and resubmitted.

4.3.2 A batch of broadgoods which fails only one of the specified tests for vendor certification or McDonnell incoming inspection may be retested for the failed test only. If the retest fails, then the provisions of Paragraph 3.12 are in effect. If the retest is acceptable, then the broadgoods batch is acceptable.

4.4 TEST METHODS - Specimens for destructive tests are to be machined using diamond tools only. Diamond particule size shall be 120 grit or finer. Cutting speed is 1800-4000 surface feet per minute at a feed rate of 4.0 inch/min. The following nomenclature is to be used for calculations of mechanical properties:

P = Load, lbs.
L = Test Span, inches
B = Specimen Width, inches
T = Metal Skin Thickness, inches
E = Modulus, lbs/in.^{2}
σ_f = Ultimate Flexural Strength, lbs/in.^{2}
σ_t = Ultimate Tensile Strength, lbs/in.^{2}
τ = Shear Strength, lbs/in.^{2}
Y = Deflection, inches
W = Weight, grams
C = Honeycomb Core Height, inches
t = Graphite/Polyimide Thickness, inches

4.4.1 Longitudinal Flexural Strength and Modulus

4.4.1.1 The specimen configuration and load support method shall be as shown in Figure 1.

4.4.1.2 The specimen shall be loaded with 0.125 inch radius supports at a rate of 0.05 in/min.

4.4.1.3 Place a deflectometer capable of measuring deflection to 0.001 inch under the span midpoint.

4.4.1.4 While the specimen is loaded record the deflection and load continuously. Remove the deflectometer prior to failure. Record the load at failure.

4.4.1.5 Calculate the longitudinal flexural strength as follows:

$$\sigma_f = \frac{1.5\ PL}{Bt^2}$$

4.4.1.6 Calculate the longitudinal flexural modulus as follows:

$$E = \frac{(P_1 - P_2)\ (L^3)}{4\ Bt^3\ (Y_1 - Y_2)}$$

4.4.1.7 Deflection data (Y_1, Y_2) are taken at the respective load (P_1, P_2) data points. Subscript 1 data is numerically larger than subscript 2 data. Do not take P or Y data near either end of the curve. Report the arithmetic mean of at least 3 values.

4.4.2 Horizontal Shear Strength

4.4.2.1 The specimen configuration and load support method shall be as shown in Figure 2.

4.4.2.2 The specimen shall be loaded with 0.0625 inch radius supports at 0.05 inch/min. Record the failure load.

4.4.2.3 Calculate the horizontal shear strength as follows:

$$\tau = \frac{0.75\ P}{Bt}$$

4.4.2.4 Report the arithmetic mean of at least three values.

4.4.3 Longitudinal and Transverse Tension Properties

4.4.3.1 The specimen configuration shall be as shown in Figure 3. This specimen is used for longitudinal tensile strength and transverse tensile strength.

4.4.3.2 Filaments shall be parallel to the beam length for longitudinal tests and perpendicular to the beam length for transverse tests. The tolerance on alignment is $\pm$ 1°.

4.4.3.3 The loading rate shall be 0.05 inch/min. using knife edge supports.

4.4.3.4 Record the failure load for all specimens. Report the arithmetic mean of at least three values.

4.4.3.5 Calculate the longitudinal (0°) and transverse (90°) tensile strength as follows:

$$\sigma_t = \frac{4P}{Bt\left(C + \frac{t + T}{2}\right)}$$

4.4.4 "A" Stage Prepreg Resin Solids and Resin Volatile Content

4.4.4.1 Take duplicate samples of boron/polyimide prepreg from the same sheet. Sample size is 2.0 in. x 2.0 in. Weigh one cleaned and dried 150 ml. Pyrex beaker to the nearest 0.0001 g and record weight as W_1.

4.4.4.2 Place a sample of prepreg in the beaker and weigh to the nearest 0.0001 g. Record as W_2.

4.4.4.3 Heat the samples to 600°F at a rate of 1-2°F/min. and hold for 1 hour. Cool to room temperature in a desiccator. Weight the sample to the nearest 0.0001 g and record as W_3.

4.4.4.4 Weigh a cleaned and dried 150 ml Pyrex beaker to the nearest 0.0001 g and record as W_4. Take the duplicate sample , place it in the beaker and weigh to the nearest 0.0001 g. Record as W_5.

4.4.4.5 Extract the polyimide resin from the prepreg with n, methylpyrolidone (NMP) solvent at 100-130°F for 1 hour. Weigh another 150 ml. Pyrex beaker to the nearest 0.0001 g and record as W_6. Transfer the resin-NMP solution to this beaker weigh to the nearest 0.0001 g and record as W_7. Weigh the beaker with fibers remaining to the nearest 0.0001 g and record as W_8.

4.4.4.6 Boil the NMP off the resin-NMP solution by heating to 500°F at 1-2°F/min. and holding for 1 hour. Cool to room temperature in a desiccator. Weigh the remaining solids and record as W_9.

4.4.4.7 Calculate the prepreg resin solids as follows:

$$\text{Prepreg Resin Solids Content (\%)} = \frac{W_9 - W_4}{W_5 - W_4}(100)$$

$$\text{Resin Total Volatile Content (\%)} = \frac{W_2 - W_3}{(W_2 - W_1) - (W_5 - W_8)}(100)$$

Report the arithmetic mean of at least 3 specimens.

4.4.5 Infrared Analysis

4.4.5.1 Place the pre-preg resin on a potassium bromide cell plate.

4.4.5.2 Place the potassium bromide plate in a Beckman IR9 Infrared Spectrophotometer or equivalent instrument and conduct the spectrum scan in direct transmission. Set the Beckman IR9 instrument as follows:

(a) Scanning Rate - 80 cm^{-1}/min.
(b) Fine gain control setting - 3
(c) Coarse gain control setting - 10
(d) Routine slit control
(e) Period setting - 2
(f) Resolution - 25 cm^{-1}

4.4.6 Resin Tack

4.4.6.1 Prepare a steel test tool 4.0 in. x 4.0 in. with Mold Wiz F-57, Axel Plastics Research Laboratories.

4.4.6.2 Cut two (2) pieces of test material three (3) x four (4) inches long.

4.4.6.3 Condition the tool and pre-preg at room temperature for 30 minutes.

4.4.6.4 Place the test tool on end in a vertical position and place one ply of pre-preg on the tool with a squeegee. Remove the separator paper from the first ply.

4.4.6.5 Place the second ply against the first ply and squeegee for intimate contact between the two plies.

4.4.6.6 Determine whether the pre-preg material adheres to itself and the tool.

4.4.7 Drapability

4.4.7.1 Cut sufficient material to obtain a sample 2 inches long by 3 inches wide maximum.

4.4.7.2 Bend specimen in the fiber (0^o) direction over a 1/16 inch radius mandrel.

4.4.7.3 Repeat test for at least three separate samples.

4.4.7.4 Report results as "pass" or "fail". All three tests must pass or material has failed to meet the drapability requirement.

4.4.8 Tow Count

4.4.8.1 Back light a 3 inch wide section of broadgoods and count the number of tows in that 3 inch width.

4.4.8.2 Divide that number by 3 and report the tow count as the number of tows per inch.

4.4.9 Thickness Per Ply - Measure the thickness of the cured laminate in at least 5 locations, spaced to represent the laminate, with a flat-nosed micrometer, to the nearest 0.001 inch. Average the readings and divide by the number of plies. Report as "thickness per ply".

4.4.10 Reports and Specimen Disposition - One copy of all test results shall be sent to the Material and Process Development Department. Retain all specimens a minimum of two (2) weeks after issuance of the test report. Disposal at that time is at the discretion of the Test Laboratory Supervisor.

5.0 PREPARATION FOR DELIVERY

5.1 MARKING OF CONTAINER - Each container shall be permanently marked with the following information:

5.1.1 Title, number and revision letter of this specification.

5.1.2 Vendor's name and address.

5.1.3 Date of impregnation.

5.1.4 Size and number of sheets of broadgoods material.

5.1.5 Resin manufacturer's batch number and trade name.

5.1.6 Vendor's pre-preg lot/batch number and trade name.

5.1.7 CAUTION: Ship and store at or below 0°F.

5.2 MARKING OF PLASTIC SHEET CONTAINERS - Each plastic bag shall be permanently marked with the vendor's lot/batch number, sheet number and trade name.

5.3 PACKAGING OF CONTAINER

5.3.1 Package each sheet of pre-preg material in individual sealed moisture proof plastic bags.

5.3.2 Place silica gel (or equivalent disiccant) inside each sealed plastic bag.

5.3.3 The sealed plastic bags are to be packed in an insulated shipping container, that will be accepted for safe transportation by common carriers. Construct the containers so that sufficient room is available for the dry ice necessary to maintain the temperature inside the container at or below $0^{o}F$ for a minimum of three days.

5.5 SHIPPING - The container containing the pre-preg graphite/polyimide broadgoods shall be shipped by air express from the vendor to McDonnell. Upon receipt of the container at McDonnell, it shall be opened to ascertain that solid dry ice remains in the container.

5.6 STORAGE AT MCDONNELL

5.6.1 Immediately upon receipt of the material at McDonnell, the pre-preg material shall be forwarded to the using department, removed from the shipping container, and placed in $0^{o}F$ storage.

5.6.2 The material information on the shipping container (see Paragraph 5.1) shall be recorded with the date of arrival included.

6.0 NOTES

Not applicable.

7.0 APPROVED PRODUCTS

Products which have been qualified under the requirements of this specification are listed below. Revision of the list will be made as necessary. The listing of a product does not release the supplier from compliance with the specification requirements. Reproduction or reference to the list for advertising purposes is expressly forbidden.

MCAIR DESIGNATION	SUPPLIERS DESIGNATION	TAPE	BROAD GOODS	SUPPLIERS NAME ADDRESS
MMS-523	Modmor II Graphite/Skybond 703 Polyimide Resin		X	Whittaker Research and Development 3540 Aero Court San Diego, California

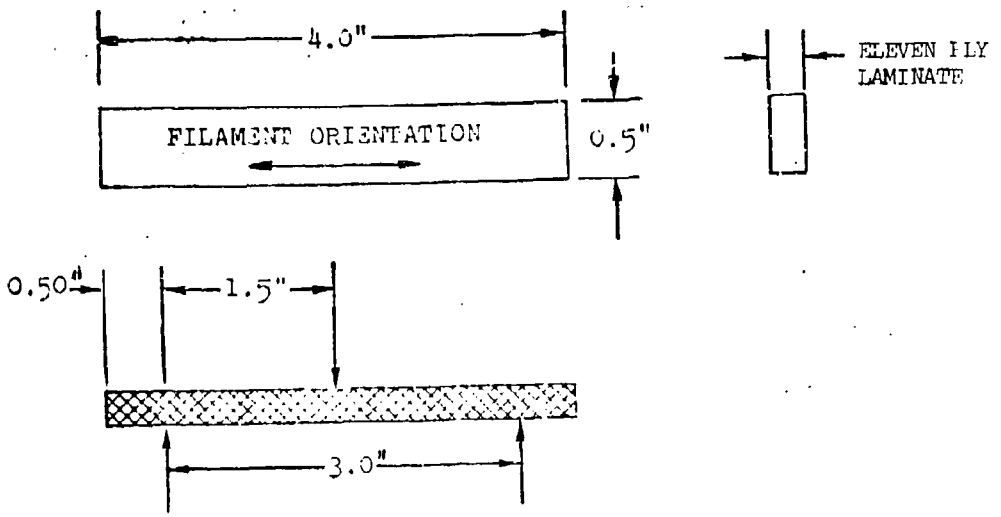


FIGURE I - LONGITUDINAL FLEXURE TEST

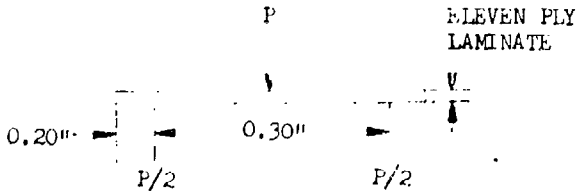
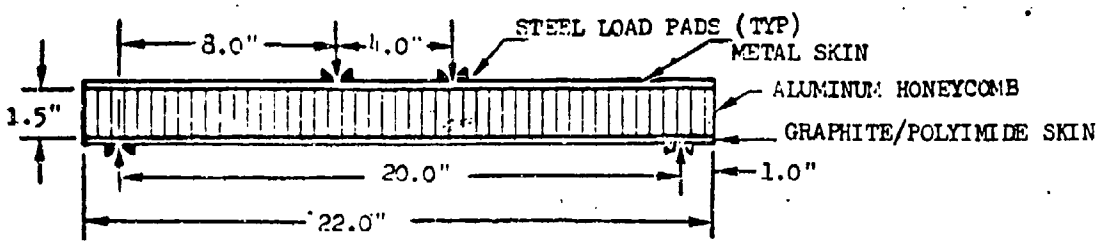


FIGURE 2 - HORIZONTAL SHEAR TEST



NOTES: (1) For 0° tension test, the metal skin is 1 x 22 x 0.090 inch thick (minimum) 6Al-4V annealed titanium. Aluminum honeycomb is 1.5 inch thick 5052-H39 non-perforated, 1/8 inch cell - 0.003 inch foil gage (minimum).

(2) For 90° tension test, the metal skin is 1 x 22 x 0.063 inch thick 2024-T62 aluminum. Aluminum honeycomb is 1.5 inch thick 5052-H39 non-perforated, 3/16 inch cell - 0.0015 inch foil gage (minimum).

(3) Graphite/Polyimide skin is approximately 1 inch wide x 22 inch x 4 ply thick for the 0° tension specimens and 1.00 inch wide x 22 inch x 6 ply thick for the 90° tension test specimens.

(4) The honeycomb core width shall be a minimum of one cell size (3/16" or 1/8" as applicable) wider on each side than the graphite/polyimide skin and the ribbon direction parallel to the 22 inch direction.

(5) Bond specimens with FM-400 per P.S. 14081 except curing pressure may be 85 ± 5 psig.

(6) Measure and record the metal skin thickness, honeycomb core height, and the graphite/polyimide skin width. Make measurements in the 4.0 inch center span.

(7) The steel load pads must have length sufficient to overhang the specimen edges, a minimum width of 0.75 inch, and a minimum radius of 0.125 inch at edges where they contact the specimen surface.

FIGURE 3

LONGITUDINAL (0°) AND TRANSVERSE (90°) TENSION TESTS

PROPERTY	TEST METHOD PARAGRAPH	REQUIREMENT
Resin Solids Content	4.4.4	37 ± 3% by weight
Total Volatile Content of Resin in Pre-Preg	4.4.4	40 ± 3% by weight
Infrared Analysis	4.4.5	For information only
Tack	4.4.6	Adhere to a vertical surface
Drapability	4.4.7	Shall bend on a 1/16 inch radius mandrel with no evidence of filament damage
Tow Count	4.4.8	As specified in Purchase Order
Working Life at 65-75°F	-	240 hours minimum
Storage Life at 0°F	-	12 months minimum

TABLE I - PHYSICAL PROPERTY REQUIREMENTS OF UNCURED PRE-PREG MATERIAL

MECHANICAL PROPERTY	TEMPERATURE (°F)	MINIMUM AVERAGE (PSI)	TEST METHOD PARAGRAPH
Longitudinal Flexural Strength, psi	R.T. 550	170,000 110,000	4.4.1
Longitudinal Flexural Modulus, psi	R.T. 550	15×10^6 14×10^6	4.4.1
Horizontal Shear Strength, psi	R.T. 550	8,000 5,000	4.4.2
Longitudinal Tensile Strength, psi	R.T.	*	4.4.3
Transverse Tensile Strength, psi	R.T.	*	4.4.3
Thickness Per Ply	-	For Information Only	4.4.9

*Values to be negotiated

TABLE II - MECHANICAL PROPERTY REQUIREMENTS OF CURED LAMINATES

PREPARED Ron Kellmansberger APPROVED _____
Engineering

APPROVED _____
AFPRO, Engineering

ISSUED 11 June 1971 REVISION A	GRAPHITE/POLYIMIDE PRE-PREG MATERIAL	MMS-523 PAGE 13 OF 13
	STANDARD MATERIAL SPECIFICATION	CODE IDENT.NO. 76301

MAC 2956C (2 JUL 70)

MCDONNELL DOUGLAS
ST. LOUIS

P.S. 14224

FABRICATION PROCEDURES

SKINS, STRUCTURAL, BORON/POLYIMIDE, FABRICATION AND ACCEPTANCE OF

1.0 APPLICATION

1.1 This process specification defines the procedures for fabricating boron/polyimide structural skins which incorporate a bonded titanium splice skin around the periphery. This specification also pertains to fabrication of flat composite laminates without splice bonded edge members.

1.2 This specification is effective upon issue and when specified on an Engineering Drawing.

2.0 APPLICABLE DOCUMENTS

2.1 The following specifications (or documents) form a part of this specification to the extent specified herein.

P.S. 12030 - Alkaline Cleaning

P.S. 12045 - Cleaning, Liquid Hone

P.S. 14081 - Sandwich Material, Aluminum Honeycomb - Boron/Epoxy Skin, Fabrication and Acceptance Procedure for

P.S. 14225 - Rudder Assembly, Boron/Polyimide Skin - Fiber Glass/Polyimide Honeycomb Sandwich, Fabrication and Inspection of

P.S. 20002 - Safety Standards and Information on the Use and Handling of Hazardous Materials

P.S. 20503 - Calibration Laboratories, Facility and Operational Requirements for

P.S. 20509 - Requirements for Class 2 Clean Room

P.S. 21233 - Nondestructive Testing of Adhesive Bonds

P.S. 21239 - Coating Thickness Measurement

P.S. 23401 - Certification of Furnaces and Temperature Control Systems for Heat Treating and Thermal Processing

MMS-304 - High Temperature Resistant Structural Adhesives

MMS-335 - Sealant Material, Vacuum Bag

MMS-522 - Boron/Polyimide Pre-Preg Material

MMS-528 - Glass Fabric, Teflon Coated

3.0 MATERIALS AND/OR SOLUTIONS

3.1 "A" Staged Boron/Polyimide Pre-Preg Material per MMS-522, Whittaker Research and Development, San Diego, California.

3.2 Metlbond 840 Adhesive Film per MMS-304, 0.135 ± 0.015 lb/ft^2, Whittaker Corporation, Narmco Materials Division, Costa Mesa, California.

3.3 Narmco 800 II Primer per MMS-304, Whittaker Corporation, Narmco Materials Division Costa Mesa, California.

3.4 Permacel No. 02C Double Sided Tape, 1.0 inch wide, Permacel Tape Co., or equal.

3.5 DK-153 cork dam-0.5 in. wide, thickness determined by laminate, Armstrong Cork Co., St. Louis, Mo.

3.6 Y-9050 Lead Foil Tape, 3M Company, or equal.

3.7 9151 Vacuum Bag Sealant Tape per MMS-335, pink, 3/32" thick, 1" wide, Schnee-Morehead Polymer Corporation, Irving, Texas.

3.8 Mylar Film, Type A, 0.0015 inch thick, E. I. duPont Film Division, Wilmington, Delaware.

3.9 Nylon Film, Capran Type 512-H or 80 Gage 2, Allied Chemical Corporation, or equal.

3.10 Teflon Squeegee, 3.0 inch x 5.0 inch.

3.11 Acrylic Plastic Sheet, minimum of 0.125 inch thick, Commercial.

3.12 Silicone Rubber Sheet, 1/32 inch thick, per ZZ-R-765 Class II, Grade 50.

3.13 Glass Cloth, Style 181 and 1000, Commercial.

3.14 Teflon Coated Glass Fabric per MMS-528, Connecticut Hard Rubber Co.; 3TLL Teflon Coated Fabric, Paliflex Products Company, or equal.

3.15 Mold Wiz F-57 Mold Release, or equal.

3.16 Ram 225 and Plastilease 334 Mold Release, Ram Chemical Company, Los Angeles, California.

3.17 M380-10 TFE Coated Glass Fabric, 50-54 inches wide, Dodge Industries; CHR 6TB TFE Coated Glass Fabric, 36 inches wide (minimum), Connecticut Hard Rubber Company, or equal.

3.18 White cotton and rubber gloves.

3.19 Solutions

(a) Pasa-Jell 107-C7, Semco Sales and Service Inc.

(b) Nitric Acid 42° Be' (O-N-350), Technical.

(c) Chromic Acid (O-C-303), Flake.

(d) Methyl Ethyl Ketone (TT-M-261).

(e) Deionized water.

3.20 Untreated Kraft Wrapping Paper per UU-P-268.

3.21 0.050 inch thick 6Al-4V annealed titanium sheet per MIL-T-9046, Type III, Composition C (required for fabrication of finger panels).

4.0 EQUIPMENT

4.1 Autoclave capable of 400°F (minimum) and 200 psig (minimum) nitrogen and/or air pressurized with pressure control capability of ± 5 psig and a minimum heat up rate of 3°F/min. A continuous temperature recording system and a 50 CFM vacuum pumping system capable of maintaining 200 microns or less of vacuum are required. The autoclave must be certified to P.S. 23401, Class 2C, except as noted in Paragraph 7.1.1.

4.2 Cold trap, capable of condensing all volatiles removed by the vacuum pumping system.

4.3 Ultrasonic NDT equipment per P.S. 21211.4.

4.4 Air circulating oven with 250°F capability certified per P.S. 23401 Class 3A for drying both cleaned and primed parts. Also, air circulating oven with 600°F capability certified per P.S. 23401 Class 3C for post curing composite parts.

4.5 Oven, air circulating type, capable of 550°F (minimum), certified per P.S. 23401, Class 1B, for performing elevated temperature tests on process control specimens.

4.6 Test equipment for testing process control specimens at room temperature and 550°F.

4.7 Refrigeration facilities for storing pre-preg material, adhesive, and primer at 0 ± 10°F.

4.8 Liquid honing equipment per P.S. 12045.

4.9 Alkaline cleaning equipment per P.S. 12030.

4.10 Intermediate lay-up fixture.

4.11 Final lay-up and curing fixture.

4.12 Diamond cutting wheels and routers with 200 grit or finer diamond particles.

4.13 X-acto knives, "pizza" cutters or equivalent.

4.14 Thermostatically controlled heat iron (150°F maximum).

4.15 Equipment per P.S. 21239 for measuring dry primer thickness.

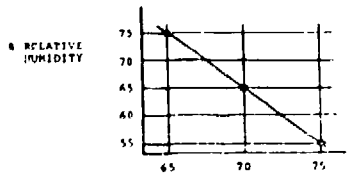
5.0 REQUIREMENTS

5.1 GENERAL

5.1.1 The initial fabrication of any composite part shall be witnessed by the Material and Process Development Department. Fabrication as witnessed by Material and Process Development shall not be changed without notifying Material and Process Development Department.

5.1.2 Personnel who fabricate composite parts shall be qualified. They must demonstrate, by passing applicable written and/or practical proficiency tests, that they possess the skills and knowledge necessary to ensure acceptable workmanship on the part to be fabricated. A list of qualified personnel shall be maintained.

5.1.3 The lay-up area shall meet the requirements of P.S. 20509 except the temperature shall be 65 to 75°F and the relative humidity shall not exceed the following values.



5.1.4 Thermocouples

(a) Production Part - A minimum of 4 thermocouples shall be used for each production part. They shall be located at opposite ends and sides of the part, and on the bonded titanium splice components or trim area of the laminate as applicable. For production parts of varying thickness,

the necessary number of additional thermocouples shall be used to insure uniform heating in all sections of the part.

(b) Process Control Specimens - A single thermocouple shall be placed adjacent to the process control laminate lay-up. A single thermocouple shall be attached to the center of the double lap shear process control panel.

NOTE: Under no circumstances shall thermocouples be placed directly on the surface of the pre-preg lay-up.

5.1.5 The leakage rate shall not exceed 0.5 inches of mercury per minute when leak checking the bagged lay-up both in the lay-up room and in the autoclave.

5.2 LAY-UP AND CURING FIXTURES

5.2.1 An intermediate lay-up fixture shall be used to lay-up the initial half of a composite laminate when the laminate incorporates a bonded titanium splice skin around the periphery.

5.2.2 The top surface of the final lay-up and curing fixture shall have a finish of 80 RHR or less.

5.2.3 The final lay-up and curing fixture shall be a minimum of 3.0 inches larger in all edge directions than the dams or joined peripheral members of the structural part, plus ample area for the 6.0 inch x 4 inch process control laminate. For parts with splice bonded metal components, the fixture shall also have ample area for the 9 inch x 10 inch double lap shear process control panel.

5.3 ADHESIVE AND BONDING

5.3.1 Titanium components of the composite laminate shall be cleaned and primed prior to splice bonding. Priming shall be accomplished within 8 hours after cleaning and bonding within 21 days after priming.

5.3.2 Each batch or shipment of Metlbond 840 adhesive and Narmco 300 II primer shall be qualified upon receipt at MCAIR per MMS-304. These materials shall also be requalified prior to their usage according to the criteria established in MMS-304.

5.3.3 Qualified Metlbond 840 adhesive and Narmco 300 II primer shall be stored at 0 ± 10°F upon receipt and when not in use. Each shall be packaged in a manner similar to the supplier's packaging technique. They shall not be removed from their sealed package or container until moisture ceases to condense on the package or container surface.

5.3.4 A record shall be kept of the batch and roll number of the adhesive and primer batch and container number used in the composite assembly.

5.3.5 Adhesive splice joints shall not overlap or have a gap greater than 1/32 inch.

5.3.6 All personnel shall wear clean white cotton gloves when handling cleaned or primed unwrapped parts, film adhesives or parts with film adhesives applied.

5.4 PRE-PREG AND LAY-UP

5.4.1 The boron/polyimide pre-preg sheet material shall be shipped in the "A" stage condition with Teflon coated glass fabric on one side and plastic release material on the other side. The boron/polyimide material shall be packaged in individual sealed moisture proof plastic bags with enough dry ice to keep the pre-preg at 0 ± 10°F.

5.4.2 The boron/polyimide pre-preg ("A" stage and "B" stage) material shall be packaged in individual sealed moisture proof plastic bags and stored at 0 ± 10°F when not in use.

5.4.3 The boron/polyimide pre-preg ("A" stage and "B" stage) material shall remain inside the sealed moisture proof plastic bag a minimum of 30 minutes after removal from 0°F storage to room temperature working area.

5.4.4 Butt joints of the pre-preg material within the composite (end to end type) are not allowed, unless required by the Engineering Drawing.

5.4.5 The edge of any individual piece of pre-preg material in one layer shall not be placed directly over the edge of another piece of pre-preg in a previous layer. Joints such as this shall be overlapped a minimum of 0.5 inch.

5.4.6 Cut edges of pre-preg terminating inside the laminate shall be uniform and defect free.

5.4.7 Where steel dams are used, cut edges of the pre-preg shall be within 0.10 inch of the inside edge of the dam.

5.4.8 Composite laminates without bonded splice members around the periphery shall be fabricated a minimum of 0.5 inches oversize on all sides for trimming purposes.

5.4.9 Any individual piece of pre-preg material in the composite lay-up shall not be exposed to room temperature in excess of 240 cumulative hours prior to cure.

5.4.10 Tools, clamps, etc. shall not be placed on top of the lay-up unless required by the fabrication procedure.

5.4.11 Manufacturing and Quality Assurance personnel working on or inspecting the lay-up at any time prior to the start of the cure cycle shall wear clean shop coats and caps.

5.5 PROCESS CONTROL SPECIMENS

5.5.1 Cleaning control coupons per Figure 1 (lap shear specimens used to evaluate the effectiveness of cleaning and etching solutions) shall be fabricated and tested, with test results meeting the lap shear requirements in Table II.

5.5.2 A boron/polyimide process control laminate shall be fabricated and cured at the same time and under the same vacuum bag as each production part. This laminate shall be evaluated per Section 7.0 and conform to the mechanical property requirements of Table II.

5.5.3 One double lap shear process control panel of the construction illustrated in Figure 2 shall be fabricated and cured with each production part (under the same vacuum bag) which incorporates splice bonded titanium components. This panel shall be evaluated per Section 7.0 and conform to the mechanical property requirements of Table II.

5.6 CURE CYCLE

5.6.1 The "A" staged pre-preg shall be "B" staged for 3 hours $\pm$ 2 minutes at 215 $\pm$ 2°F.

5.6.2 A minimum of 29" Hg vacuum is required throughout the pre-preg cure cycle.

5.6.3 Heat up rate shall be 3-4°F/min.

6.0 PROCEDURES

6.1 "B" STAGING BORON/POLYIMIDE SHEETS

6.1.1 Remove the "A" staged boron/polyimide pre-preg from 0° storage. Keep the pre-preg material in the plastic bag a minimum of 30 minutes after removal from the freezer.

6.1.2 Identify the orientation of each ply in the composite laminate by marking Mylar templates. Identify each template with progressive numbers (1, 2, 3, 4 . . etc.) as shown on the Engineering Drawing. Do not use grease pencil.

6.1.3 Remove the "A" staged pre-preg from the plastic bag and then orient the "A" staged sheet, sandwiched between Teflon coated glass fabric on one side and plastic release material on the other side, in the designated direction and cut to the dimensions of the Mylar templates. Identify each cut pre-preg ply on the Teflon backing with the corresponding number from the Mylar template.

6.1.4 Place the numbered plies of boron/polyimide pre-preg, with the plastic release sheet removed from the pre-preg (Teflon coated glass fabric remains on bottom surface of pre-preg), in an oven certified to P.S. 23401, Class 3A. Tape the pre-preg in place if necessary.

6.1.5 Place a thermocouple near the pre-preg (sandwiched between CHR 3TLL and Mylar also). Heat the pre-preg to 215 $\pm$ 2°F in 10 to 30 minutes, hold at that temperature for 180 $\pm$ 5 minutes.

6.1.6 At the end of the "B" stage cycle, shut off the oven blowers and heater and then open the oven doors.

6.1.7 Remove the "B" staged sheets from the oven, place in plastic bags and then reseal the bags.

6.1.8 Store the "B" staged sheets in sealed plastic bags at 0 $\pm$ 10°F if not used within 8 hours on the lay-up.

6.2 CLEANING OF TITANIUM SPLICE PLATE (IF REQUIRED)

NOTE: Fabricate and test cleaning control coupons (Paragraph 5.5.1) before bonding parts.

6.2.1 Solvent wipe the part (titanium splice skin, finger panels and cleaning control coupons) with MEK to remove grease or oil contamination. Do not vapor degrease.

6.2.2 Liquid hone the part per P.S. 12045. If alkaline cleaning will not be performed within 2 hours after liquid honing, protect the part by wrapping with wax-free Kraft paper.

6.2.3 Alkaline clean the part per P.S. 12030, Type III. Immerse the part immediately in Pasa-Jell solution or the part may be air dried for 20 minutes maximum prior to immersion in Pasa-Jell.

6.2.4 Immerse the part in a room temperature Pasa-Jell solution of the composition defined in Table I for 15 to 20 minutes. Remove and rinse the part thoroughly in room temperature deionized water. Check for water-break free surface. If water-break occurs, reclean parts from Para. 6.2.2 to 6.2.4.

MATERIAL	SMALL BATCH	LARGE BATCH
Pasa-Jell 107-C7	544 ml.	14.3 gal.
Concentrated Nitric Acid (HNO_3)	1,163 ml.	30.7 gal.
Deionized Water	1 gal.	100.0 gal.
Chromic Acid	165 grams	36.5 lbs.

TABLE I - PASA-JELL SOLUTION COMPOSITION

6.2.5 Dry the part at 100 to 150°F for 30 minutes in an oven certified per P.S. 23401, Class 3A. Protect the part by wrapping with wax-free Kraft paper if priming is not to be performed immediately after drying. If priming is not performed within 8 hours after drying, reclean the part per Paragraph 6.2.

6.2.6 Apply Narmco 800 II Primer to the cleaned metal bonding surfaces to a thickness of 1-2 mils and air dry 60 minutes and then force dry at 200 ± 25°F for 30 minutes in a well ventilated oven certified per P.S. 23401, Class 3A. Attach thermocouples to surfaces which will not be bonded.

6.3 PREPARATION FOR FABRICATION

6.3.1 Cut a sheet of CHR 6TB Teflon coated glass fabric (or equivalent) to a dimension which equals the area formed by the outer perimeter of the dams or titanium splice skin assembly.

6.3.2 Cut a sheet of CHR 3TLL (or equivalent) release fabric to the same dimensions as the CHR 6TB material. Where necessary, splice the CHR 3TLL (or equivalent) with an FEP Teflon tape to obtain sufficient area.

6.3.3 Cut one layer of 181 dry glass cloth to the same dimensions as the CHR 3TLL (or equivalent) for every five plies of boron/polyimide material. Use one extra layer of 181 dry glass cloth when the lay-up has two to four extra plies of pre-preg over multiples of 5 (i.e., 5, 10, 15, 20, etc.). For tapered boron/polyimide parts, the number of 181 glass cloth layers will vary with the location on the part.

6.3.4 Remove the "B" staged pre-preg from 0° storage and keep in the plastic bag a minimum of 30 minutes, if necessary. Pre-preg material may be stored at room temperature overnight if the remainder is completely used the following day. However, do not allow the pre-preg material to be exposed to temperatures over 0 ± 10°F unnecessarily.

6.3.5 Remove the Metlbond 840 adhesive (if required) from 0° refrigeration and allow to warm to room temperature. Verify that the adhesive has been qualified per MMS-304. Record the adhesive batch and roll number.

6.3.6 Proceed with fabrication of the structural composite part per Paragraph 6.4.1 or 6.4.2 as applicable, but first make certain (if the fabrication procedure is per Paragraph 6.4.1) that the lap shear cleaning control coupons of Paragraph 6.2 have been tested and meet the requirements of Table II. If they do not, reclean all titanium components of the structural part and again evaluate cleaning control coupons per Paragraph 6.2.

6.4 FABRICATION PROCEDURE

6.4.1 Structural Skins with a Bonded Titanium Splice Skin Around the Periphery

(a) Clean the surface of the intermediate lay-up fixture with an MEK dampened cheesecloth. Then cover the surface with a sheet of CHR 6TB Teflon coated glass fabric (or equivalent).

(b) Position the primed titanium splice skin assembly on the intermediate lay-up fixture.

(c) Apply a single layer of Metlbond 840 film adhesive to all exposed titanium surfaces which have been primed and will be bonded to pre-preg. Follow the procedures described below using clean white cotton gloves at all times.

(1) Cut the adhesive to the approximate shape of the surface to be bonded on a table which has been previously cleaned with MEK or equivalent. If splice joints are required, they shall not overlap or have a gap greater than 1/32 inch.

(2) Remove the protective wrapping from one side of the adhesive film and apply the film smoothly to the primed surface. Tack the film adhesive in place over 100% of its area using hand pressure. If necessary use a thermostatically controlled heat iron (150°F max.) to aid in tacking the film, but maintain the temperature of the adhesive below 150°F. Trim the film adhesive flush with the perimeter of the bond joint surface and then remove the remaining protective sheet from the adhesive.

(d) Lay-up the initial half of the laminate by collating the plies of pre-preg on the lay-up fixture. Use a warm (140-160°F) iron to momentarily soften and smooth out each layer of the composite laminate. Keep the warm iron moving at all times, taking care not to degrade the pre-preg by over-heating. Remove the top Teflon backing sheet from the pre-preg sheet prior to adding the next ply of pre-preg.

(e) Place the sheet of CHR 6TB (or equivalent) cut per Paragraph 6.3.1 on top of the lay-up and then place a sheet of 1/32 inch thick silicone rubber on top of the bottom release material.

(f) Clean the surface of the final lay-up and curing fixture with an MEK dampened cheese-cloth.

(g) Locate the final lay-up and curing fixture on the surface of the intermediate lay-up fixture by means of the positioning pins. Visually inspect the lay-up for proper alignment and fit on the final lay-up fixture. If acceptable, turn over the entire fixture package taking extreme care not to shift or alter the lay-up in any manner. Again inspect the lay-up for proper alignment and fit on the final lay-up and curing fixture. If acceptable, slowly remove the intermediate lay-up fixture from the surface of the final lay-up fixture. Again take extreme care not to damage the lay-up.

(h) Repeat procedure (c).

(i) Lay-up the remaining half of the composite laminate by collating the plies of pre-preg per the instructions in Paragraph 6.4.1 (d).

(j) Attach steel dams to all required areas of the titanium splice skin assembly as illustrated in Figure 3 using Permacel No. 02C double sided tape. Make sure all joints are tightly sealed. Make the dams the thickness $^{+0.020}_{-0.000}$ of the expected cured laminate thickness.

(k) Complete the lay-up per Paragraph 6.5 and fabricate the required process control specimens per Paragraphs 6.4.3 and 6.4.4.

6.4.2 Structural Parts Without Splice Bonded Edge Members

(a) Clean the surface of the final lay-up and curing fixture with an MEK dampened cheesecloth.

(b) Place the sheet of CHR 6TB (or equivalent), cut per Paragraph 6.3.1 ,on top of the fixture.

(c) Place the cork dams on top of the CHR 6TB with the outside edge of the dam directly over the outside edge of the CHR 6TB. Make sure all joints are tightly sealed. Make the dams the thickness $^{+0.020}_{-0.000}$ of the expected cured laminate thickness.

(d) Lay-up the composite laminate by collating the pre-preg plies on the fixture using the processing procedures described in Paragraph 6.4.1 (d).

(e) Complete the lay-up per Paragraph 6.5 and fabricate the process control specimens per Paragraph 6.4.3.

6.4.3 Process Control Laminate

Lay-up a process control laminate on the same tool as the structural part using the processing procedures described in Paragraph 6.4.2. Lay-up one laminate 6.0" x 4.0" x 15 plies with filaments parallel to the 6" direction.

6.4.4 Double Lap Shear Process Control Panel

(a) Chem mill both sides of the 0.050 inch thick annealed 6Al-4V titanium finger panels to provide a panel thickness of 0.045 ± .005 inches and surface finish of 125 RHR.

(b) Clean and prime the finger panels per Paragraph 6.2 at the same time and in the same manner as the titanium components of the structural part.

(c) Fabricate the double lap shear panel per Figure 2 and place on the same fixture as the structural part.

6.5 COMPLETION OF LAY-UP

6.5.1 Attach thermocouples to the lay-up per the requirements of Paragraph 5.1.4.

6.5.2 Place the top release material (CHR 3TLL or equivalent) cut per Paragraph 6.3.2 over the completed lay-up and attach to the steel dams and/or titanium splice skin assembly with Permacel #02C Double Sided Tape. Make sure the CHR 3TLL, Permacel Tape and dam and/or splice skin are in intimate contact.

6.5.3 Place the correct number of 181 glass cloth layers over the CHR 3TLL (or equivalent) per the requirements of Paragraph 6.3.3.

6.5.4 Place a Mold Wis F-57 coated 0.040" aluminum sheet perforated with 1/16 inch diameter holes on 1/2 inch centers on top of the production assembly to act as a pressure plate. Place Mold Wis F-57 coated 0.040" perforated aluminum sheets over each of the process control panels. Tape the pressure plates to the steel dams to insure that the plates will not ride on the dams during the cure cycle. Place 2-5 layers of style 1000 dry glass cloth over the top of the lay-up and extend beyond the dam.

6.5.5 Place a single layer of Mylar Type A on top of the entire lay-up (i.e., structural part and process control specimens) and extend several inches beyond the dams and/or splice skin assembly. The vacuum and static lines shall be located adjacent to the lay-ups and inside the sealed edge of the vacuum bag. Seal the vacuum bag to the fixture with 9151 Sealant Tape.

6.5.6 Run a leak check per Paragraph 6.6.1 (a).

6.5.7 Place a layer of style 1000 dry glass cloth over the top of the Mylar bag, and then place a single layer of nylon film on top of the entire lay-up. Place a vacuum line between the bags and seal the Nylon bag as described in Para. 6.5.5.

6.5.8 See Figure 3 and 4 for typical laminate lay-up cross sections.

6.6 LEAK CHECKING

NOTE: When leak checking, apply vacuum to the interior of the vacuum bag slowly. As the air is evacuated, make the bag conform to the shape of the lay-up and fixture. Make certain the bag does not bridge any areas.

6.6.1 A leak check is required first in the lay-up room and then in the autoclave prior to application of heat to the lay-up. Conduct these checks with a mercury manometer or suitable vacuum gage as follows:

(a) Pull 29 inches (minimum) mercury vacuum on the bagged lay-up and close off the vacuum source. Take a pressure reading 2 minutes after isolation of the system. The maximum allowable leakage rate is 0.5 inches of mercury per minute.

(b) Maintain at least 29 inches of mercury vacuum on the bagged lay-up and place in the autoclave. Connect the required plumbing (thermocouples, vacuum and static lines and cold trap) and repeat the leak check of the previous paragraph.

(c) At the conclusion of the second leak check, close the autoclave door while maintaining 29 inches (minimum) of mercury vacuum on the bagged lay-up.

(d) Apply 200 psi autoclave pressure. Run a leak check per Paragraph 6.6.1(a). A reading of 50 microns or less of vacuum and a minimum manometer reading of 29 inches of Hg is required.

6.7 CURE SCHEDULE

6.7.1 Heat the lay-up to 215°F at rate of 3-4°F/min. under 29" (minimum) Hg vacuum. Hold at 215 ± 10°F for 60-70 minutes.

6.7.2 Raise the temperature to 235°F at rate of 3-4°F/min. under 29" (minimum) Hg vacuum. Hold at 235 ± 10°F for 60-70 minutes; after 15 minutes at 235°F, start pressurizing the autoclave to 200 ± 5 psig.

6.7.3 Raise the temperature to 350°F at rate of 3-4°F/min. under 29" Hg (minimum) vacuum and 200 psig pressure.

6.7.4 Cool to 125°F or less at rate of 3-4°F/min. under full vacuum and pressure.

6.7.5 Record the autoclave temperature and pressure throughout the cure cycle. Also, record the static vacuum bag readings every fifteen minutes.

6.8 POST CURING

6.8.1 Post Curing Laminates

6.8.1.1 Post cure the cured skin(s), process control laminate and double-lap shear control panel (if required) in an oven.

6.8.1.2 Heat the laminates to 350°F at 3-4°F/min. Hold at 350 ± 10°F for 60-70 minutes, then heat to 600°F at rate of 1°F/min., with 24 ± 1/2 hour holds at 400 ± 10°F, 450 ± 10°F, 500 ± 10°F, 550 ± 10°F and 600 ± 10°F.

6.8.1.3 Cool down to 125°F or less at 2-3°F/min.

6.9 TRIMMING

6.9.1 General - Trim the structural part and process control specimens after removal from the final lay-up and curing fixture.

6.9.2 Structural Part

(a) Parts with a bonded titanium splice skin around the periphery do not require trimming.

(b) Trim boron/polyimide parts without edge members with a diamond impregnated cutoff wheel or router to the dimensions of the Engineering Drawing. Use water or carbon dioxide for cooling the diamond tool, but do not use any oil based compound. Use 200 grit or finer diamond particle impregnated tooling, and operate at a speed of 1800-4000 SFM and a feed rate of 3.0 to 5.0 inch/min. depending on thickness. Shearing the edges of composites to 7 plies thick, in lieu of diamond tool cutting, is acceptable if allowed by the Engineering Drawing.

6.9.3 Process Control Specimens

(a) Cut the process control laminate fabricated per Paragraph 6.4.3 into six 4.0" (filament direction) x 0.5" x thickness flexural test specimens and six 0.70" (filament direction) x 0.25" x thickness horizontal shear test specimens. Use a thin (0.060 inch or less) diamond cut-off wheel at 4000 surface feet per minute (minimum) and 0.5 inch/min. maximum feed rate. Attach acrylic plastic back-up to the bottom of the laminate with double sided tape. Use a 200 grit or finer diamond tool.

7.0 QUALITY ASSURANCE

7.1 RAW MATERIAL ACCEPTANCE

7.1.1 Each batch of Metlbond 840 adhesive and Narmco 800 II primer shall be qualified upon receipt at MCAIR per MMS-304. The materials shall be requalified prior to their usage according to the criteria established in MMS-304.

7.1.2 Each batch of boron/polyimide pre-preg material shall meet the requirements of MMS-522.

7.2 EQUIPMENT

7.2.1 Instrumentation used to control processes shall be calibrated per P.S. 20503.

7.2.2 Ovens, autoclaves, furnaces and temperature control systems shall be certified to P.S. 23401. The autoclave shall be certified to P.S. 23401, Class 3C, process control specimen testing oven to P.S. 23401, Class 1B, and the oven to P.S. 23401, Class 3A.

7.3 PROCESS CONTROL - Quality Assurance shall verify conformance to the following:

(a) Materials used have a current Quality Assurance acceptance tag affixed which shows date for requalification.

(b) Metlbond 840 adhesive and Narmco 800 II primer are stored in their original sealed shipping containers at 0 ± 10°F until ready for use.

(c) Applicable process equipment has a current Quality Assurance calibration tag affixed.

(d) Adhesive is conditioned to ambient temperature in closed containers before use.

(e) All adhesive splices are butt splices with a maximum 1/32 inch gap.

(f) The temperature in the lay-up room is held between 65-75°F and the relative humidity in the lay-up room is held within the limits shown in Paragraph 5.1.3.

(g) During cure there was a minimum of three thermocouples functioning at all times.

(h) The top surface of the final lay-up and curing fixture has a finish of 80 RHR or less.

(i) Titanium splice plate is properly prepared for bonding and then immediately wrapped in wax-free Kraft paper.

(j) Priming is accomplished within 8 hours after cleaning and bonding within 21 days after priming.

(k) All personnel handling cleaned, unwrapped parts and/or adhesives through the lay-up operation, and all support personnel coming in direct contact with cleaned parts and/or adhesives wear clean, white hats and coats, and clean white cotton gloves.

(l) The boron/polyimide material is packaged in moisture proof plastic bags and stored at 0 ± 10°F when not in use.

(m) The boron/polyimide pre-preg material remains inside the sealed moisture proof plastic bag a minimum of 30 minutes after removal from 0°F storage to room temperature working area.

(n) The "A" staged boron/polyimide pre-preg material with release sheets on both surfaces is cut with templates in the orientation required.

(o) The "A" staged boron/polyimide pre-preg material with the plastic release sheet removed from the pre-preg (Teflon coated glass fabric remains on bottom surface of pre-preg) is "B" staged at 215 ± 2°F for 3 hours ± 2 minutes.

(p) No end to end type butt joints (perpendicular to filament direction) of boron/polyimide pre-preg material are used within the composite laminate unless required by the Engineering Drawing.

(q) The edge of any individual piece of pre-preg in one layer is not placed directly over the edge of another piece of pre-preg in a previous layer, but overlapped a minimum of 0.5 inch.

(r) Cut edges of the pre-preg where steel dams are used are within 0.10 inch of the inside edge of the dam.

(s) Structural parts without splice bonded members around the periphery are fabricated a minimum of 0.5 inches oversize on all sides for trimming purposes.

(t) Any individual piece of pre-preg material is not exposed to room temperature in excess of 240 cumulative hours prior to cure.

(u) Manufacturing and Quality Assurance personnel working on or inspecting the lay-up at any time prior to the start of the cure cycle are wearing clean shop coats and caps.

(v) A minimum of 29" Hg vacuum was kept during the pre-preg cure cycle.

7.4 STRUCTURAL ASSEMBLY ACCEPTANCE

7.4.1 Nondestructive Testing - Nondestructively inspect the structural composite part for foreign material inclusions per P.S. 21233 and unbonded areas and/or voids per P.S. 21233, Class 2A.

7.4.2 Control Panel Testing

7.4.2.1 Cleaning Control Coupons - Cleaning control coupons shall be fabricated per Paragraph 6.2 and tested per Figure 1 to meet the requirements in Table II.

7.4.2.2 Process Control Laminate - Test six longitudinal flexural specimens, three at room temperature and three at 550°F, in accordance with Figure 5. Test six horizontal shear specimens, three at room temperature and three at 550°F, in accordance with Figure 6. The specimens shall meet the requirements of Table II.

7.4.2.3 Double Lap Shear Specimens - Perform double lap shear testing with three specimens at room temperature and three specimens at 550°F per Figure 2. The specimens shall meet the requirements of Table II.

7.4.2.4 Perform all elevated temperature tests in an air circulating oven. Specimens that are tested at elevated temperatures shall be at temperature 10 minutes prior to test. The test specimen temperature shall be controlled to ± 5°F.

8.0 SAFETY

8.1 ORGANIC SOLVENTS

8.1.1 Description - The following solvents and/or solvent bearing materials are used in small quantities per this specification.

(a) Methyl Ethyl Ketone: A colorless, thin solvent. Flammable and toxic.

(b) Narmco 800 II primer: A brownish thin liquid, flammable and toxic.

8.1.2 First Aid - In event of eye or skin contamination:

(a) Flush the exposed area with large amounts of water.

(b) Remove contaminated clothing.

(c) Secure first aid.

8.1.3 Toxicology - Avoid prolonged breathing of solvent vapors.

8.1.4 Fire Hazard - Flammable solvents are extremely easy to ignite and shall not be used in the vicinity of smoking, sparks or open flames.

8.1.5 Handling and Storage

(a) Wear Safety glasses (or goggles) and rubber gloves while working with solvents.

(b) Do not use solvents in confined areas unless specifically authorized, since fumes are generally toxic, flammable, and explosive.

(c) Store and handle solvents in properly labelled safety containers.

(d) Dispose of all rags in flammable solvent areas in special containers used only for this purpose.

8.1.6 NOTE: The preceding are minimum safety precautions. Personnel using these materials should be familiar with the more detailed precautions provided in P.S. 20002.

8.2 NON-SOLVENT ORGANIC MATERIALS

8.2.1 Description - The following non-solvent organic materials are used per this specification.

(a) Metlbond 840 Film Adhesive: A glass fabric supported, grey film. Not a fire hazard, but is toxic.

8.2.2 Precautions - The safety precautions specified in Paragraph 8.1 shall apply to this material with the exception of those precautions related to fire hazards.

8.3 ACIDS

8.3.1 General Precautions

(a) Acid resistant gloves, aprons, chemical goggles and boots must be worn when handling hazardous chemicals.

(b) Use chemicals and solutions only as stated in this specification.

(c) Always add acid to water when diluting or mixing chemicals or solutions.

(d) Avoid inhaling vapors. Wear McDonnell approved respirators when necessary.

(e) Correctly label and maintain the identity of all containers as to their contents.

(f) Incompatible Materials: Isolate from incompatible materials as specified per McDonnell P.S. 20002 under both specific and general precautions.

8.3.2 First Aid - In the event of eye or skin contaminations:

(a) Flush the exposed area with large amounts of water for 15 minutes.

(b) Remove contaminated clothing.

(c) Secure first aid.

8.3.3 Pasa-Jell 107-C7 Solution

(a) Description: A thin, orange liquid.

(b) Toxicology: Liquid contains acid and dichromate which can irritate and burn eyes and skin.

(c) Fire Hazard: May cause ignition when in contact with flammable or combustible materials.

(d) Storage: Store in original shipping containers.

CAUTION: Do not allow Pasa-Jell 107-C7 or rinse water containing Pasa-Jell 107-C7 to come in contact with MEK or other solvents.

8.3.4 Nitric Acid - HNO_3

(a) Description: Transparent, colorless or yellowish fuming, suffocating, corrosive liquid.

(b) Toxicology: Nitric acid vapor is highly irritating to the skin, eyes, and mucous membranes.

(c) Fire-Hazard: Nitric acid is a powerful oxidizing agent. Avoid contact with reducing agents such as alcohol or petroleum solvents, as the mixture is explosive.

8.3.5 Chromic Acid

(a) Description: Red crystals

(b) Toxicology: Chromates attack the skin and mucous membranes.

(c) Handling and Storage: Isolate chromates from flammable solvents, and other organic matter such as wood, peper, alcohol and petroleum compounds as the mixture is flammable and explosive.

9.0 NOTES

9.1 Vendors or subcontractors needing this specification should direct their request for copies to the attention of McDonnell Aircraft Company (MCAIR) St. Louis, Purchasing or Subcontracting, as applicable.

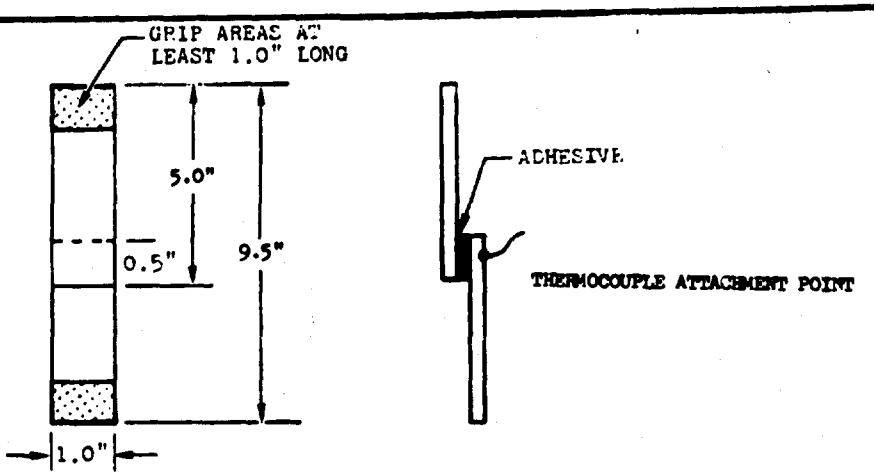
Information pertaining to the technical aspect of this specification can be obtained from MCAIR Material and Process Development Department (Dept. 372).

10.0 REFERENCE PUBLICATIONS

None applicable.

MECHANICAL PROPERTY	MINIMUM AVERAGE (PSI)		SPECIMEN MINIMUM (PSI)	
	R.T.	550°F	R.T.	550°F
Lap Shear Strength (Cleaning Coupon)	2,250	1,000	2,000	750
Double Lap Shear Strength	3,000	2,000	2,750	1,750
0° Flexural Strength, Boron/Polyimide	190,000	155,000	180,000	140,000
0° Flexural Modulus, Boron/Polyimide	26.0×10^6	21.0×10^6	24.0×10^6	20.0×10^6
Horizontal Shear Strength	11,000	6,000	10,000	5,000

TABLE II - PROCESS CONTROL MECHANICAL PROPERTY REQUIREMENTS



NOTES:

(1) Specimens are 6Al-4V annealed titanium 0.045 ± .005 x 1.0" x 6.0". Clean and prime per Section 6.0 of this specification with the production parts that will be bonded with the specimens.

(2) Apply Metlbond 840 adhesive and cure per Section 6.0 of this specification.

(3) Use self-aligning grips and load at 600-700 lbs/min.

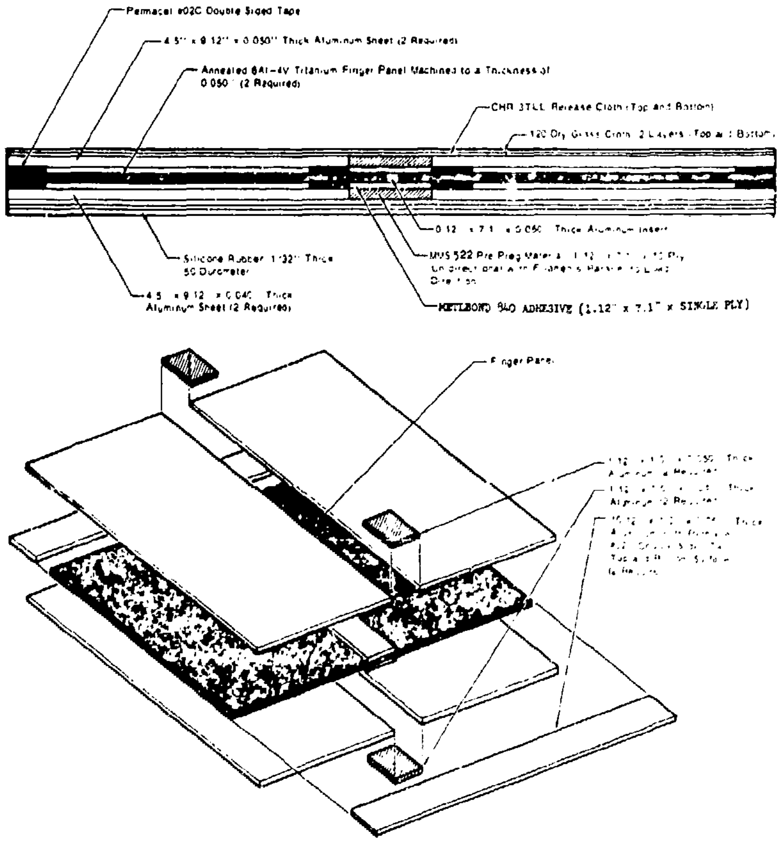
(4) Six (6) specimens required. Test three (3) at room temperature and three at 550 ± 5°F after holding at temperature for 10 minutes. Heat specimens in air circulating oven only.

(5) Calculate lap shear strength as follows:

$$F_s = \frac{P}{A}$$

Where F_s = Lap Shear Strength (psi)
P = Failing Load (lb)
A = Bond Line Area (in^2)

FIGURE 1 -- LAP SHEAR SPECIMEN AND TEST (CLEANING CONTROL COUPON)



Notes: (1) Place a metal insert (10 required) in each of the [illegible] out areas of the finger panel [illegible]
(2) Load each specimen at a rate of 600-700 Lbs per minute. Calculate failing shear stress [illegible] using the following Formula:

$F_s = \frac{L}{2lw}$ Where F_s = Failing Shear Stress
L = Failure Load
l = [illegible]
w = [illegible]

FIGURE 2 LAY-UP CROSS SECTION AND EXPLODED VIEW OF DOUBLE LAP SHEAR PROCESS CONTROL PANEL

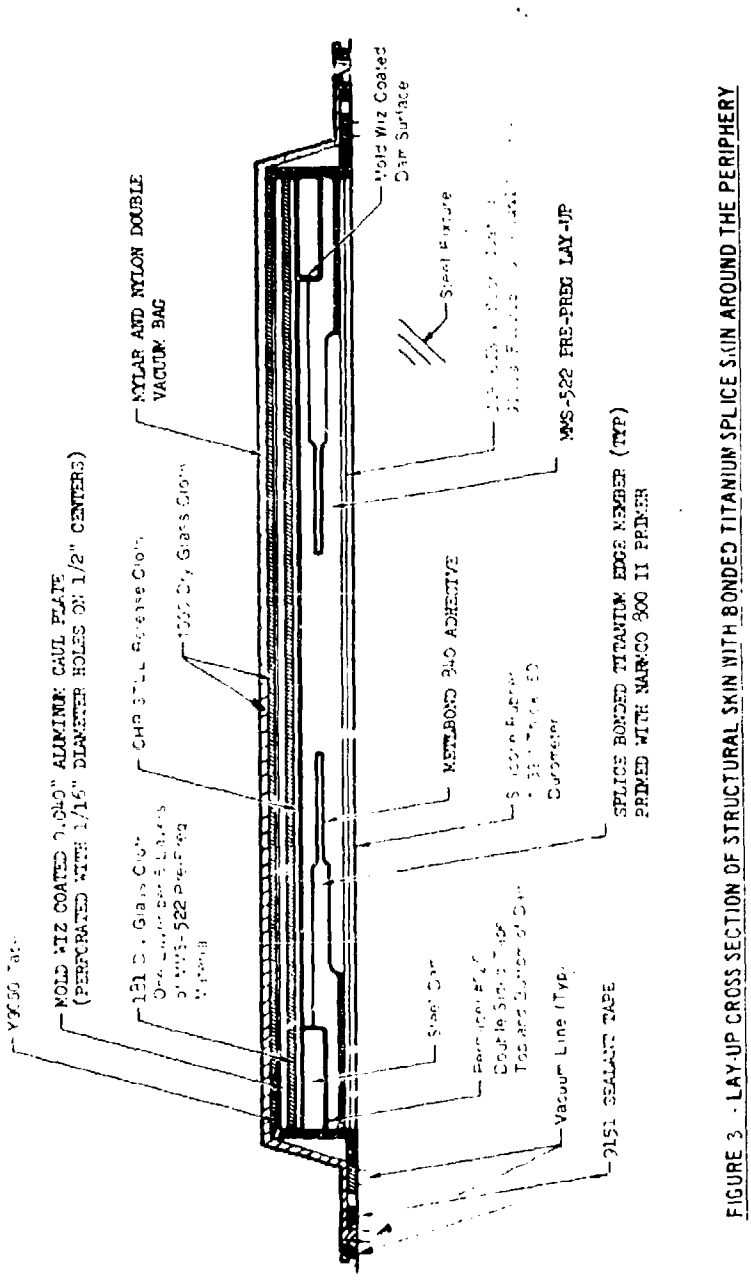


FIGURE 3 - LAY-UP CROSS SECTION OF STRUCTURAL SKIN WITH BONDED TITANIUM SPLICE SKIN AROUND THE PERIPHERY

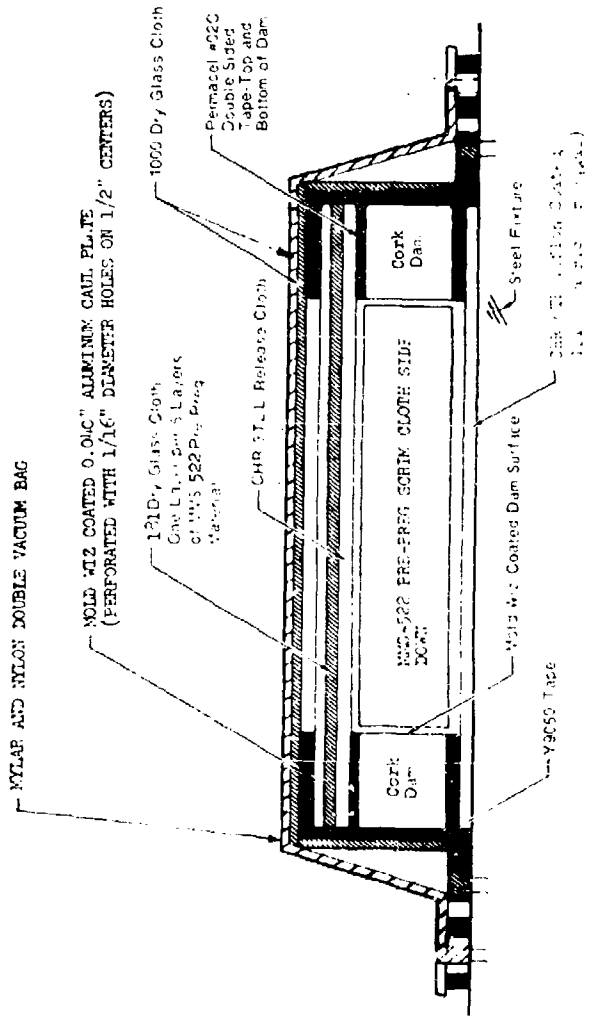
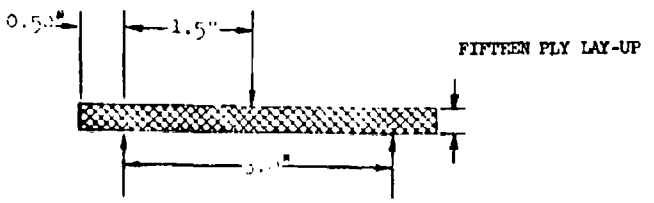


FIGURE 4 - LAY-UP CROSS SECTION OF LAMINATE WITHOUT EDGE MEMBERS



NOTES:

(1) Width of specimen is 0.5 inch.

(2) Load the specimen with 0.125 inch radius supports at a rate of 0.05 in./min.

(3) Place a deflectometer capable of measuring deflection to 0.001 inch under the span midpoint.

(4) While the specimen is loaded record the deflection and load continuously. Remove the deflectometer prior to failure.

(5) Run 550°F temperature test in air circulating oven, holding at 550° for 10 minutes prior to test.

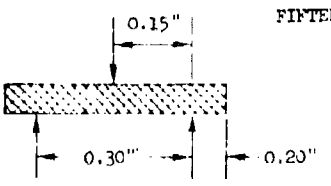
(6) Calculate the ultimate flexural strength as follows:

$$F = \frac{1.5\ PL}{wt^2}$$, where F = Flexural Strength, psi, P = Load at failure, lb., L = Test span, 3 in., W = Specimen width, in., t = Specimen thickness, in.

(7) Calculate the flexural modulus as follows:

$$E = \frac{P(L^3)}{4w(t^3)Y}$$, where E = Flexural modulus, psi, P = Load at failure, lb., L = Test span, 3 in., w = Specimen width, in., t = Specimen thickness, in., Y = Deflection, in.

FIGURE 5 - LONGITUDINAL FLEXURE SPECIMEN AND TEST



NOTES:

(1) Width of specimen is 0.25 inches.

(2) Load the specimens with 0.125 inch radius supports at a rate of 0.05 in./min.

(3) Run 550°F temperature test in air circulating oven, holding at 550°F for 10 minutes prior to test.

(4) Calculate the interlaminar shear strength as follows:

$F = \frac{0.75P}{wt}$, where F = Interlaminar shear strength, psi
P = Load at failure, lb.
w = Specimen width, in.
t = Specimen thickness, in.

FIGURE 6 - INTERLAMINAR SHEAR SPECIMEN AND TEST

PREPARED _____ APPROVED _____
Manufacturing

APPROVED _____ APPROVED _____
Engineering Quality Assurance

APPROVED _____ APPROVED _____
AFTRO, Engineering Safety and Medical

P.S. 14225

FABRICATION PROCEDURES

BONDED STRUCTURE, POLYIMIDE MATRIX COMPOSITE, FABRICATION AND INSPECTION OF

1.0 APPLICATION

1.1 This process specification provides procedures for the fabrication and inspection of both the F-4 Polyimide Composite Rudder and the F-15 Wing Compression Panel.

1.2 This process specification is effective upon issue and when specified on an Engineering drawing.

2.0 APPLICABLE DOCUMENTS

2.1 The following documents, of the latest revision, form a part of this specification:

P.S. 12030 - Alkaline Cleaning

P.S. 12045 - Cleaning, Liquid Hone

P.S. 14224 - Skins, Structural, Boron/Polyimide, Fabrication and Acceptance Of

P.S. 14227 - Skins and Substructural Shapes, Structural, Graphite/Polyimide, Fabrication and Acceptance Of

P.S. 20002 - Safety Standards and Information on the Use and Handling of Hazardous Materials

P.S. 20503 - Calibration Laboratories, Facility and Operational Requirements for

P.S. 20509 - Requirements for Class 2 Clean Room

P.S. 21206.3 - Radiographic Inspection of Honeycomb Assemblies and Composite Structures

P.S. 21211.4 - Ultrasonic Inspection of Honeycomb Assemblies and Composite Structures

P.S. 21233 - Nondestructive Testing of Adhesive Bonds

P.S. 21239 - Coating Thickness Measurement

P.S. 23401 - Certification of Furnaces and Temperature Control Systems for Heat Treating and Thermal Processing

MMS-304 - High Temperature Resistant Structural Adhesives

MMS-335 - Sealant Material, Vacuum Bag

MMS-528 - Glass Fabric, Teflon Coated

MMS-705 - Core Material, Polyimide Honeycomb, High Temperature, Glass Reinforced

Federal Specification TT-M-261, Methyl Ethyl Ketone for Use in Organic Coating

3.0 MATERIALS AND/OR SOLUTIONS

3.1 PRODUCTION MATERIALS

3.1.1 Boron/polyimide skins fabricated per P.S. 14224 and the Engineering drawing.

3.1.2 Graphite/polyimide spar and upper closure (for Rudder), and hat stiffeners (for Wing Compression Panel) fabricated per P.S. 14227.

3.1.3 HRH-327 polyimide honeycomb core per MMS-705, 3/16" cell, 3.0 lb/ft^3, Hexcel, Grand Prairie, Texas.

3.1.4 Titanium rib, hinges and fairing and steel rib per the Engineering drawing.

3.1.5 Metlbond 840 supported film adhesive per MMS-304, 0.135 ± 0.015 lb/ft^3, Whittaker Corporation, Materials Division, Costa Mesa, California.

3.1.6 FM-34 supported film adhesive per MMS-304, Bloomingdale Rubber Department, American Cyanamid Company, Havre de Grace, Maryland.

3.1.7 FM-29 unsupported foaming adhesive per MMS-304, 0.1 inch thick, Bloomingdale Rubber Department, American Cyanamid Company, Havre de Grace, Maryland.

3.1.8 Narmco 800 II primer per MMS-304, Whittaker Corporation, Narmco Materials Division, Costa Mesa, California.

3.1.9 BR-34 primer per MMS-304, 80% solids, Bloomingdale Rubber Department, American Cyanamid Company, Havre de Grace, Maryland.

3.2 AUXILIARY MATERIALS

3.2.1 Boron/polyimide 6 ply panels fabricated per P.S. 14224 with ply orientation of 0, +45, -45, -45, +45, 0°. Used for skins on control panels.

3.2.2 HRH-327 polyimide honeycomb core, 3/16" cell, 8 lb/ft^3, Hexcel, Grand Prairie, Texas.

3.2.3 Methyl ethyl Ketone (MEK), Federal Specification TT-M-261.

3.2.4 Materials and solutions for alkaline cleaning per P.S. 12030, Type III.

3.2.5 Materials for liquid honing per P.S. 12045.

3.2.6 Pasa-Jell 107-C7, Semco Sales and Service Inc.

3.2.7 Nitric acid 42° Be¯ (O-N-350), Technical.

3.2.8 Chromic acid (O-C-303), Flake.

3.2.9 Aluminum sheet, 0.040" thick.

3.2.10 Deionized water, Commercial.

3.2.11 FM 641, Verifilm, Glass Supported, .075± .005 lb/ft^2, green, American Cyanamid Company, Bloomingdale Department, Havre de Grace, Maryland.

3.3.12 CHR 3TLL Teflon Coated Glass Fabric per MMS-528, Connecticut Hard Rubber Company; TX1050 Teflon Coated Glass Fabric, Pallflex Products Company; or equal.

3.2.13 Mylar film, Type A, 0.0015 inch thick, E. I. DuPont Film Division, Wilmington, Delaware, or HS-6262 film, Noland Paper Co., Los Angeles, California, or equivalent.

3.2.14 Wax-free Kraft paper, commercial.

3.2.15 White cotton gloves, commercial.

3.2.16 200 grit or finer sandpaper, commercial.

3.2.17 Glass cloth, Style 1000, commercial.

3.2.18 9151 Vacuum Bag Sealant Tape per MMS-335, pink, 3/32" thick, 1" wide, Schnee-Morehead Polymer Corporation, Irving, Texas.

3.2.19 P-211 tape, or equal, Permacel Tape Corp., New Brunswick, New Jersey, or equivalent.

3.2.20 N-methyl pyrrolidone solvent.

3.2.21 Y-9133 Mylar Tape, 3M Co., St. Paul, Minnesota.

3.2.22 Lab coats and hats, DuPont Tyvek disposables or Dacron cleanables.

4.0 EQUIPMENT

4.1 Autoclave certified to P.S. 23401, Class 3C except the working zone of the autoclave shall extend to within 3 inches of the wall and 20 thermocouples which continue to function during the entire certification procedure shall be the minimum number required to certify the autoclave. The autoclave shall also be capable of 600°F and 85 psig with pressure control capability of ± 5 psig with nitrogen and/or air atmosphere, and a minimum heat up rate of 3°F/min. Pressure gages are required to monitor autoclave pressure and pressure build-up in the vacuum bag throughout the cure cycle.

4.2 Vacuum pumping system and vacuum gage to perform leak checks inside and outside the autoclave.

4.3 Air circulating oven with 250°F capability certified per P.S. 23401 Class 3A for drying both cleaned and primed parts. Air circulating oven with 400°F capability certified per P.S. 23401 Class 1A for bonding the spar assembly. Also, air circulating oven with 600°F capability certified per P.S. 23401 Class 3C for post curing composite parts.

4.4 Oven, air circulating type, capable of 550°F (minimum), certified per P.S. 23401, Class 1B, for performing elevated temperatrue tests on process control specimens.

4.5 Mechanical property test equipment for testing process control specimens at room temperature and 550°F.

4.6 Refrigeration facilities for storing adhesives and primers at 0 ± 10°F.

4.7 Liquid honing equipment per P.S. 12045.

4.8 Alkaline cleaning equipment per P.S. 12030.

4.9 Bonding fixtures which permit direct fluid pressure on at least one side of the assembly and allows even heating of the assembly from both top and bottom. The maximum thermal expansion allowed for the bonding fixture is 7.0 x 10^{-6} in/in/°F over a temperature range of 70 to 600°F.

4.10 Diamond cutting wheels and routers.

4.11 Thermostatically controlled heat iron (150°F maximum).

5.0 REQUIREMENTS

5.1 GENERAL

5.1.1 The initial fabrication of any composite part shall be witnessed and approved in writing by the Material and Process Development Department. Fabrication as approved by Material and Process Development shall not be changed without written approval from Material and Process Development Department.

5.1.2 Personnel who fabricate composite parts must demonstrate by passing applicable written and/or practical proficiency tests, that they possess the skills and knowledge necessary to ensure acceptable workmanship on the part to be fabricated. A list of personnel qualified to the above requirements shall be maintained.

5.1.3 Thermocouples

(a) Production part - A minimum of 4 thermocouples shall be used for each production part. They shall be located at opposite ends and sides of the part. For production parts of varying thickness, the necessary number of additional thermocouples shall be used to insure uniform heating in all sections of the part. When the number and location of thermocouples has been established using the above criteria, a sketch shall be made and used for each successive part.

(b) Process control specimens - A minimum of one thermocouple shall be placed adjacent to each process control panel. A single thermocouple shall be attached to the center of the lap shear process control panel.

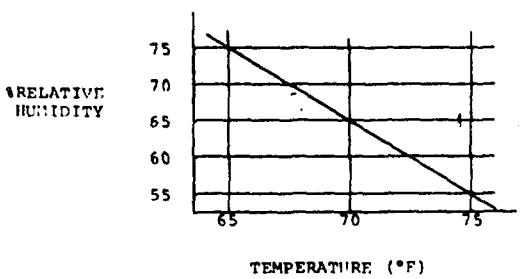
5.1.4 Cleaning control coupons (test specimens used to evaluate the effectiveness of cleaning and etching solutions) shall be fabricated per Figure 1 to meet the R.T. requirements in Table I. Parts cleaned at the same time as the cleaning control coupons cannot be used in the assembly until cleaning control coupons pass Table I requirements.

5.1.5 Extreme caution must be exercised throughout the fabrication sequence to prevent damage or contamination to any individual material used in the assembly or the completed assembly.

5.2 MANUFACTURING ENVIRONMENTS

5.2.1 Cleaning, priming and oven drying operations shall be performed where there is no fume producing machinery or combustion engines in the immediate area. In addition, the presence of these fumes shall be minimized in the cleaning, priming and oven drying areas by performing these operations in areas which are completely enclosed or isolated from the surrounding shop area. Sufficient make-up air shall be introduced to maintain a slight positive pressure. Doors shall be kept closed when not in use.

5.2.2 The assembly area shall meet the requirements of P.S. 20509 except the temperature shall be 65 to 75°F and the relative humidity shall not exceed the following values.



5.3 ADHESIVES AND BONDING

5.3.1 Titanium components of the composite laminate shall be cleaned and primed prior to splice bonding. Priming shall be accomplished within 48 hours after cleaning and bonding within 21 days after priming.

5.3.2 Boron polyimide skins shall be grit blasted prior to bonding. The grit blasted surface shall have a dull, matte surface but shall not remove all the resin and expose boron fibers.

5.3.3 Each batch or shipment of Metlbond 840, FM-34 and FM-29 adhesives and Narmco 800 II and BR-34 primers shall be qualified upon receipt at MCAIR to meet the requirements of Table I. These materials shall be requalified prior to their usage if more than 90 days have elapsed since the materials were qualified. This requalification shall be good for 90 days.

5.3.4 Qualified Metlbond 840, FM-29 and FM-34 adhesives shall be stored at 0 + 10°F upon receipt and when not in use. Each shall be packaged in a manner similar to the supplier's packaging technique. They shall not be removed from their sealed package, after removal from refrigeration, until moisture ceases to condense on the package surface.

5.3.5 Qualified Narmco 800 II and BR-34 adhesive primers shall be stored at 0 + 10°F upon receipt and when not in use. The primer container shall not be opened or the primer mixed, after removal from refrigeration, until moisture ceases to condense on the container surface.

5.3.6 A record shall be kept of the batch and roll number for each adhesive used in any individual composite part. A record shall also be kept of the primer batch and container number.

5.3.7 Adhesive splice joints shall not overlap or have a gap greater than 1/32 inch.

5.3.8 All personnel shall wear clean white cotton gloves when handling cleaned or primed unwrapped parts, film adhesives or parts with film adhesive applied.

5.3.9 Manufacturing and Quality Assurance personnel working on or inspecting the assembly at any time prior to the start of each cure cycle shall wear clean shop coats and caps.

5.3.10 An acceptable verifilm check shall be run prior to bonding the bottom and top skins to the assembly.

5.3.11 The thermal lag between the top and bottom skins can be a maximum of 30°F.

5.3.12 The quality control specimens bonded with the assembly shall meet the requirements of Table I.

5.3.13 Qualification and quality control testing at elevated temperature shall be accomplished in an air circulating oven.

6.0 PROCEDURES

6.1 PREPARATION FOR BONDING - Perform the operations listed in this paragraph when specified in Paragraphs 6.2, 6.3 and 6.5.

6.1.1 Preparation of Titanium Parts for Bonding

NOTE: Fabricate and test cleaning control coupons (Paragraph 5.1.4) before bonding parts .

6.1.1.1 Solvent wipe the part with MEK to remove grease or oil contamination. Do not vapor degrease.

6.1.1.2 Liquid hone the part per P.S. 12045. If alkaline cleaning will not be performed within 2 hours after liquid honing, protect the part by wrapping with wax-free Kraft paper.

6.1.1.3 Alkaline clean the part per P.S. 12030, Type III. Immerse the part immediately in Pasa-Jell solution or the part may be air dried for 20 minutes maximum prior to immersion in Pasa-Jell.

6.1.1.4 Immerse the part in a room temperature Pasa-Jell solution of the composition defined below for 15 to 20 minutes. Remove and rinse the part thoroughly in room temperature tap water. Follow with a thorough room temperature deionized water rinse. Check for water-break free surface. If water-break occurs, reclean the part per Para. 6.1.1.

MATERIAL	SMALL BATCH	LARGE BATCH
Pasa-Jell 107-C7	544 ml.	14.3 gal.
Concentrated Nitric Acid (HNO_3)	1,163 ml.	30.7 gal.
Deionized Water	1 gal.	100.0 gal.
Chromic Acid	165 grams	36.5 lbs.

6.1.1.5 Dry the part at 100 to 150°F for 30 minutes in an oven certified per P.S. 23401, Class 3A. Protect the part by wrapping in wax-free Kraft paper if priming is not to be performed immediately after drying. If priming is not performed within 48 hours after drying, reclean the part per Paragraph 6.1.1.

6.1.1.6 Make sure the Narmco 800 II primer is at room temperature before opening the container or mixing the primer. Record the primer batch and container number.

6.1.1.7 Agitate or stir the Narmco 800 II adhesive primer by mechanical means to insure a homogeneous mixture. Do not use primer containing lumps or indication of gelation.

6.1.1.8 Brush apply a smooth uniform film of Narmco 800 II adhesive primer on all surfaces of the part intended for bonding. A dried film thickness of 0.001 to 0.002 inch is required. Check the thickness by thickness measurements of the dried film per P.S. 21239.

6.1.1.9 Rack the primed part in such a manner as to avoid contamination of any surface intended for bonding. If several parts are involved, also provide for adequate ventilation between the parts. Air dry the primer a minimum of 60 minutes at room temperature.

6.1.1.10 Oven dry the air dried part at 200 + 25°F for 30 minutes in a well ventilated oven certified per P.S. 23401, Class 3A. Attach thermocouples to surfaces which will not be bonded. If several parts are involved, attach thermocouples to parts at opposite ends of the rack. Start dry time after coolest thermocouple reaches the required temperature. Record time versus temperature data for the part.

NOTE: The use of thermocouples may be waived after a heat cycle has been established for the particular oven used.

6.1.1.11 Protect the oven dried part from contamination and bond within 21 days after completion of the oven drying operation. If the part is not bonded within 21 days consult Material and Process Development.

6.1.2 Preparation of Boron/Polyimide Skin for Bonding

6.1.2.1 Clean the boron/polyimide skins immediately prior to bonding by grit blasting with 220 grit or finer particles at a line pressure of 15-20 psi holding the nozzle 8-12 inches from the part. USE EXTREME CARE SO THAT THE RESIN IS NOT REMOVED UNEVENLY. Use sand, glass beads or aluminum oxide particles. Keep the grit blasting nozzle (or the part) moving constantly during this operation. Continue until the boron/polyimide surface takes on a dull, gray, matte appearance.

6.1.2.2 Remove from the grit blasting facility and rinse with tap water.

6.1.2.3 Force dry at 175 + 25°F for 30-45 minutes. After the drying operation, wrap the boron/polyimide skin in clean, wax-free kraft paper. Handle only with clean cotton gloves from the start of cleaning through completion of all bonding operations affecting that particular skin.

6.1.3 Preparation of Graphite/Polyimide Components for Bonding

6.1.3.1 Sand the bonding surface of the graphite/polyimide components with 140 to 200 grit sandpaper.

6.1.3.2 Clean the sanded surface with a clean rag dampened with clean MEK. If cheesecloth is used rinse cheesecloth with MEK before use.

6.1.3.3 Allow the cleaned surface to air dry for a minimum of 30 minutes.

6.1.4 Preparation of Fiber Glass/Polyimide Core for Bonding

6.1.4.1 Dilute the BR-34 primer as follows:

BR-34 primer	100 parts by volume
N-Methyl Pyrrolidone (NMP)	25 parts by volume

6.1.4.2 Apply a heavy brush coat, 0.002 to 0.003 inch thick, of BR-34 primer to the fiber glass/polyimide core, brushing in four directions to ascertain that a thorough coat of primer is applied to the core.

6.1.4.3 Air dry the primer a minimum of 30 minutes. Then oven dry as follows:

Heat to 210-230°F and hold for 30-40 minutes.
Heat to 400-420°F at 5-10°F/min.
Hold at 400-420°F for 40-50 minutes.

6.1.4.4 Wrap the primed core in wax-free Kraft paper until ready for use.

6.1.5 Preparation of Steel Rib for Bonding

6.1.5.1 Liquid hone the bonding surface of the steel rib per Paragraphs 6.1.1.1 and 6.1.1.2.

6.1.5.2 Dry the part at 100 to 150°F for 30 minutes in an oven certified per P.S. 23401, Class 3A.

6.2 BONDING OF SPAR SUB-ASSEMBLY

6.2.1 Install the titanium drive rib and upper balance weight skins, graphite/polyimide spar and upper closure, steel rib, and the three titanium hinge fittings on the bonding jig per the Engineering drawing. Fasten the bonding jig clamps and check for uniform and complete contact of the bonding surfaces.

6.2.2 Disassemble the sub-assembly, clean and prime the titanium drive rib, upper balance weight skins, three titanium hinge fittings and six lap shear specimens (Figure 1) per Paragraph 6.1.1.

6.2.3 Clean the bonding surfaces of the graphite/polyimide spar and upper closure per Paragraph 6.1.3.

6.2.4 Clean the steel rib per Paragraph 6.1.5.

6.2.5 Place one ply of Metlbond 840 adhesive on the bonding surface of the drive rib, spar, skins, upper closure, rib and three hinge fittings and then assemble on the bonding jig per the Engineering drawing. Fasten all bonding jig clamps and check for complete and uniform contact of all bonding surfaces.

6.2.6 Assemble 6 single lap shear specimens per Figure 1. Insert the specimens in a bonding jig and set the pressure at 60 psi.

6.2.7 Attach one thermocouple to the titanium drive rib, one thermocouple to the graphite/polyimide spar and one thermocouple to one of the lap shear specimens.

6.2.8 Heat the assembly and lap shear specimens in an oven as follows:

Heat to 340-360°F at 3-4°F/min.
Hold at 340-360°F for 120-130 minutes
Cool to 125°F or less at 3-4°F/min.

6.2.9 Post-cure the lap shear panels as follows:

Place in an autoclave or other suitable closed container and purge with nitrogen.

Heat to 340-360°F at 3-4°F/min.

Hold at 340-360°F for 30-40 minutes

Heat to 390-410°F at 3-4°F/min.

Hold at 390-410°F for 60-70 minutes

Heat to 440-460°F at 3-4°F/min.

Hold at 440-460°F for 60-70 minutes

Heat to 490-510°F at 3-4°F/min.

Hold at 490-510°F for 60-70 minutes

Heat to 565-585°F at 3-4°F/min.

Hold at 565-585°F for 110-120 minutes

Cool to 125°F or less at 3-4°F/min.

6.2.10 Test the lap shear panels per Paragraph 7.4.2.

6.3 BONDING OF SPAR ASSEMBLY

6.3.1 Install the spar sub-assembly on the bonding jig and then fit the core insert and the titanium fairing on the spar sub-assembly. Fasten the bonding jig clamps and check for uniform and complete contact of all bonding surfaces. Disassemble and clean the details.

6.3.2 Clean and prime the titanium fairing and six lap shear specimens (Figure 1) per Paragraph 6.1.1.

6.3.3 Prime the polyimide core insert per Paragraph 6.1.4.

6.3.4 Place one ply of FM-29 adhesive on the bonding surface of the core insert and one ply of Metlbond 840 adhesive on the bonding surface of the titanium fairing and assemble on the spar sub-assembly per the Engineering drawing. Fasten all clamps on the bonding jig and then check for complete and uniform contact of all bonding surfaces.

6.3.5 Assemble 6 single lap shear specimens per Figure 1. Insert the specimens in a bonding jig and set the pressure at 60 psi.

6.3.6 Attach one thermocouple to the titanium fairing and one thermocouple to one of the lap shear specimens.

6.3.7 Heat the assembly and lap shear specimens in an oven per Paragraph 6.2.8.

6.3.8 Post-cure the lap shear specimens per Paragraph 6.2.9.

6.3.9 Test the lap shear specimens per Paragraph 7.4.2.

6.4 BONDING OF BOTTOM SKIN AND CORE TO SPAR ASSEMBLY

6.4.1 Cut one piece of Verifilm 641 to the size of the bonded assembly. Place a sheet of 0.0015 in. thick Mylar on each side of the Verifilm 641, and extend it 0.25 in. beyond the edge of the Verifilm 641. The Mylar coated Verifilm is hereafter referred to as Verifilm 641.

6.4.2 Put the Verifilm 641 film on top of the bottom skin where the adhesive would normally be placed and then place the skin on the bonding fixture. Place the spar assembly and the honeycomb core over the Verifilm 641 in the same position relative to the bottom boron/polyimide skin as required by the Engineering drawing. Cover the honeycomb with perforated aluminum sheets and build up edgemember areas so that autoclave pressure is evenly distributed on the Verifilm 641. Bag the assembly with Mylar film using fiberglass to prevent the Mylar from being punctured by the assembly. Attach thermocouples to the assembly prior to bagging.

6.4.3 Pull an 8-10 in. Hg vacuum on the bag and correct any leaks. Place the assembly in the autoclave at 8-10 in. Hg vacuum and check for any leaks. Correct any leaks found. Pressurize the autoclave to 50 ± 5 psig. Vent the bag to atmospheric pressure when 5 psig autoclave pressure is reached. Heat the autoclave to 290 ± 10°F at 3.0-4.0°F/min. Hold at temperature for 60 ± 10 min. and cool down under pressure to 125°F or less at 3-4°F/min.

6.4.4 Inspect the Verifilm 641 for imprint of the core. Verify that the Verifilm 641 has not left a residue on any assembly components. If there is any residue, remove it with an MEK dampened cheesecloth.

6.4.5 Clean the boron-polyimide skin per Paragraph 6.1.2.

6.4.6 Prime the fiber glass/polyimide core per Paragraph 6.1.4.

6.4.7 Clean the bonding surfaces of the graphite/polyimide spar and upper closure per Paragraph 6.1.3.

6.4.8 Heat tack FM-34 adhesive on the bottom skin bonding surface which will mate against the core. Place the bottom skin on the tool and locate properly. Place one ply of Metlbond 840 adhesive on the edgemember to skin bonding surface of the spar assembly.

6.4.9 If the Verifilm 641 check per Paragraph 6.4.1 through 6.4.4 showed a need for one additional layer of adhesive, place a layer of Metlbond 840 and/or FM-34 adhesive, as applicable, in the appropriate place(s). Requirement for more than one extra ply of adhesive is subject to Engineering disposition. Record areas of more than one layer of adhesive for later use in analysis of NDT results.

6.4.10 Place one layer of FM-29 adhesive on the edge member bonding surfaces of the core.

6.4.11 Locate the core on the assembly per the Engineering drawing, splicing the core with FM-29 adhesive.

6.4.12 Place a 0.040 in. aluminum sheet (perforated with 1/16 in. diameter holes on 1/2 in. centers) on top of the assembly to act as a pressure plate. Assemble 6 single lap shear specimens per Figure 1, a titanium skin-fiberglass/polyimide honeycomb core splice test panel per Figure 2, and a boron/polyimide skin - fiberglass (polyimide honeycomb core flatwise tension panel per Figure 3 on the surface of the bonding fixture. Place a minimum of six thermocouples on the assembly. Place one thermocouple on each quality control panel, one thermocouple on the tool, and three thermocouples as near the bond areas to the assembly as possible without contaminating areas to be bonded and about equally spaced from each other. Install style 1000 dry glass cloth over the lay-up, making it especially heavy in areas where vacuum lines are located and where metal parts may puncture the vacuum bag. Place a sheet of vacuum bag material, Paragraph 3.2.10, over the entire lay-up and seal to the bonding fixture with 9151 Sealant Tape. Pull 27-29 in. Hg vacuum on the assembly to check for any bag leaks. Correct any leaks found.

6.4.13 Place the bonding fixture in the autoclave and attach the thermocouples, vacuum lines and static lines to the connections inside the autoclave. Pull 27-29 in. Hg vacuum inside the bag and check for any leaks. Correct any leaks found. Close the autoclave and pressurize to 50 ± 5 psig. Heat the sub-assembly per Paragraph 6.2.8, maintaining full vacuum plus 50 psig autoclave pressure until 125°F is reached during the cool down. Record the autoclave pressure and all thermocouple readings every 15 minutes from the start of autoclave pressurization to assembly removal from the autoclave.

6.4.14 Remove the sub-assembly and quality control panels from the bonding fixture. Handle only with clean cotton gloves until completion of all bonding operations.

6.4.15 Prime the honeycomb core in the trailing edge per Paragraphs 6.1.4.1 and 6.1.4.2.

6.4.16 Chop FM-29 adhesive into small pieces and loosely fill into the core trailing edge as specified on the Engineering drawing.

6.4.17 Place a sheet of CHR 3TLL release cloth, then one ply of 1000 dry glass cloth, a 0.040 in. aluminum sheet (perforated with 1/16 inch diameter holes on 1/2 inch centers), and finally metal weights (to prevent the foaming adhesive from moving the aluminum sheet) over the core surface filled with FM-29 adhesive.

6.4.18 Cure the primed core and FM-29 adhesive per Paragraph 6.1.4.3.

6.4.19 Post-cure the quality control specimens and the rudder assembly per Paragraph 6.2.9.

6.5 CORE MACHINING

6.5.1 Machine the honeycomb core to the correct height and shape as designated by the Engineering drawing. Be careful to prevent contamination, especially with oil, of any area that still requires bonding. Contaminated surfaces are subject to Engineering disposition.

6.5.2 Vacuum clean the sub-assembly to remove all loose chips and dust.

6.6 BONDING OF TOP SKIN TO SUB-ASSEMBLY

6.6.1 Assemble the verifilm 641 sheet per Paragraph 6.4.1. Place over the top of the bonded sub-assembly, and align it so that areas that are to be bonded are covered with Verifilm 641.

6.6.2 Place the top boron/polyimide skin in place, and bag the assembly using Style 1000 dry glass cloth bleeder and a Mylar bag. Seal the Mylar bag to the tool with 9151 Sealant Tape.

6.6.3 Repeat Paragraph 6.4.3.

6.6.4 Repeat Paragraph 6.4.4.

6.6.5 Prime the honeycomb core per Paragraphs 6.1.4.1 thru 6.1.4.3.

6.6.6 Clean the graphite/polyimide bonding surfaces of the sub-assembly per Paragraph 6.1.3.

6.6.7 Clean the top skin per Paragraph 6.1.2.

6.6.8 Place the bonded sub-assembly on the bonding fixture in the proper location.

6.6.9 Place one ply of Metlbond 840 adhesive on the edgemember to skin bonding surface of the spar assembly. If the Verifilm 641 check per Paragraph 6.6.1 through 6.6.4 showed a need of one additional layer of adhesive, place a layer of Metlbond 840 in the appropriate place(s). Requirement of more than one extra ply of adhesive is subject to Engineering disposition. Record areas of more than one layer of adhesive for later use in analysis of NDT results.

6.6.10 Heat tack FM-34 adhesive on the core bonding surface. If the Verifilm 641 check per Paragraph 6.6.1 through 6.6.4 showed a need for one additional layer of adhesive, place a layer of FM-34 adhesive in the appropriate place(s). Requirement for more than one extra ply of adhesive is subject to Engineering disposition. Record areas of more than one layer of adhesive for later use in analysis of NDT results.

6.6.11 Locate the top skin in its proper place over the subassembly. Hold in place with Mylar tape, or equal.

6.6.12 Repeat Paragraph 6.4.12 (no aluminum sheet required).

6.6.13 Repeat Paragraphs 6.4.13 and 6.4.14, and then post-cure the quality control panels and the assembly per Paragraph 6.2.9.

6.7 WING COMPRESSION PANEL

6.7.1 The Wing Compression Panel is fabricated using the same fabrication and quality assurance procedures as the F-4 Polyimide Composite Rudder. The only difference occurs in the timing of several of the fabrication procedures.

6.7.2 The fabrication sequence is as follows:

6.7.2.1 Machine the HRH-327 honeycomb core to the dimensions of the Engineering drawing and per the requirements of Para. 6.5, and prepare for bonding per Para. 6.1.4.

6.7.2.2 Prepare the graphite/polyimide spacer blocks for bonding per Para. 6.1.3.

6.7.2.3 Bond the graphite/polyimide spacer blocks to the periphery of the HRH-327 honeycomb core with FM-29 adhesive per Para. 6.3 and the Engineering drawing.

6.7.2.4 Prepare both boron/polyimide skins for bonding per Para. 6.1.2.

6.7.2.5 Bond both boron/polyimide skins to the graphite/polyimide edgemember - HRH-327 honeycomb core subassembly with FM-34 adhesive per Para. 6.4 and the Engineering drawing. Ultrasonic NDT per P.S. 21211.4.

6.7.2.6 Prepare the outside surface of the boron/polyimide skins and the bottom surface of the graphite/polyimide hat flanges for bonding per Para. 6.1.2 and 6.1.3 respectively.

6.7.2.7 Bond the graphite/polyimide hats to the boron/polyimide sandwich panel with Metlbond 840 adhesive per Para. 6.2 and the Engineering drawing. Ultrasonic NDT per P.S. 21211.4.

6.7.8 Perform all pertinent process control tests as required by Para. 6.1 through 6.6.

7.0 QUALITY ASSURANCE PROVISIONS

7.1 RAW MATERIAL ACCEPTANCE

7.1.1 Each batch of Metlbond 840, FM-29 and FM-34 adhesives and Narmco 800 II and BR-34 primers shall be tested and accepted per Table I.

(a) Acceptable material shall have a dated Quality Assurance acceptance tag affixed which shows date for requalification.

(b) The materials shall be requalified prior to their usage if more than 90 days have elapsed since the materials were qualified. This requalification shall be good for 90 days.

7.2 EQUIPMENT

7.2.1 Instrumentation used to control processes shall be calibrated per P.S. 20503.

7.2.2 Ovens, autoclaves, furnaces and temperature control systems shall be certified to P.S. 23401. The bonding oven shall be certified to P.S. 23401, Class 1A, the autoclave to P.S. 23401, Class 3C, the drying oven to P.S. 23401, Class 3A, and the testing oven to P.S. 23401, Class 1B.

7.3 PROCESS CONTROL - Quality Assurance shall verify conformance to the following:

(a) Materials used have a current Quality Assurance acceptance tag affixed which shows date for requalification.

(b) Matlbond 840, PM-29 and PM-34 adhesives and Narmco 800 II and BR-34 primers are stored in their original sealed shipping containers at 0 ± 10°F until ready for use.

(c) Applicable process equipment has a current Quality Assurance calibration tag affixed.

(d) Adhesive is conditioned to ambient temperature in closed containers before use.

(e) All adhesive splices are butt splices with a maximum 1/32 inch gap.

(f) The Verifilm 641 does not leave a residue that was improperly cleaned from the assembly.

(g) Deficiencies shown by the Verifilm 641 prefit check are corrected.

(h) All materials are properly prepared for bonding and then immediately wrapped in wax-free kraft paper.

(i) All personnel handling cleaned, unwrapped parts and/or adhesives through the layup operation, and all support personnel coming in direct contact with cleaned parts and/or adhesives wear clean, white hats and coats, and clean white cotton gloves.

(j) The grit blast operation on the boron/polyimide skins results in a dull, matte surface, but does not remove all resin and expose boron fibers.

(k) Cure pressure was maintained at 50 ± 5 psi autoclave pressure and 27-29 inches of Hg vacuum.

(l) Cure temperature was 350 ± 10°F, and the heat-up and cool down was at a rate of 3-4°F/min.

(m) During cure there was a minimum of three thermocouples functioning at all times.

(n) Cure time didn't start until all thermocouples reached minimum temperature and was a total of 110-120 minutes long.

(o) Assembly was cooled to 125°F or less under full pressure.

(p) Thickness of Narmco 800 II primer was checked per P.S. 21239.

(q) All bonding operations were performed by qualified personnel (Paragraph 5.1.2).

(r) The temperature in the bonding room is held between 65-75°F and the relative humidity in the bonding room is held within the limits shown in Paragraph 5.2.2.

7.4 STRUCTURAL ASSEMBLY ACCEPTANCE

7.4.1 Nondestructive Testing

(a) X-ray inspect per P.S. 21206.3, quality level per P.S. 21233, Class C, for node bond separation.

(b) X-ray inspect per P.S. 21206.3 and ultrasonic inspect per P.S. 21211.4, quality level per P.S. 21233, Class C, for voids between core and edge member and voids between the core-to-core bond.

(c) X-ray inspect per P.S. 21206.3 and ultrasonic inspect per P.S. 21211.4, quality level per P.S. 21233, Class C, for voids between skin to edge member bond and voids between skin to core bond.

(d) Defects found by NDT are subject to Engineering disposition.

7.4.2 Control Panel Testing

7.4.2.1 The spar sub-assembly bond and the spar assembly bond each requires six (6) lap shear specimens per Figure 1 to be fabricated at the same time as the production assembly. These specimens shall be fabricated with the same adhesive batch and cured at the same time as the production assembly. The lap shear specimens shall be tested per Figure 1 to meet the requirements of Table I.

7.4.2.2 Each sandwich assembly bonding operation requires process control test panels to be fabricated at the same time. These panels are to be fabricated with the same adhesive batch and on the same bonding fixture as the production assembly. The following specimens are required for each sandwich assembly bond:

(a) Six (6) lap shear specimens per Figure 1 to meet the requirements of Table I.

(b) Six (6) flatwise tension specimens per Figure 3 and Figure 4 to meet the requirements of Table I.

(c) Six (6) honeycomb core shear specimens per Figure 2 to meet the requirements of Table I.

7.4.2.3 Perform all elevated temperature tests in an air circulating oven. Specimens that are tested at elevated temperatures shall be at temperature 10 minutes prior to test. The test specimen temperature shall be controlled to ± 5°F.

7.4.2.4 Failure to meet the requirements of Table I will require Engineering disposition of the bonded assembly cured with the specimens. All use of the adhesive batch shall cease until the adhesive is tested and requalified.

8.0 SAFETY

8.1 ORGANIC SOLVENTS

8.1.1 Description: The following solvents and/or solvent bearing materials are used in small quantities per this specification.

(a) Methyl Ethyl Ketone: A colorless, thin solvent. Flammable and toxic.

(b) Narmco 800 II primer: A brownish thin liquid, flammable and toxic.

(c) BR-34 primer: A thick liquid, flammable and toxic.

(d) N-Methyl Pyrrolidone: A colorless, thin solvent. Flammable and toxic.

8.1.2 First Aid: In event of eye or skin contamination:

(a) Flush the exposed area with large amounts of water.

(b) Remove contaminated clothing.

(c) Secure first aid.

8.1.3 Toxicology: Avoid prolonged breathing of solvent vapors.

8.1.4 Fire Hazard: Flammable solvents are extremely easy to ignite and shall not be used in the vicinity of smoking, sparks or open flames.

8.1.5 Handling and Storage:

(a) Wear safety glasses (or goggles) and rubber gloves while working with solvents.

(b) Do not use solvents in confined areas unless specifically authorized, since fumes are generally toxic, flammable, and explosive.

(c) Store and handle solvents in properly labelled safety containers.

(d) Dispose of all rags in flammable solvent areas in special containers used only for this purpose.
NOTE: The preceding are minimum safety precautions. Personnel using these materials should be familiar with the more detailed precautions provided in P.S. 20002.

8.2 NON-SOLVENT ORGANIC MATERIALS

8.2.1 Description: The following non-solvent organic materials are used per this specification.

(a) Metlbond 840 Film Adhesive: A glass fabric supported, grey film. Not a fire hazard, but is toxic.

(b) FM-34 Film Adhesive: A glass fabric supported, greenish brown film. Not a fire hazard, but is toxic.

(c) FM-29 Adhesive: A non-supported, tan film. Not a fire hazard, but is toxic.

8.2.2 Precautions: The safety precautions specified in Paragraph 8.1 shall apply to these materials with the exception of those precautions related to fire hazards.

8.3 ACIDS

8.3.1 General Precautions

(a) Acid resistant gloves, aprons, chemical goggles and boots must be worn when handling hazardous chemicals.

(b) Use chemicals and solutions only as stated in this specification.

(c) Always add acid to water when diluting or mixing chemicals or solutions.

(d) Avoid inhaling vapors. Wear McDonnell approved respirators when necessary.

(e) Correctly label and maintain the identity of all containers as to their contents.

(f) Incompatible Materials - Isolate from incompatible materials as specified per McDonnell P.S\ 20002 under both specific and general precautions.

8.3.2 First Aid: In the event of eye or skin contaminations:

(a) Flush the exposed area with large amounts of water for 15 minutes.

(b) Remove contaminated clothing.

(c) Secure first aid.

8.3.3 Pasa-Jell 107-C7 Solution

(a) Description: A thin, orange liquid.

(b) Toxicology: Liquid contains acid and dichromate which can irritate and burn eyes and skin.

(c) Fire Hazard: May cause ignition when in contact with flammable or combustibel materials.

(d) Storage: Store in original shipping containers.
CAUTION: Do not allow Pasa-Jell 107-C7 or rinse water containing Pasa-Jell 107-C7 to come in contact with MEK or other solvents.

8.3.4 Nitric Acid - HNO_3

(a) Description - Transparent, colorless or yellowish fuming, suffocating, corrosive liquid.

(b) Toxicology - Nitric acid vapor is highly irritating to the skin, eyes and mucous membranes.

(c) Fire-Hazard - Nitric acid is a powerful oxidizing agent. Avoid contact with reducing agents such as alcohol or petroleum solvents, as the mixture is explosive.

8.3.5 Chromic Acid

(a) Description - Red crystals

(b) Toxicology - Chromates attach the skin and mucous membranes.

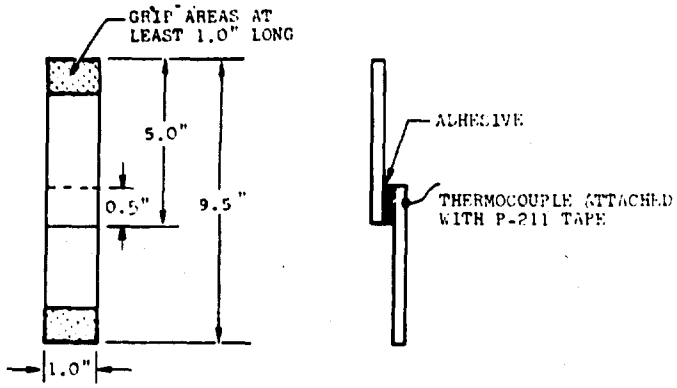
(c) Handling and storage - Isolate chromates from flammable solvents, and other organic matter such as wood, paper, alcohol and petroleum compounds as the mixture is flammable and explosive.

9.0 NOTES

9.1 Vendors or subcontractors needing this specification should direct their request for copies to the attention of McDonnell Aircraft Company (MCAIR), St. Louis, Purchasing or Subcontracting, as applicable. Information pertaining to the technical aspect of this specification can be obtained from MCAIR Material and Process Development Department (Dept. 372).

10.0 REFERENCE PUBLICATIONS

None applicable



NOTES:

(1) Specimens are 6Al-4V annealed titanium 0.040" (min.) x 1.0" x 5.0". Clean and prime per Section 6.0 of this specification with the production parts that will be bonded with the specimens.

(2) Apply Metlbond 840 adhesive and cure per Section 6.0 of this specification.

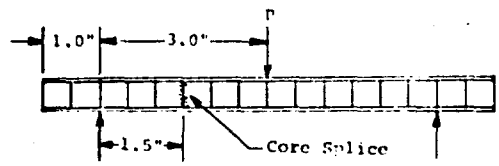
(3) Use self-aligning grips and load at 600-700 lbs/min.

(4) Six (6) specimens required. Test three (3) at room temperature and three at 550°F ± 5°F after holding at temperature for 10 minutes. Heat specimens in air circulating oven only.

(5) Calculate lap shear strength as follows:

$$F_s = \frac{P}{A}$$ Where F_s = Lap Shear Strength (psi)
P = Failing Load (lb)
A = Bond Line Area (in^2)

FIGURE 1 - LAP SHEAR SPECIMEN AND TEST



NOTES:

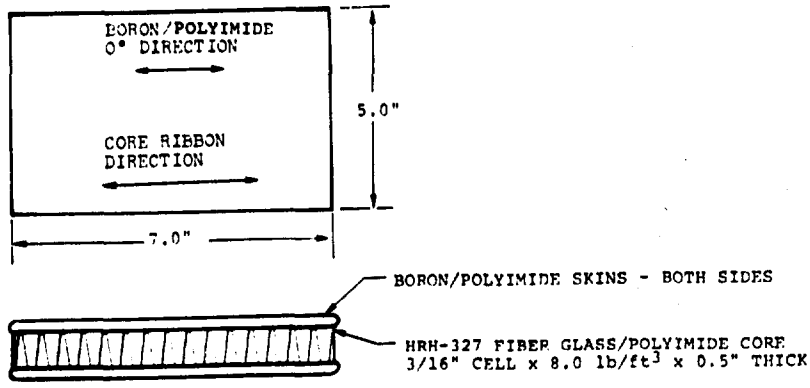
(1) Sandwich panel consists of 0.010" x 1.0" x 8.0" 6Al-4V annealed titanium skins cleaned per Section 6.0 of this specification, .5" thick x 1" wide, 3/16" cell, 8.0 lb/ft^3, HRH-327 fiber glass/polyimide core cleaned and primed per Section 6.0 of this specification, with FM-34 adhesive for core to skin bond and FM-29 adhesive for core splice bond. Sandwich to be cured per Section 6.0 of this specification.

(2) Six (6) specimens required. Test three (3) at room temperature and three (3) at 550°F after holding at temperature for 10 minutes. Heat specimens in air circulating oven only.

(3) Calculate sandwich shear strength as follows:

$$F_{cs} = \frac{P}{2wh}$$, where F_{cs} = Sandwich shear strength, psi
P = Failing load, lb.
w = Width, in.
h = Distance between skin centroids, in.
= C + T ; where C = core thickness, in.
T = Skin thickness, in.

FIGURE 2 - SANDWICH SHEAR SPECIMEN AND TEST



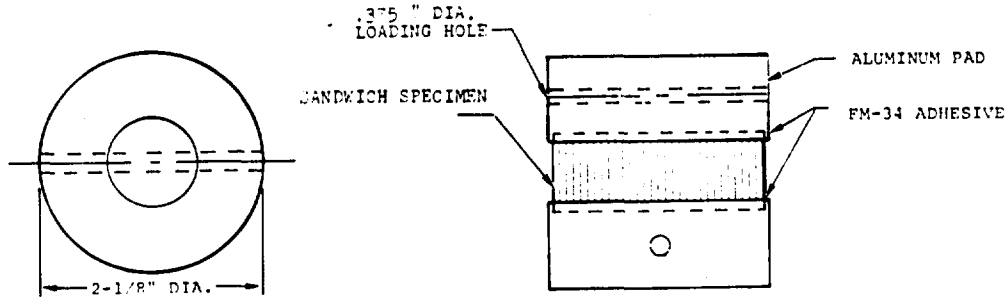
NOTES: (1) Fabricate the boron/polyimide skins per P.S. 14224 with orientation of 0, +45, -45, -45, +45, 0.

(2) Clean and prime per Section 6.0 with the production parts that will be bonded with the panel.

(3) Bond the skins to the honeycomb with FM-34 adhesive per Section 6.0.

(4) Cut out six (6) flatwise tension specimens 2.0" dia. with diamond tooling and test per Figure 4.

FIGURE 3 - FLATWISE TENSION PANEL SPECIMEN



NOTES: (1) Load the specimen through the two (2) 0.375" dia. holes with a self aligning clevice device.

(2) Load at a rate of 1000 - 1200 lb/min. to failure. Record the failure load and the type of failure.

(3) Calculate flatwise tension strength as follows:

$$F_t = \frac{P}{A}$$, where F_t = Flatwise tensile strength, psi
P = Failure load, lb.
A = Bond area, in^2

(4) Use air circulating oven as heat source for 550°F tests.

FIGURE 4 - FLATWISE TENSION SPECIMEN AND TEST

PROPERTY	FAILURE STRESS (SPI)	
	ROOM TEMPERATURE	550°F
Metlbond 840 Adhesive and Narmco 800 II Primer Single Lap Shear (Figure 1)(Minimum Average)	2250	1500
Metlbond 840 Adhesive and Narmco 800 II Primer, Single Lap Shear (Minimum Strength any one Specimen)	2000	1250
FM-34 Adhesive and BR-34 Primer, Flatwise Tension (Figure 4)(Minimum Average)	400	75
FM-34 Adhesive and BR-34 Primer, Flatwise Tension (Figure 4)(Minimum Strength any one Specimen)	300	50
FM-29 Foaming Adhesive, Adhesive Core Splice (Figure 2)(Minimum Average)	200	100
FM-29 Foaming Adhesive, Adhesive Core Splice (Figure 2)(Minimum Strength any one Specimen)	150	75

TABLE I - ADHESIVE INCOMING INSPECTION AND CONTROL SPECIMEN REQUIREMENTS

PREPARED R C Adams — APPROVED _____ Manufacturing

APPROVED _____ Engineering — APPROVED _____ Quality Assurance

APPROVED _____ AFPRO, Engineering — APPROVED _____ Safety and Medical

PRELIMINARY

P.S. 14227

SKINS AND SUBSTRUCTURAL SHAPES, STRUCTURAL, GRAPHITE/POLYIMIDE, FABRICATION AND ACCEPTANCE OF

FABRICATION PROCEDURES

1.0 APPLICATION

1.1 This process specification defines the procedures for fabricating graphite/polyimide non-integrally stiffened skin type structure and structural shapes for use to 550°F.

1.2 This specification is effective upon issue and when specified on an Engineering drawing.

2.0 APPLICABLE DOCUMENTS

2.1 The following specifications (or documents) form a part of this specification to the extent specified herein.

P.S. 20002 - Safety Standards and Information on the Use and Handling of Hazardous Materials

P.S. 20503 - Calibration Laboratories, Facility and Operational Requirements for

P.S. 20509 - Requirements for Class 2 Clean Room

P.S. 21206 - Radiographic Inspection - General

P.S. 21233 - Nondestructive Testing of Adhesive Bonds

P.S. 23401 - Certification of Furnaces and Temperature Control Systems for Heat Treating and Thermal Processing

MMS-335 - Sealant Material, Vacuum Bag

MMS-523 - Graphite/Polyimide Pre-Preg Material

MMS-528 - Glass Fabric, Teflon Coated

3.0 MATERIALS AND/OR SOLUTIONS

3.1 Graphite/Polyimide Pre-preg Material per MMS-523.

3.2 "A" Staged Skybond 703 Polyimide Resin, Whittaker Research and Development, San Diego, Calif.

3.3 Permacel No. 02C Double Sided Tape, 1.0 inch wide, Permacel Tape Co., or equal.

3.4 DK-153 Synthetic Rubber and Sponge Cork (cork dam), 0.5 inch wide, thickness determined by [illegible], Armstrong Cork Co., St. Louis, Missouri

3.5 Y-9054 Lead Foil Tape, 3M Company, or equal.

3.6 9151 Vacuum Bag Sealant Tape per MMS-335, [illegible], 1/32" thick, 1" wide, Schnee-Morehead Polymer Corporation, Irving, Texas.

3.7 Mylar Film, Type A, 0.0015 inch thick, E. I. DuPont Film Division.

3.8 Nylon Film, Capran Type 512-H or 80 Gage 2, Allied Chemical Corp., or equal.

3.9 Teflon squeegee, 3.0 in. x 5.0 in.

3.10 Acrylic plastic sheet, minimum of 0.125 inch thick, Commercial.

3.11 Methyl Ethyl Ketone (MEK), TT-M-261.

3.12 Glass Cloth, Style 120 and 1000, Commercial.

3.13 Teflon Coated Glass Fabric per MMS-528, CHR 3TLL, Connecticut Hard Rubber Co.; TX1050 Teflon Coated Fabric, Fallflex Products Co.; or equal.

3.14 Mold Wiz F-57 Mold Release, or equal.

3.15 Ram 225 and Plastilease 334 Mold Release, Ram Chemical Co., Los Angeles, California.

3.16 M380-10 TFE coated glass fabric, 50-54 in. wide, Dodge Industries; CHR 6TB TFE coated glass fabric, 36 in. wide (minimum), Connecticut Hard Rubber Co.; or equal.

3.17 N-Methyl Pyrrolidone Solvent, Eastman Kodak Chemical Co., New York, New York.

4.0 EQUIPMENT

4.1 Autoclave capable of 400°F (minimum) and 100 psig (minimum) nitrogen and/or air pressurized with pressure control capability of ± 5 psig and a minimum heat up rate of 3°F/min. A continuous temperature recording system and a 50 CFM vacuum pumping system capable of maintaining 200 microns or less of vacuum are required. The autoclave must be certified to P.S. 23401, Class 3C, except as noted in Paragraph 7.1.1.

4.2 Cold trap, capable of condensing all volatiles removed by the vacuum pumping system.

4.3 Oven, air circulating type, capable of 600°F (minimum), certified per P.S. 23401, Class 3C for post curing composite parts.

4.4 Oven, air circulating type, capable of 550°F (minimum), certified per P.S. 23401, Class 1B, for performing elevated temperature tests on process control specimens.

4.5 Diamond cutting wheels and routers with 120 grit or finer diamond particles.

4.6 Carbide tipped carbide drills.

4.7 Paper cutter, X-acto knives, "pizza" cutter or equivalent.

4.8 Steel or aluminum lay-up and curing tool.

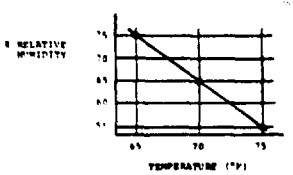
4.9 Thermostatically controlled heat iron (150°F maximum).

5.0 REQUIREMENTS

5.1 The initial fabrication of any component shall be witnessed and approved in writing by the Material and Process Development Department. Fabrication as approved by Material and Process Development shall not be changed without written approval from Material and Process Development Department.

5.2 Fabrication of composite parts shall be performed by qualified personnel. Qualified personnel are those who have demonstrated by passing written and/or practical proficiency tests, that they possess the skills and job knowledge necessary to ensure acceptable workmanship. A list of qualified personnel shall be maintained by Quality Assurance.

5.3 The lay-up area shall meet the requirements of P.S. 20509 except the temperature shall be 65 to 75°F and the relative humidity shall not exceed the following values:



5.4 The top surface of the steel or aluminum tool used for final lay-up and cure of the composite laminate shall have a maximum 80 RHR finish.

5.5 The steel tool plate shall be a minimum of 3.0 inches larger in all edge directions than the dams surrounding the lay-up, plus ample area for the process control panel.

5.6 Graphite/polyimide pre-preg material shall be packaged in a moisture proof plastic bag and stored at 0 ± 10°F when not in use.

5.7 Graphite/polyimide pre-preg material shall remain inside the moisture proof plastic bag a minimum of 30 min. after removing from 0° storage to room temperature working area.

5.8 A record shall be kept of time in and out of 0°F storage for each container of pre-preg material.
NOTE: The container referenced here is the container used by the vendor to facilitate shipment of broadgoods.

5.9 A record shall be kept of the identification number of each individual container of pre-preg material used for any individual composite lay-up.

5.10 The graphite/polyimide pre-preg material shall be placed in the orientation designated on the Engineering drawing.

5.11 Fiber butt joints of the pre-preg material within the composite (end to end type) are forbidden, unless required by the Engineering drawing.

5.12 The edge of any individual piece of pre-preg material in one ply shall not be placed directly over the edge of another piece of pre-preg of a previous ply. Edge joints such as those shall be overlapped a minimum of 0.5 inch unless otherwise required by the Engineering drawing.

5.13 Cut edges of pre-preg terminating inside the laminate shall be uniform and defect free.

5.14 Prepreg material for each ply shall be placed on Mylar templates to the dimensions and orientations specified on the Engineering drawing, and then transferred to the lay-up tool.

5.15 The uncured pre-preg material and composite lay-up shall not be exposed to room temperature in excess of 240 cumulative hours.

5.16 Tools, clamps, etc. shall not be placed on top of the lay-up unless required by the fabrication procedure.

5.17 Manufacturing and Quality Assurance personnel working on or inspecting the lay-up at any time prior to the start of the cure cycle shall wear clean shop coats and caps.

5.18 Edges of composite laminates shall be fabricated a minimum of 0.25 inches oversize for trimming purposes.

5.19 Cut edges of the pre-preg shall be within 0.10 inch of the inside edge of the dam.

5.20 A graphite/polyimide process control laminate shall be fabricated and cured at the same time and under the same vacuum bag as each production part. Size of the laminate shall be 6.0" x 4.0" x 11 plies with fibers parallel to the 6" direction. This laminate shall be evaluated per Section 7.0 and conform to the mechanical property requirements of Table I.

5.21 A minimum of 29" Hg vacuum is required throughout the pre-preg cure cycle.

5.22 Cure cycle heat up rate shall be 3-4°F/min.

6.0 PROCEDURES

6.1 PREPARATION FOR FABRICATION

6.1.1 Identify the orientation of each ply in the composite laminate by marking Mylar templates or referencing the Engineering drawing.

6.1.2 Clean the top of the steel or aluminum tool with an MEK dampened cheese cloth. Allow to air dry for a minimum of 30 minutes.

6.1.3 Cover the tool surface within an area formed by the outer perimeter of the dams with M380-10 or CHR 6TB. If excessive tool curvature is a problem consult Material and Process Development Department for appropriate release material.

6.1.4 Cut a sheet of CHR 3TLL (or equivalent) release film to the same dimensions as the CHR 6TB material. Where necessary, splice the CHR 3TLL (or equivalent) with an FEP Teflon tape to obtain sufficient area.

6.1.5 Cut one layer of 120 dry glass cloth to the same dimensions as the CHR 3TLL (or equivalent) for every three plies of pre-preg material. Use one extra layer of 120 dry glass cloth when the lay-up has one extra ply of pre-preg over multiples of 3 (i.e., 3, 6, 9, 12 etc.). For tapered pre-preg lay-ups the size of the 120 glass cloth will vary with the location of the pre-preg material.

6.1.6 Make sure that the pre-preg cutters (paper cutter, X-acto knives, "pizza" cutter, etc.) are sharp prior to their use.

6.1.7 Remove the pre-preg containers required for each day's fabrication from 0° storage. Keep the pre-preg material in the plastic bag a minimum of 30 minutes after removal from the freezer. Pre-preg material may be stored at room temperature overnight if the remainder is completely used the following day. However, do not allow the pre-preg material to be exposed to temperatures over 0 ± 10°F unnecessarily. Record the cumulative time out of 0 ± 10°F storage for each pre-preg container. Also, record the batch and container number for the pre-preg material used in each lay-up.

6.1.8 Orient the pre-preg broadgoods in the designated direction and cut to the dimensions of the Mylar templates if applicable.

6.2 LAY-UP PROCEDURE FOR FLAT LAMINATES

6.2.1 Remove the adhesive backing sheet off of the cork dams and then place the cork dams on top of the release film with the outside edge of the dam directly over the outside edge of the release material. Make sure all joints are tightly sealed. Make the dams the thickness $^{+0.020}_{-0.000}$ of the expected cured laminate thickness.

6.2.2 Collate each individual ply of pre-preg on the lay-up tool, adding small amounts (approximately 1/8" x 1/8" x 0.001 - 0.002" thick) of Skybond 703 resin at strategic spots on each layer to prevent ply to ply slippage. Use a warm (140-160°F) iron and an intermediate Mylar film to momentarily soften and smooth out each layer of the composite laminate. Keep the warm iron moving at all times, taking care not to degrade the pre-preg by overheating. Place a clean Mylar sheet over areas of the lay-up not being worked on to protect it from damage and contamination.

6.2.3 If a production part is being fabricated, lay-up a process control laminate on the same tool and/or under the same vacuum bag as the structural part using the processing procedures described in Paragraphs 6.2.1 and 6.2.2. Lay-up one laminate 6.0" x 4.0" x 11 plies with filaments parallel to the 6" direction.

6.2.4 Place the CHR 3TLL (or equivalent) film over the top of the completed lay-up and attach to the cork dams with Permacel Double Sided Tape. Make sure the CHR 3TLL, Permacel Tape and dam are in intimate contact.

6.2.5 Place the correct number of 120 glass cloth layers as specified in Paragraph 6.1.5 over the CHR 3TLL (or equivalent). Attach the 120 glass cloth to the tool with Y9050 tape.

6.2.6 Place a Mold Wiz F-57 coated 0.040" aluminum sheet perforated with 1/16 inch diameter holes on 1/2 inch centers on top of the production assembly to act as a pressure plate. Place Mold Wiz F-57 coated 0.040" perforated aluminum sheets over each of the process control panels. Tape the pressure plates to the cork dams to insure that the plates will not ride on the dams during the cure cycle. Place 2-5 layers of style 1000 dry glass cloth over the top of the lay-up and extend beyond the dam.

6.2.7 Place a layer of Mylar film on top of the entire lay-up (i.e., structural part and process control laminates) and extend several inches beyond the dams. Locate the vacuum and static lines adjacent to the lay-ups and inside the sealed edge of the vacuum bag. Seal the vacuum bag to the tool with 9151 Pink Sealing Tape. See Figure 1 for typical laminate lay-up cross section.

6.2.8 Use a minimum of 4 thermocouples for each part. Locate the thermocouples at opposite ends and sides of the part next to the trim area of the laminate. For production parts of varying thickness, add additional thermocouples to insure uniform heating in all sections of the part. Place a minimum of one thermocouple adjacent to each process control laminate lay-up.
NOTE: Do not place any thermocouples directly on the surface of the pre-preg lay-up.

6.2.9 Leak check the lay-up per Paragraph 6.4. At the conclusion of the leak check add two layers of 1000 glass cloth and a second nylon bag over the assembly per Paragraph 6.2.7; add one vacuum line. Leak check per Paragraph 6.4.

6.3 LAY-UP PROCEDURE FOR STRUCTURAL SHAPES

6.3.1 Clean the steel or aluminum die with an MEK dampened cheese cloth.

6.3.2 Heat the die to 250°F and apply a heavy coat of Ram 225. Then bake the mold for fifteen minutes at 350°F. Repeat the operation three times.

6.3.3 Apply a thin coat of Plastilease 334 to the glossy finished mold and bake the mold for one hour at 350°F.

6.3.4 Cool the mold to room temperature, then buff the mold surface to a smooth, non-sticky surface. This procedure is performed only once and need not be repeated for the duration of the mold.

6.3.5 Before each lay-up operation, lightly wipe the mold surface with Plastilease 334 with the mold surface at least 80°F. This coating should be uniform, smooth and non-streaky.

6.3.6 Identify the orientation of each ply in the composite shape by marking Mylar templates.

6.3.7 Remove the pre-preg containers required for each day's fabrication from 0° storage per the requirements in Paragraph 6.1.7.

6.3.8 Lay-up the pre-preg on the Mylar templates as described in Paragraph 6.1.8.

6.3.9 If the pre-preg is not pliable enough to be shaped on the tool, spray a thin coat of N-methyl pyrrolidone solvent - to be done under the direction of the Material and Process Development Department - to the individual plies of pre-preg, sandwich the sprayed pre-preg between Nylon film, and then allow the pre-preg to stand approximately 6 - 12 hours at room temperature until it becomes pliable.

6.3.10 Transfer each individual ply of pre-preg onto the tool according to the Engineering drawing.

6.3.11 Lay-up a process control panel per the requirements in Paragraph 6.2.3.

6.3.12 Bag the lay up as described in Paragraph 6.2.4 through 6.2.9.

6.4 LEAK CHECKING

6.4.1 When leak checking apply vacuum to the interior of the vacuum bag slowly. As the air is evacuated, make the bag conform to the shape of the lay-up and tool. Make certain the bag does not bridge any areas.

6.4.2 Pull 29 inches (minimum) mercury vacuum on the bagged lay-up and close off the vacuum source. Take a pressure reading 2 minutes after isolation of the system. The maximum allowable leakage rate is 0.5 inches of mercury per minute.

6.4.3 Maintain at least 29 inches of mercury vacuum on the bagged lay-up and place in the autoclave. Connect the required plumbing (thermocouples, vacuum, static lines and cold trap) and repeat the leak check of the previous paragraph with 100 psi autoclave pressure.

6.5 CURE SCHEDULE

6.5.1 Cure flat laminates and structural shaped parts which have not been sprayed with N-methyl pyrrolidone solvent as follows:

(a) While maintaining at least 29 inches of mercury vacuum on the bagged lay-up, raise the temperature to 200°F at a rate of 3-4°F/min.

(b) Hold at 200 ± 10°F for 60-70 minutes under at least 29 inches of mercury vacuum.

(c) Raise the temperature to 350°F at a rate of 3-4°F/min. When the temperature reaches 250°F, pressurize the autoclave to 100 ± 5 psig.

(d) Hold at 350 ± 10°F for 2 hours ± 10 minutes.

(e) Cool to 125°F or less, while maintaining 100 psig pressure, at a rate of 3-4°F/min.

6.5.2 Cure flat laminates and structural shaped parts which have been sprayed with N-methyl pyrrolidone solvent as follows:

(a) While holding 1-3 inches of mercury vacuum on the bagged lay-up, raise the temperature to 175°F at a rate of 3-4°F/min.

(b) Hold at 175°F for 4 hours ± 10 minutes under 1-3 inches of mercury vacuum.

(c) While holding 1-3 inches of mercury vacuum, raise the temperature to 200°F at a rate of 3-4°F/min. Hold at 200 ± 10°F for 110-130 minutes, then raise the bag vacuum to 27-29 inches of mercury vacuum.

(d) Raise the temperature to 350°F at a rate of 3-4°F/min. When the temperature reaches 250°F, pressurize the autoclave to 100 ± 5 psig.

(e) Hold at 350 ± 10°F for 2 hours ± 10 minutes.

(f) Cool to 125°F or less, while maintaining 100 psig pressure, at a rate of 3-4°F/min.

6.5.3 Post-cure the production part and the process control panel in an oven as follows:

NOTE: Structural shapes must be restrained during post-cure to prevent warpage.

(a) Raise oven temperature to 350°F at a rate of 3-4°F/min. Hold at 350 ± 10°F for 30 - 40 minutes.

(b) Raise oven temperature to 600°F at a rate of 1/4°F/min.

(c) Hold temperature at 600 ± 10°F for 6 ± 1/2 hours.

(d) Cool to 125°F or less at a rate of 3-4°F/min.

6.5.4 Record the autoclave temperature and pressure throughout the cure cycle and record the oven temperature throughout the post-cure cycle. Continuously record pressure build-up in the vacuum bag with a suitable instrument. Also record the time the lay-up is at room temperature prior to cure. Include all this information in the permanent record for the part being fabricated.

6.6 TRIMMING

6.6.1 Structural Part - Trim the structural part with a diamond impregnated cutoff wheel or router to the dimensions of the Engineering drawing. Use water or carbon dioxide for cooling the diamond tool, but do not use any oil based compound. Use 120 grit or finer diamond particle impregnated tooling, and operate at a speed of 1800 to 4000 SFPM and a feed rate of 4 inch/min.

6.6.2 Process Control Specimens - Cut the process control laminate fabricated per Paragraph 6.2 into six 4.0" (filament direction) x 0.5" x thickness flexural test specimens and six 0.70" (filament direction) x 0.25" x thickness horizontal shear test specimens. Use a thin (0.060 inch or less) diamond cut-off wheel at 1800 to 4000 SFPM and 4 inch/min. maximum feed rate. Attach acrylic plastic back up to the bottom of the laminate with double sided tape. Use a 120 grit or finer diamond tool. From the offal machine three 1/2" x 1/2" x thickness resin content specimens.

6.7 DRILLING

6.7.1 Drill the post-cured graphite/polyimide part with a cerrated edged carbide drill with a cutting speed of 40-80 surface feet/minute. Apply a light hand feed pressure and no coolant.

7.0 QUALITY ASSURANCE PROVISIONS

7.1 Quality Assurance shall maintain surveillance to assure adequate compliance to the following:

7.1.1 The autoclave shall be certified per P.S. 23401, Class 3C except working zone of the autoclave shall extend to within 3 inches of the wall and 20 thermocouples which continue to function during the entire certification procedure shall be the minimum number required to certify the autoclave. Calibrate instrumentation per P.S. 20503. The post-curing oven shall be certified per P.S. 23401, Class 3C.

7.1.2 The composite lay-up work is performed in a room that meets the requirements of Paragraph 5.3.

7.1.3 The surface finish of the top of the tool is a maximum of 80 RHR.

7.1.4 Only pre-preg material qualified per the requirements in Table I is used in the fabrication of parts.

7.1.5 The composite pre-preg material is packaged in a moisture proof plastic bag and stored at 0 ± 10°F when not in use.

7.1.6 The composite pre-preg material remains inside the moisture proof plastic bag for a minimum of 30 min. after removal from 0°F storage.

7.1.7 The composite pre-preg material is placed in the orientation designated by the Engineering drawing.

7.1.8 No end to end type butt joints of composite pre-preg material are used inside the composite laminate unless required by the Engineering drawing.

7.1.9 The edge of any individual piece of pre-preg in one ply is not placed directly over the edge of another piece of pre-preg of a previous ply, but that edge joints overlap a minimum of 0.5 inch.

7.1.10 No visible foreign material contamination exists on the lay-up.

7.1.11 Edges of composite laminates are fabricated a minimum of 0.25 inches oversize, and composite pre-preg material terminates within 0.10 inches of the dam and does not overlap the dam.

7.1.12 Fabrication procedures are performed by qualified personnel per the requirements of Paragraph 5.2.

7.2 Quality Assurance shall monitor records of the following information:

7.2.1 The time in and out of 0°F storage for each individual composite pre-preg container used in Manufacturing.

7.2.2 The time-temperature-pressure history of each autoclave run.

7.2.3 The time-temperature history of each oven run.

7.2.4 The total amount of time that each composite laminate lay-up is at room temperature prior to curing per the requirements of Paragraph 5.10.

7.2.5 The composite pre-preg batch and container numbers used for each composite lay-up.

7.3 NONDESTRUCTIVE INSPECTION - Radiographically inspect per P.S. 21206.1 and ultrasonically inspect per P.S. 21211.4. Any defects found are subject to Engineering disposition.

7.4 PROCESS CONTROL TESTS

7.4.1 Perform all elevated temperature tests in an air circulating oven. Specimens tested at elevated temperature shall be at temperature 10-20 minutes prior to test. The specimen temperature shall be controlled to ± 5°F. Monitor the temperature by attaching a single thermocouple to the test specimen.

7.4.2 Test six longitudinal flexural specimens, three at room temperature and three at 550°F, in accordance with Figure 2. The specimens shall meet the requirements of Table I.

7.4.3 Test six interlaminar shear specimens, three at room temperature and three at 550°F, in accordance with Figure 3. The specimens shall meet the requirements of Table I.

7.4.4 Perform resin content determinations on three 1/2" x 1/2" x thickness test specimens as follows:

(a) Weigh the test sample to nearest 0.0001 gm (W_1).

(b) Place sample in glass beaker containing 200 ml. of concentrated HNO_3 at 140 ± 10°F and boil at 140 ± 10°F for 2 - 2-1/2 hours.

(c) Filter (hot) the mixture through a previously dried, tared (to nearest 0.0001 gm) fritted crucible (W_2). Wash with concentrated HNO_3. Rinse fibers with a minimum of 600 ml. of distilled water or until filtrate is neutral. Dry in a drying oven at 220°F for 1 hour.

(d) Cool to room temperature in a desiccator and weigh the crucible to 0.0001 gm (W_3).

(e) Calculate resin content as follows:

$$\% \text{ resin content} = \frac{W_1 - (W_3 - W_2)}{W_1} \times 100$$

(f) Report the resin content value to the nearest of three specimens.

8.0 SAFETY

8.1 PRE-PREG MATERIAL

8.1.1 Personnel shall wear eye protection when (1) cutting the pre-preg material or the finished composite, and (2) observing mechanical property tests. The composite filaments shatter and can be a danger to the eyes.

8.1.2 Composite filament particles that become embedded in the skin shall be removed immediately by pulling straight out. Bending or flexing the broken filaments will only cause further breakage of the filament. If not removed immediately, the filaments tend to work themselves further into the skin.

8.2 ORGANIC SOLVENTS

8.2.1 Description - The following solvents and/or solvent bearing materials are used in small quantities per this specification.

(a) Methyl Ethyl Ketone: A colorless, thin liquid, flammable and toxic.

(b) Mold Wiz F-57 Mold Release: A colorless, thin liquid, flammable and toxic.

(c) N-Methyl Pyrrolidone: A colorless thin liquid, flammable and toxic.

8.2.2 First Aid - In the event of eye or skin contamination:

(a) Flush the exposed area with large amounts of water.

(b) Remove contaminated clothing.

(c) Secure first aid.

8.2.3 Toxicology: Avoid prolonged breathing of solvent vapors.

8.2.4 Fire Hazard: Flammable solvents are extremely easy to ignite and shall not be used in the vicinity of smoking, sparks or open flames.

8.2.5 Handling and Storage

(a) Wear safety glasses (or goggles) and rubber gloves while working with solvents.

(b) Do not use solvents in confined areas unless specifically authorized, since fumes are generally toxic, flammable and explosive.

(c) Dispose of all rags in flammable solvent areas in special containers used only for this purpose.

(d) Store and handle solvents in properly labeled safety containers.

8.2.6 NOTE: The preceding are minimum safety precautions. Personnel using these materials should be familiar with the more detailed precautions provided in McDonnell Specification P.S. 20002.

9.0 NOTES

9.1 Vendors or subcontractors needing this specification should direct their request for copies to the attention of McDonnell Aircraft Company (MCAIR) St. Louis, Purchasing or Subcontracting, as applicable. Information pertaining to the technical aspect of this specification can be obtained from MCAIR Material and Process Development Department (Dept. 372).

10.0 REFERENCE PUBLICATIONS

Not applicable

MECHANICAL PROPERTY	MINIMUM AVERAGE (PSI)	SPECIMEN MINIMUM (PSI)
0° Flexural Strength, R.T.	170,000	155,000
0° Flexural Strength, 550°F	110,000	90,000
0° Flexural Modulus, R.T.	①	①
0° Flexural Modulus, 550°F	①	①
Interlaminar Shear Strength, R.T.	8,000	7,000
Interlaminar Shear Strength, 550°F	5,000	4,000
% Resin Content (By Weight)	①	①

① - For Information Only

TABLE I - GRAPHITE/POLYIMIDE PROCESS CONTROL MECHANICAL PROPERTY REQUIREMENTS

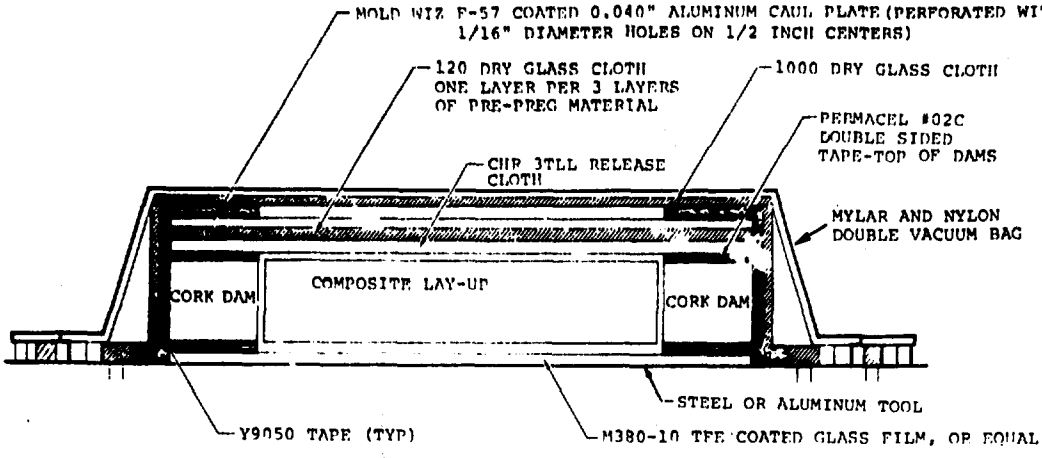
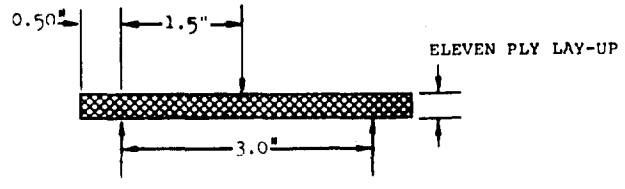


FIGURE 1 - LAY-UP CROSS SECTION OF LAMINATE FABRICATED WITH CORK DAMS



NOTES:

(1) Width of specimen is 0.5 inch.

(2) Load the specimen with 0.125 inch radius supports at a rate of 0.05 in./min.

(3) Place a deflectometer capable of measuring deflection to 0.001 inch under the span midpoint.

(4) While the specimen is loaded record the deflection and load continuously. Remove the deflectometer prior to failure.

(5) Run 550°F temperature test in air circulating oven, holding at 550°F for 10 minutes prior to test.

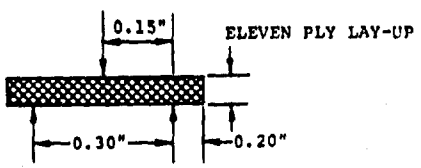
(6) Calculate the ultimate flexural strength as follows:

$$F = \frac{1.5\ PL}{wt^2}$$, where F = Flexural Strength, psi
P = Load at failure , lb.
L = Test span, 3 in.
w = Specimen width, in.
t = Specimen thickness, in.

(7) Calculate the flexural modulus as follows:

$$E = \frac{P(L^3)}{4w(t^3)Y}$$, where E = Flexural modulus, psi
P = Load at failure, lb.
L = Test span, 3 in.
w = Specimen width, in.
t = Specimen thickness, in.
Y = Deflection, in.

FIGURE 2 - LONGITUDINAL FLEXURE SPECIMEN AND TEST



NOTES:

(1) Width of specimen is 0.25 inches.

(2) Load the specimens with 0.125 inch radius supports at a rate of 0.05 in./min.

(3) Run 550°F temperature test in air circulating oven, holding at 550°F for 10 minutes prior to test.

(4) Calculate the interlaminar shear strength as follows:

$F = \frac{0.75P}{wt}$, where F = Interlaminar shear strength, psi
P = Load at failure, lb.
w = Specimen width, in.
t = Specimen thickness, in.

FIGURE 3 - INTERLAMINAR SHEAR SPECIMEN AND TEST

PREPARED R. C. Allen

APPROVED _____ Manufacturing

APPROVED _____ Engineering

APPROVED _____ Quality Assurance

APPROVED _____ AFPRO, Engineering

APPROVED _____ Safety and Medical

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10. DISTRIBUTION

Distribution limited to United States Government agencies and designated recipients only, since this report concerns the test and evaluation of technology directly applicable to Military hardware (03 January 1972). Request for additional copies or further distribution of this document must be referred to the Air Force Materials Laboratory (AFML/LC), Wright-Patterson Air Force Base, Ohio 45433.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
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13 ABSTRACT

The objectives of this program were to place polyimide matrix advanced composite processing technology on a production basis, and to demonstrate its acceptability for structural applications by fabrication and test of representative structure. Under this program polyimide matrix advanced composites were cured in a one shift autoclave operations (~8 hours) at 350°F and 200 psi maximum while maintaining epoxy matrix-type properties.

These process parameters were confirmed by the successful fabrication and test of an F-4 polyimide rudder, which demonstrated the flight worthiness of thin gauge boron and graphite/polyimide in this type structure. Design allowable data for tension, compression, and inplane shear were developed at both room temperature and 550°F.

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Security Classification

Security Classification

KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Composite Structural Materials						
High Temperature Composite Material						
Resin Matrix Materials						
High Temperature Polyimide Materials						
Polyimide Structural Materials						
Material Properties for Polyimides						

DD FORM 1473 (BACK)

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