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NASA Contractor Report 3601

Design, Fabrication and Test of Graphite/Polyimide Composite Joints and Attachments

Summary

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Design, Fabrication and Test of Graphite/Polyimide Composite Joints and Attachments

Summary

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and S. H. Ward
Boeing Aerospace Company
Seattle, Washington

Prepared for
Langley Research Center
under Contract NAS1-15644

National Aeronautics
and Space Administration

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Information Branch

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FOREWORD

This document was prepared by the Boeing Aerospace Company for the National Aeronautics and Space Administration, Langley Research Center in compliance with Contract NAS1-15644, "Design, Fabrication and Test of Graphite/Polyimide Composite Joints and Attachments for Advanced Aerospace Vehicles."

This report is one of five that fully document contract results. It is the Summary of Task 1.0 "Design, Fabrication and Test of Graphite/Polyimide Joints."

Dr. Paul A. Cooper was the contracting officer's technical representative for the full contract and Gregory Wichorek was the technical representative for design allowables testing of Celion 6000/PMR-15. Boeing performance was under the management of Mr. J. E. Harrison. Mr. D. E. Skoumal was the technical leader. Major participants in this program were James B. Cushman. Stephen F. McCleskey, and Stephen H. Ward from the Structural Development organization and Sylvester G. Hill of Materials and Processes.

The use of commercial products or names of manufacturers in this report does not constitute official endorsement of such products or manufacturers either expressed or implied by the National Aeronautics and Space Administration.

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1.0 SUMMARY

This report summarizes the design, analysis and test activities performed under TASK 1.0 of NASA Contract NAS1-15644 to develop four types of graphite/polyimide (Gr/PI) bonded and bolted composite joints. Design data were established for building Gr/PI lightly loaded control surface structures for advanced space transportation systems that operate at temperatures up to 561K (550°F).

A detailed screening of joint designs was conducted to select the most promising concepts. Material properties and "Small Specimen" tests were conducted to establish design data and to evaluate specific design details. "Static Discriminator" tests were conducted on preliminary designs to verify structural adequacy. These tests led to improvements which were incorporated into the final designs. Scaled-up specimens of the final joint designs, representative of production size requirements, were subjected to a series of static and fatigue tests to evaluate joint strength. Effects of environmental conditioning were determined by testing aged (125 hr @ 589K (600°F)) and thermal cycled (116K to 589K (-250°F to 600°F), 125 times) specimens.

Analyses and tests have demonstrated that bonded and bolted Gr/PI joints can be designed and fabricated to carry loads up to 560 kN/m (3200 lb/in), and moments up to 3.0 kN-m/m (684 in-lb/in) at temperatures up to 561K (550°F). Tests also demonstrated that bolted Gr/PI to titanium joints can be designed to carry loads up to 2100 kN/m (12000 lb/in). Bonded Gr/PI to titanium joints designed to carry this load level require further developing with respect to cocured bond processing. However, a load carrying capability of 875 kN/m (5000 lb/in) was demonstrated for a Gr/PI to titanium "3-step" symmetric step lap joint under Task 2.0 of this contract. Test results also indicated a loss of resin and degradation of laminates and adhesive bonds after exposure to 589K (600°F) for 125 hours as evidenced by a decrease in laminate strengths. This has been attributed to resin chemistry and adhesive processing problems which were identified by post-test analysis.

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2.0 INTRODUCTION

Advanced designs for high-speed aircraft and space transportation systems require structures for operation in the 116K (-250°F) to 589K (600°F) temperature range. Design data are needed for bonded and bolted composite joints to support design of structural concepts.

The program discussed herein was designed to extend the current epoxy matrix composite technology in joint and attachment design to include high-temperature polyimide matrix composites. It provides an initial data base for designing and fabricating graphite/polyimide (Gr/PI) flight components for advanced space transportation systems and high-speed aircraft. The objectives of this program were two-fold. The first objective was to develop and evaluate bonded and bolted design concepts for joints applicable to specific rib to skin, spar to skin, and panel to panel configurations subjected to loads typical of those expected in lifting surfaces of high-speed aircraft and space transportation systems during re-entry. The second objective was to explore advanced design concepts for bonded composite to composite and composite to metal joints. These objectives were pursued concurrently—TASK 1 was focused on the first objective and TASK 2 on the second. The overall program flow for the two tasks is shown in Figure 2-1. The technical activities and results of the TASK 1 investigation, shown enclosed in a dashed box in the figure, are reported in this document.

The generic joint concepts developed under TASK 1 are shown in Figure 2-2. Several concepts were designed and analyzed for each bonded and each bolted attachment type. Concurrent with this a series of material properties and "Small Specimen" tests were conducted to support the concept designs. The analytical results and design data were used to select the most promising bonded and bolted joint concepts.

The most promising concepts for each joint type were fabricated, tested, and evaluated. Test results were used to define any design changes that would improve the joint performance.

Design changes were incorporated and the final joint concepts were fabricated on a scaled-up basis (1.5 m (5 ft) minimum length) to assure that attachments could be fabricated for full-scale components. A series of static tests were performed on specimens cut from the scaled-up attachments to verify the validity of the scaled-up manufacturing process and the final designs. Other specimens were environmentally conditioned and subjected to a series of static and fatigue tests to evaluate joint strength. Test results were compared with the analytical predictions to verify design and analysis procedures.

This is one in a series of five reports that fully document the results of design, analysis and test activities performed under NASA contract NAS1-15644. The other reports are:

1. Cushman, J. B.; and McCleskey, S. F.: Design Allowables Test Program, Celion 3000/PMR-15 and Celion 6000/PMR-15 Graphite/Polyimide Composites, NASA CR-165840, 1982.

2. Cushman, J. B.; McCleskey, S. F.; and Ward, S. H.: Design, Fabrication and Test of Graphite/Polyimide Composite Joints and Attachments - Data Report, NASA CR-165955, 1982.

3. Cushman, J. B.; McCleskey, S. F.; and Ward, S. H.: Test and Analysis of Celion 3000/PMR-15, Graphite/Polyimide Bonded Composite Joints - Summary, NASA CR-3602, 1982.

4. Cushman, J. B.; McCleskey, S. F.; and Ward, S. H.: Test and Analysis of Celion 3000/PMR-15, Graphite/Polyimide Bonded Composite Joints - Data Report, NASA CR-165956, 1982.

Measurement Units

All measurement values in this report are expressed in the International System of Units and in U.S. Customary Units. Actual measurements and calculations were made in U.S. Customary Units.

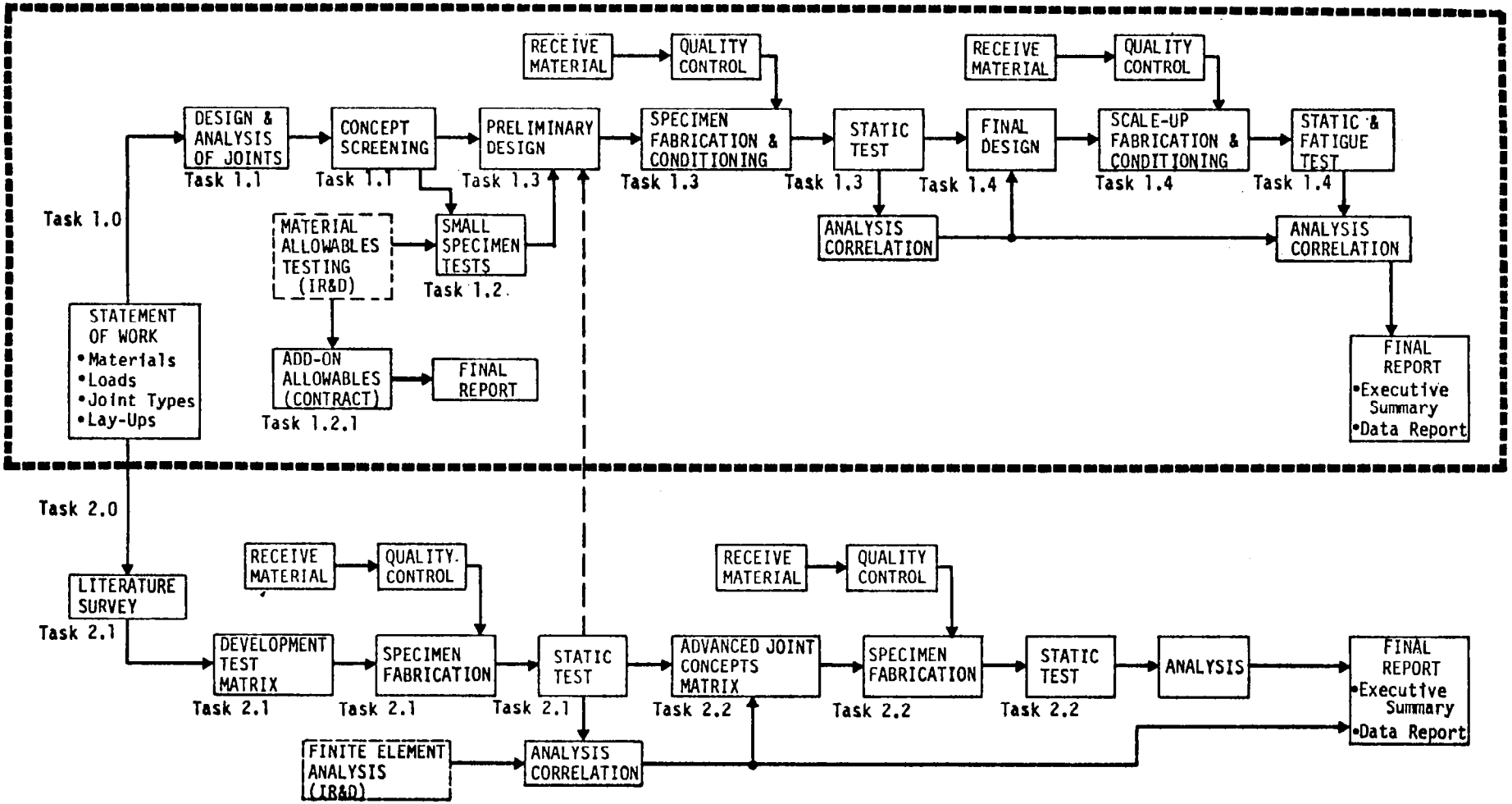

Figure 2-1: TASK 1 and TASK 2 PROGRAM FLOW

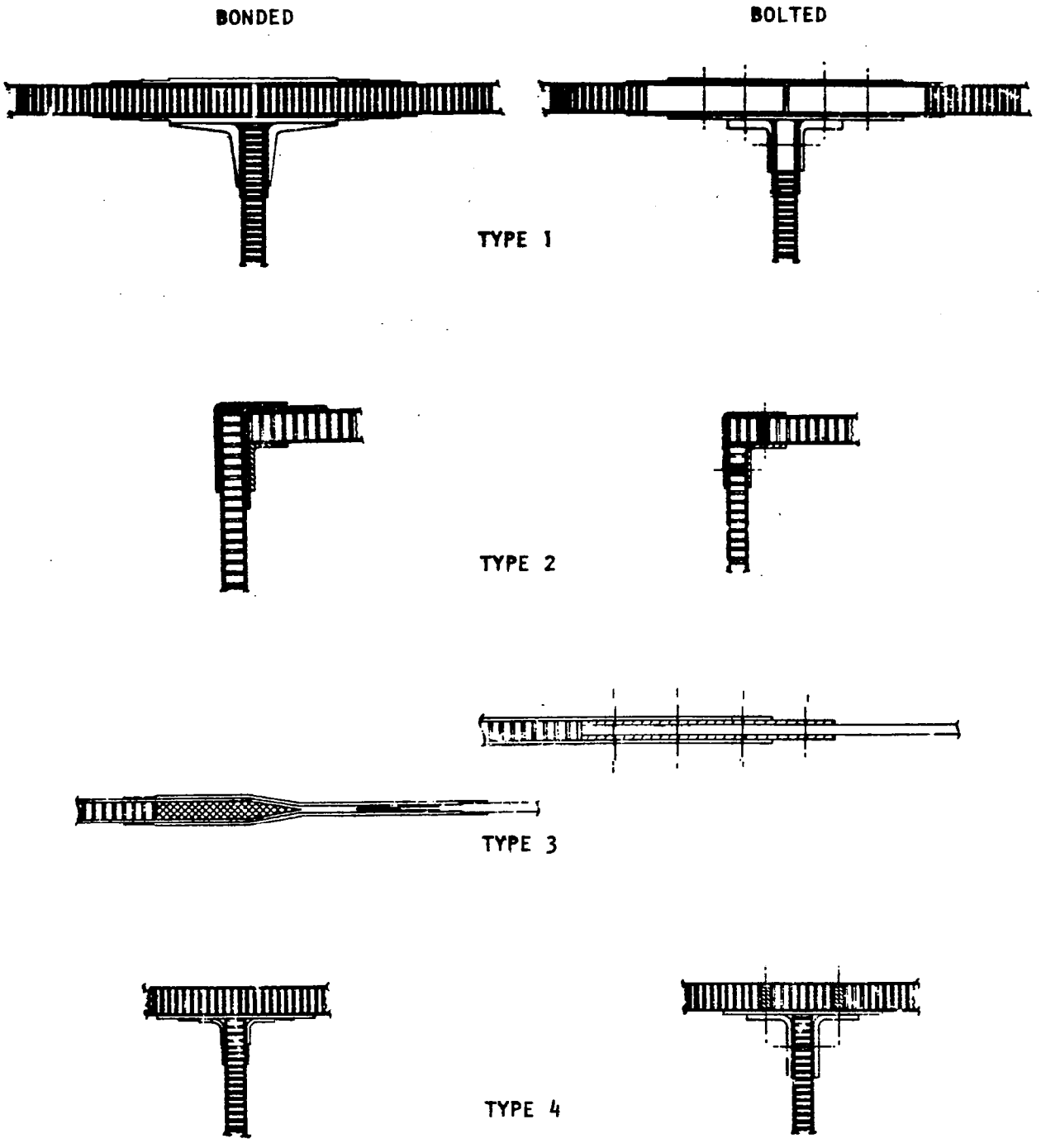

Figure 2-2: Generic Joint Concepts for 4 Attachment Types

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3.0 JOINT DESIGN REQUIREMENTS AND ANALYSIS

TASK 1 of this program was to design, analyze and test specific joint concepts for each of the generic attachment types shown in Figure 2-2. This section presents joint design requirements specified in the contract statement of work along with analysis procedures and concept screening procedures and results.

3.1 Joint Design Requirements

Joint Configurations

Basic configurations for each attachment type are given in Figures 3-1 through 3-4 and are described below.

Laminate lay-ups and honeycomb core thicknesses used are described in Table 3-1.

Type 1 Attachment—The Type 1 attachment, shown in Figure 3-1, is typical of an attachment in the sandwich shell at a rib or spar interface of an aerodynamic surface such as a wing or control surface.

Type 2 Attachment—The Type 2 attachment, shown in Figure 3-2, is typical of the attachment occurring at an unloaded edge of a wing or aerodynamic surface.

Type 3 Attachment—The Type 3 attachment, shown in Figure 3-3, is typical of a localized attachment of a metallic plate to a composite sandwich structure. The attachment is subjected to relatively large inplane forces which must be distributed to the sandwich face sheets.

Type 4 Attachment—The Type 4 attachment, shown in Figure 3-4, is similar to the Type 1 attachment in that it connects members that are perpendicular; however, the cover panels are not spliced in the Type 4 attachment. The applied load levels are well below those required for the Type 1 attachments.

Environmental Conditioning

The effects of the following environmental conditioning were evaluated for each joint type.

(1) As cured/post-cured
(2) Thermally aged 125 hours at 589K (600°F) in a one atmosphere environment
(3) Thermally cycled 125 times from 116K (-250°F) to 589K (600°F) in a one atmosphere environment

Design Loads

Loading conditions and load ratios specified for each attachment type represent internal loads from the Space Shuttle Orbiter aft body flap. The loads were scaled to produce the design allowable stress state in at least one lamina of the cover panel outside the joint area. Loads for each joint type are given in Tables 3-2 through 3-5.

3.2 Analysis Procedures

The primary objective of this program was to demonstrate that Gr/PI joints could be designed and built to carry the required loads. Since resources were limited it was desired to avoid a series of testing and redesign. Therefore the design philosophy was to design the joints such that they would fail in the basic covers outside of the joint. The only exception was the Type 2 joints, which due to their configuration were designed to fail in the joint but above the required load.

Bonded Joints

Sizing of Type 1, 2 and 4 bonded joints was based on design curves giving lap length verus failure load. Preliminary analyses were based on existing design curves selected from available literature. Final designs were based on bonded

joint data generated under TASK 2.0 of this program. Design of bonded attachment angles loaded in tension was based on "Small Specimen" tests discussed in Section 4.0.

Analyses of the Type 3 joint showed that a simple double lap bonded joint was not adequate to carry the design load. However, a symmetrical step-lap bonded joint was designed using the A4EG computer code (Ref. 1). This code uses both elastic and elastic-plastic analyses to predict ultimate joint strength.

Bolted Joints

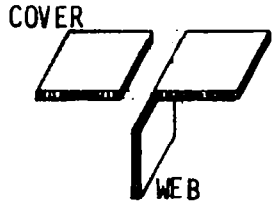
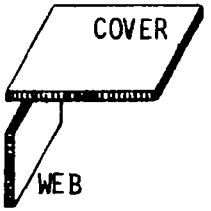
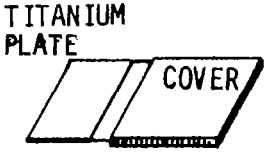
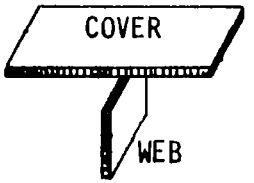
Bolted joints were sized by the three basic failure modes of bearing, shear-out, and net area tension. Basic material properties for Gr/PI were not initially available, thus preliminary sizing was based on estimated properties from the literature and from data derived from Boeing IR&D programs. Final analyses were performed using the material properties determined from the "Small Specimen" test presented in Section 4.0. The design philosophy was to approach bearing ultimate in the joint while stressing the cover skin(s) to their ultimate load capability.

3.3 Joint Concept Screening

Ten to fourteen concepts were defined for each of the bonded and bolted joint types. These concepts were subjected to a first cut screening that was a qualitative assessment based on the three selection criteria and evaluation parameters shown in Figure 3-5. This screening resulted in deletion of some concepts and modification of others. The remaining concepts were then subjected to a more detailed second cut screening. The second cut screening used the same three selection criteria as the first; however, each concept was evaluated using the nineteen evaluation parameters shown in Figure 3-6. The weighting factors shown account for the relative importance of each selection criteria. The sum of these scores was the final rating score. Joint design concepts with the highest rating scores are shown in Figures 3-7 through 3-16. These were the baseline concepts used to define preliminary joint designs that

were subjected to "Static Discriminator" tests. Two concepts were evaluated for the Type 3 bolted joint, one with Gr/PI splice plates and one with titanium splice plates. Results of the "Static Discriminator" tests were the last screening step that selected the final joint designs to be used for the "Final Evaluation" tests discussed in Section 9.0.

Table 3-1: STATEMENT-OF-WORK REQUIREMENTS FOR LAMINATE LAYUPS AND HONEYCOMB CORE THICKNESSES

	TYPE 1	TYPE 2	TYPE 3	TYPE 4
				
COVER PANEL FACE SHEETS	8 plies in 0°,$+45^o$, -45°,90° directions, symmetric laminate.	8 plies in 0°,$+45^o$, -45°,90° directions, symmetric laminate.	Symmetric, quasi-isotropic laminate.	Minimum gage, plies in 0°,$+45^o$,-45°,90° directions, unsymmetric laminate, symmetric panel.
COVER PANEL HONEYCOMB CORE	Glass/polyimide, 19.1 mm (.75 in) thick.	Glass/polyimide, 19.1 mm (.75 in) thick.	Glass/polyimide, 12.7 mm (.50 in) thick	Glass/polyimide, 12.7 mm (.50 in) thick.
WEB PANEL FACE SHEETS	6 plies in 0°,$+45^o$, -45° directions, symmetric laminate.	6 plies in 0°,$+45^o$, -45° directions, symmetric laminate.	—	Minimum thickness, plies in 0°,$+45^o$,-45° directions, symmetric sandwich panel
WEB PANEL HONEYCOMB CORE	Glass/polyimide 12.7 mm (.50 in) thick.	Glass/polyimide 12.7 mm (.50 in) thick.	—	Glass/polyimide 12.7 mm (.50 in) thick.
METAL PLATE	—	—	Ti-6Al-4V, 6.35 mm (.25 in.) thick.	—

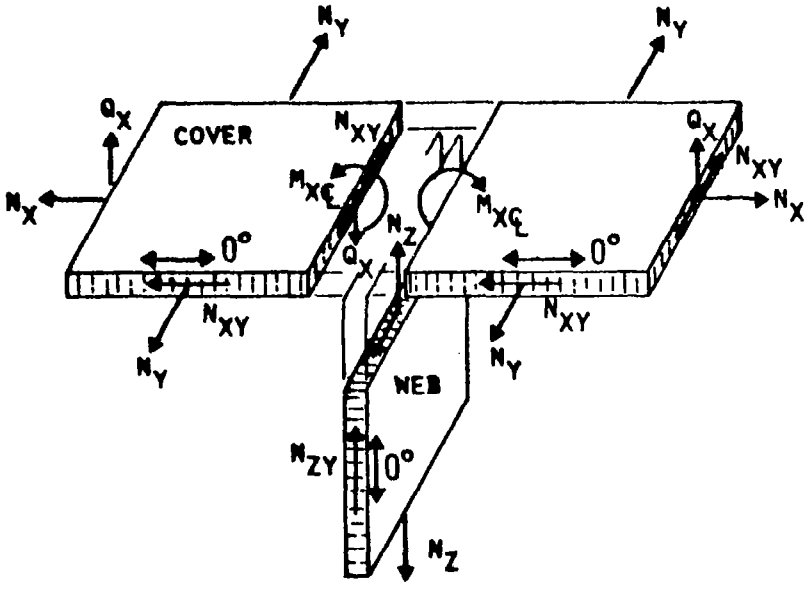


FIGURE 3-1: ATTACHMENT TYPE NO. 1

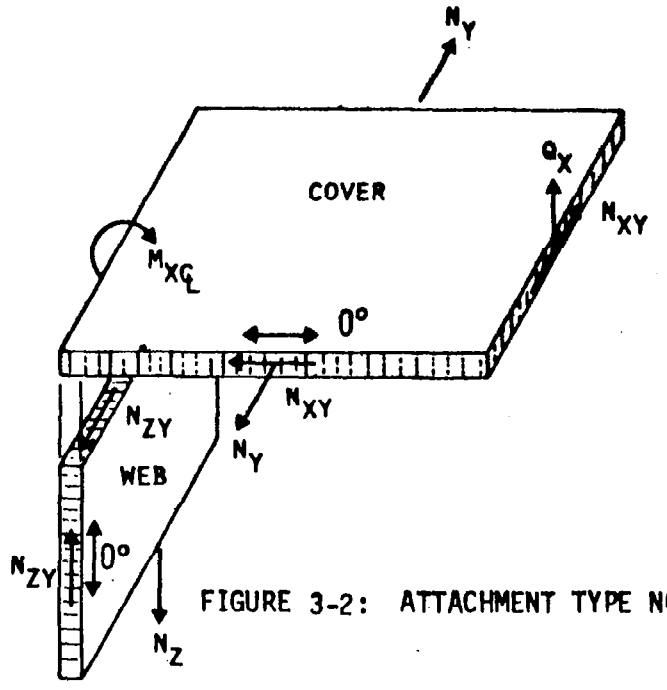


FIGURE 3-2: ATTACHMENT TYPE NO. 2

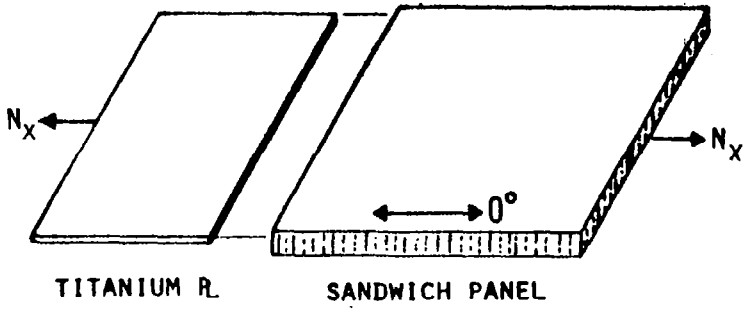


FIGURE 3-3: ATTACHMENT TYPE NO. 3

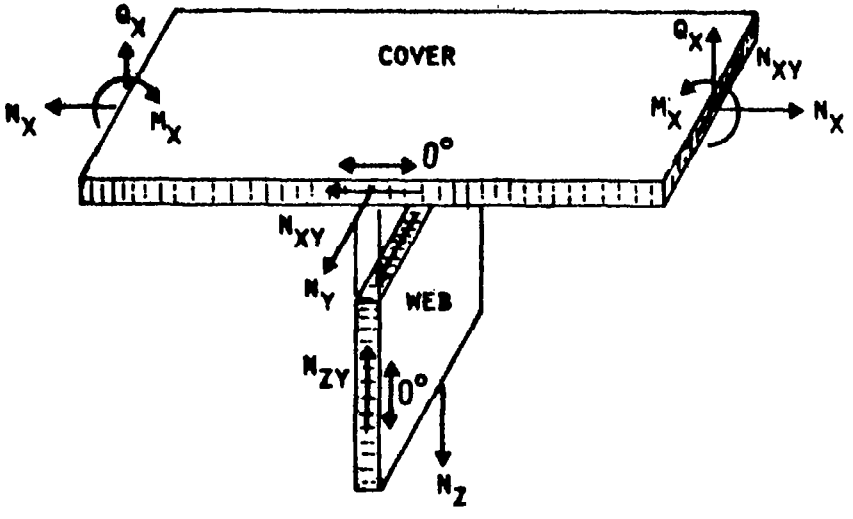


FIGURE 3-4: ATTACHMENT TYPE NO. 4

Table 3-2 : PRELIMINARY LOADS FOR TYPE 1 JOINTS

CASE	LOAD CONDITION 1	RATIO	VALUE kN/m (lb/in)
1	N_x	1	442.3 (2526)
	N_{xy}	0.03 N_x	13.3 (76)
	N_y	0.15 N_x	66.4 (379)
	N_{zy}	0.10 N_x	44.3 (253)
	N_z	0.02 N_x	8.9 (51)
	$M_{x\ell}$	(0.10 in) N_x	1.1* (253*)
	Q_x	0.01 N_x	4.4 (25.3)
2	N_x	1	239.6 (1368)
	N_{xy}	0.03 N_x	3.2 (41)
	N_y	0.15 N_x	35.9 (205)
	N_{zy}	0.10 N_x	24.0 (137)
	N_z	0.12 N_x	28.7 (164)
	$M_{x\ell}$	(0.50 in) N_x	3.0* (684*)
	Q_x	0.06 N_x	14.4 (82)

LAYUP: COVER $(0/\pm45/90)_S$ WEB $(0/\pm45)_S$

*kN-m/m (lb-in/in)

1 See Figure 3-1

Table 3-3 : PRELIMINARY LOADS FOR TYPE 2 JOINTS

CASE	LOAD CONDITION 2	RATIO	VALUE kN/m (lb/in)
1	N_x	0	
	N_y	1	560.4 (3200)
	N_{xy}	0.10 N_y	56.0 (320)
	N_{zy}	0.10 N_y	56.0 (320)
	N_z	0.02 N_y	11.2 (64)
	$M_{x\ell}$	(0.01 in)N_y	0.1* (32*)
	Q_x	0.02 N_y	11.2 (64)
2	N_x	0	
	N_y	0.40 N_{y1}	224.1 (1280)
	N_{xy}	0.10 N_y	22.4 (128)
	N_{zy}	0.10 N_y	22.4 (128)
	N_z	0.10 N_y	22.4 (128)
	$M_{x\ell}$	(0.05 in) N_y	0.3* (64*)
	Q_x	0.10 N_y	22.4 (128)

LAYUP: COVER $(0/\pm45/90)_S$ WEB $(0/\pm45)_S$

*kN-m/m (lb-in/in)

2 See Figure 3-2

Table 3-4: PRELIMINARY LOAD FOR TYPE 3 JOINTS

CASE	LOAD CONDITION [1]	RATIO	VALUE kN/m (lb/in)
1	N_x	1	2100 (12,000)

LAYUP: Cover $(0/\pm 45/90)_{2S}$

[1] See Figure 3-3

Table 3-5: PRELIMINARY LOADS FOR TYPE 4 JOINTS

CASE	LOAD CONDITION [2]	RATIO	VALUE kN/m (lb/in)
1	N_x	1	200.2 (1143)
	N_{xy}	0.03 N_x	6.0 (34)
	N_y	0.15 N_x	30.1 (172)
	N_{zy}	0.10 N_x	20.0 (114)
	N_z	0.02 N_x	5.0 (28)
	$M_{x\mathrm{℄}}$	(0.10 in) N_x	0.5* (114*)
	Q_x	0.01 N_x	2.0 (11.4)
2	N_x	1	93.3 (533)
	N_{xy}	0.03 N_x	2.8 (16)
	N_y	0.15 N_x	14.0 (80)
	N_{zy}	0.10 N_x	9.3 (53.3)
	N_z	0.12 N_x	11.2 (64)
	$M_{x\mathrm{℄}}$	(0.50 in) N_x	1.2* (266.5*)
	Q_x	0.06 N_x	5.6 (32)

LAYUP: COVER $(0/\pm 45/90)$ WEB $(0/\pm 45)$

*kN-m/m (lb-in/in)

[2] See Figure 3-4

SELECTION CRITERIA	EVALUATION PARAMETER
LOAD TRANSFER ABILITY	DIRECTNESS OF LOAD PATH
FABRICATION COMPLEXITY	EASE OF FABRICATION
SERVICE LIFE	REDUNDANT LOAD PATHS

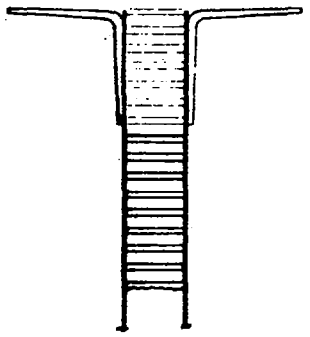
Qualitative Assessment Only

Figure 3-5 : 1st CUT SCREENING EVALUATION PARAMETERS

- o Best given rating of 1.0.
- o Others ratioed down.
- o Total rating for each selection criteria factored to baseline of 10 points.

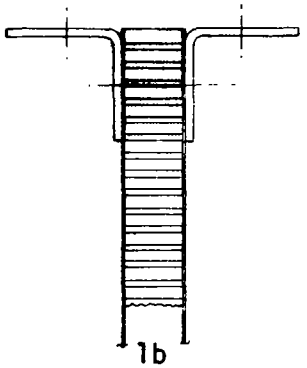
SELECTION CRITERIA	WEIGHTING FACTOR	EVALUATION PARAMETER	RATING		
			CONCEPT 1a	CONCEPT 1b	CON
LOAD TRANSFER ABILITY	2	Directness of load path	1.00	1.00	
		Stress concentration areas	1.00	.66	
		Thermal balance	.50	.50	
		Stiffness balance	.75	.75	
		Abrupt change of section	1.00	1.00	
		AVERAGE X 10	8.50	7.82	
		AVERAGE x 10 x WEIGHTING FACTOR	17.00	15.64	
FABRICABILITY	5	Number of parts	1.00	1.00	
		Ease of detail fabrication	.80	1.00	
		Ease of component assembly	1.00	1.00	
		Ease of joint assembly	.50	.50	
		Inspectability	.50	.50	
		Special tooling	.90	.90	
		New development	.77	1.00	
		AVERAGE X 10	7.81	8.43	
		AVERAGE x 10 x WEIGHTING FACTOR	39.05	42.15	
SERVICE LIFE	3	Redundant load path	1.00	1.00	
		Crack propagation	.50	.50	
		Damage tolerance	1.00	1.00	
		Inspectability	.50	.75	
		Repairability	1.00	1.00	
		Length of bonded joint	1.00	1.00	
		Number of exposed edges	1.00	1.00	
		AVERAGE X 10	8.57	8.93	
		AVERAGE x 10 x WEIGHTING FACTOR	25.71	26.79	
		TOTAL RATING SCORE (Sum of averages x weighting factors)	81.76	84.58	

Figure 3-6: 2nd CUT SCREENING EVALUATION PARAMETERS



1b

Figure 3-7: SELECTED TYPES 1 & 4 BONDED JOINT CONCEPT - WEB TO COVER ATTACHMENTS



1b

Figure 3-8: SELECTED TYPES 1 & 4 BOLTED JOINT CONCEPT - WEB TO COVER ATTACHMENTS

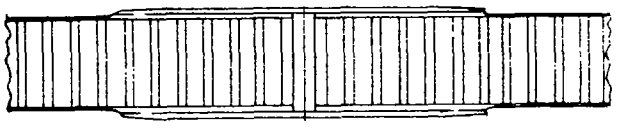
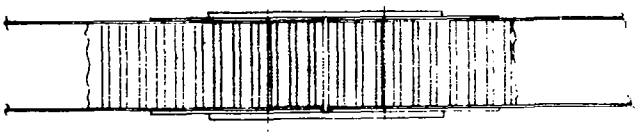


Figure 3-9: SELECTED TYPE 1 BONDED JOINT CONCEPT - COVER

1a



1a

Figure 3-10: SELECTED TYPE 1 BOLTED JOINT CONCEPT - COVER

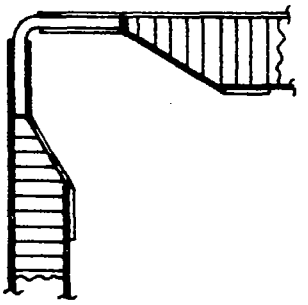


Figure 3-11: SELECTED TYPE 2 BONDED JOINT CONCEPT

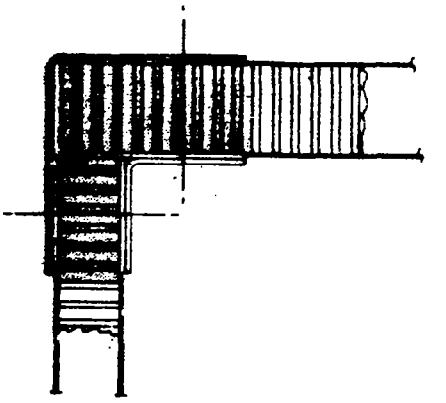
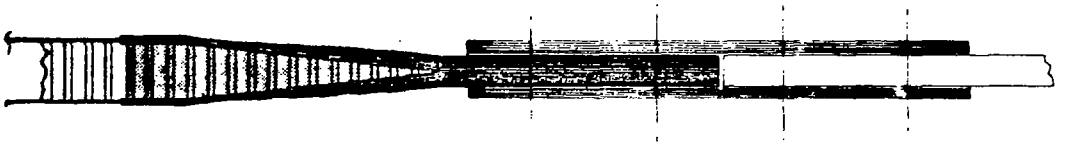


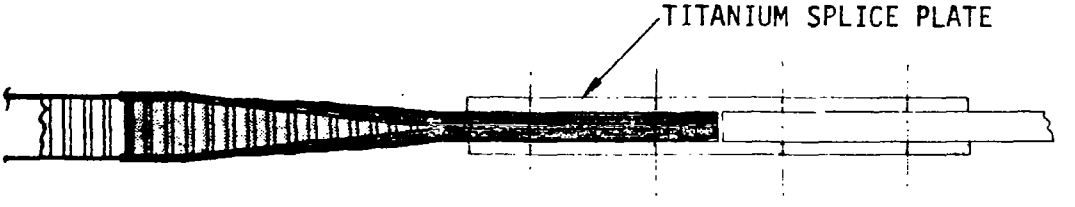
Figure 3-12 : SELECTED TYPE 2 BOLTED JOINT CONCEPT

3a

Figure 3-13 : SELECTED TYPE 3 BONDED JOINT CONCEPT

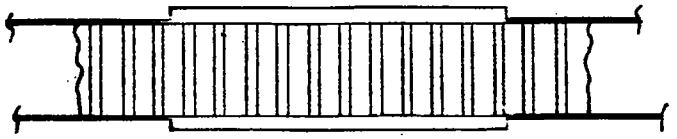


3b



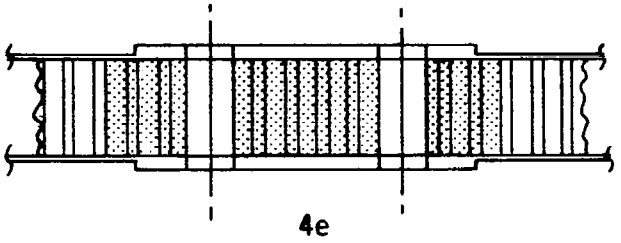
3b modified

Figure 3-14 : SELECTED TYPE 3 BOLTED JOINT CONCEPT



4b

Figure 3-15: SELECTED TYPE 4 BONDED JOINT CONCEPT--COVER



4e

Figure 3-16: SELECTED TYPE 4 BOLTED JOINT CONCEPT--COVER

4.0 MATERIALS AND SMALL COMPONENT CHARACTERIZATION

This section describes materials used for joint fabrication and presents results of design allowables and "Small Specimen" tests conducted to establish material properties and to support detail design of specific joint areas.

4.1 Materials

Composites

The composite joints characterized under this program were made from graphite/polyimide tape materials. Based on previous experience from the CASTS* composites for Advanced Space Transportation Systems (Contracts NAS1-15009 and NAS1-15644) program research, Boeing and NASA chose the Celion/PMR-15 material system. The graphite fibers were Celion 3000 and Celion 6000 with NR150B2G polyimide sizing. Preimpregnated tape was procured from US Polymeric, Inc. to a material specification contained in Reference 3. Laminate processing was specified to be according to procedures developed under NASA Contract NAS1-15009 (Ref. 3).

Adhesive

The high temperature adhesive used was designated A7F. A7F is a 50:50 resin solids copolymer blend of NASA's LARC-13 adhesive (supplied by NASA, Langley) (Ref. 2) and AMOCO's AI-1130 L Amide-imide. Sixty percent by weight aluminum powder and 5% by weight Cab-O-Sil are added. The adhesive was applied to 112 E-glass scrim to form a .25mm (.01 in) thick adhesive film.

Titanium

Titanium used was 6Al-4V (Standard) purchased to MIL-T-0946, Type III Comp. C.

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*Composites for Advanced Space Transportation Systems (Contracts NAS1-15009 and NAS1-15644

Mechanical Fasteners

Fasteners used were NAS 1303, with NAS 679 self-locking nuts.

Potting Compound

Two types of potting compounds were used in the joint areas. They were BMS 8-126 (Boeing Materials Spec.) high temperature structural foam, and BR34 polyimide resin with 6% aluminum powder filler from American Cynamid.

Honeycomb Core

Honeycomb core used was a bias weave glass/polyimide purchased from Hexcel Corporation to Boeing material specification XBMS 8-125. The Hexcel designation was HRH-327. Core used was 12.7 mm (0.50 in.) and 19 mm (.75 in.) thick, had a cell size of 4.76 mm (3/16 in.) and densities of 64.1 kg/m^3 (4 lb/ft^3) and 128.2 kg/m^3 (8 lb/ft^3).

4.2 Design Allowables Testing

A design allowables test program was conducted to evaluate graphite/polyimide composites over a temperature range of 116K (-250°F) to 589K (600°F). This program used a limited number of replications to establish an initial data base and identify performance trends. Statistically based allowables, "A" and "B" basis, were not determined. A total of 225 tests were conducted on Celion 6000/PMR-15 composites. A total of 189 tests were also conducted on Celion 3000/PMR-15 composites under Boeing IR&D funds. These tests measured tension, compression, flatwise (out-of-plane) tension, in-plane shear, interlaminar shear and coefficient of thermal expansion (CTE) properties. Typical average material properties for a quasi-isotropic laminate are given in Table 4-1. Effects of environmental conditioning are shown in Table 4-2. Test procedures and results are reported in detail in Reference 4. Material properties from these tests were used for final analysis and test correlation of each joint type.

4.3 Small Component Characterization

The small component characterization, "Small Specimen", test program was developed to provide design data of specific joint areas to support detail design of the joint concepts defined by the screening process. Tests were conducted to measure bolted joint strengths for net area tension, bearing and shear-out, shear strength of bonded versus co-cured doublers, and tension tests of bonded attachment angles. Effects of elevated temperatures (561K (550°F)) and environmental conditioning (cured/post-cured, aged, thermal cycled) were evaluated. Test results are summarized in the following sections.

Bolted Joints

Bolted joint testing was conducted for Celion 3000/PMR-15 laminates with $(0/\pm 45/90)_{8S}$ layups. Tests were conducted to measure shearout, bearing, and net area tension strength for a loaded hole, and net area tension strengths for an unloaded but filled hole. All tests were pin loaded type specimens (except for the unloaded hole). Results are summarized in Figure 4-1. The data show no significant change in strength for the three environmental conditions tested. There is, however, a significant drop in bearing strength at elevated temperature.

Comparison of the net area tension strengths of the Gr/PI with corresponding data for Gr/Ep from the literature (Ref. 5) shows the Gr/PI laminates are more sensitive (higher effective stress concentration factors) to holes than Gr/Ep laminates. This was as expected because of the brittleness of the Gr/PI system.

Bonded Versus Co-Cured Doublers

The basic skins of the covers being joined must have additional material added at the joint area to account for stress concentrations at bolt holes and

to enable the bonded joint to carry the design load. Tests were conducted to evaluate the relative efficiency of secondarily bonded doublers versus stacked co-cured doublers. Test setup and results are summarized in Figure 4-2. It was concluded that the stacked co-cured doubler was slightly stronger, and potentially less costly to fabricate, so this concept was used for the "Static Discriminator" specimens (Section 5.0).

Bonded Attachment Angles

Tension tests of bonded attachment angles were conducted to determine the strengths of the bonded concepts defined during the screening process. Three attachment concepts were evaluated; a single 90° angle, double 90° angles and a "T" section. Specimen loading and results are summarized in Figure 4-3. Results for the single 90° angle were below the minimum design requirement of 11.2 kN/m (64 lbs/in). Both the double 90° angle and the "T" section exceeded the maximum design requirement of 28.7 kN/m (164 lbs/in). Since the 90° angles are easier to fabricate than a "T" section, they were selected for the Type 1 and Type 4 bonded joints.

Table 4-1: QUASI-ISOTROPIC LAMINATE STRENGTHS CELION 3000/PMR-15 AND CELION 6000/PMR-15

MATERIAL	TEMPERATURE K(°F)	TENSION STRENGTH MPa (ksi)	TENSION MODULUS GPa (10^6 psi)	COMPRESSION STRENGTH MPa (ksi) ▷	COMPRESSION MODULUS GPa (10^6 psi)
CELION 3000/PMR-15 $[0/\pm45/90]^*_{NS}$ CURED/POST CURED	294 (70)	509 (73.8)	49.1 (7.13)	532 (77.2)	41.8 (6.1)
	589 (600)	482 (69.9)	44.4 (6.45)	470 (68.1)	44.2 (6.4)
CELION 6000/PMR-15 $[0/+45/90/-45]^{**}_{NS}$ DRYED	294 (70)	525 (76.1)	48.3 (7.0)	545 (79.0)	41.9 (6.1)
	589 (600)	516 (74.8)	46.2 (6.7)	380 (55.1)	50.7 (7.4)

Table 4-2: EFFECT OF ENVIRONMENTAL CONDITIONING CELION 3000/PMR-15 $[0/\pm45/90]^*_{NS}$

PROPERTY	TEMPERATURE K (°F)	CURED/POSTCURED MPa (ksi)	AGED MPa (ksi)	THERMAL CYCLE MPa (ksi)
TENSION STRENGTH	294 (70)	509 (73.8)	477 (69.2)	401 (58.1)
	561 (550)	482 (69.9)	452 (65.6)	376 (54.5)
COMPRESSION STRENGTH ▷	294 (70)	532 (77.2)	512 (74.2)	534 (77.4)
	561 (550)	470 (68.1)	413 (59.9)	448 (65.0)

* 51.4% F.V.

** 65.3% F.V.

▷ Celion 3000/PMR-15 Lay-up Was $(90/\pm45/0)_{NS}$

CELION 3000/PMR-15: Cured/Post-Cured, Aged - Average of 3 Specimens
Thermal Cycled - Average of 5 Specimens

CELION 6000/PMR-15: All - Average of 10 Specimens

NS: Symmetric Laminate, 2N Total Ply Groups

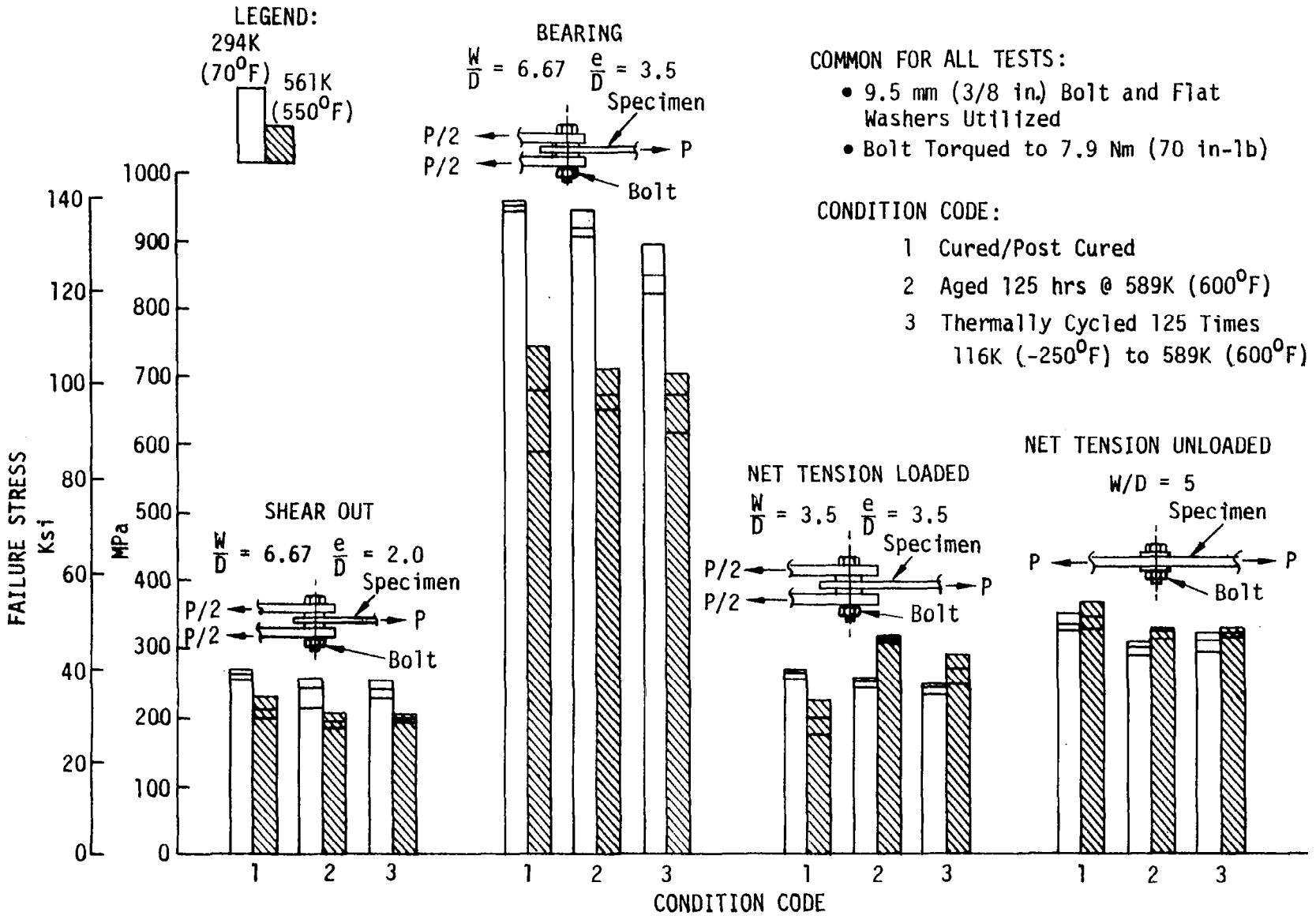

Figure 4-1: SUMMARY OF SMALL COMPONENT BOLTED JOINT TESTS CELION 3000/PMR-15 $(0/\pm 45/90)_{8S}$

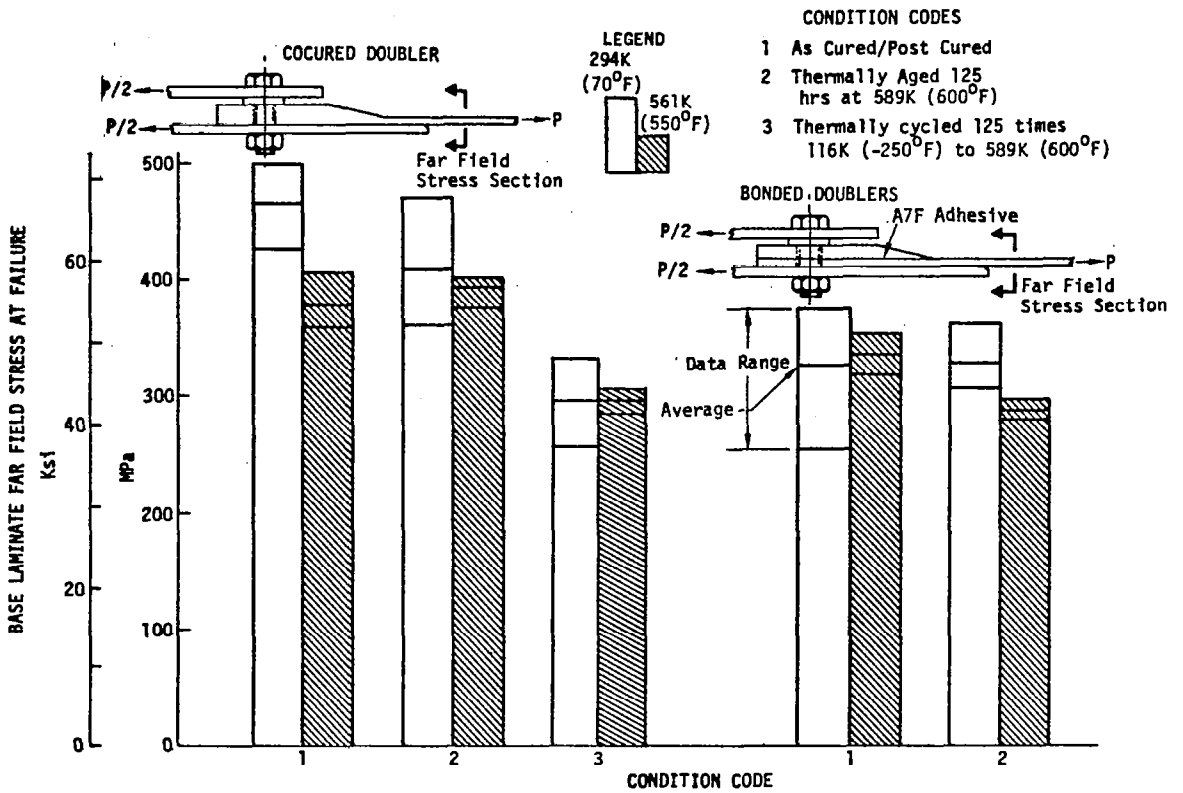

Figure 4-2: SMALL COMPONENT TEST RESULTS OF COCURED AND BONDED DOUBLERS
CELION 3000/PMR-15

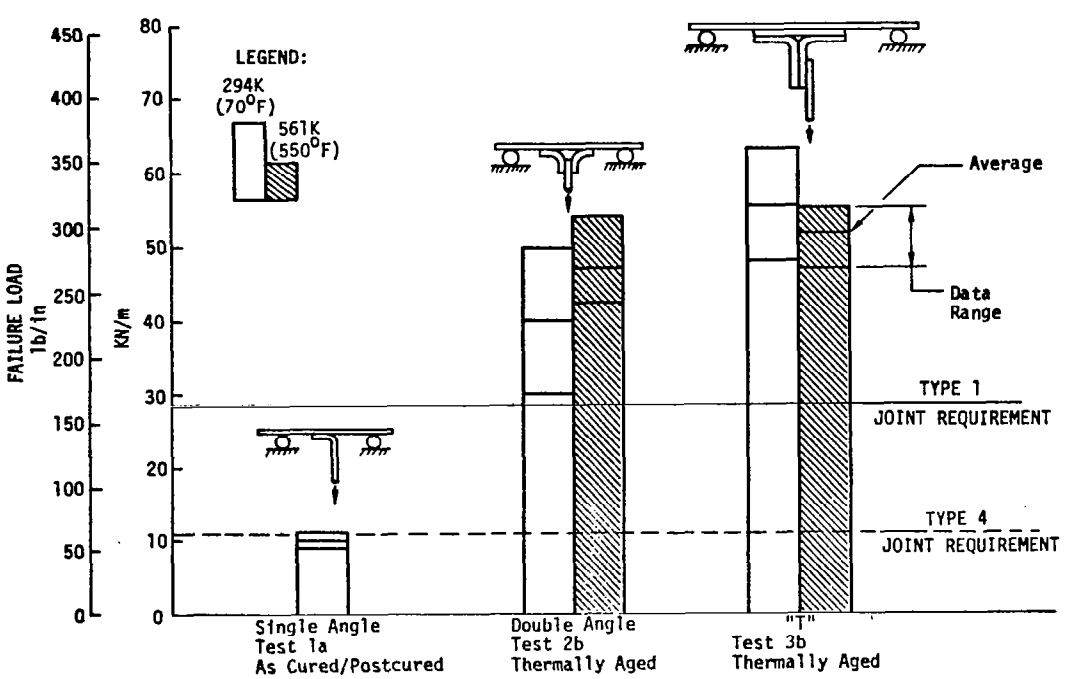

Figure 4-3: SMALL COMPONENT BONDED ATTACHMENT ANGLE PULL-OFF TESTS
CELION 3000/PMR-15

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5.0 STATIC DISCRIMINATOR TESTS

Starting with specified joint requirements and the basic joint concepts resulting from the screening process (see Figs. 3-7 through 3-16), preliminary joint designs were developed for each of the joint types. Each joint was sized using material properties and test results from the "Design Allowables" and "Small Specimen" tests presented in Section 4.0. These preliminary designs were subjected to "Static Discriminator" tests to verify structural adequacy. Each joint type was subjected to a single axis critical design load condition and loaded to failure. Only cured/post-cured specimens were tested. The test matrix and loading conditions are shown in Table 5-1.

Static Discriminator Test Results

Results for the "Static Discriminator" tests are summarized in Table 5-2. These tests demonstrated that the Type 2 and Type 4 joints would carry the design loads without requiring any design changes. However, since the Type 2 bolted joints greatly exceeded the design load, the corner angles were reduced in thickness for the "Final Evaluation" tests to reduce design conservatism.

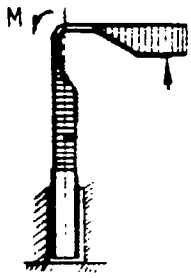
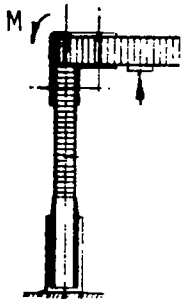
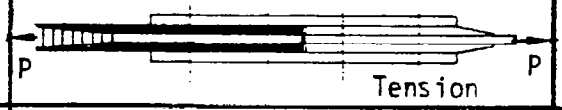
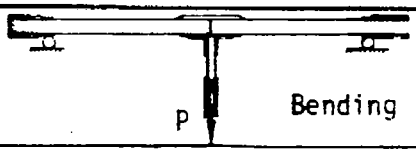
The Type 3 bolted joints failed at loads below the design load; however, this was due to premature failures in the grip. Since one specimen failed at 92% of the design load without failure of the joint, it was concluded that no design changes to the joint were required except to improve fabrication as discussed in Section 6.0. The load grips were altered for the "Final Evaluation" tests to improve their load transfer capability.

Two of the Type 1 bolted joints had cover tension failures outside the joint area at an average of 97% of the design load. All the other Type 1 joints experienced interlamina shear failures of the co-cured doublers at 67% to 79% of the design load (see Figs. 5-1 and 5-2). As a result, an interleaved doubler design, as shown in Figure 5-3, was incorporated into the Type 1 joints for the "Final Evaluation" tests. Special tests of the interleaved doubler design had shown that it would eliminate the interlamina shear failures.

During testing of bonded joints under TASK 2.0 of this contract, a "3-step" symmetric step-lap bonded joint was successfully fabricated and tested (Ref. 6). These joints sustained loads up to 875 kN/m (3000 lb/in) at 561K (550°F). This was the basic concept to be used for a Type 3 bonded joint except that it would have 6 steps instead of 3 to carry the higher design load (2100 kN/m (12000 lbs/in)). During the "Static Discriminator" tests three attemps were made to fabricate and test a symmetric step-lap bonded joint as the Type 3 Bonded preliminary design. The three attempts evaluated three different bond processing techniques in an effort to obtain a satisfactory bond. All three attempts were unsuccessful. C-scans of the bonds showed there were bond line voids on several of the steps for each of the processing techniques attempted. Because of program schedules and cost constraints further development of bond processing techniques was not possible. With NASA concurrence the Type 3 bonded joint was deleted from the "Final Evaluation" phase of the contract.

Subsequently, work on this program indicated that the bonding problem with the step-lap joints was due to the uneven heating of the titanium and Gr/PI. Since the cure pressure was applied after the entire panel reached the cure temperature, the thinner prepreg sections had cured or advanced past the gell stage before the pressure was applied resulting in poor bonds to the titanium. During cure of the "Final Evaluation" panels the cure cycle was altered to apply the pressure when the thinnest panel section reached the cure temperature. This procedure proved to be successful. It is also expected that it would lead to successful fabrication of thick step-lap joints of the kind required for the Type 3 Bonded joints.

Table 5-1: PRELIMINARY EVALUATION TEST MATRIX

JOINT TYPE	PRIMARY LOAD CONDITION	TASK 1.3 PRELIMINARY EVALUATION STATIC DISCRIMINATOR MATRIX 5 CURED/POSTCURED	
		294K (70°F)	561K (550°F)
TYPE 1 BONDED & BOLTED		●	●
TYPE 2 BONDED		●	●
TYPE 2 BOLTED		●	●
TYPE 3 BOLTED		●	●
TYPE 4 BONDED & BOLTED		●	●

Note: Nominally 3 specimens for each test condition.

Table 5-2: MATRIX 5 STATIC DISCRIMINATOR TEST RESULTS

TEST NO.	JOINT TYPE	TEMPERATURE K (°F)	AVERAGE** FAILURE LOAD kN/m (lb/in)	DESIGN LOAD kN/m (lb/in)	FAILURE MODE
1A	Type 1 Bonded	294 (70)	445 (2542)	560 (3200)	Doubler Shear
		561 (550)	378 (2159)	560 (3200)	Doubler Shear
1B	Type 1 Bolted	294 (70)	546 (3117) 438 (2500)	560 (3200)	Cover Tension (2)* Doubler Shear (1)*
		561 (550)	434 (2476)	560 (3200)	Doubler Shear
2A	Type 2 Bonded	294 (70)	418 [1] (94) [2]	285 [1] (64) [2]	Angle Delamination
		561 (550)	632 (142)	285 (64)	Angle Delamination
2B	Type 2 Bolted	294 (70)	1293 [1] (291) [2]	285 [1] (64) [2]	Angle Delamination
		561 (550)	1453 (327)	285 (64)	Angle Delamination
3B	Type 3 Bolted	294 (70)	1690(9653) [3]	2100 (12000)	Grip Failure
		561 (550)	1667(9520)	2100 (12000)	Grip Failure
4A	Type 4 Bonded	294 (70)	19.4 (111)	11.2 (64)	Attach Angle Pull Off (2)* Cover Compression (1)*
		561 (550)	12.3 (70)	11.2 (64)	Attach Angle Pull Off (1)* Cover Compression (2)*
4B	Type 4 Bolted	294 (70)	22.2 (127)	11.2 (64)	Cover Compression
		561 (550)	14.7 (84)	11.2 (64)	Cover Compression

[1] mm-N/mm [2] in-lbs/in [3] One specimen had titanium splice plates

*Number of Specimens (Nominally 3 Per Test)

** Load in direction shown in Table 5-1 per unit width.

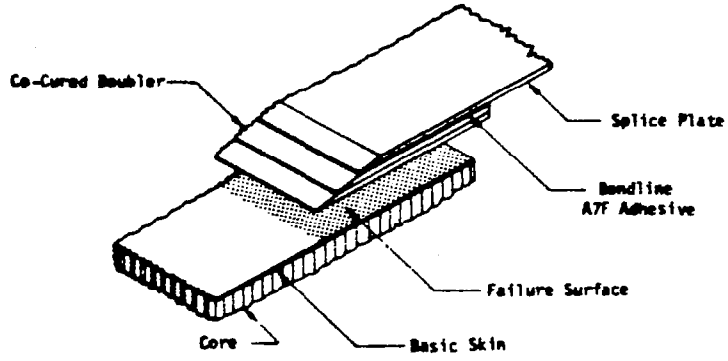


Figure 5-1 : STATIC DISCRIMINATOR TESTS, TYPE 1 BONDED, FAILURE MODE

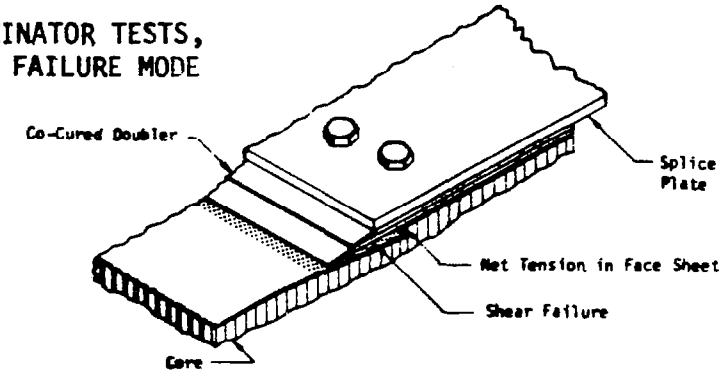


Figure 5-2 : STATIC DISCRIMINATOR TESTS, TYPE 1 BOLTED, FAILURE MODE

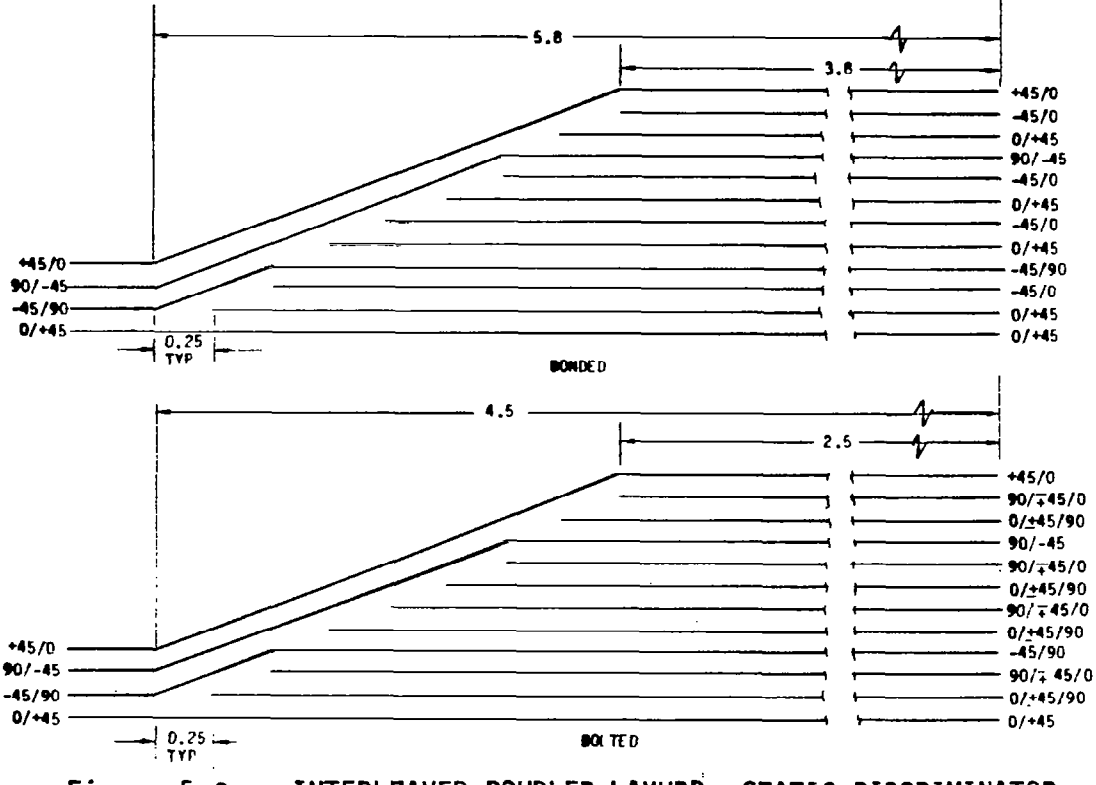


Figure 5-3 : INTERLEAVED DOUBLER LAYUPS, STATIC DISCRIMINATOR TYPE 1 JOINTS - ALTERNATE DESIGN

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6.0 FINAL JOINT DESIGNS

Results of the "Static Discriminator" tests of the preliminary joint designs were used to determine any design deficiencies. These were corrected and incorporated into the final designs for each joint type. The final design configurations were then subjected to a series of "Final Evaluation" tests. This section presents the designs for each joint type and identifies the design changes incorporated as a result of the "Static Discriminator" tests.

All joints, with the exception of Type 2, were designed to fail in the basic cover skins outside the joint area. The critical load for Type 2 joints was load case 2 (see Table 3-3). This gives the maximum corner moment and tension in the web but does not produce an ultimate load condition in the cover skin. Type 2 joints would therefore fail in the joint area. Designs for each joint type are discussed below.

Type 1 Bonded & Bolted Joints

As a result of the concept screening a double-lap joint with the inner adherend being a laminate and honeycomb core sandwich construction (see Fig. 3-9) was selected for the Type 1 joints. The bonded joint lap length was selected to result in failure of the basic cover outside the joint. This meant the basic cover had to be reinforced in the joint area. Results of the Task 2.0 double-lap standard bonded joints were used to select the lap length and adherend thickness required. Results of standard double-lap bonded joint tests had also shown a significant increase in joint performance was achieved by tapering the outer adherends and by increasing the laminate axial and flexural stiffness.

The bolted joints were also designed to fail in the cover outside of the joint. The splice plates were designed to fail initially in bearing to prevent a "two part" catastrophic failure of the plates. Bolt bearing, net

tension and shear-out strengths for the quasi-isotropic laminate were determined from the bolted joint tests described in Section 4.0 and were used to finalize local joint geometry.

Stacked co-cured doublers were used for the preliminary design specimens based on the results of the "Small Specimen" tests (Section 4.0). When subjected to "Static Discriminator" tension tests the specimens had premature failures due to interlaminar shear at the doubler to skin interface (see Section 5.0). Interleaved doublers were incorporated to eliminate the premature shear failure by distributing the load transfer from the basic skin over several shear interfaces instead of just one.

Double 90^{0} web attachment angles were used because of manufacturing simplicity and because the "Small Specimen" tests (see Section 4.0) showed they were adequate for the design loads.

Final designs for the Type 1 Bonded and Bolted joints are shown in Figures 6-1 and 6-2.

Type 2 Bonded and Bolted Joints

Results of the screening study showed the Type 2 Joints should be the basic concepts shown in Figures 3-11 and 3-12. For the bonded joint design, the cover and web are bonded to the corner angle in separate operations so proper bonding pressure can be maintained. The corner angle was sized to carry the moment and resulting bending loads around the corner. Bonded lap lengths were selected to carry the equivalent line loads resulting from the moments. For the bolted joint design the inner and outer corner angles were also sized to carry the design moment around the corner and the resulting bending loads. The corner angles of the bolted joint were reduced in thickness from the preliminary designs because of the higher than required failure loads from the "Static Discriminator" tests (see Section 5.0). These changes were incorporated to simplify fabrication and reduce joint weight.

The Type 2 bonded joint is more flexible than the bolted joint design due to its thinner corner cross-section. The bonded joint could produce undesirably large deflections. Deflection limited design criteria would probably require revision of this design.

Final designs for the Type 2 Bonded and Bolted joints are shown in Figures 6-3 and 6-4.

Type 3 Bolted Joints

There were two concepts selected during screening for the Type 3 Bolted Joint as indicated in Figure 3-14. They were basically double-lap joints with one concept having a Gr/PI splice plate and the other a titanium splice plate. Splice plate and cover reinforcement areas were sized using net-tension and bearing allowables determined from the "Small Specimen" tests presented in Section 4.0. The reinforcement area consisted of continuous plies from the basic cover away from the joint interleaved with filler plies to provide the required pad-up thickness. For simplicity the total pad-up thickness was increased to match the basic cover total thickness thus avoiding costly tapered lay-up tooling.

The "Static Discriminator" specimens had extensive delamination in the bolt pad-up area. For the final design this area was made as three pieces secondarily bonded together instead of two. This allowed the laminates to be layed-up in thinner sections and provided for escape of volatiles during curing and precluded delaminations.

The final design for the Type 3 Bolted joint had GR/PI splice plates and is shown in Figure 6-5.

Type 4 Bonded and Bolted Joints

The basic Type 4 Joint selected from the screening process is shown in Figure 3-15. Laminates are unsymmetric lay-ups in order to provide a minimum gage

design; however, sandwich midplane symmetry was maintained. The cover was reinforced in the joint area for both the bonded and bolted joints. Reinforcement was put on both sides of the sandwich to maintain stiffness balance and assure uniform transfer of in-plane load to both skins. Bolted joints were also reinforced in the joint area to account for reduction in strength due to the bolt holes. Bonded double 90° web attachment angles were used for the web because of simplicity in manufacturing. The "Small Specimen" tests showed they were adequate for the design loads (see Section 4.0). The ± 45 plies were on the outer surface of the attachment angles and cover reinforcement to provide maximum shear strength of the bonds. Double 90° web attachment angles were also used for the bolted web.

Final designs for the Type 4 Bonded and Bolted joints are shown in Figures 6-6 and 6-7.

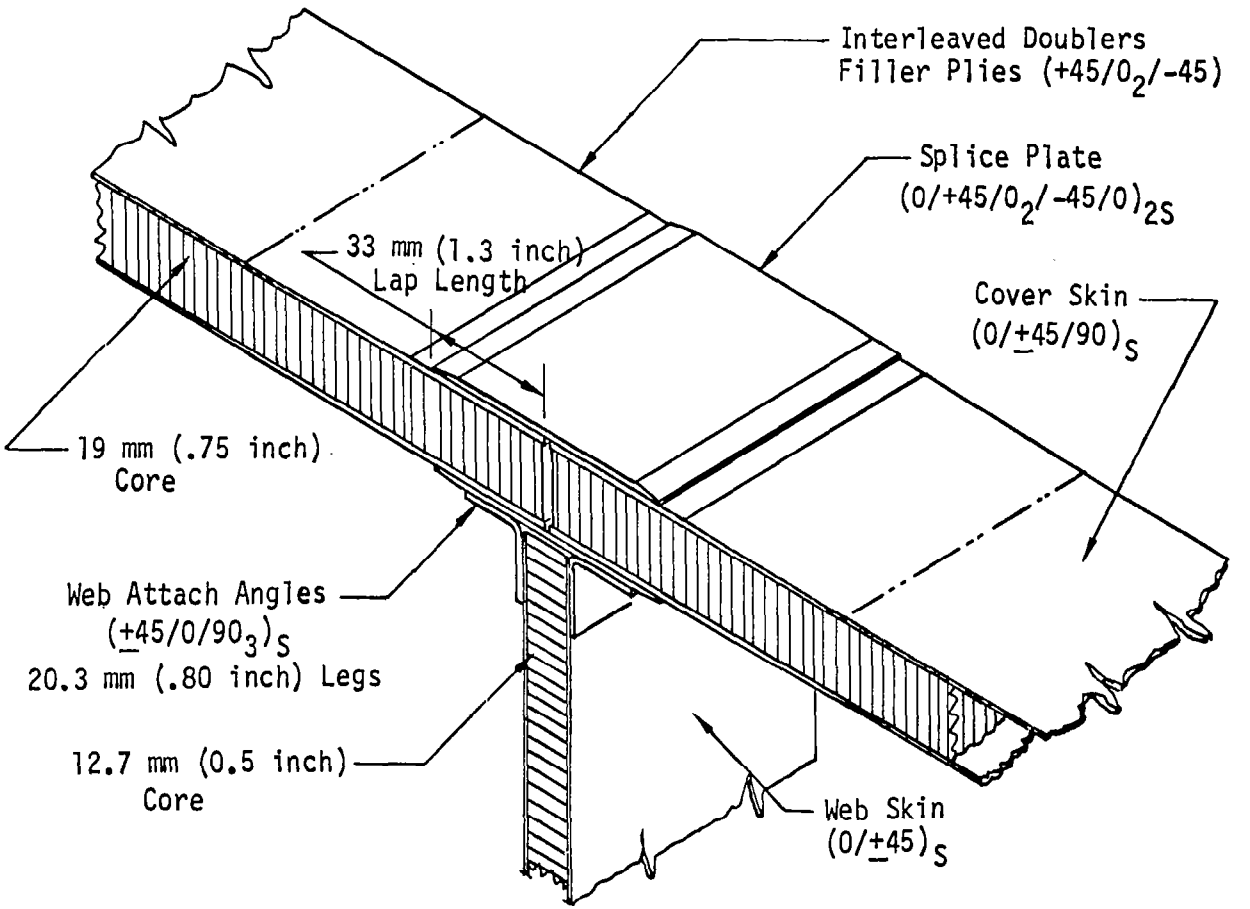


Figure 6-1: FINAL DESIGN TYPE 1 BONDED JOINT

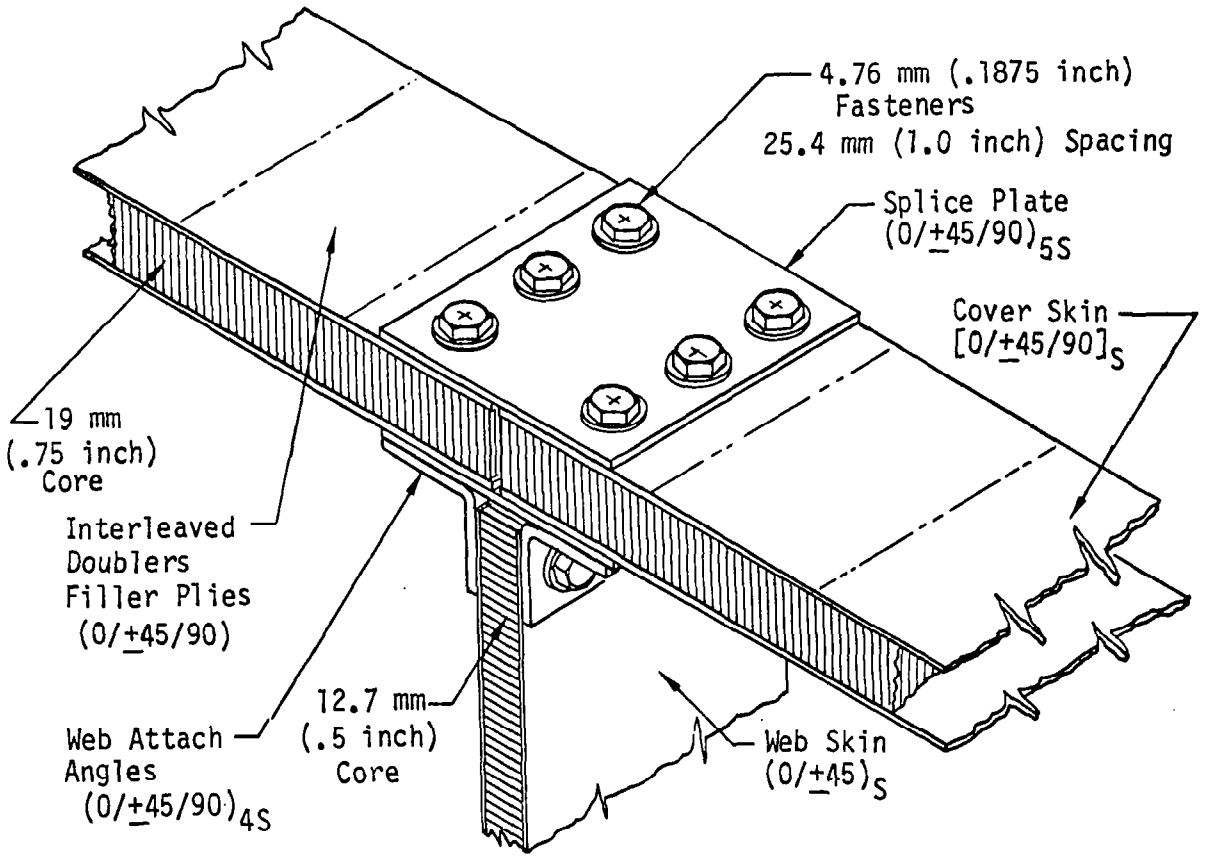


Figure 6-2: FINAL DESIGN TYPE 1 BOLTED JOINT

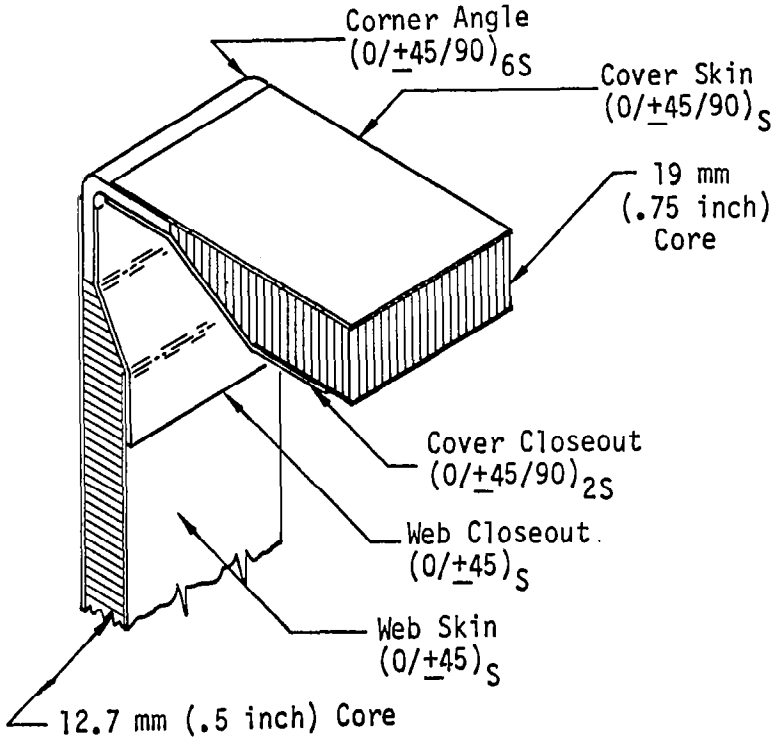


Figure 6-3: FINAL DESIGN TYPE 2 BONDED JOINT

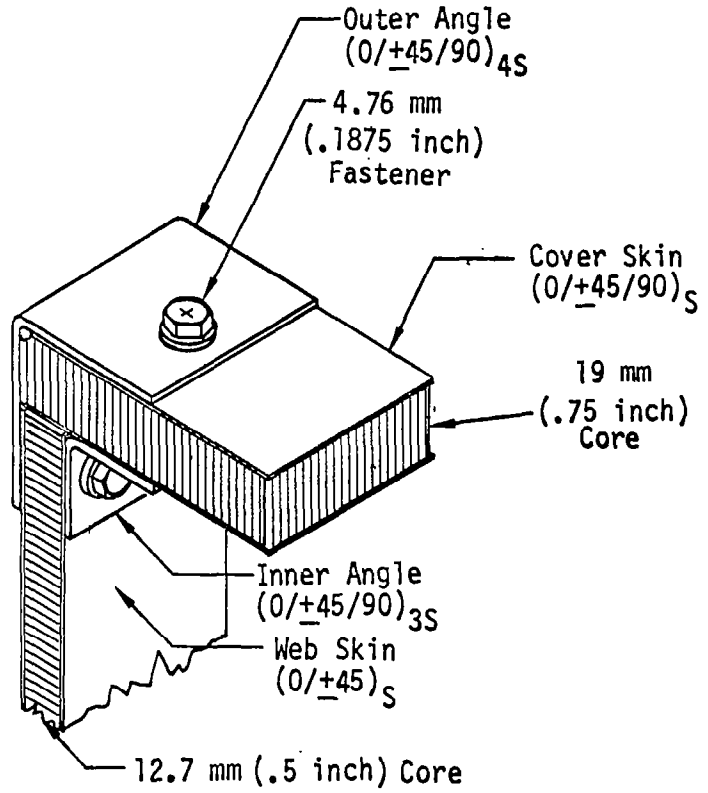


Figure 6-4: FINAL DESIGN TYPE 2 BOLTED JOINT

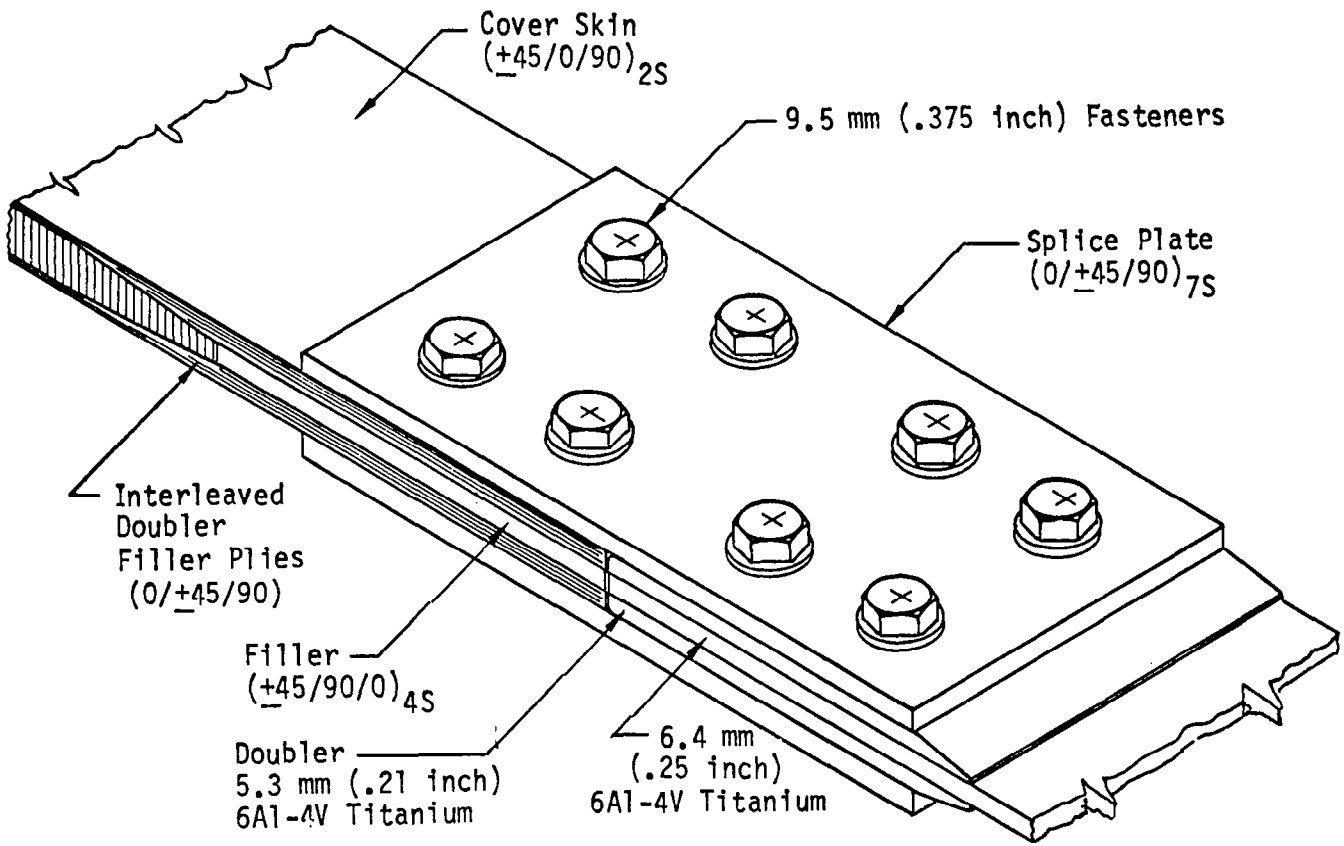


Figure 6-5: FINAL DESIGN TYPE 3 BOLTED JOINT

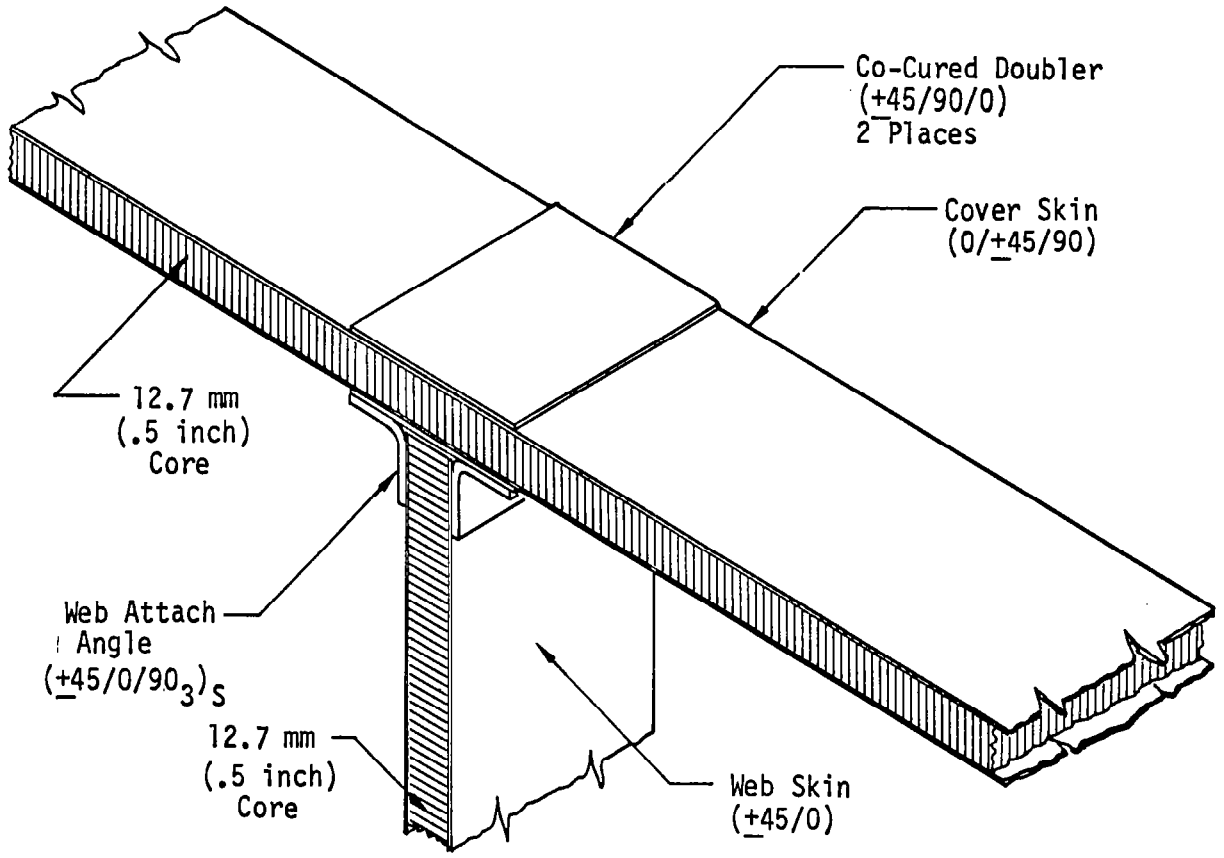


Figure 6-6: FINAL DESIGN TYPE 4 BONDED JOINT

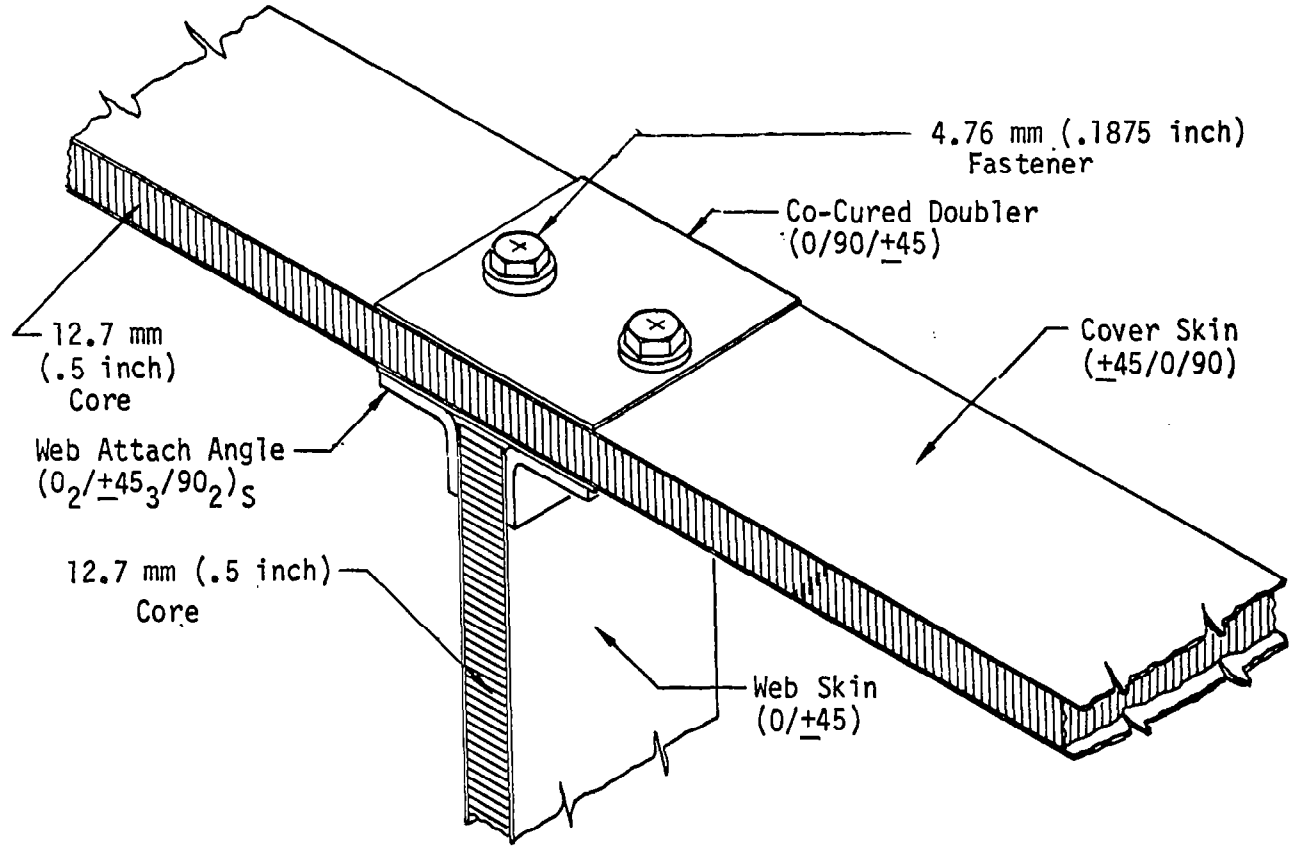
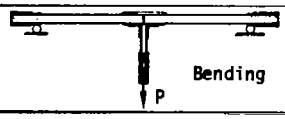
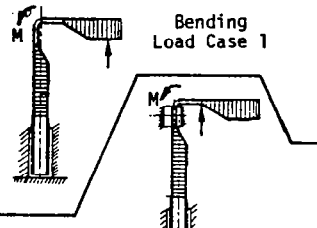
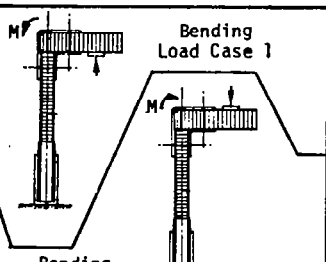
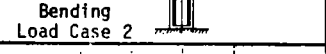
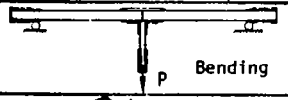
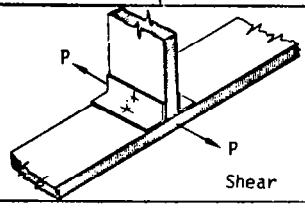


Figure 6-7: FINAL DESIGN TYPE 4 BOLTED JOINT

7.0 TEST MATRICES AND PROCEDURES

The final joint designs (see Section 6.0) were subjected to "Final Evaluation" testing to verify the validity of the scaled-up manufacturing process and to evaluate the structural integrity of the joint designs. A series of static tests (Matrix 7.1 of Table 7-1), identical to the "Static Discriminator" tests were conducted on specimens cut from large panels. These tests were to demonstrate that there was no degradation in joint strength due to the scaled-up manufacturing process and to validate the final designs. Specimens cut from the remaining portion of the scaled-up joint were thermally conditioned and tested in a series of static (Matrix 7.2 of Table 7-1) and fatigue (Matrix 7.3 of Table 7-1) tests to evaluate the structural integrity of each joint design. Two types of tests-to-failure were performed for each joint type, except for the Type 3 joints where only one load condition was required. Test matrices and loading conditions for the Scale-up Verification, Static Strength and Fatigue tests are summarized in Table 7-1. Test results for each joint type are discussed in Section 9.0.

Table 7-1: FINAL EVALUATION TEST MATRIX

JOINT TYPE	LOAD CONDITION	TASK 1.4 FINAL EVALUATION PHASE					
		SCALE-UP VERIFICATION MATRIX 7.1	STATIC STRENGTH EVALUATION MATRIX 7.2			FATIGUE EVALUATION MATRIX 7.3	
		CURED/POSTCURED	AGED		THERMAL CYCLED	AGED	
		561K (550°F)	294K (70°F)	561K (550°F)	561K (550°F)	294K (70°F)	561K (550°F)
TYPE 1 BONDED & BOLTED		●	●	●	●	●	●
			●	●			
TYPE 2 BONDED		●	●	●			
			●	●	●	●	●
TYPE 2 BOLTED		●	●	●	●	●	●
			●	●			
TYPE 3 BOLTED		●	●	●	●	●	●
TYPE 4 BONDED & BOLTED		●	●	●	●	●	●
			●	●			

Note: Nominally 3 specimens for each test condition.

8.0 SPECIMEN FABRICATION

All specimens for this program were fabricated in the Boeing Materials technology Laboratories. Specimens for "Small Specimen" tests and "Static Discriminator" tests were fabricated using small laboratory size panels nominally up to 0.6 m (2 ft) wide. Specimens for the "Final Evaluation" phase were fabricated in scaled-up configurations to demonstrate that the parts could be made in sizes required for production type programs. These parts were made in lengths up to 2.1 m (7 ft). An overall flow diagram showing the fabrication procedure is given in Figure 8-1.

Prior to making panels for the test specimens, prepreg received from the vendor was subjected to Quality Control (Q.C.) tests to assure its acceptability. Tests included mechanical property tests and chemical characterization tests as specified in the material specification (Ref. 3). In some cases, material with Q.C. mechanical properties slightly lower than the specification requirements was accepted. This was because of the experimental nature of this material system and the fact that the specification requirements were based on a sample size. The primary control for acceptance or rejection of the prepreg was the chemical characterization test of the prepreg resin using high pressure liquid chromatography. Liquid chromatography has the sensitivity required to detect small amounts of undesirable resin constituents (reaction products) that affect processing. Results of these tests were considered the principal indicator of material processability.

The Gr/PI prepreg was laid up and processed using autoclave processing procedures defined in Reference 3. The material processing was developed and studied by Boeing under the NASA, LaRC sponsored CASTS program, contract NAS1-15009. Cured laminates were non-destructively inspected using C-scan at 5.6 MHz sweep at 4 dB loss above the water path. Panels containing voids or other defects were rejected and new panels made. Typical C-scan results for an unacceptable and a rejected laminate are shown in Figure 8-2.

The curing of panels with variable thickness (i.e., such as thin panels with doublers) required slight deviations from standard procedures. The temperature at which pressure was applied was controlled based on the temperature of the thin section of the laminate rather than the thick portion. This was to assure the resin had not started to gel while the thick portion was reaching temperature and thus preventing proper resin flow when the pressure was applied. This procedure was successful.

Figures 8-3 and 8-4 show typical scaled-up joint detail parts prior to cutting into specimens.

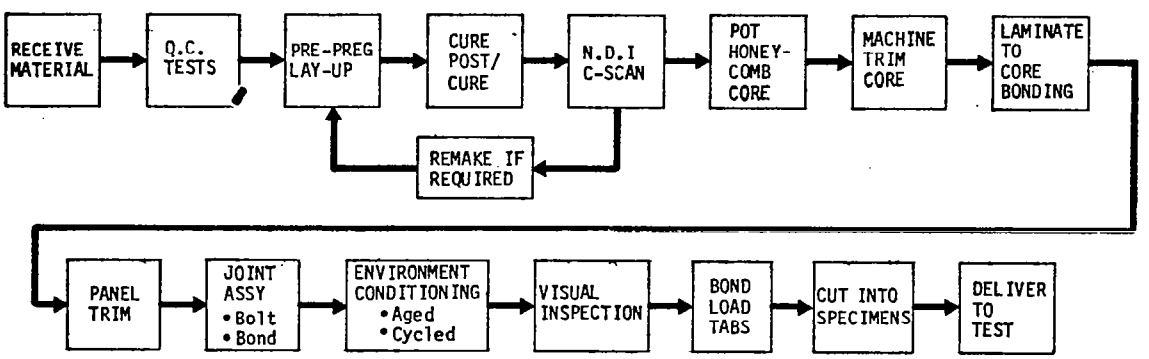
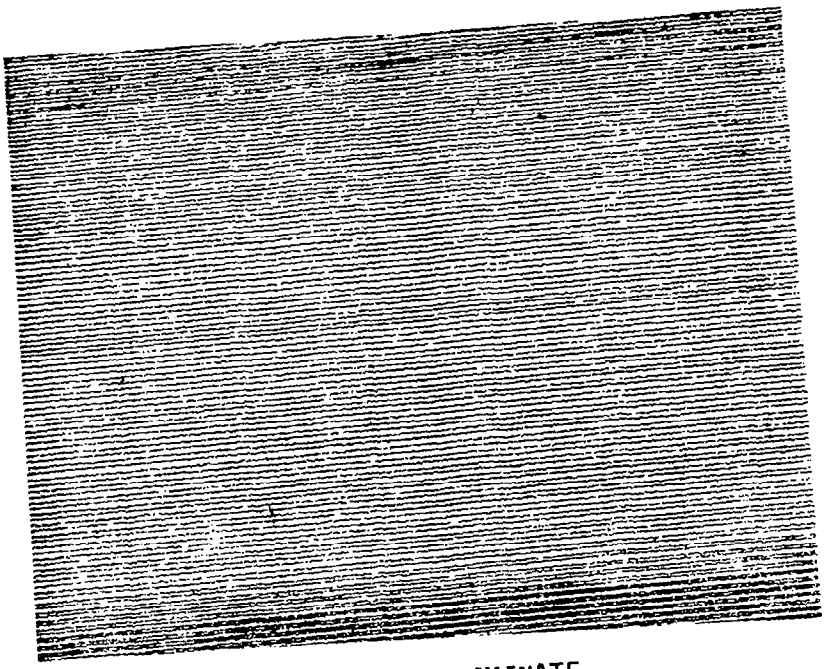
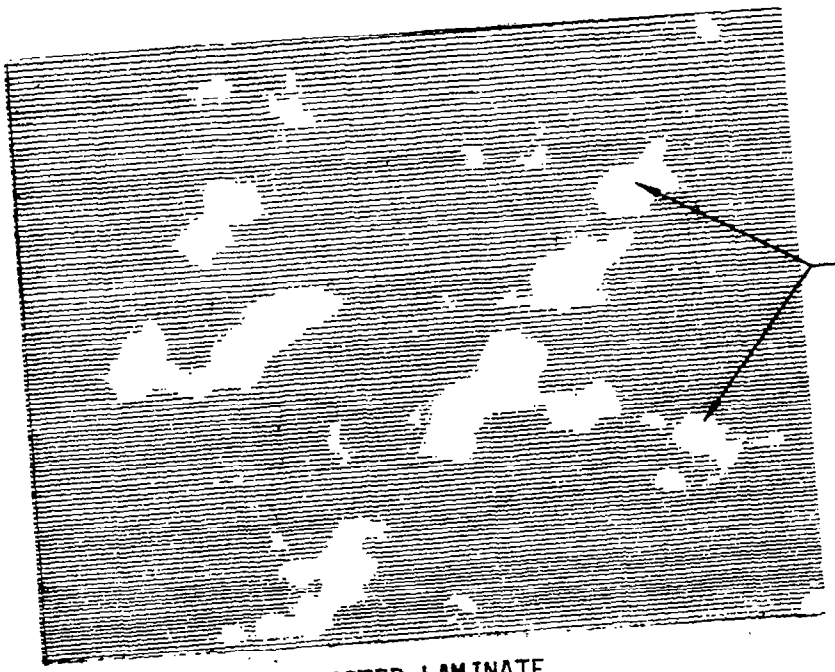


Figure 8-1: SPECIMEN FABRICATION FLOW DIAGRAM



ACCEPTABLE LAMINATE



REJECTED LAMINATE

Figure 8-2: TYPICAL C-SCAN RESULTS

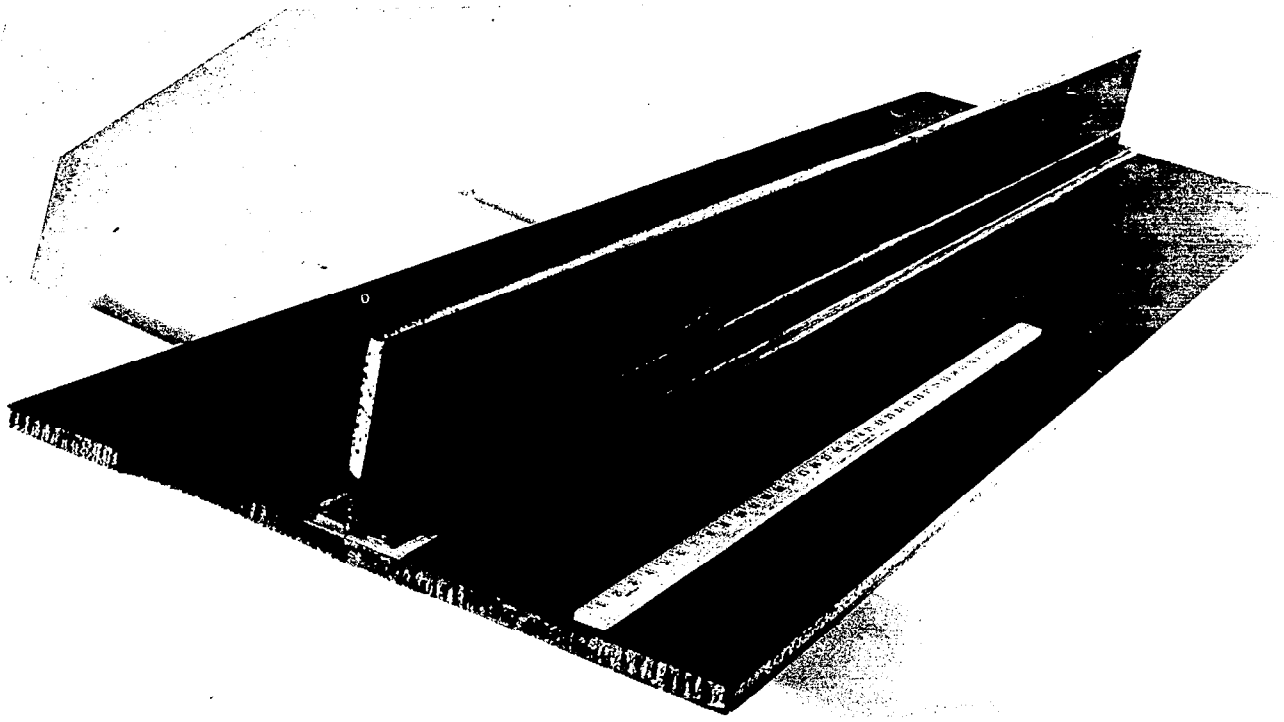


Figure 8-3: TYPE 1 BONDED JOINT SCALED-UP ASSEMBLY

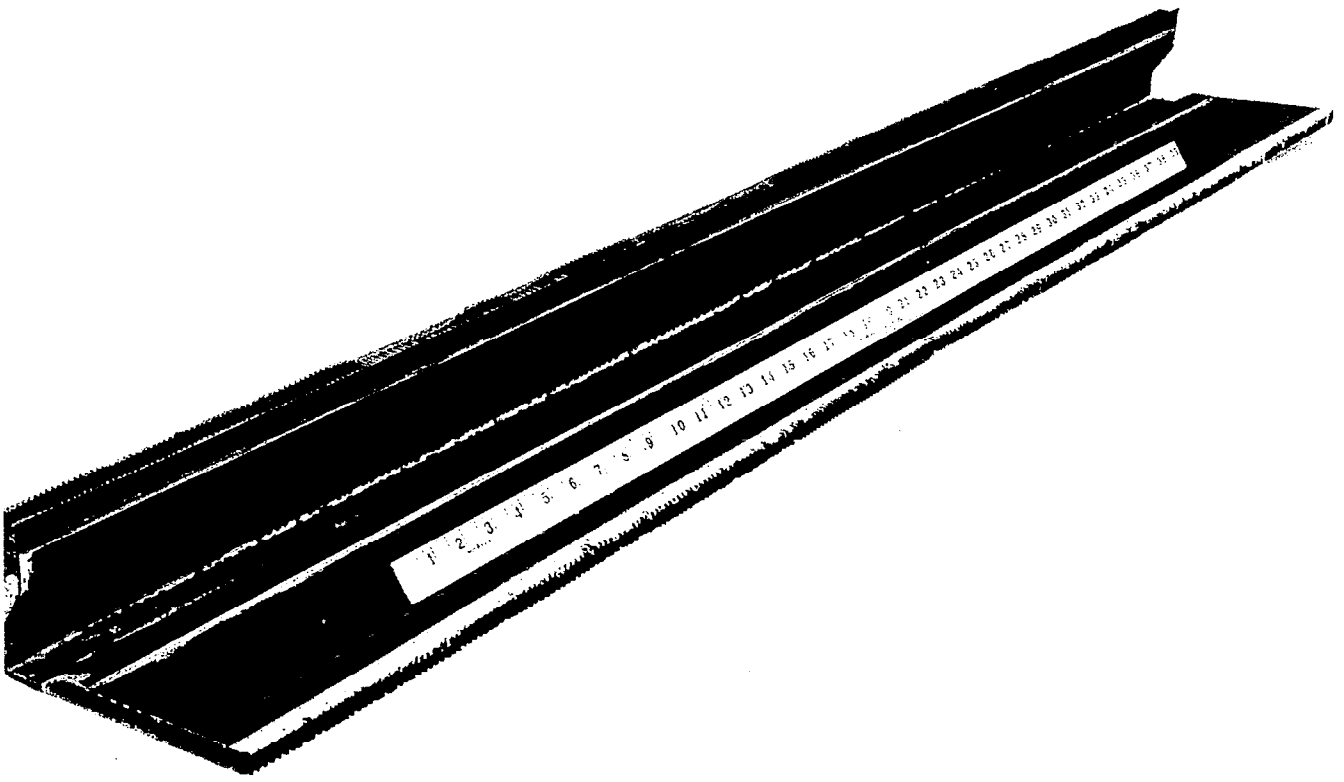


Figure 8-4: TYPE 2 BONDED JOINT SCALED-UP ASSEMBLY

9.0 FINAL EVALUATION TEST RESULTS

This section discusses "Final Evaluation" static and fatigue test results and the resin chemistry and adhesive processing problems experienced.

9.1 Final Evaluation Test Results

Static Strength Tests

Results of the "Final Evaluation" static tests for each joint type are summarized and compared to the design loads and predicted failure loads in Figures 9-1 through 9-7. The design loads were based on preliminary material properties, while the predicted strengths were based on material properties from the design allowables testing (Ref. 4). Each figure shows the average failure load and data range for each temperature, specimen conditioning and load case tested. Residual strengths after fatigue testing are also shown if applicable.

For all joint types there were large variations in failure loads and failure modes. Despite the large variations, in all cases there were some specimens that met or exceeded the design load, except for Type 4 bonded joints (see Fig. 9-6). The identical Type 4 bonded joint design exceeded the design load during the "Static Discriminator" tests (see Table 5-2). The low failure loads for the "Final Evaluation" Type 4 bonded are attributed to bad laminates and adhesive bonds as discussed in Section 9.2.

A summary of maximum failure loads for each joint type is given in Table 9-1. Failures occurred outside the joint area except for Type 2 joints. Typical failures of specimens that met the design load for each joint type are shown in Figures 9-8 through 9-17. It is concluded that these type joints can be fabricated from a Celion 3000-6000/PMR-15 material system and that they will sustain the load levels specified in this program for control surfaces on advanced aerospace vehicles and space transportation systems.

The low failure loads and failure modes experienced during testing are attributed to grip problems and in some cases to resin chemistry and adhesive processing problems which are discussed in Section 9.2. The polymide resin problem was demonstrated by extensive delamination of laminates that failed in tension and that had outer play buckling and peeling of laminates under compression, with corresponding low failure loads. Typical grip failures and specimens with excessive delaminations and laminate buckling/peeling are shown in Figures 9-18 through 9-22. There were some specimens which were not tested that had laminates or adhesive bonds which were badly damaged during aging or thermal cycling. Typical bad adhesive bonds are shown in Figures 9-23 and 9-24.

Because of the large variations in failure loads and modes, no firm conclusion can be drawn regarding the effects of aging and thermal cycling on joint performance. Trends do indicate, however, that the effects are small for tension loading conditions (see Fig. 9-1). This is consistent with the results of design allowables testing reported in Reference 4. Results for Type 2 bonded joints indicate a significant loss in strength due to thermal cycling if the failure mode is transverse tension or peel (see Fig. 9-3). The large loss in strength may be attributable to microcracking observed in thermally cycled laminates during design allowables testing (Ref. 4).

Fatigue Tests

Each joint type was subjected to fatigue testing using the critical load condition for the static tests. Maximum fatigue loads were 67% of the ultimate static load determined from the static evaluation tests. The load ratio was +.05 at a frequency of 7 cps (except for Type 3 joints which were tested at 6 cps). Specimens that sustained 10^6 cycles without failure were tested statically to determine their residual strength. Results of fatigue tests for each joint type are summarized in Table 9-2.

The Type 1 bonded joints has premature failures in the grip area, however, one room temperature specimen did go 953,000 cycles without a joint failure. it

is concluded that the Type 1 bonded joints are good for 10^6 fatigue cycles at room temperature. At elevated temperature the joints can sustain at least 553,000 cycles. All of the Type 1 bolted joints sustained 10^6 cycles without failure. The ply delaminations are attributed to the resin problem discussed in Section 9.2. Residual strength tests showed that the joint itself was not degraded due to fatigue cycling.

The Type 2 joints sustained 10^6 cyles at room temperature without failure but not at elevated temperature; however, even though three specimens had angles delaminate at elevated temperature, they were still able to carry the design load up to 10^6 cycles.

Two room temperature Type 3 bolted joints sustained 10^6 cycles, while the third failed at 914,000 cycles. Only a small decrease in residual strength was experienced, indicating that these joints can withstand the fatigue environment at room temperature. However, at elevated temperature the Type 3 joints sustained a maximum of only 383,000 cycles. It is believed that these premature failures are due to the material problems discussed below.

No firm conclusions can be drawn regarding the Type 4 bolted joints because of the large variations in failure loads and failure modes. Although the attachment angles delaminated in all cases, they were still able to sustain the design load. Two specimens sustained 10^6 cycles without cover failures which indicate the laminates are adequate for the fatigue environment.

The large differences in fatigue test results are attributed to the resin chemistry and adhesive processing problems discussed below.

Residual strengths of those specimens that did sustain 10^6 cycles showed both a +39% increase and a -10% decrease as compared to non-fatigued specimens; however, the changes were well within the range of data scatter. The data indicate the joints can sustain the fatigue environment without a catastrophic reduction in residual strength. It should be noted that the potting compound used to reinforce the honeycomb core around the bolts in the joints

was placed in a band across the entire joint along the line of bolts. In full-scale production hardware potting would most likely be only placed locally around each hole. It is possible that fatigue lives may be reduced when the potting is applied only locally around a hole.

9.2 Material Processing Problems

During specimen conditioning and testing for the "Final Evaluation" phase of this program several problems and anomalies occurred which appear to be material processing problems. In several cases, there were extensive laminate delaminations under tension or compression loads with resultant failures at much lower loads than predicted. Visual examination of specimens after aging and thermal cycling showed a much darker appearance than cured/post-cured specimens indicating a loss of resin. Some specimens had "fuzzy" surface areas which were actually bare fibers. Specimens from earlier "Design Allowables" and "Small Specimen" tests, which had also undergone aging and thermal cycling, were reexamined to see if they had any evidence of delamination or resin loss. There was no evidence of material change due to the conditioning environments, nor did the test results indicate any changes in material performance during these earlier tests (see Fig. 4-1).

In some cases, conditioning of the "Final Evaluation" specimens resulted in a complete loss of the A7F adhesive bond. All of the adhesive resin was destroyed leaving a residue of scrim cloth and aluminum powder. The adhesive loss occurred at laminate-to-core bonds (see Fig. 9-23) and laminate-to-laminate bonds (see Fig. 9-24) but was not consistent. Specimens cut from the same full scale joint assembly panel lost adhesive during thermal cycling but not during aging while others lost the adhesive during both aging and thermal cycling. No specimens lost adhesive during cure/post-cure.

The degradation of the Gr/PI laminates due to thermal aging and cycling is attributed to a low percentage of Nadic Ester (NE) in the PMR-15 polyimide resin. Based on past experience PMR-15 resin which contains 2.8% NE has a shelf life of 60 days when kept at a temperature of $0^{o}C$. However, the later

batches of prepreg used for the "Final Evaluation" panels had an initial NE content of 2.5%. Quality control panels which were made from these batches had good mechanical properties after both thermal aging and cycling. Based on the mechanical property results this material was accepted for use. However, as discussed above, laminates which were made from this material experienced a loss of matrix resin after thermal aging and cycling. Upon review of liquid chromatography data on NE from fresh and 45 day aged PMR-15 resins, it is apparent that the PMR-15 resin loses NE with time, even while stored at $0^{o}C$. Compare the NE content shown in Figures 9-31 and 9-32 for fresh and 45 aged PMR-15 resin respectively. Thus, even though the PMR-15 resin with the low NE content showed good mechanical properties initially, its shelf life was reduced, resulting in the use of material which had an insufficient NE content to achieve proper cure of the PMR-15 resin. Therefore it is recommended that a shelf life be determined from the initial NE content of the resin, and upon expiration of that life quality control tests be repeated before the material is used for fabrication.

The degradation of the A7F adhesive has been attributed to overheating of the adhesive during the DMF solvent stripping process prior to coating onto the 112 E-glass scrim. The adhesive film for the "Final Evaluation" panels was prepared by US Polymeric. The DMF solvent was not used in the process used by the Boeing Materials Technology labs to prepare the previous batches of adhesive used in this program and thus the stripping difficulties were not encountered earlier. Only one of the two batches of A7F adhesive prepared by US Polymeric resulted in bad bonds. During the stripping of the DMF solvent from the second batch of adhesive the AI-1130L amide-imide resin was advancing (partially curing) due to overheating. Therefore the properties of the adhesive were degraded, resulting in a loss of bond strength after thermal aging and cycling.

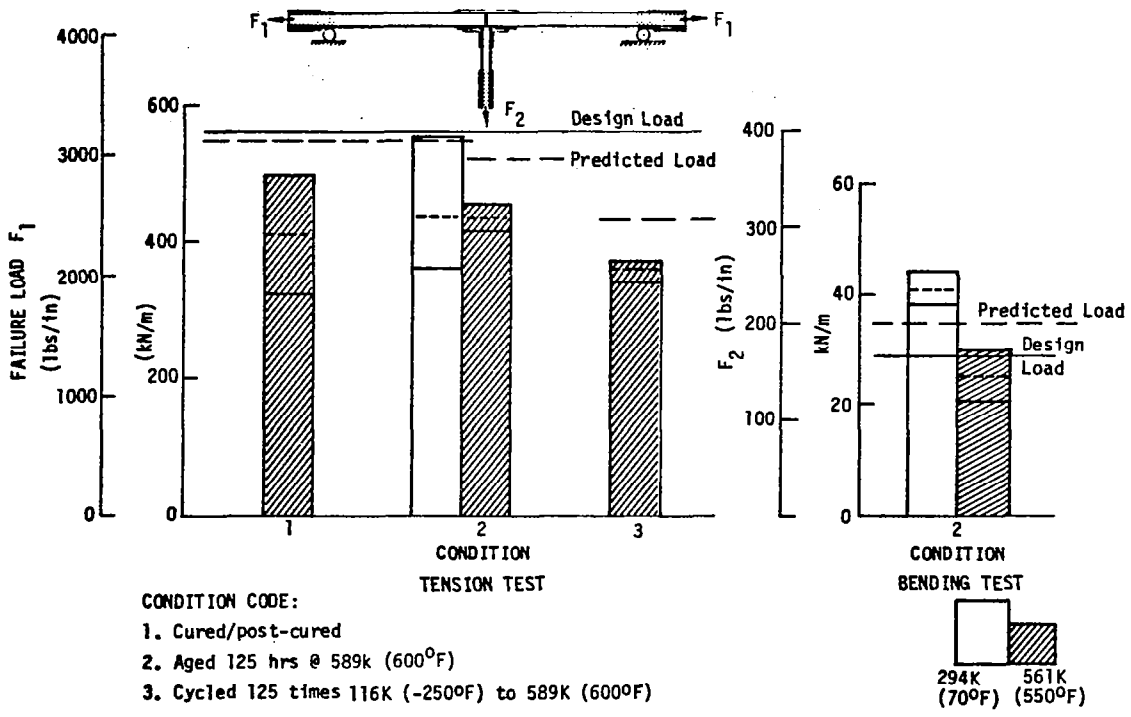


Figure 9-1: TYPE 1 BONDED JOINT STATIC TEST RESULTS

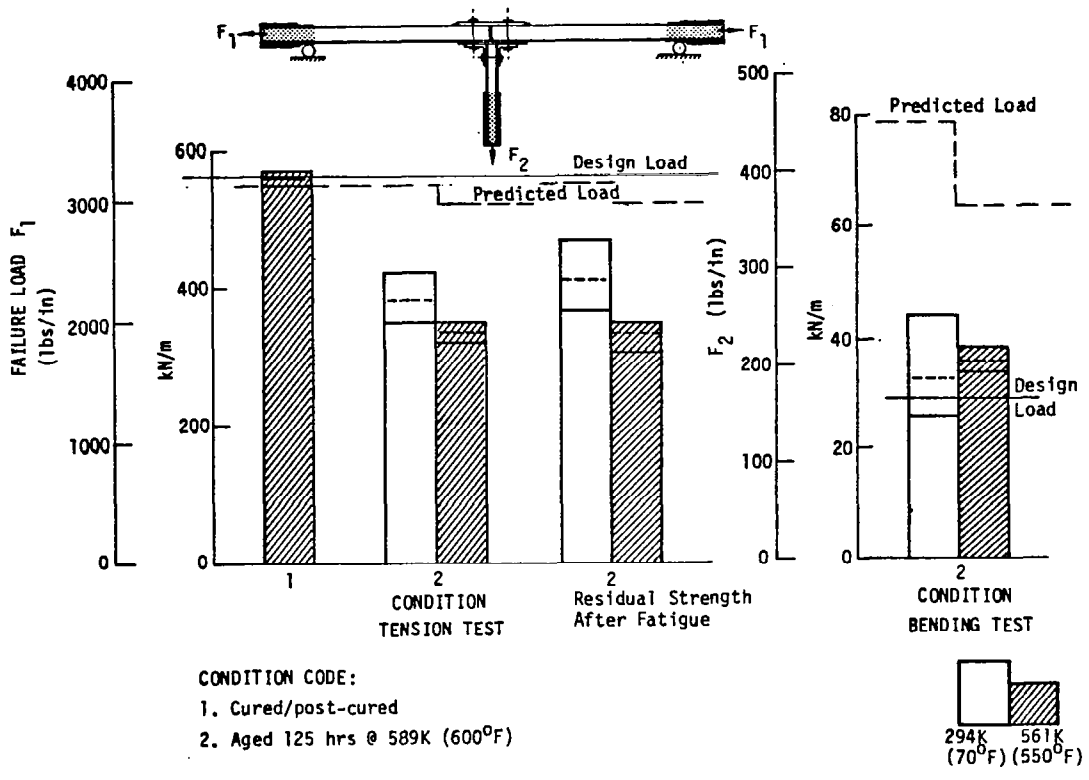


Figure 9-2: TYPE 1 BOLTED JOINT STATIC TEST RESULTS

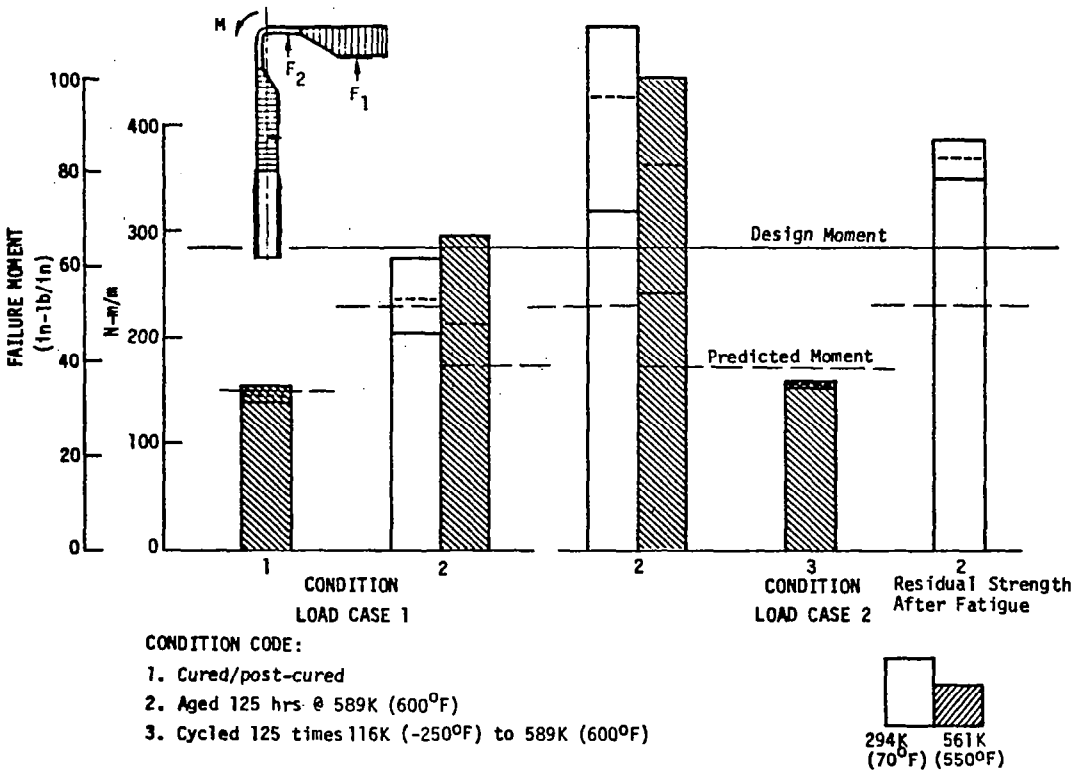


Figure 9-3: TYPE 2 BONDED JOINT STATIC TEST RESULTS

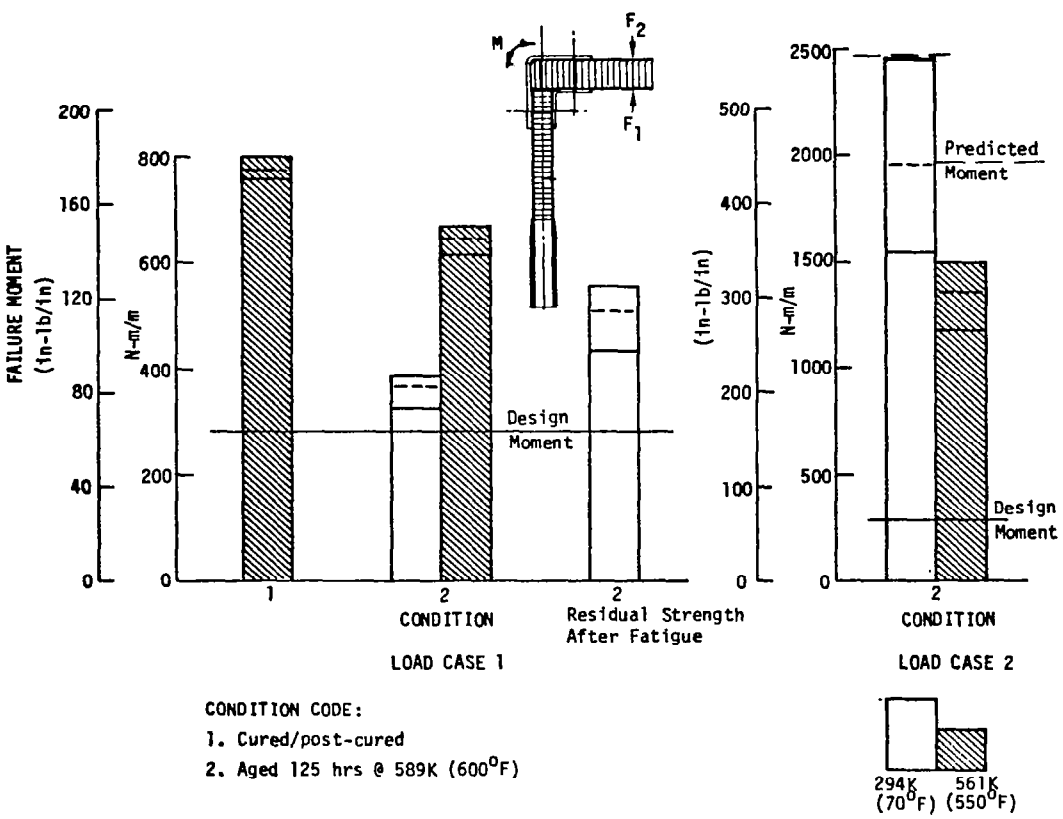


Figure 9-4: TYPE 2 BOLTED JOINT STATIC TEST RESULTS

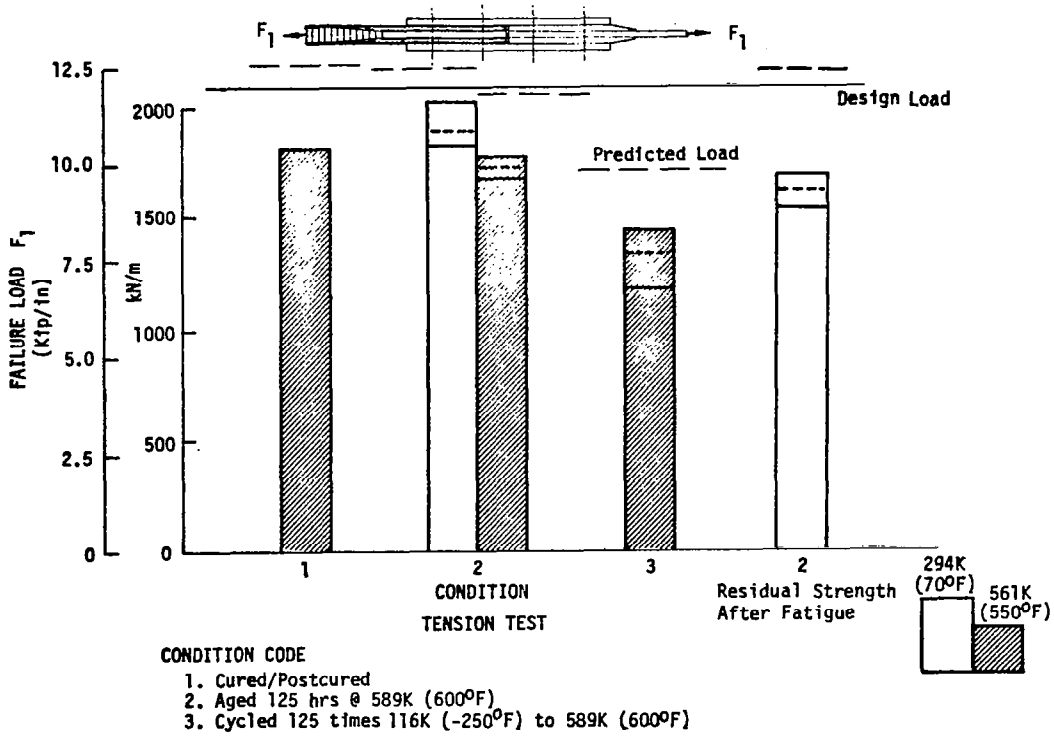


Figure 9-5: TYPE 3 BOLTED JOINT STATIC TEST RESULTS

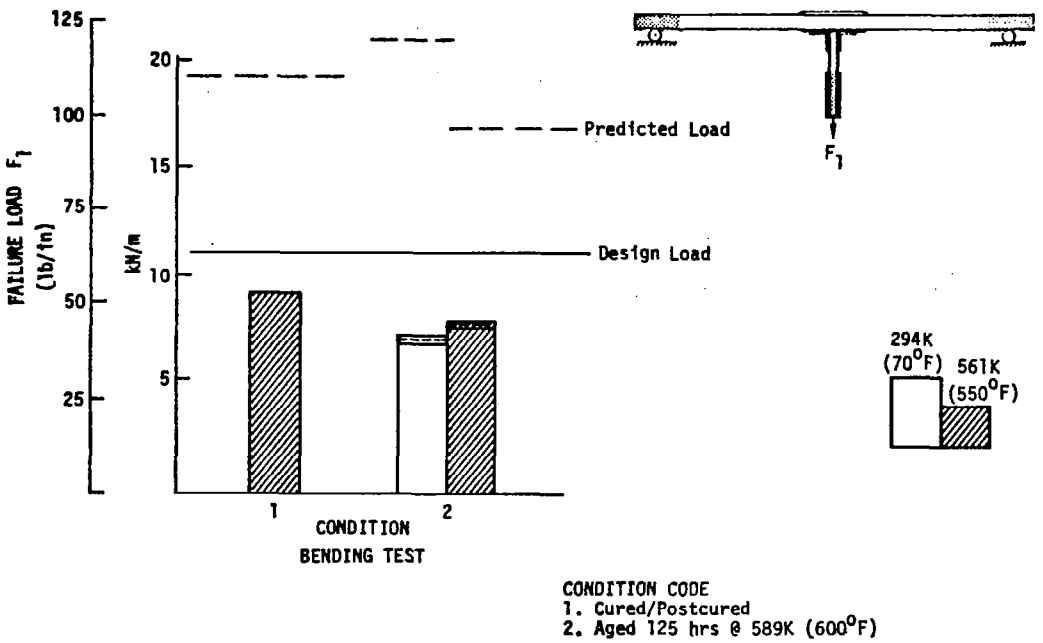


Figure 9-6: TYPE 4 BONDED JOINT STATIC TEST RESULTS

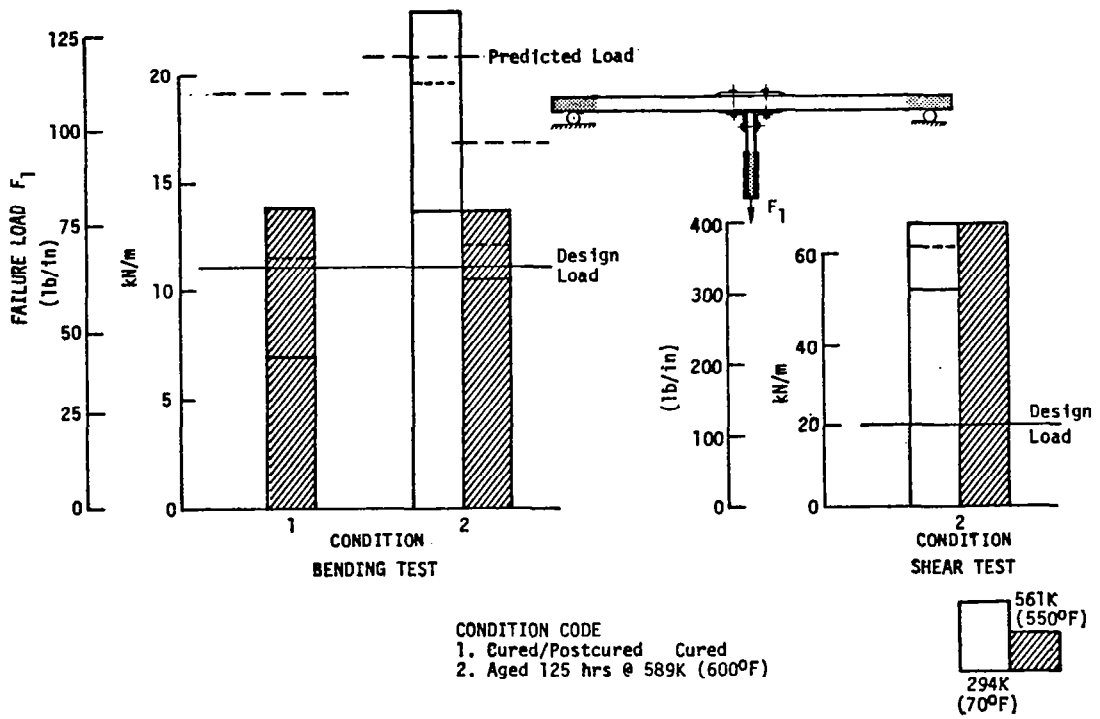
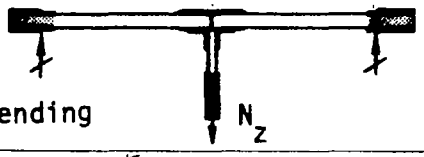
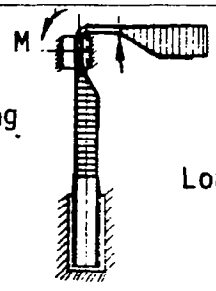
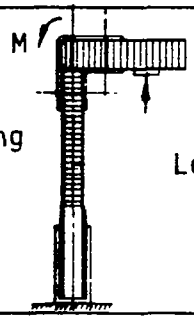
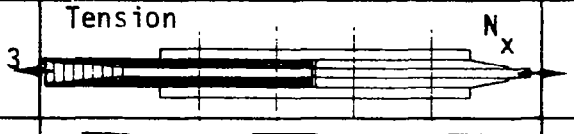
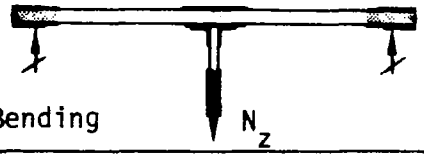
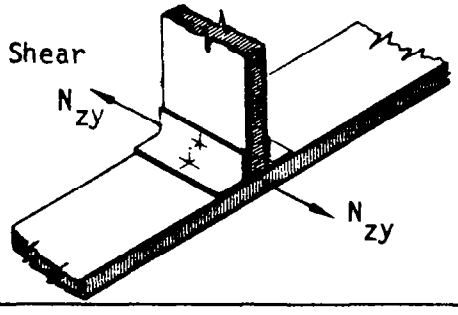


Figure 9-7: TYPE 4 BOLTED JOINT STATIC TEST RESULTS

Table 9-1 : SUMMARY OF MAXIMUM FAILURE LOADS

JOINT TYPE	LOAD CONDITION	MAXIMUM FAILURE LOAD kN/m (lb/in)		DESIGN LOAD kN/m (lb/in)
		BONDED	BOLTED	
TYPE 1		N_x 556 (3174)	N_x 562 (3208)	N_x 560 (3200)
		N_z 44.5(254)	N_z 44.1(252)	N_z 28.7 (164)
TYPE 2		M 494 (111)*		M 285* (64)*
			M 792 (178)*	M 285* (64)*
TYPE 3		—	2021 N_x (11,540)	N_x 2100 (12,000)
TYPE 4		N_z 9.2 (52.8)	N_z 23.1 (132)	N_z 11.2 (64)
		Not Tested	N_{zy} Greater Than 55.9 (319)	N_{zy} 20.0 (114)

* m-N/m (in-lb/in)

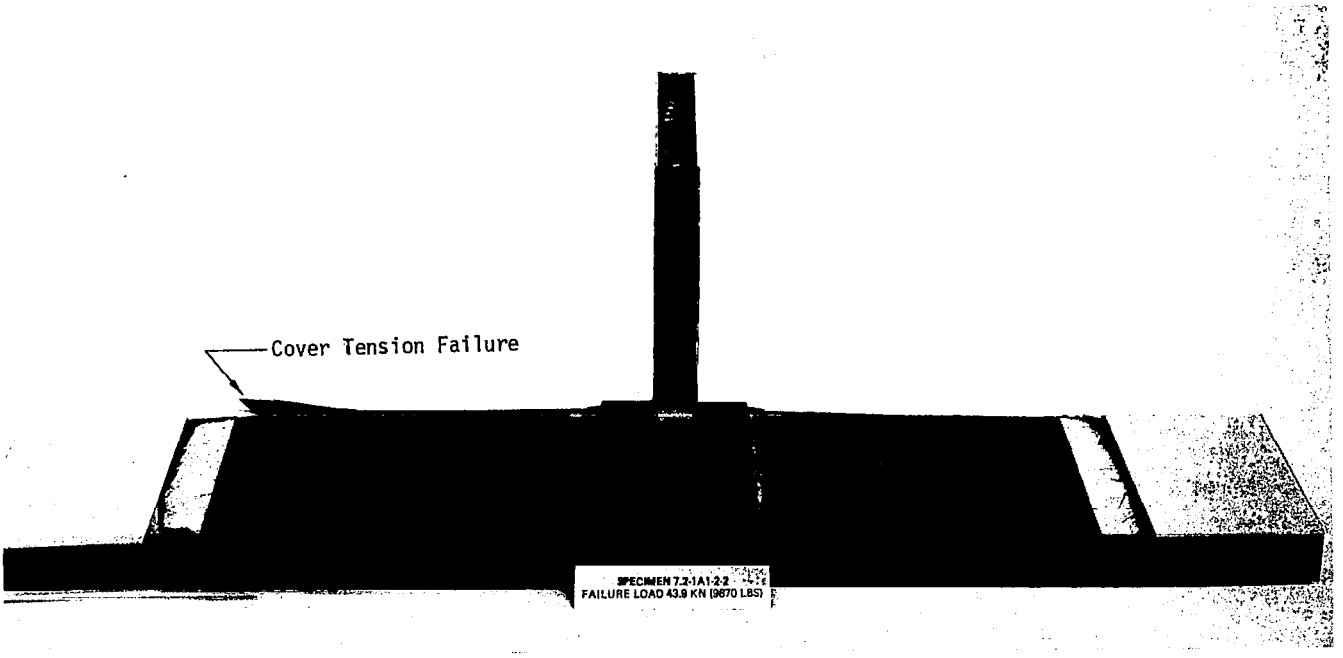


Figure 9-8: TYPE 1 BONDED-TENSION TEST, AGED, 294K (70°F)

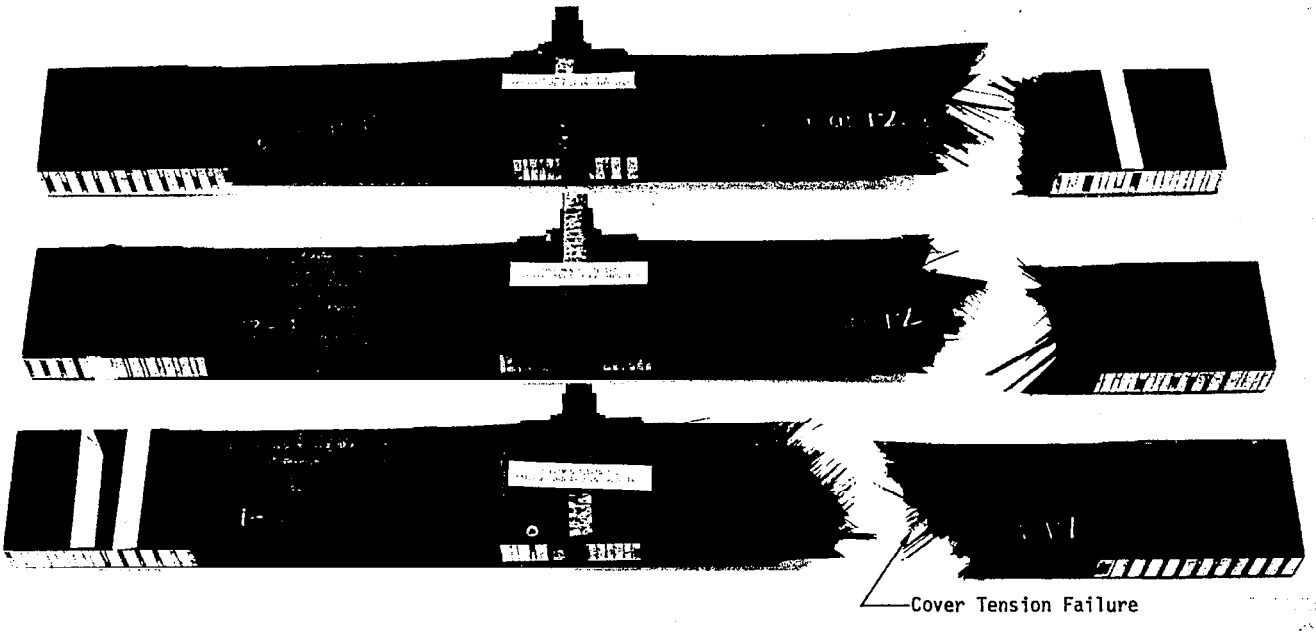


Figure 9-9: TYPE 1 BOLTED-TENSION TEST, CURED/POST-CURED, 561K (550°F)

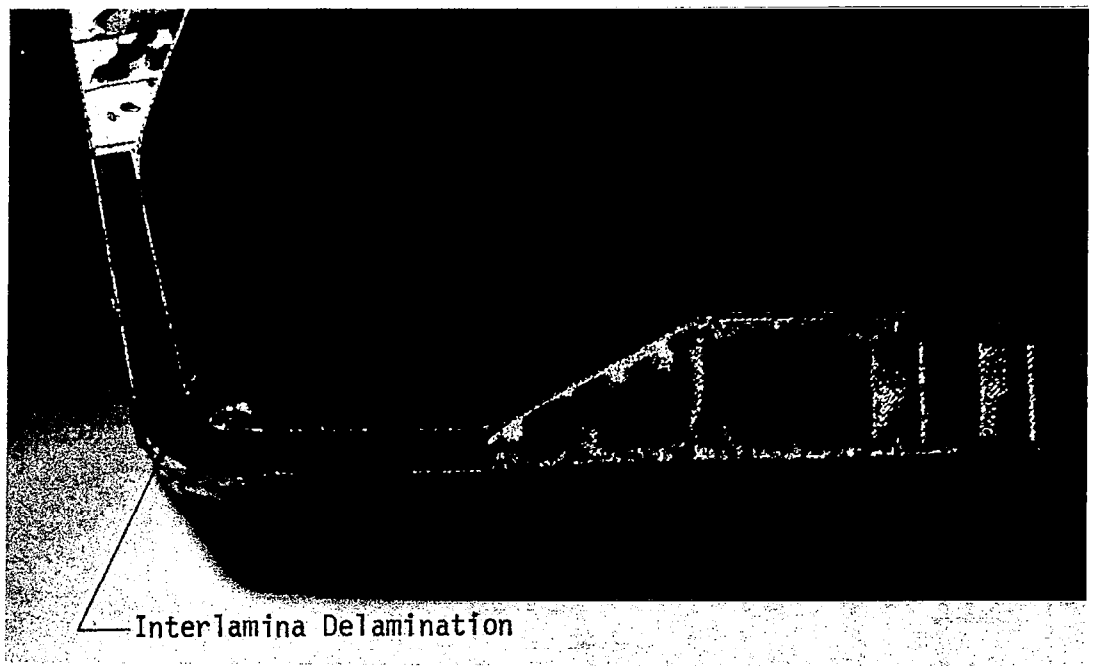


Figure 9-10: TYPE 2 BONDED-LOAD CASE 2, TYPICAL CORNER ANGLE DELAMINATION

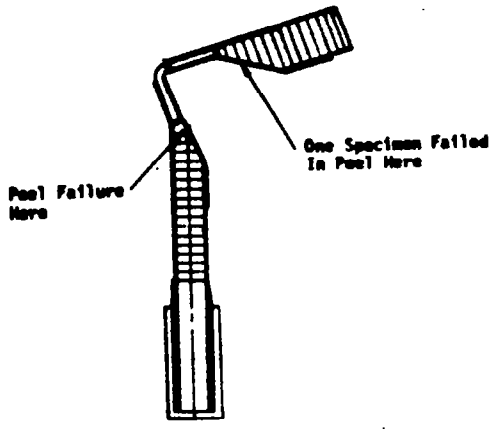


Figure 9-11: TYPE 2 BONDED JOINT
LOAD CASE 1 FAILURE MODE

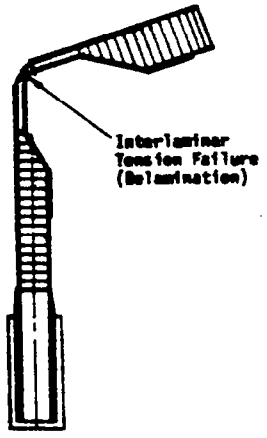


Figure 9-12: TYPE 2 BONDED JOINT
LOAD CASE 2 FAILURE MODE

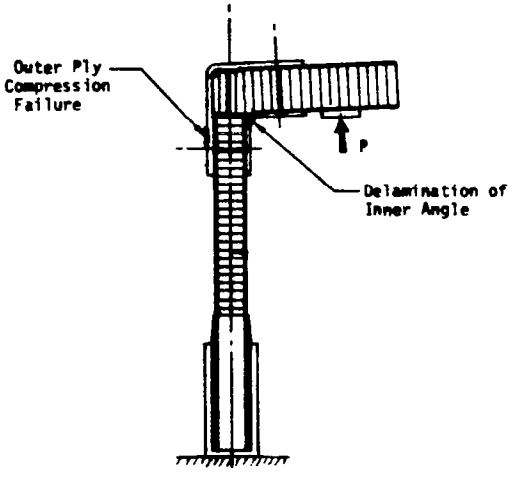


Figure 9-13: TYPE 2 BOLTED JOINT, FAILURE MODE - LOAD CASE 1

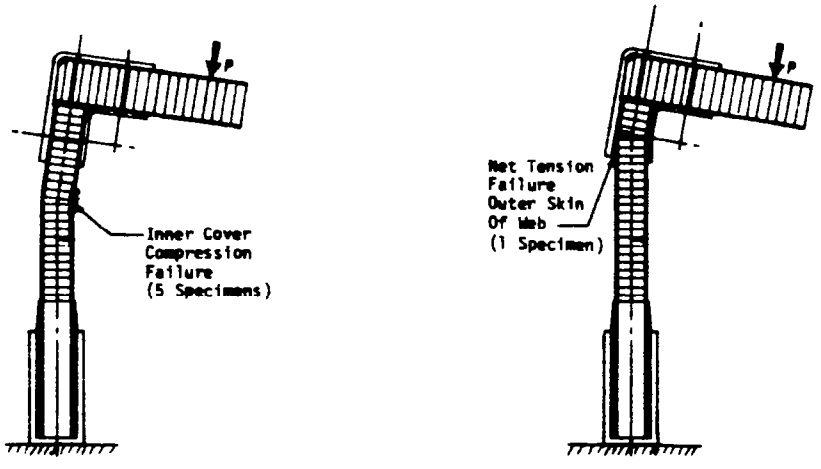
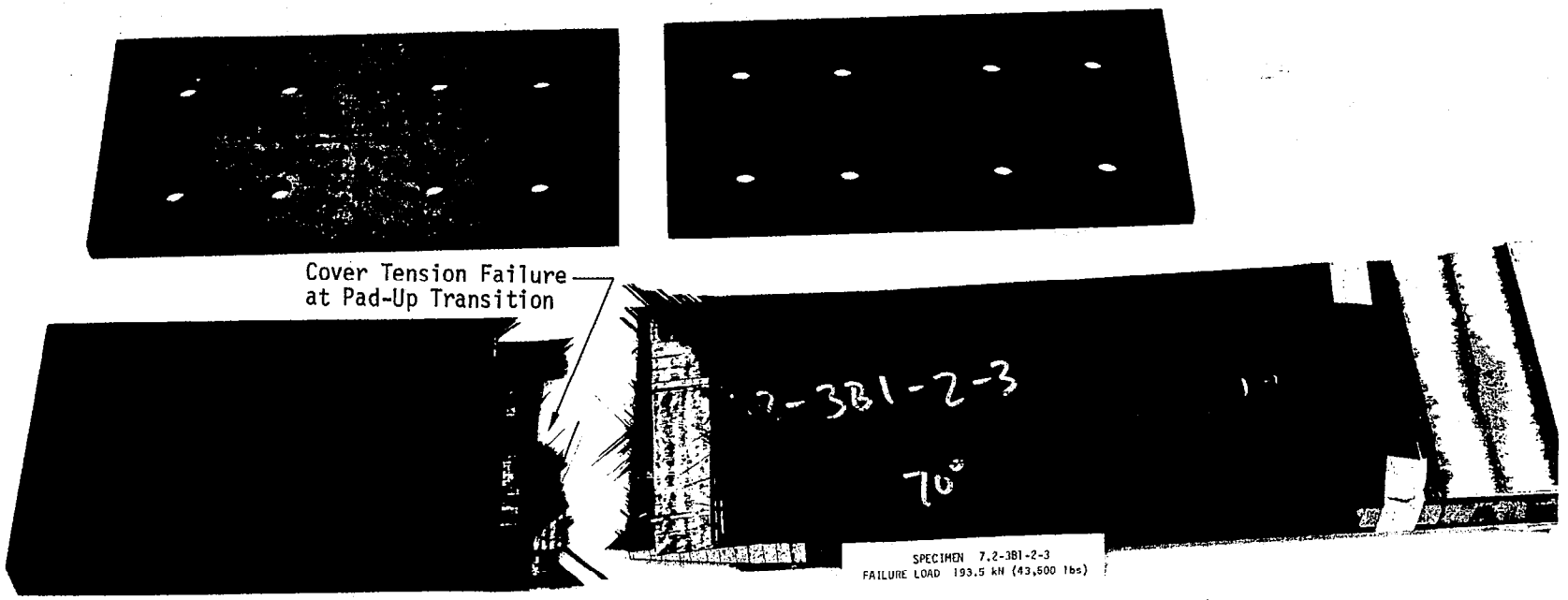


Figure 9-14: TYPE 2 BOLTED JOINT, FAILURE MODE - LOAD CASE 2



Matrix 7.2 Static Strength Test

Type 3 Bolted 294 K (70°F)

Figure 9-15: TYPE 3 BOLTED-TENSION TEST, AGED, 294K (70°F)

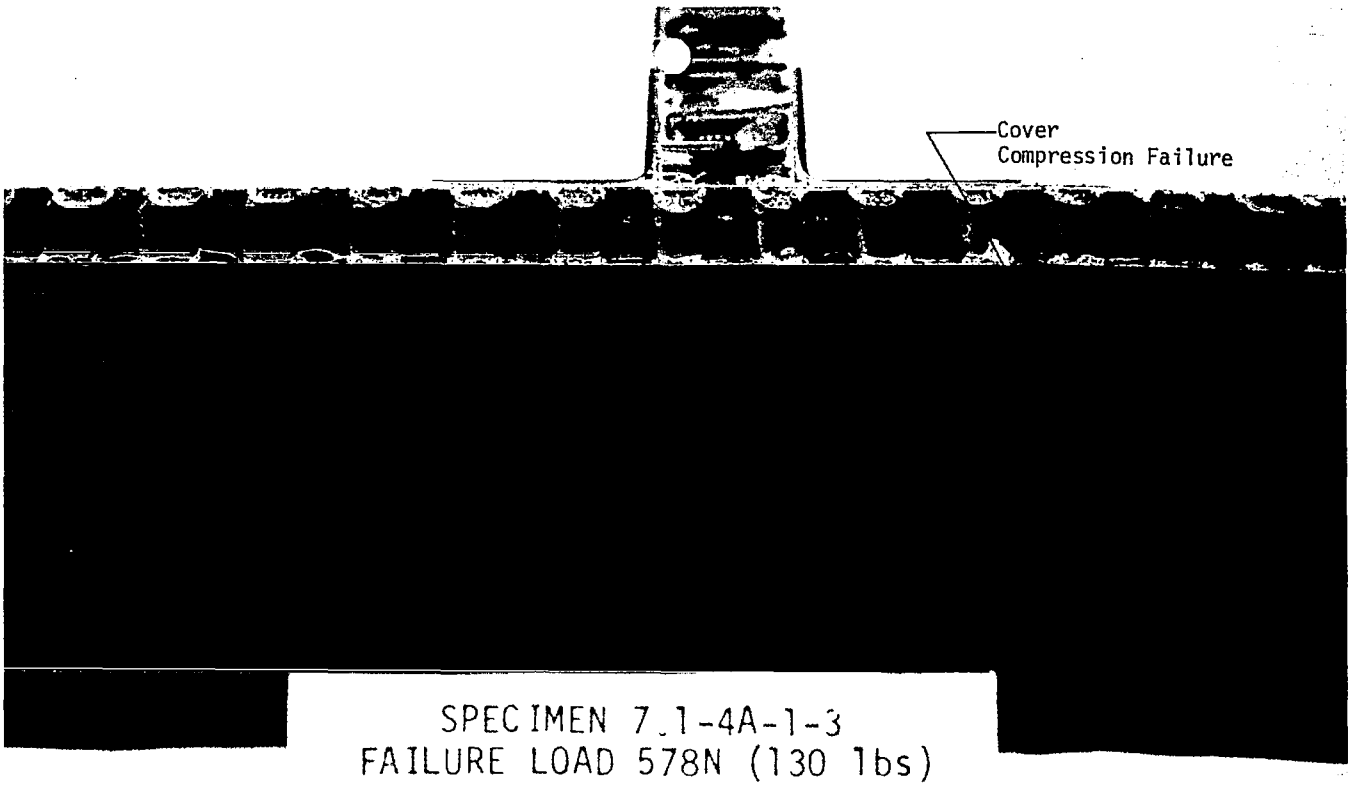


Figure 9-16: TYPE 4 BONDED-BENDING TEST, CURED/POST-CURED, 561K (550°F)

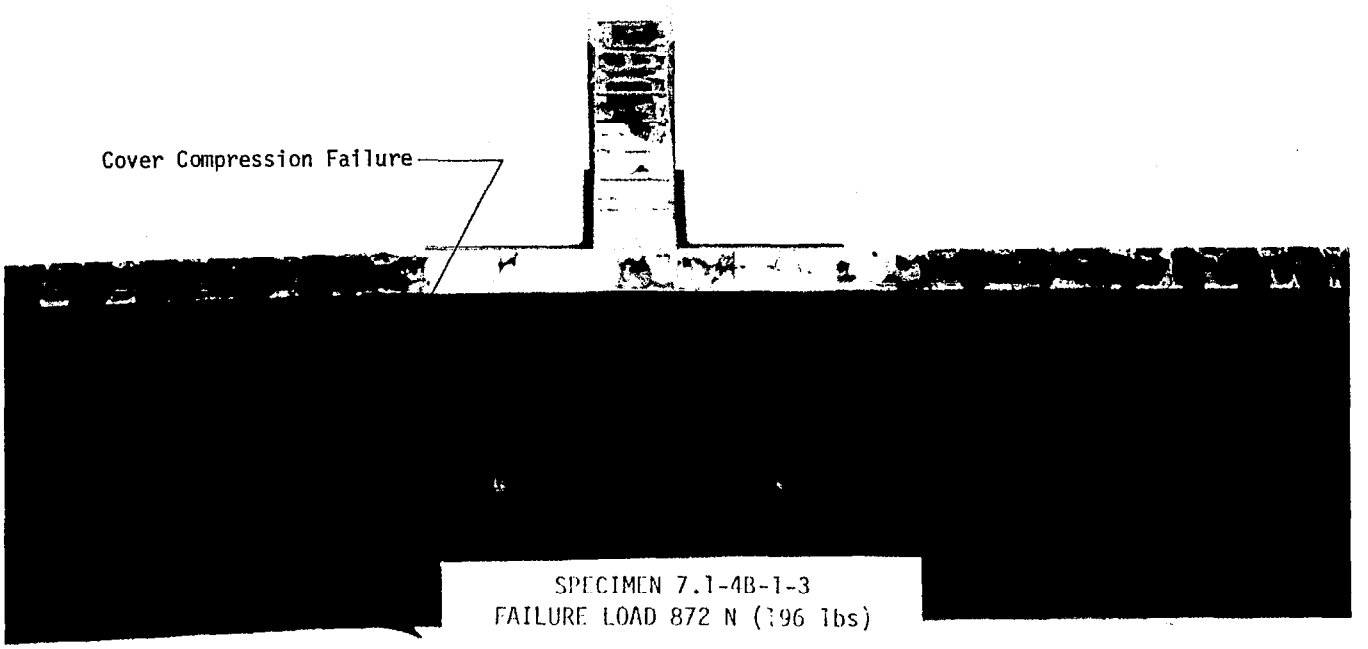


Figure 9-17: TYPE 4 BOLTED-BENDING TEST, CURED/POST-CURED, 561K (550°F)

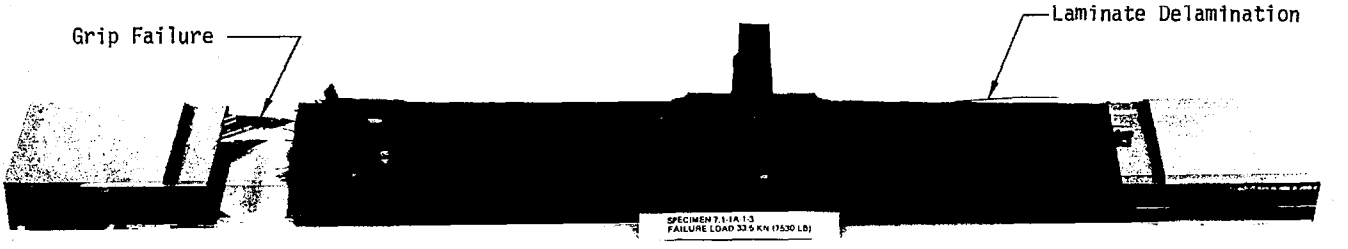


Figure 9-18: TYPE 1 BONDED-TENSION TEST, CURED/POST-CURED, 561K (550°F)

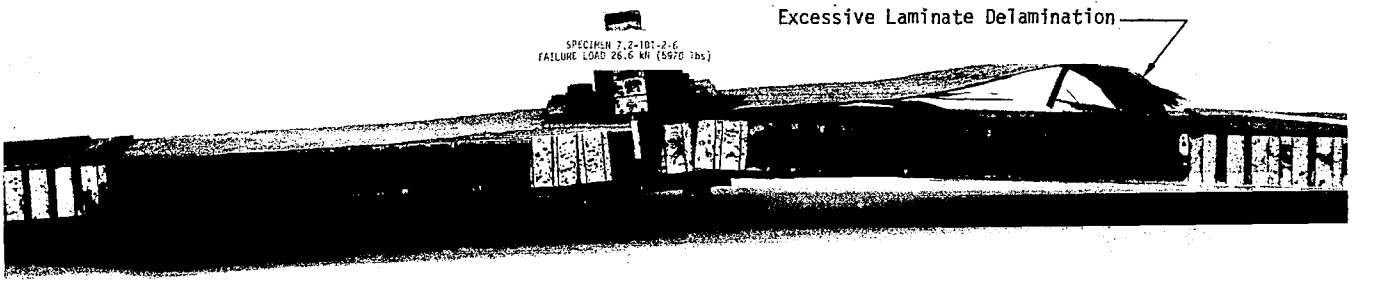


Figure 9-19: TYPE 1 BOLTED-TENSION TEST, AGED, 561K (550°F)

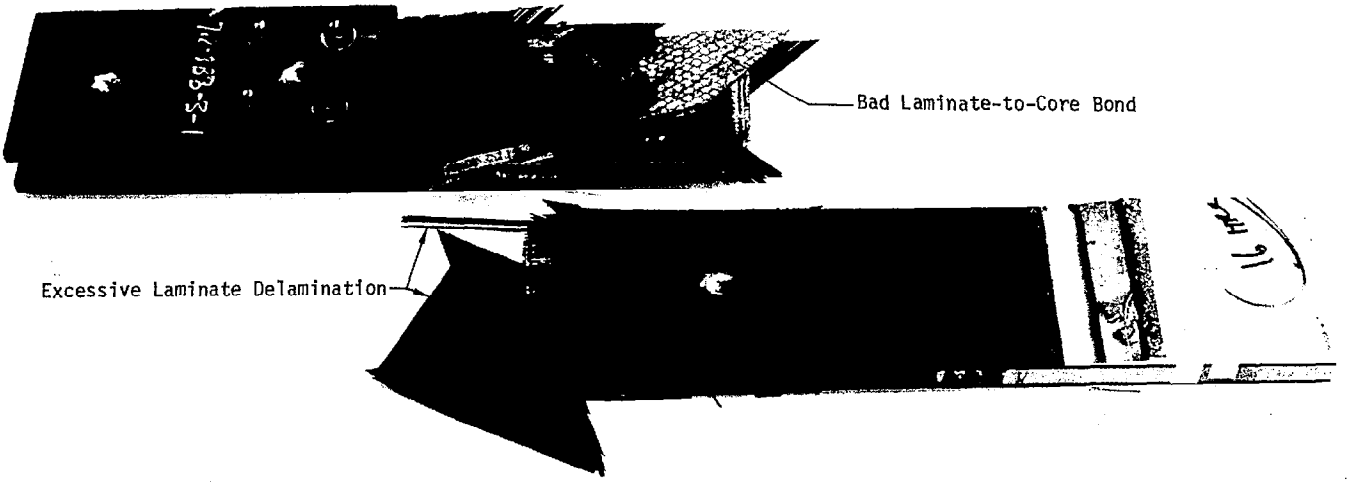


Figure 9-20: TYPE 3 BOLTED-TENSION TEST, THERMALLY CYCLED, 561K (550°F)

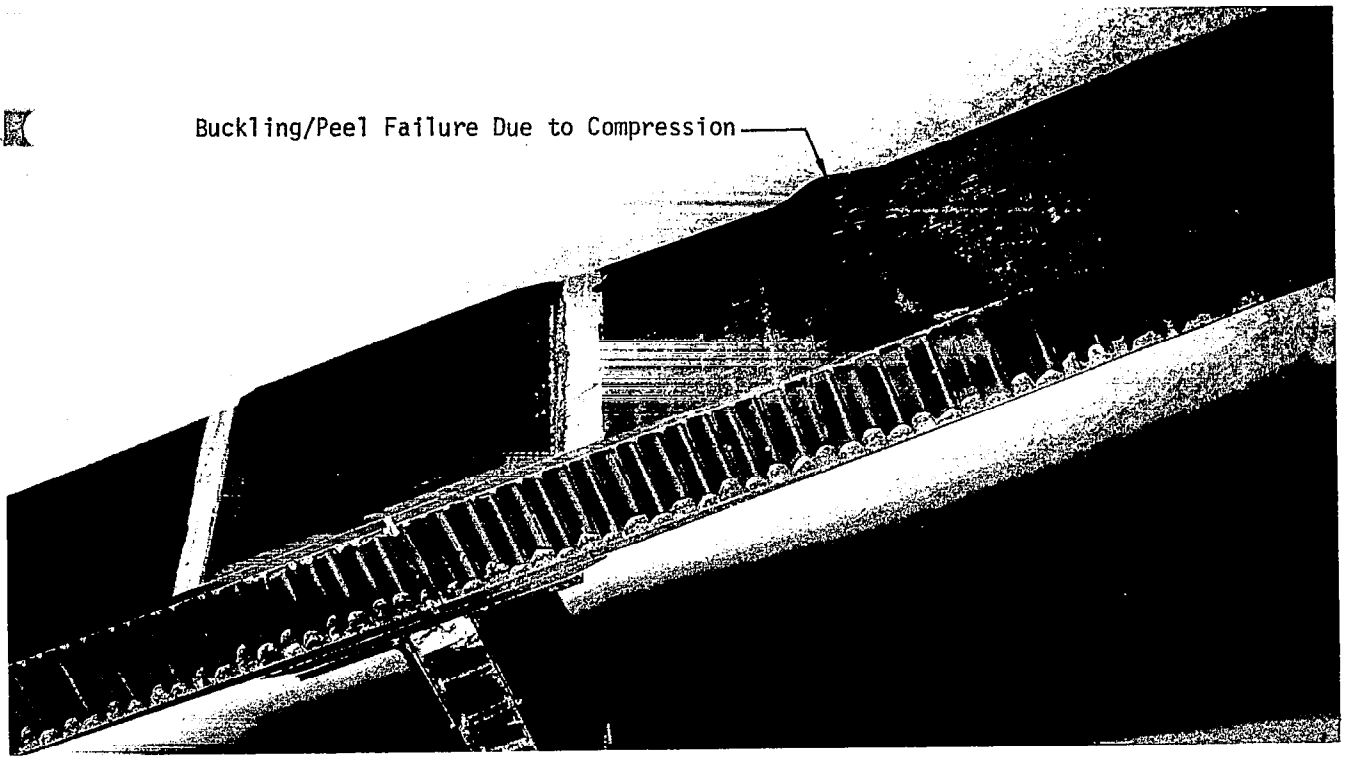


Figure 9-21: TYPE 1 BONDED-BENDING TEST, AGED, 294K (70°F)

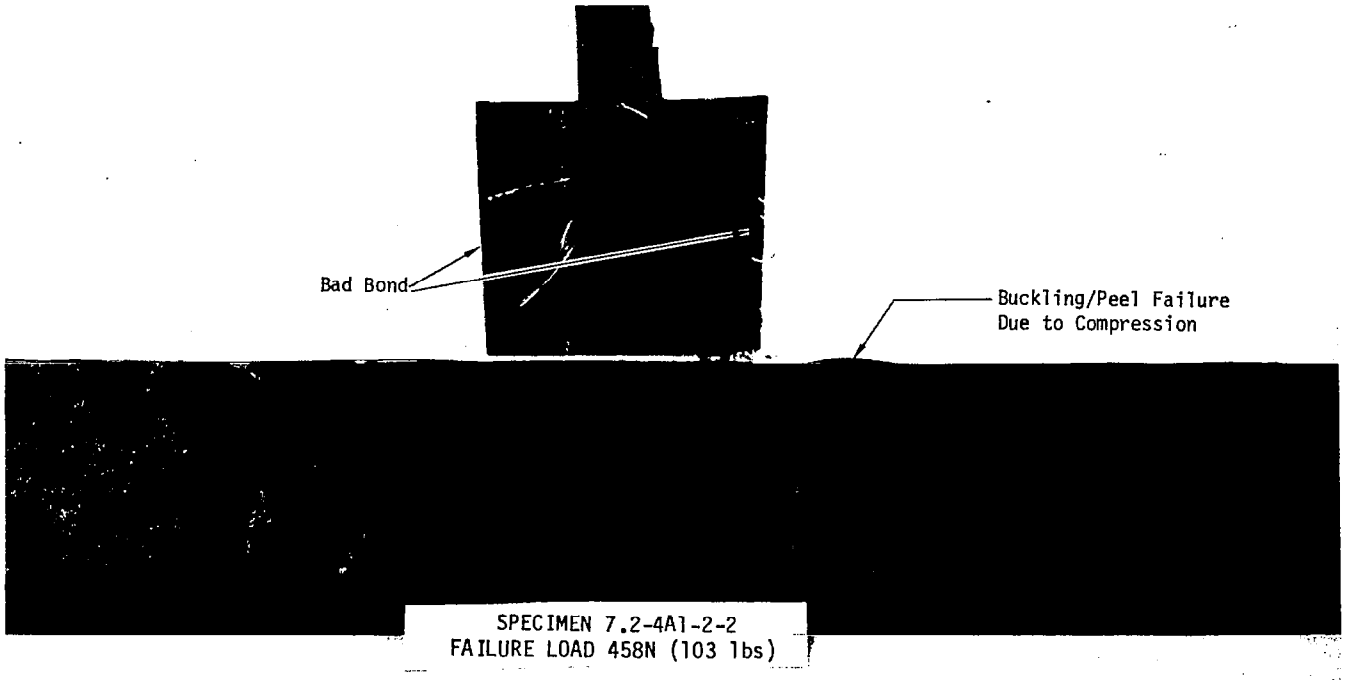


Figure 9-22: TYPE 4 BONDED-BENDING TEST, AGED, 294K (70°F)

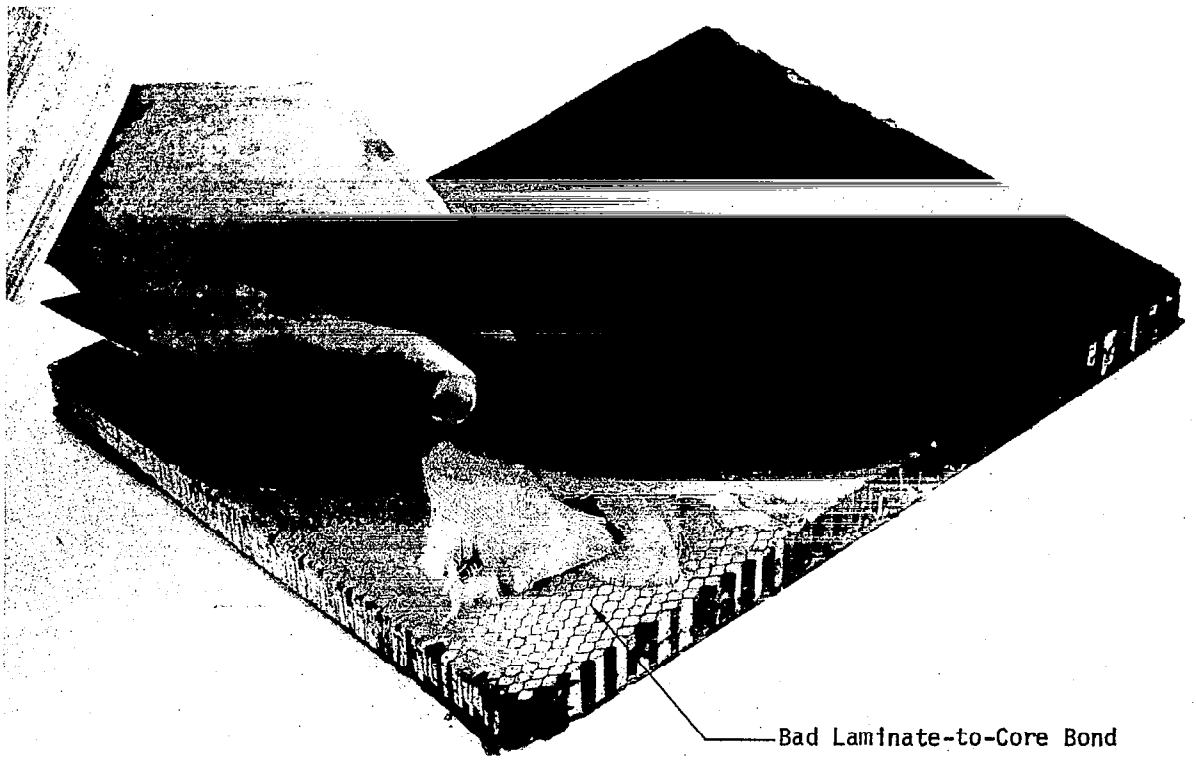


Figure 9-23: TYPE 1 BOLTED, THERMALLY CYCLED PANEL

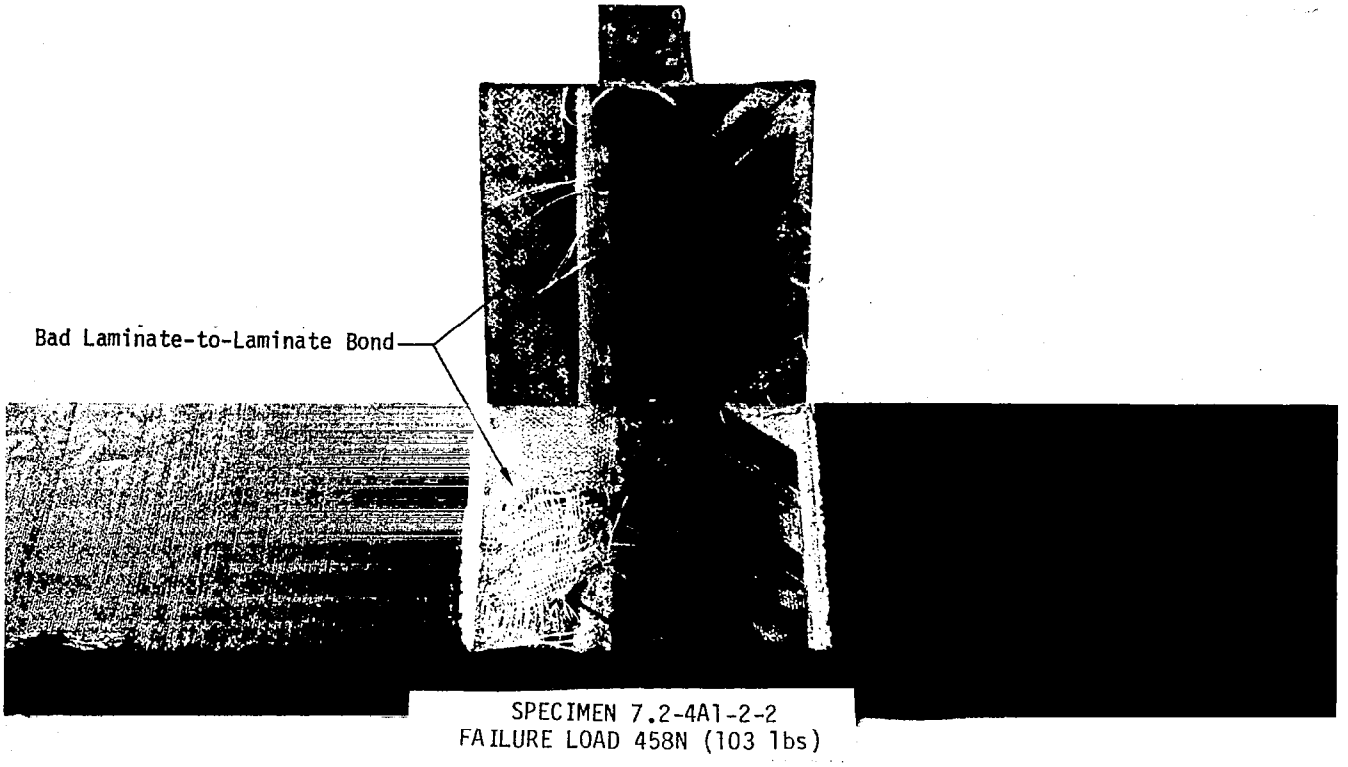
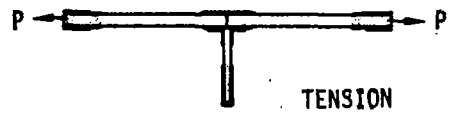
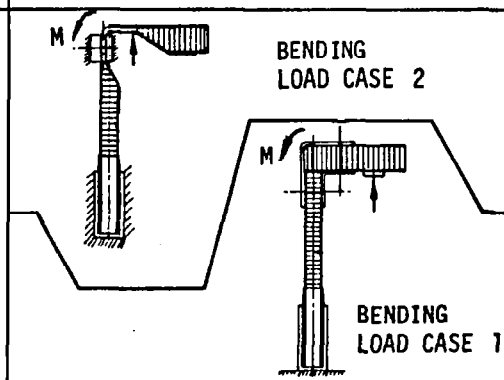
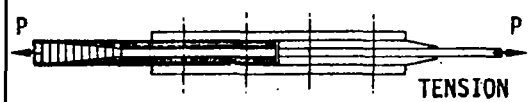
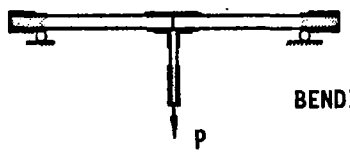


Figure 9-24: TYPE 4 BONDED-BENDING TEST, AGED, 294K (70°F)

Table 9-2: SUMMARY OF FATIGUE TEST RESULTS (AGED SPECIMENS)

JOINT TYPE	LOAD CONDITION	CONFIGURATION	TEMPERATURE K (°F)	NUMBER OF CYCLES*	FAILURE MODE	RESIDUAL STRENGTH % CHANGE
TYPE 1		BONDED	294 (70)	953,000(1) 573,000(1) 303,000(1)	Grips (See Fig.9-25)	NA
			561 (550)	553,000(1) 30,000(1) 100(1)	Grips (See Fig. 9-25)	NA
		BOLTED	294 (70)	10^6 (3)	No 2 Part Failures Plies Delaminated	+7% (See Fig. 9-26)
			561 (550)	10^6 (3)	No 2 Part Failures	-1%
TYPE 2		BONDED	294 (70)	10^6 (3)	No Failure	-14% (See Fig. 9-10)
			561 (550)	10^6 (2) 3,000 (1)	Corner Angle Delaminated (See Fig.9-10)	NA
		BOLTED	294 (70)	10^6 (3)	No Failure	+39% (See Fig. 9-10)
			561 (550)	10^6 (1) 100 (1)	Corner Angle Delaminated (See Fig.9-10)	NA
TYPE 3		BOLTED	294 (70)	10^6 (2) 914,000(1)	No Failure Cover Tension at Pad-up	-15 % (See Fig. 9-27)
			561 (550)	383,000 (1) 117,000 (1) 307,000 (1)	Cover Tension at Pad-up	N.A.
TYPE 4		BONDED	294 (70)	Not Tested	———	—
			561 (550)	Not Tested	———	—
		BOLTED	294 (70)	10^6 (1)	[1]	-10% [2]
			561 (550)	53,000 (1) 10^6 (1) 49,600 (1)	Outer Cover Compression [1] Outer Cover Compression	NA +19% [3] NA

* Number of Spec. shown in ( ).

[1] No 2 Part Failure. Attach Angles Delaminated (See Fig. 9-28)

[2] Failed Attach Angles (See Fig. 9-29)

[3] Failed Attach Angles (See Fig. 9-30)

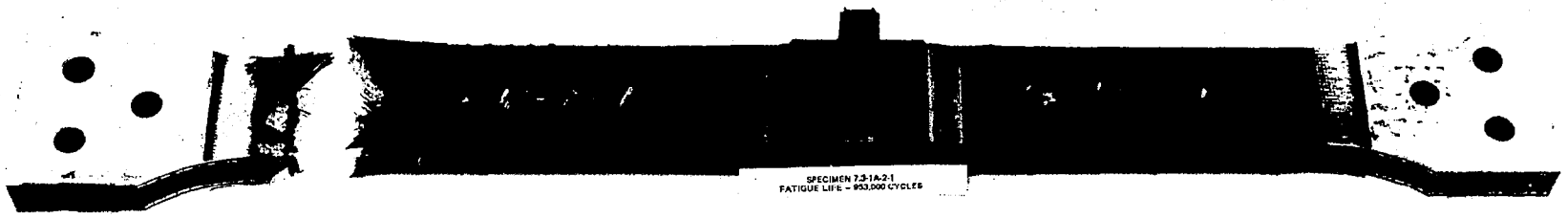
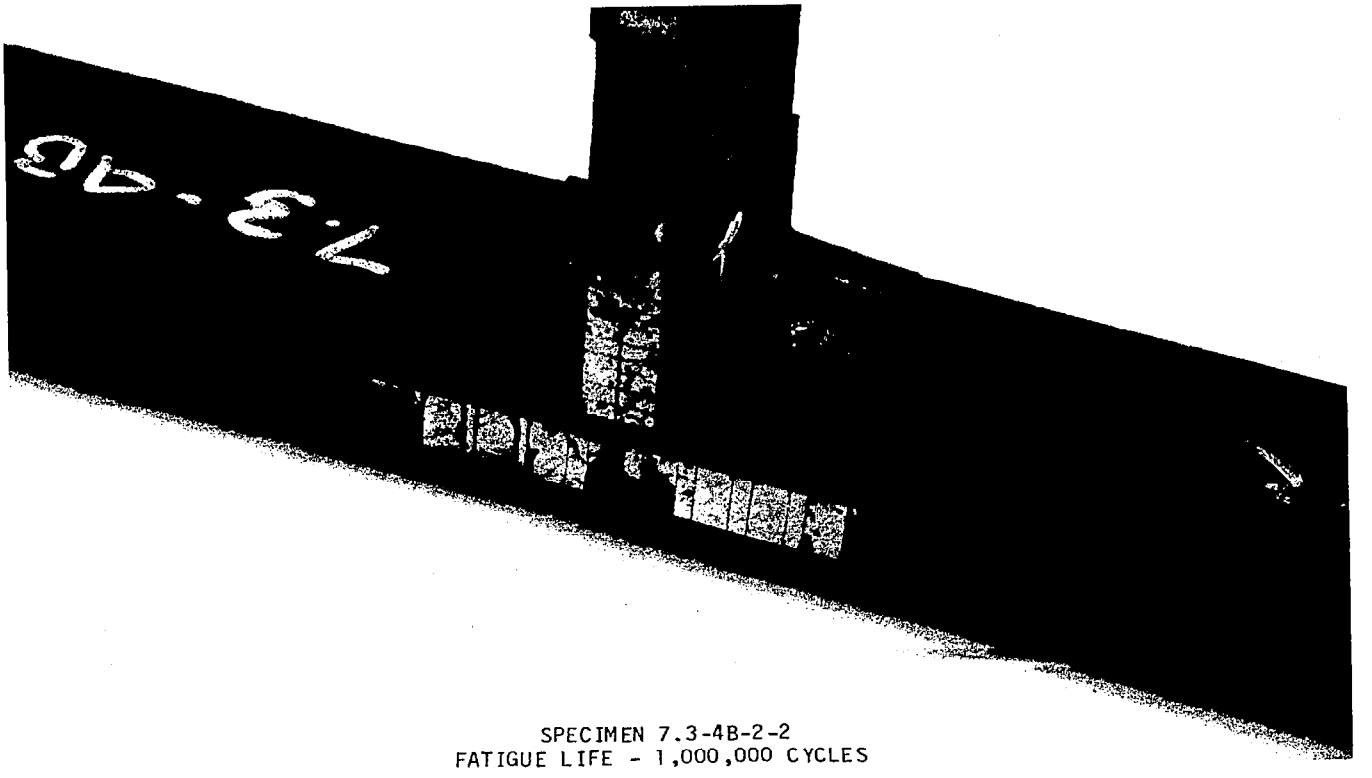


Figure 9-25: TYPE 1 BONDED-FATIGUE TEST, AGED, 294K (70°F)

Figure 9-26: TYPE 1 BOLTED-FATIGUE TEST AND RESIDUAL STRENGTH TEST, AGED, 294K (70°F)

SPECIMEN 7.3-3B-2-1
1,000,000 FATIGUE CYCLES
STATIC FAILURE LOAD 74.7 kN (16 800 lbs)

Figure 9-27: TYPE 3 BOLTED FATIGUE TEST AND RESIDUAL STRENGTH TEST, AGED, 294K (70°F)



SPECIMEN 7.3-4B-2-2
FATIGUE LIFE - 1,000,000 CYCLES

Figure 9-28: TYPE 4 BOLTED-FATIGUE TEST, AGED, 294K (70°F)

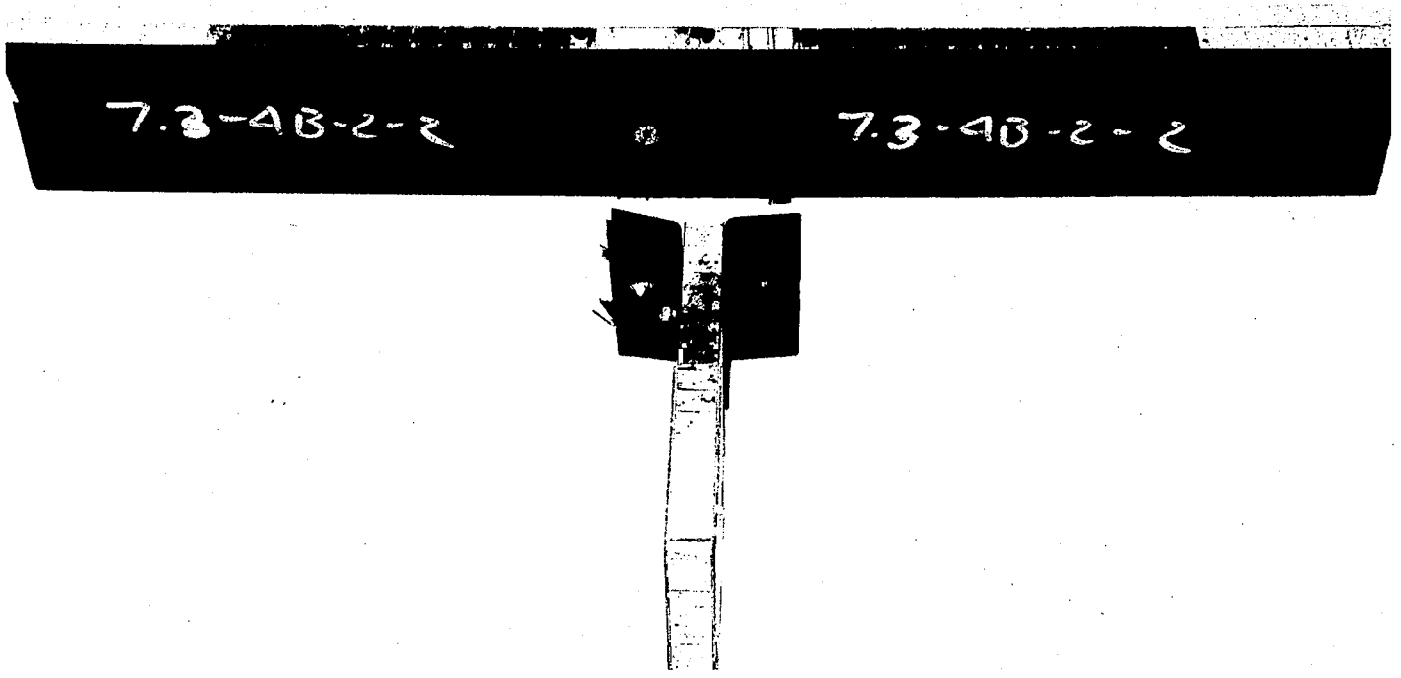


Figure 9-29: TYPE 4 BOLTED-FATIGUE TEST & RESIDUAL STRENGTH TEST, AGED, 294K (70°F)

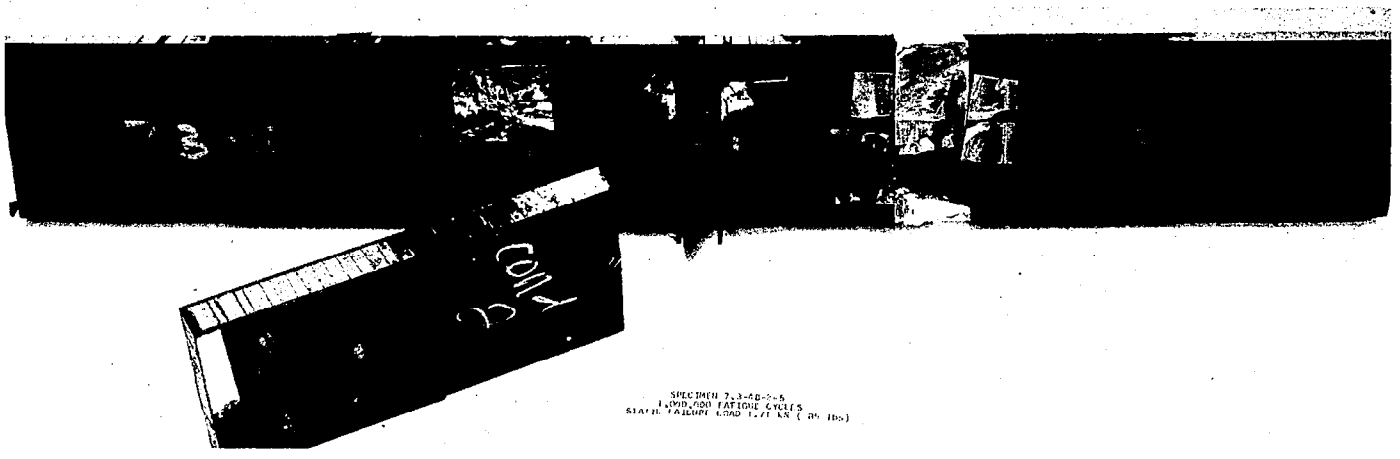


Figure 9-30: TYPE 4 BOLTED-FATIGUE TEST & RESIDUAL STRENGTH TEST, AGED, 561K (550°F)

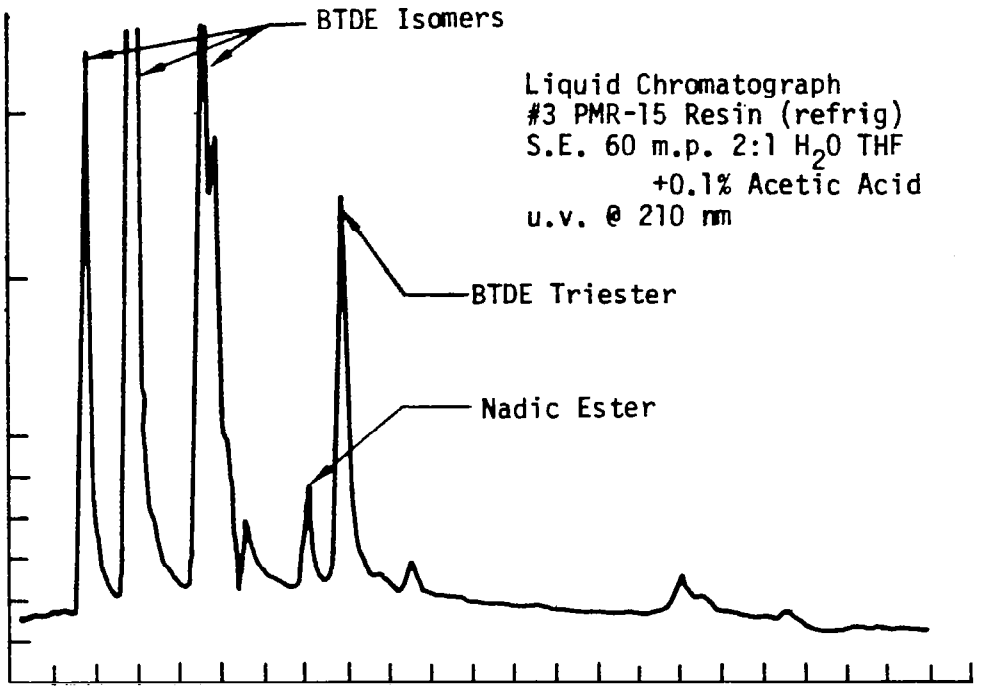


Figure 9-31: LIQUID CHROMATOGRAPHY OF PMR-15 FRESH RESIN

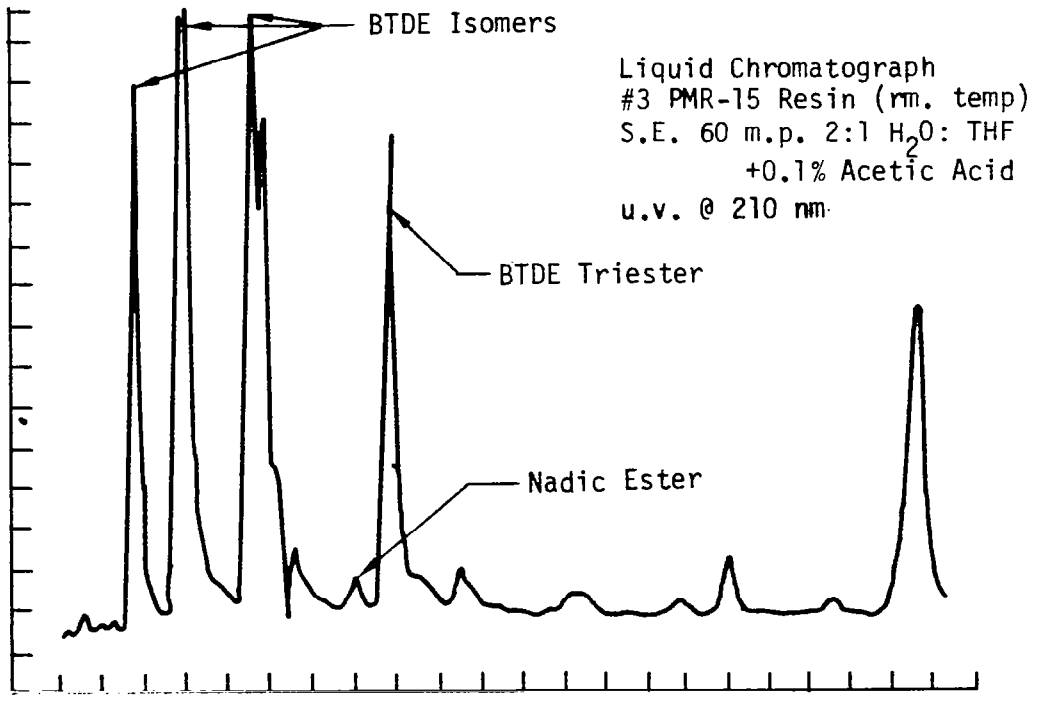


Figure 9-32: LIQUID CHROMATOGRAPHY OF PMR-15 AGED 45 DAYS

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10.0 TEST/ANALYSIS CORRELATION

The Type 1, 3 and 4 joints were designed to fail outside the joint area; therefore, prediction techniques for the actual failure modes experienced were for tension or compression failures of the cover laminates. The Type 2 joints failed, as expected, in the joint region. Strength predictions for these joints by hand analyses was much more difficult because of the complex load paths and transverse stresses due to bending of angles. Table 10-1 shows the prediction methods used. Material strengths used in the analyses are given in Table 10-2. The joint strength predictions are shown on Figures 9-1 through 9-7, which also shows the static strength results for the "Final Evaluation" testing. In some cases, the predicted strengths fall below the design loads. This is because the design loads were based on preliminary material properties, while the predicted strengths were based on material properties from the design allowables testing (Ref. 4). The design loads were based on a quasi-isotropic laminate tension and compression strength of 552 MPa (80 ksi) for all conditions and temperatures. In most cases, the predicted loads were greater than the actual failure loads. This can be attributed to grip problems (Type 1 joints), the resin chemistry and adhesive processing problems discussed in Section 9.2, and to the fact that the material strengths used for the predictions were averages from the design allowables testing and not statistically based allowable strengths.

Table 10-1: SUMMARY OF JOINT STRENGTH PREDICTION TECHNIQUES

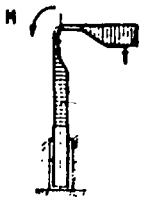
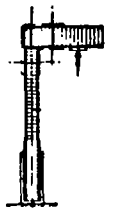
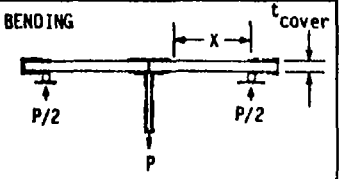
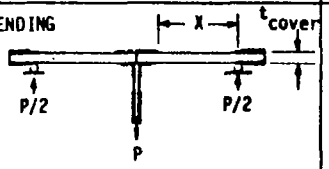
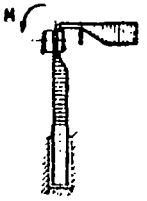
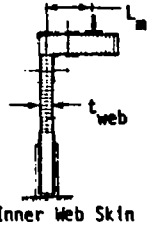
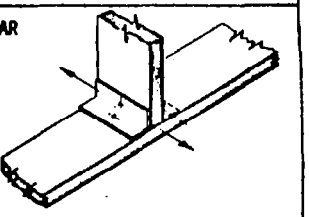
LOAD CASE	TYPE 1 JOINTS	TYPE 2 JOINTS		TYPE 3 JOINTS	TYPE 4 JOINTS
		BONDED	BOLTED		
1	TENSION $P_{ult} = 2F^{tu}t_{skin}$	BENDING  M_{ult} based on radial (interlaminar) stresses in corner angle calculated from curved beam theory.	BENDING  M_{ult} based on radial (interlaminar) stresses in corner angle calculated from curved beam theory.	TENSION $P_{ult} = 2F^{tu}t_{skin}$	BENDING  Cover Compression $P_{ult} = \frac{2t_{cover}t_{skin}F^{cu}}{x}$
2	BENDING  Cover Compression $P_{ult} = \frac{2t_{cover}t_{skin}F^{cu}}{x}$ Angle Pull-Off P_{ult} based on "Static Discriminator" Type 4 loads.	BENDING (Angle Only)  M_{ult} based on radial (interlaminar) stresses in corner angle calculated from curved beam theory.	BENDING  Inner Web Skin Compression $P_{ult} = \frac{F^{cu}t_{skin}}{[\frac{1}{2} + \frac{L_m}{t_{web}}]}$	—	SHEAR  Joints not critical for shear. Tests terminated based on observed deflections.

Table 10-2 : ULTIMATE STRENGTHS USED FOR JOINT STRENGTH PREDICTIONS

Celion 3000/PMR-15, Normalized to 58% Fiber Volume $(0/\pm 45/90)_{NS}$ Laminate

	TEMPERATURE	ULTIMATE STRESS, MPa (ksi)		
		CURED/ POST-CURED	THERMALLY AGED	THERMALLY CYCLED
TENSION (F^{TU})	294K (70°F)	572 (83.5)	539 (78.2)	453 (65.7)
	561K (550°F)	544 (78.3)	510 (74.0)	424 (61.5)
COMPRESSION (F^{CU})	294K (70°F)	601 (87.2)	578 (83.8)	599 (86.9)
	561K (550°F)	530 (76.9)	466 (67.6)	506 (73.3)
FLATWISE TENSION LAMINATE TO LAMINATE	294K (70°F)	26.43 (3.833)	13.32 (1.932)	—
	561K (550°F)	8.84 (1.282)	9.97 (1.447)	—

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11.0 BONDED VERSUS BOLTED JOINT COMPARISONS

This program has demonstrated that both bonded and bolted Gr/PI composite joints can be designed and fabricated to carry loads up to 500 kN/m (3200 lb/in). Futhermore, bolted Gr/PI to titanium joints can be designed and fabricated to carry loads up to 2100 kN/m (12000 lb/in). Bonded joints currently cannot be fabricated to carry this load level, due to bonding difficulties. However, Gr/PI to titanium "3-step" symmetric step lap joints were fabricated under Task 2.0 and achieved a load carrying capability of 875 kN/m (5000 lb/in) at 561K (550°F).

As expected, bolted joints have weights 2 to 7 times that of the corresponding bonded joint. Computed weights per unit width for each joint type are shown in Table 11-1.

Although the bolted designs are heavier than the bonded versions, they give improvements in reliability and repairability. Bolted joints can be disassembled far easier than a bonded joint, thus allowing for efficient repair of damaged parts. The occurrence of bad bonds in bonded joint fabrication is difficult to detect and can lead to reliability problems, whereas bolted joints maintain structural integrity.

Bonded attachment angles, used on the Type 1 and Type 4 bonded joints are susceptible to peel stresses when the joints experience large deflections under bending, resulting in premature failure. Bolted attachment angles can withstand these large deflections without two part failures.

The Type 2 bonded design is more flexible than the bolted version because of a thinner corner cross-section. This could lead to undesirably large deflections. Deflection limited design criteria would probably require revision of this design.

Bonded joints demonstrated fabrication advantages over bolted joints in this program. Bonding proved to be faster and less costly than the procedures required for a bolted joint. In addition bonded joints have a lower part count than the corresponding bolted versions.

Due to the variability of the test results, no firm conclusions can be drawn about the relative fatigue resistance of bonded and bolted joints.

Table 11-1: COMPARISON OF JOINT WEIGHTS FOR THE VARIOUS JOINT TYPES

JOINT TYPE	JOINT WEIGHT		PRIMARY DESIGN LOAD
	BONDED kg/m (lb/in)	BOLTED kg/m (lb/in)	
Type 1	.98 (.055)	3.77 (.211)	560 kN/m (3200 lb/in) Tension
Type 2	.77 (.043)	1.78 (.100)	285 N-m/m (64 in-lb/in) Moment
Type 3	-- --	14.3 (.811)	2100 kN/m (12,000 lb/in) Tension
Type 4	.142 (.008)	.96 (.054)	11.2 kN/m (64 lb/in) Bending

12.0 CONCLUSIONS AND RECOMMENDATIONS

The following conclusions and recommendations have resulted from this program.

Conclusions

- Bonded and bolted graphite/polyimide composite joints can be designed and fabricated to transfer the loads commensurate with the loads experienced on lightly load control surfaces for advanced space transportation systems and high-speed aircraft. This load carrying capability is maintained at temperatures up to 561K (550°F). It is also maintained after 125 hours of thermal aging and thermal cycling, except for the Type 2 bonded joints which, as designed, are susceptible to failures resulting from microcracking experienced during thermal cycling. The joints can withstand a fatigue environment of 10^6 cycles without a catastrophic loss in strength, although the fatigue results are limited due to the material problems experienced.

- Fabrication of joints in scaled-up sizes that would be required for production type programs can be accomplished with state-of-the-art tooling. No degradation in joint load carrying capability results from fabricating large scale panels.

- Bonded joints are significantly lighter in weight than bolted joints designed for the same load transfer requirement. Bonded joints are cheaper to fabricate and have lower part counts than the corresponding bolted joints. However, bolted joints offer advantages in reliability and repairability.

- While initial attempts at cocuring bonded Gr/PI-titanium joints to carry loads up to 2100 kN/m (12,000 lb/in) were unsuccessful, it appears that a hybrid cure and sequenced pressure application would result in successful fabrication. It was demonstrated under Task 2.0 of this program that loads up 875 kN/m (5000 lb/in) could be carried by a bonded Gr/PI-titanium step-lap joint.

- The time and temperature at which pressure is applied during laminate cure is critical to laminate processing and varies with part thickness.

- The cured PMR-15 resin is susceptible to degradation after exposure to 589K (600°F) for periods of time well under 125 hr. when the amount of nadic ester in the PMR-15 resin is low prior to laminate cure.

Recommendations

- Strict quality control procedures should be imposed to insure that the chemical composition of the PMR-15 resin is correctly maintained. Shelf lives should be determined from the initial chemical composition of the resin, with quality control tests to be repeated upon expiration of these lives before the material is used for fabrication.

- Conduct studies of cocured bonded composite to titanium joints to increase load carrying capabilities.

- Develop better NDI techniques for the acceptance of production hardware. Detection of bad bonds, delaminations and poorly cured laminates should be stressed.

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15. Supplementary Notes

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16. Abstract

This report summarizes the design, analysis and testing performed to develop four types of graphite/polyimide (Gr/PI) bonded and bolted composite joints for lightly loaded control surfaces on advanced space transportation systems that operate at temperatures up to 561K (550°F). Material properties and "Small Specimen" tests were conducted to establish design data and to evaluate specific design details. "Static Discriminator" tests were conducted on preliminary designs to verify structural adequacy. Scaled-up specimens of the final joint designs, representative of production size requirements, were subjected to a series of static and fatigue tests to evaluate joint strength. Effects of environmental conditioning were determined by testing aged (125 hours @ 589K (600°F)) and thermal cycled (116K to 589K (-250°F to 600°F), 125 times) specimens. It is concluded Gr/PI joints can be designed and fabricated to carry the specified loads. Test results also indicate a possible resin loss or degradation of laminates after exposure to 589K (600°F) for 125 hours.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
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