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POLYIMIDE WELD BONDING FOR TITANIUM ALLOY JOINTS

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Prepared under
Contract No. NAS 1-11689

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for

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FOREWORD

This document constitutes the final report for the work accomplished between 9 June 1972 through 9 February 1974 by TRW Systems Group for the National Aeronautics and Space Administration, Langley Research Center, under Contract NAS1-11689 on the Developmental Study to Assess the Potential of the Application of Resistance Spot Welding - Polyimide Adhesive Bonding to Structural Panel Fabrication.

This work was conducted under the technical direction of Mr. R. Baucom of the Langley Research Center, Hampton, Virginia.

The Applied Chemistry Department of the Chemistry and Chemical Engineering Laboratory, Applied Technology Division was responsible for the work performed on this program. Mr. R. W. Vaughan, Head, Product Development Section was the Program Manager, Mr. R. Kurland was responsible for testing and evaluation of the structural test panels and Mr. T. V. Roszhart was responsible for testing and evaluation of fatigue properties. Dr. R. J. Jones was responsible for the preparation of all polymers and Mr. H. McCracken was responsible for resistance welding activities. Major process development work was performed by Mr. T. Braswell, weld-through process development; Mr. R. Thorpe and Mr. C. H. Choi, capillary-flow process development; and Mr. K. K. Ueda, fabrication of structural test panels. TRW Systems program administrative and review personnel were Dr. E. A. Burns and Mr. B. Dubrow.

SUMMARY

This document is the final program report describing work performed by TRW Systems for the National Aeronautics and Space Administration, Langley Research Center, under Contract NAS1-11689. The objective of this program was to perform a development study to assess the potential of the application of resistance spot welding - polyimide adhesive bonding to structural panel fabrication. This objective was accomplished by 1) evaluating adhesive resin systems and developing detailed processing procedures for fabricating welded joints by weld-through and capillary-flow techniques, 2) performing static and fatigue tests on weld bonded joints and 3) demonstrating the applicability of the weld-through process for fabricating stringer stiffened skin panels.

During Task I, an adhesive system similar to the P4/A5F system developed under Contract NAS1-9532 (Reference 1), was identified for use with a weld-through weld bonding procedure. Lap joints were fabricated by this procedure using techniques amenable to production operations. A new poly (Diels Alder) (PDA) polyimide resin developed under NASA Lewis Research Center Contract NAS3-15834 was identified for use with the capillary-flow process. This particular PDA resin system consisted of *bis*(furfuryl) benzophenone tetracarboxylic imide (BFBI) and *bis*(4-maleimidophenyl) methane (BMPM). Studies demonstrated that this resin is ideal for capillary-flow weld bonding. However, because the BFBI/BMPM resin was developed as a laminating resin, further work is required in order to develop an adhesive system from this resin.

Detailed evaluation of weld bonded joints prepared by both processes was performed during Task II. This evaluation established that a small increase in static strength was obtained with weld bonded joints over welded-only joints. Thermal aging at 561 K (550°F) and thermal cycling over a a temperature range from 219 K (-65°F) to 561 K (+550°F) did not degrade the strength of weld bonded joints significantly. The fatigue strength of weld bonded joints fabricated by the weld-through process was increased significantly over welded-only joints.

A demonstration of the utility of the weld-through weld bonding process for preparing stringer stiffened skin panels was performed during

Task III. Evaluation of the resultant panels consisted of loading the panels in compression and measuring deflection or buckling until the panels collapsed. Trends were established that indicated a contribution to the load carrying capacity of the panels was made by the weld bonding process.

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1. INTRODUCTION

This final report presents the work accomplished by TRW Systems for the National Aeronautics and Space Administration, Langley Research Center, under Contract NAS1-11689 during the period 9 June 1972 through 9 February 1974. The objective of this program was to perform a development study to assess the potential of the application of resistance spot welding - polyimide adhesive bonding to structural panel fabrication.

The need to improve the fatigue strength of resistance welded aircraft structures has been recognized by industry throughout the world. Consequently, after considerable research of the problem, techniques of reinforcing resistance welded joints with adhesives have evolved (weld bonding). These techniques currently are being used with aluminum alloy structures by the aircraft industries of the United States, Europe and the U.S.S.R. Adhesive systems used in these applications are derived from epoxy resin systems which consequently limit the service temperature of these structures to 450 K (350°F) maximum. Therefore, in order to adapt weld bonding to high performance aircraft structures where the service temperature exceeds 450 K (350°F), it is necessary to use high temperature adhesive systems instead of the epoxy resin based materials.

Most state of the art high service temperature adhesives utilize condensation cure polyimide polymer systems which have demonstrated useful properties at elevated temperatures for long periods of time. However, low void content bondlines are not readily obtained with these adhesives by techniques suitable for production processing which in turn results in low strength joints and poor bond strength reliability. Several other polymers have been considered for use in high service temperature adhesive systems such as polypyrrone, polybenzimidazole, polyquinoxaline, polyphenylquinoxaline, polyimidazoquinazoline, etc. These polymers all are condensation cured and suffer from the same deficiencies as the condensation cure polyimide systems.

In order to circumvent these deficiencies, TRW Systems developed a novel pyrolytic polymerization process under NASA Contract NAS3-7949, which utilizes alicyclic nadic end groups on polyimide prepolymers (A-type polyimides) to provide an addition reaction final cure in place of the conventional

polyimide condensation cure. Adaption of this technology to adhesive bonding was performed under NASA Contract NAS1-9532 where high strength bonded joints were obtained by autoclave bonding processes (Reference 1).

Further developmental studies by TRW Systems identified the potential of the adhesive system developed under Contract NAS1-9532 for weld bonding titanium alloy joints. The feasibility of fabricating weld bond joints with this adhesive then was demonstrated by fabricating weld bonded test panels under Purchase Order L66.028 for NASA Langley Research Center. These specimens were prepared by a weld-through weld bonding process, *i.e.*, the adhesive was applied to the joint surfaces prior to spot-welding. Evaluation of these specimens by NASA Langley Research Center demonstrated high potential for this process.

Subsequently, NASA Langley Research Center awarded Contract NAS1-11689 to TRW Systems in order to perform a detailed development study of this new process. During this program a capillary-flow spot-welding process also was investigated. This process permits spot-welding to be performed as the first operation after which the adhesive is applied to the edge of the joint. The adhesive then flows into the joint during an oven cure thus forming a weld bonded joint.

This program was performed in three tasks. During Task I, both the weld-through and capillary-flow weld bonding processes were developed. Joints formed by these two processes then were evaluated during Task II. A demonstration of the suitability of the weld-through weld bonding process for fabricating stringer stiffened skin panels was made as the Task III activity. This task also included structural test and evaluation of the resultant panels.

This report is divided into sections covering each of the three tasks. The significant conclusions reached and assessments of the results are listed together with recommendations for activities that warrant further investigations. The information presented in the main body of this report is supplemented by appendices covering detailed descriptions of procedures used in material preparation and processing.

2. WELD BONDING PROCESS DEVELOPMENT

The objective of this task was to develop two types of weld bonding procedures: 1) a weld-through process and 2) a capillary-flow process. Investigations of the weld-through process used variations of the P4/A5F adhesive system developed under Contract NAS1-9532. During the capillary-flow process studies it was necessary to evaluate other polyimide adhesive resins because the P11BA resin used in the P4/A5F adhesive system did not provide satisfactory resin flow. Satisfactory procedures were developed for both approaches which subsequently permitted the detailed evaluation of joints prepared by both procedures during Task II (see Section 3)

2.1 WELD-THROUGH PROCESS DEVELOPMENT

Two basic approaches were investigated for fabricating weld-bonded joints by a weld-through process. Both of these approaches were based on the fact that since the polyimide adhesive resins used in this program remain solid up to >422 K (>300°F), it is not possible to squeeze the adhesive out of the spot-weld nugget area during the welding operation. Consequently, it was necessary to devise methods for leaving a direct path for the electric current to flow between the two adherends, *i.e.*, leaving bare spots containing no primer or adhesive. The two approaches investigated consisted of a silk-screening adhesive application procedure and masking the spot-weld nugget area prior to applying the adhesive. The adhesive systems used throughout these studies all were based on the P4/A5F adhesive system (Reference 1).

2.1.1 Silk Screening Process Development

In order to prepare silk screens for masking bare spots in the adhesive primer P4 (see Appendix A), it was necessary to use a masking material that was resistant to the primers solvent (dimethylformamide). Several materials were evaluated including silicone elastomers, acrylic polymers and epoxy resin compounds. The epoxy resins were the most successful and consequently were used throughout the remainder of the silk-screening studies. Studies then were performed to determine the most suitable formulation for application by the silk-screening process. It was determined that the following formulation (see Appendix A for resin preparation procedures) provides the desired 0.127 mm (0.005-inch) thick coating:

P11BA (resin solids)	50 pbw
AI-1137 (resin solids)	50 pbw
Aluminum Alloy Powder	175 pbw
Cab-O-Sil	10 pbw
Dimethylformamide (DMF)	150 pbw

Lap-shear test panels then were fabricated using the bond jig shown in Figure 1. These panels were press-bonded in the following manner using adhesive film A5F (see Appendix A) with half-inch diameter holes punched through to coincide with the primer bare spots.

The panels were assembled in the NASA Langley Research Center supplied bonding jig (see Figure 1) and loaded into a cold press. Pressure of 0.7 MN/m^2 (100 psig) was applied and the press platen temperature was raised to 575 K (575°F) at the rate of 5.56 K/minute (10°F/minute). Panels were press cured for one hour, removed from the press and then oven postcured for 16 hours at 561 K (550°F).

The resultant panels were tested at room temperature and provided an average breaking load of 19037 N (4280 pounds). Examination of the failed joints showed good flow of the adhesive into the uncoated areas.

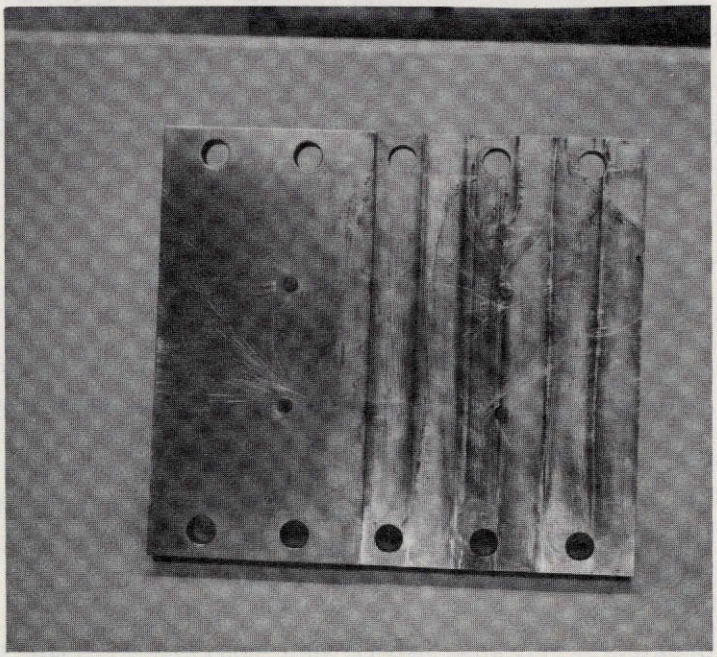


Figure 1. Bond Jig

Adherend surface preparation processes and spot-weld clearance hole diameters through the adhesive film then were evaluated; clearance hole diameters through the primer were maintained at 12.7 mm (0.5-inch) diameter. Six sets of test panels were prepared as shown in Table I using Primer P4 and Adhesive Film A5F. Each set was assembled in the NASA supplied welding jig

TABLE I.

WELD BONDING PREPARATION PROCESS SCREENING

Surface Preparation Procedure	Spot Weld Clearance Hole Diameter Through Adhesive Film: 12.7 mm (0.5 inch)	10.0 mm (0.4 inch)
Sandblast[d]	26688 N 6000 lbs	25354 N 5700 lbs [a]
Pasa-Jel[c] and Burnishing	24901 N 5600 lbs	33360 N 7500 lbs
Pasa-Jel[b]	25799 N 5800 lbs	55799 N 5800 lbs

[a] Breaking load of specimens (average of 3 tests).

[b] Titanium alloy 6Al4V was prepared for bonding with Pasa-Jel in accordance with Appendix A and then the adhesive primer was applied by the silk screen process.

[c] Titanium alloy 6Al4V was prepared for bonding with Pasa-Jel in accordance with Appendix A and then the adhesive primer was applied by brush over the complete faying surface. After drying, spot weld clearance holes were made using the trim template (see Figure 2) and a 200 grit Carborundum sanding disc.

[d] Titanium alloy 6Al4V was sand-blasted (50 micron alumina) and solvent washed with methyl ethyl ketone (MEK). The adhesive primer was applied by the silk screen process developed previously (see Section 2.1.1).

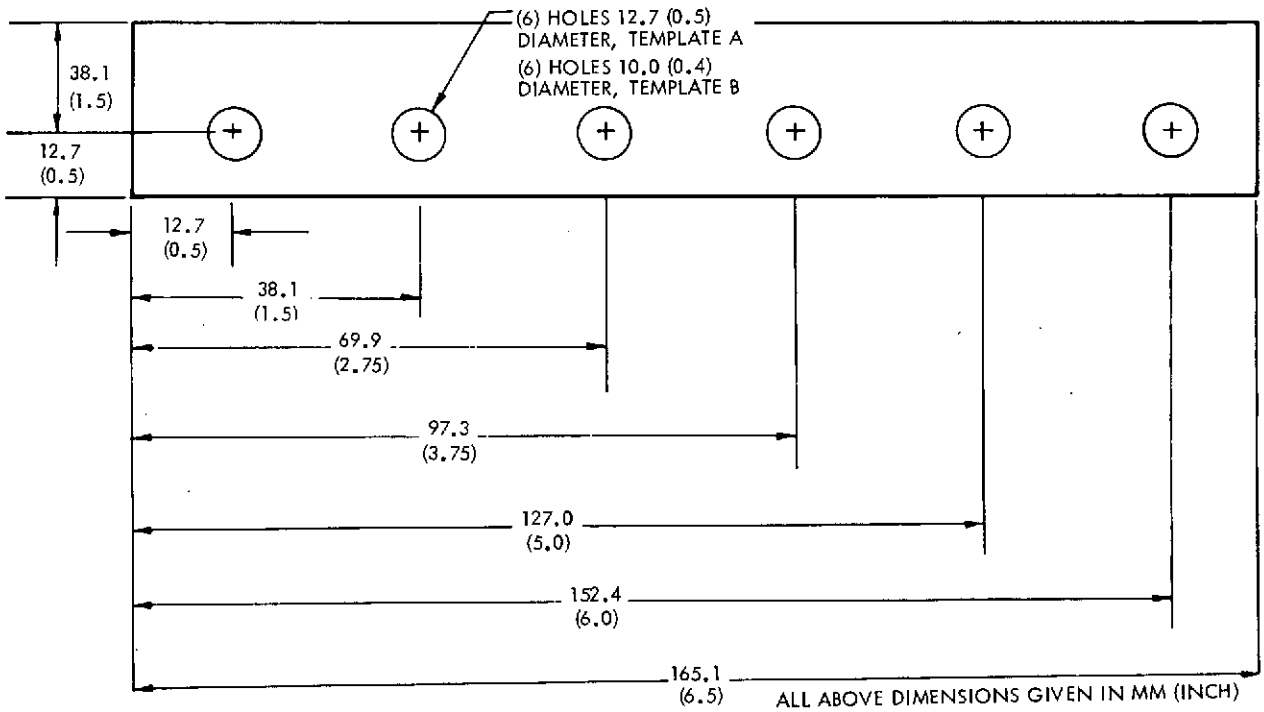


Figure 2. Trim Template

(see Figure 3) and spot welded on a Sciaky Welding Machine at the following machine settings:

Tip Pressure	-	18000 N (4000 lbs)
Tip Diameter	-	12.7 mm (0.5-inch)
Gauge #1	-	97 N/m^2 (14psig)
Gauge #2	-	234 N/m^2 (34 psig)
Squeeze Setting	-	0.3 - 50
Hold Setting	-	0.75 - 50
Weld Setting	-	0.75 - 32
Off Setting	-	0.75 - 55
Phase Shift Setting	-	27
Nugget Diameter	-	7.6 mm (0.300-inch)
Current Delay	-	None
Recompression Delay	-	None

During examination of the test specimens prior to test, it was observed that most of the spot welds appeared to be tipped. Also, the outer corners of the lap joints were curled up causing a large gap in these areas (see Figure 4). Values obtained during test showed no significant difference between the panels except for the panel produced by burnishing away the primer for the spot-weld together with a 10.0 mm (0.4-inch) diameter hole through the adhesive film which provided a breaking load twenty-five percent higher than the other panels. Post failure analysis of this panel showed that the spot welds were superior (not tipped and larger diameter) than in the other panels. Further investigation indicated that runs of the adhesive primer under the silk screen mask were the

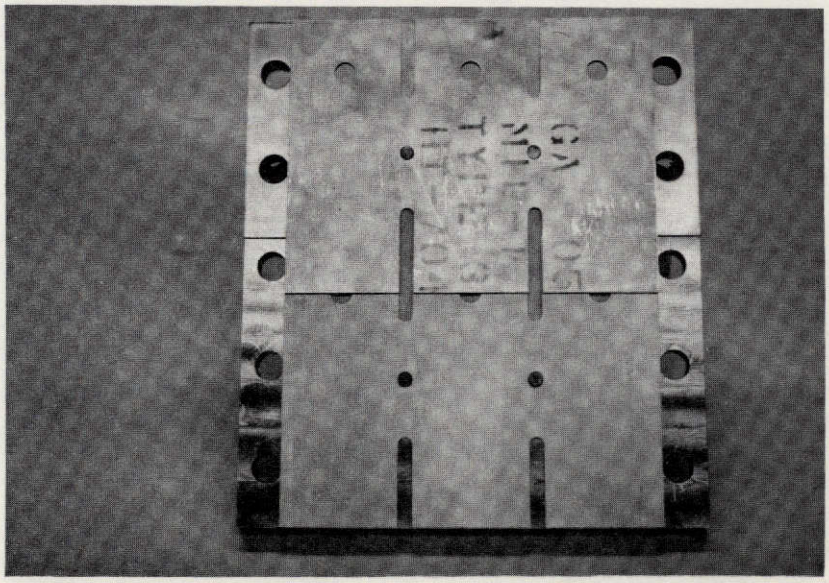


Figure 3. Weld Jig

prime cause of substandard welds in most of the above panels.

Another key observation made during post failure examinations was that the curling of the titanium alloy panels at the joint corners resulted in unbonded areas (see Figure 5). This provided an estimated 75 percent adhesive bonded area only thus reducing the adhesive efficiency considerably.

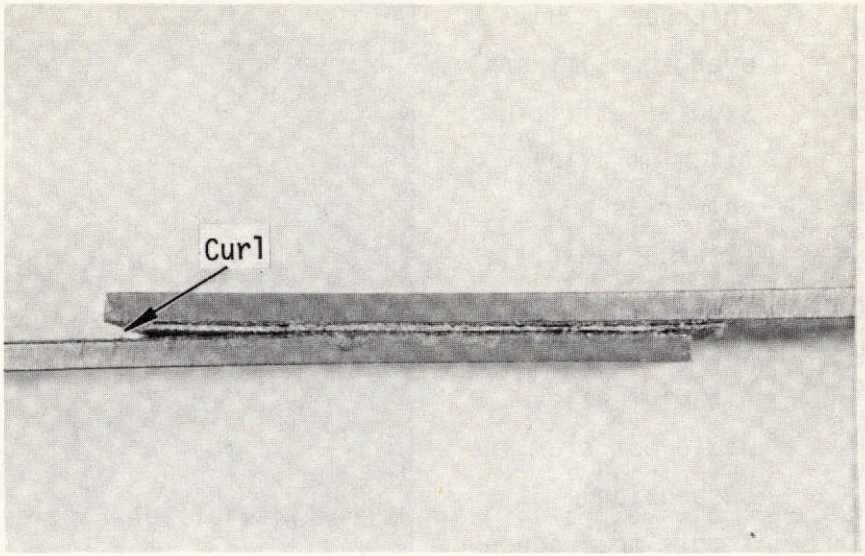


Figure 4. Uncured Resistance Welded Joint

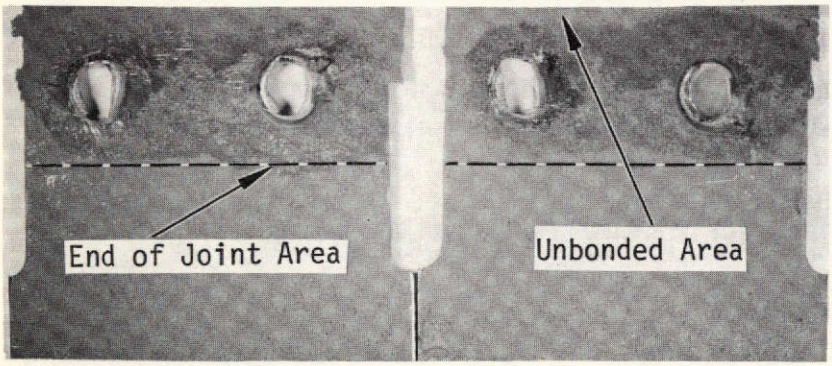


Figure 5. Unbonded Areas in Curled Joints

2.1.2 Development of Primer Masking Method

In order to eliminate the problem discussed in Section 2.1.1 pertaining to adhesive primer runs into the spot weld area, an alternative masking method was sought. During the ensuing investigations use of an adhesive backed paper mask (Avery labels) provided satisfactory results. Further development work with this approach provided a means of precisely locating round adhesive backed paper masks that also may be removed rapidly as a continuous strip. This mask configuration is shown in Figure 6 together with an adhesively primed surface with the mask removed. Tests were performed to determine whether the adhesive backing on the masking material degraded the spot weld. Results from these studies showed no significant difference in welds through panels that had been masked and those that had not.

Originally it was planned to evaluate variations in adhesive compound formulations for the weld-through process as well as variations in adhesive

thickness. However, preliminary studies utilizing titanium alloy powder as an alternative filler to aluminum alloy showed that the titanium alloy would not stay in suspension. Attempts to produce an adhesive film were negative (resin system and titanium alloy powder separated on the glass scrim carrier).

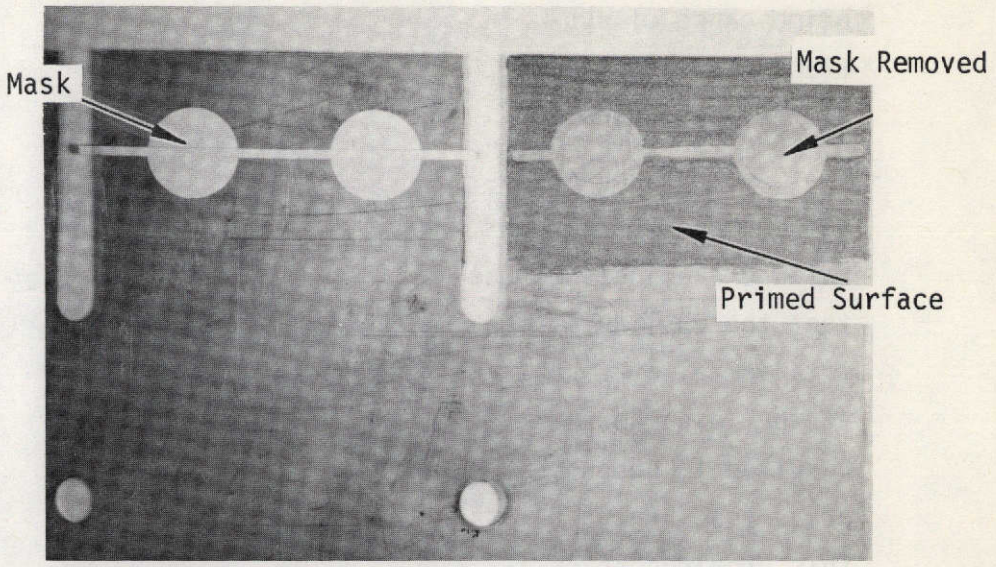


Figure 6. Primer Mask

Consequently, it was decided to concentrate on the problem of titanium alloy panel curling during the spot-welding operation. The objective of this study was to improve the adhesive bonding efficiency in weld-bonded joints.

During this study, panels were weld-bonded using different thickness adhesive films as shown in Table II, *i.e.*, 0.381 mm (0.015-inch), 0.178 mm (0.007-inch) thick and no adhesive film. Test panels also were stress relieved in order to determine whether this approach would prevent the titanium alloy from curling during the welding operation. Weld-bonded joints produced from stress relieved panels were not significantly different from those produced from as-received panels; therefore, it was decided that stress relieving was not necessary. Prior to test, the test panel without adhesive

TABLE II.

ADHESIVE THICKNESS SCREENING STUDY

Type of Specimen	Breaking[a] Load, N (lbs)	Type[b] of Failure
Adhesive primer plus adhesive film 0.381 mm (0.015-inch thick)	34250 (7700)	75% adhesive to titanium. 25% unbonded
Adhesive primer plus adhesive film 0.178 mm (0.007-inch thick)	33360 (7500)	100% unbonded
Adhesive primer only	32026 (7200)	100% unbonded

[a] Average of three specimens.

[b] As defined by visual examination.

film was flat and the test panel with 0.178 mm (0.007-inch) thick adhesive film had a small curl (see Figure 7). The panel containing 0.381 mm (0.015-inch) thick adhesive film was curled as previously described. Results from tests on these panels (see Table II) showed no significant difference between the panels containing 0.178 mm (0.007-inch) thick and 0.381 mm (0.015-inch) thick adhesive film, although they both were higher than the panel with adhesive primer-only (again not significantly). Post failure

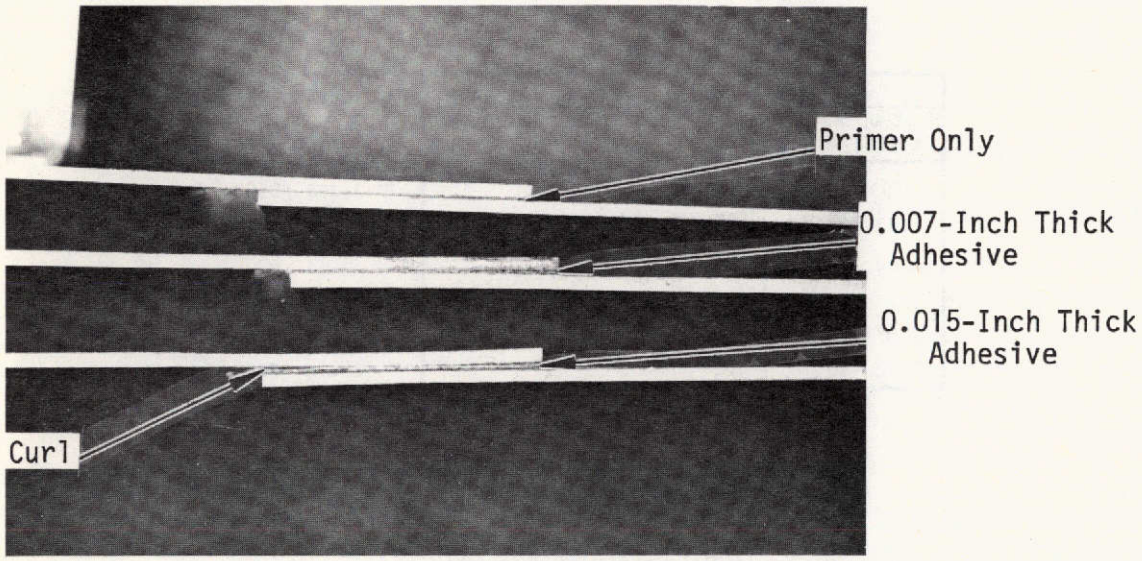


Figure 7. Bondline Thickness Effects on Skin Curl

examination of these test panels showed that only the 0.381 mm (0.015-inch) adhesive film appeared to be functioning as a bonding agent. In the bonded areas of specimens containing this film, an adhesive failure occurred (approximately 75% of the joint area). Joint areas in the other two panels appeared to be 95% unbonded which was attributed to zero applied pressure on the adhesive material as a result of the welding operation.

Adhesive bonding heat-up rates and cure temperatures then were screened (see Table III) and although lap-shear values were lower than obtained previously, it was established that a 5.56 K (10°F) per minute heat-up rate to 589 K (600°F) followed by a sixteen-hour cure at that temperature provided the best values. Because these values were significantly lower than obtained during previous studies it was decided to screen imidizing cycles in an attempt to improve adhesive flow and consequently to improve adhesion

Adhesive film A5F was prepared, dried for 5 minutes at 408 K (275°F) and then imidized in accordance with the schedule shown in Table IV. Lap-shear panels were prepared in the manner described previously and tested at room temperature. The results from this study (see Table IV) did not show any improvement from imidizing cycle variations over the established cycle of 5 minutes at 450 K (350°F).

TABLE III.
CURE CYCLE STUDIES

Cure Cycle			Breaking Load, N (lbs)
Heat-up Rate, K (°F)/Min.	Cure Temp., K (°F)	Cure Time, Hrs.	
(a)	561 (550)	16	24464 (5500)
1.67 (3)	561 (550)	16	26688 (6000)
5.56 (10)	561 (550)	16	26243 (5900)
(a)	589 (600)	16	26688 (6000)
1.67 (3)	589 (600)	16	23130 (5200)
5.56 (10)	589 (600)	16	28022 (6300)

(a) Oven preheated to cure temperature.

TABLE IV.
IMIDIZATION CURE STUDIES

Drying and Imidizing Cycle		Breaking Load[a] N (lbs)
Drying, Min/ K (°F)	Imidizing, Min/ K (°F)	
15/403 (275)	5/422 (300)	22685 (5100)
15/403 (275)	5/422 (300)	24909 (5600)
15/403 (275)	5/450 (350)	26688 (6000)

(a) Lap-shear weld-bonded specimens cured 16 hours at 589 K (600°F).

The low values reported in Tables III and IV indicated that further evaluation of the specimens tested during the preceding two studies was necessary in order to assess the causes of these lower values. After careful scrutiny of all the broken specimens it was apparent that the lower values were related to poor spot-welds. Further examination of the poor spot-welds showed that the adhesive film had slipped during assembly causing adhesive to block the electric current path partially during welding. Subsequently, it was decided to evaluate the use of adhesive paste alone using the primer masking procedure to provide positive location of the bare metal

(uncoated) areas for welding. Lap-shear specimens then were prepared using three coating thicknesses, 0.4 mm (0.016 inch), 0.25 mm (0.010 inch) and 0.15 mm (0.006 inch). These specimens were welded and then oven cured by the process discussed previously. The resultant panels were tested and the breaking loads are shown in Table V. Based upon these results it was decided to proceed with this process using the 0.25 mm (0.010-inch) thick adhesive coating for fabricating Task II test specimens and Task III structural test panels.

TABLE V.
ADHESIVE THICKNESS EVALUATION

Adhesive Thickness[(a)] mm (inch)	Breaking Load, N (lbs)
0.15 (0.006)	32470 (7300)
0.25 (0.010)	33805 (7600)
0.40 (0.016)	32026 (7200)

(a) Total thickness (including primer)

2.2 CAPILLARY-FLOW PROCESS DEVELOPMENT

Two adhesive resin systems were evaluated during this program for the capillary-flow process, *i.e.*, the BFBI/BMPM resin (developed for NASA Lewis Research Center under Contract NAS3-15834 for structural composites) and NA/TDA/BSDA resin (a new resin formulation similar to P11BA resin). Both of these resins demonstrated high promise of meeting the program's objectives as described below. However, during the period that these studies were being performed, TDA (thiodianiline) was identified by OSHA as a potential carcinogenic material. Consequently, Southern Dyestuff Company (SDC) withdrew this material from the market and ceased production. Because SDC was the only source for TDA, it was decided not to proceed with further evaluation of the NA/TDA/BSDA resin. Therefore, the BFBI/BMPM resin was selected as the capillary-flow adhesive resin for use throughout the remainder of this program.

2.2.1 Evaluation of NA/TDA/BSDA Resin

Welding schedule development studies were performed with the objective of obtaining controlled joint separations (gaps) in combination with acceptable weld-nugget strengths [*i.e.*, 12010 N (2700 lbs) breaking load] for 1.27 mm (0.05-inch) thick, 6Al4V titanium alloy joints. During these studies, it was established that adequate reproducibility and control of joint separation was established by varying the resistance spot welding tip radius, heat cycle

and tip pressure. Examples of the joint separations obtained by variations in welding schedules for preparing lap-shear specimens are provided in Table VI. All of the welds produced by these schedules provided breaking loads >12010 N (2700 lbs)/nugget.

TABLE VI.
WELDING SCHEDULES SCREENING

Control Setting	Schedule Number	
	1	2
Top Gauge, N/m^2 (psig)	234 (34)	290 (42)
Bottom Gauge, N/m^2 (psig)	97 (14)	276 (40)
Tip Radius[a], mm (inches)	152.4 (6)	50.8 (2)
Machine Power[b]		
Phase Shift	30	30
Phase Vernier	40	40
Joint Separation, mm (inch)	0.08 (0.003)	0.24 (0.009)

[a] 15.88 mm (5/8-inch) diameter electrode, 12.70 mm (1/2-inch diameter tip.
[b] Constant pressure, parallel, 3 cycle heating, 1 cycle cooling.

An evaluation of the resin system used in the P4/A5F adhesive system (*i.e.*, P11BA/AI-1137) was performed which indicated that this resin system was not suitable for the capillary-flow weld bond process. Consequently, it was decided to prepare other A-type polyimide resin formulations that provides higher resin flow. The first resin formulation prepared consisted of nadic anhydride, thiodianiline and *bis*(3,4-dicarboxyphenoxyphenyl) sulfone dianhydride (NA/TDA/BSDA) in molar ratios of 2:2:1, respectively. This resin was prepared in the imidized prepolymer form which was soluble in both DMF and MEK. A viscous resin paste subsequently was prepared from the imidized prepolymer using DMF as the solvent and applied as a bead along the edge of a spot-weld lap-shear joint. This panel then was placed in an air circulating oven and dried for two hours at 422 K (300°F) followed by two hours at 561 K (550°F) Good resin flow occurred and resin wetted both faying surfaces in the joint (see Figure 8). However a solid resinous bondline was not formed that filled the gap completely. This indicated that smaller joint separation (gap) and/or further resin formulation modification was necessary. Consequently, a screening matrix was designed which included for evaluation this new A-type resin formulation with three different solvents, *i.e.*, DMF, NMP and MEK. Earlier work had shown that A-type polyimide resins by themselves are not good metal adhesives but that copolymeric blends with amide-imide resins form excellent metal adhesive systems (Reference 1). Therefore, another factor evaluated in this screening study (see Table VII) was the copolymeric blend of the NA/TDA/BSDA resin with Amoco amide-imide resin AI-1137.

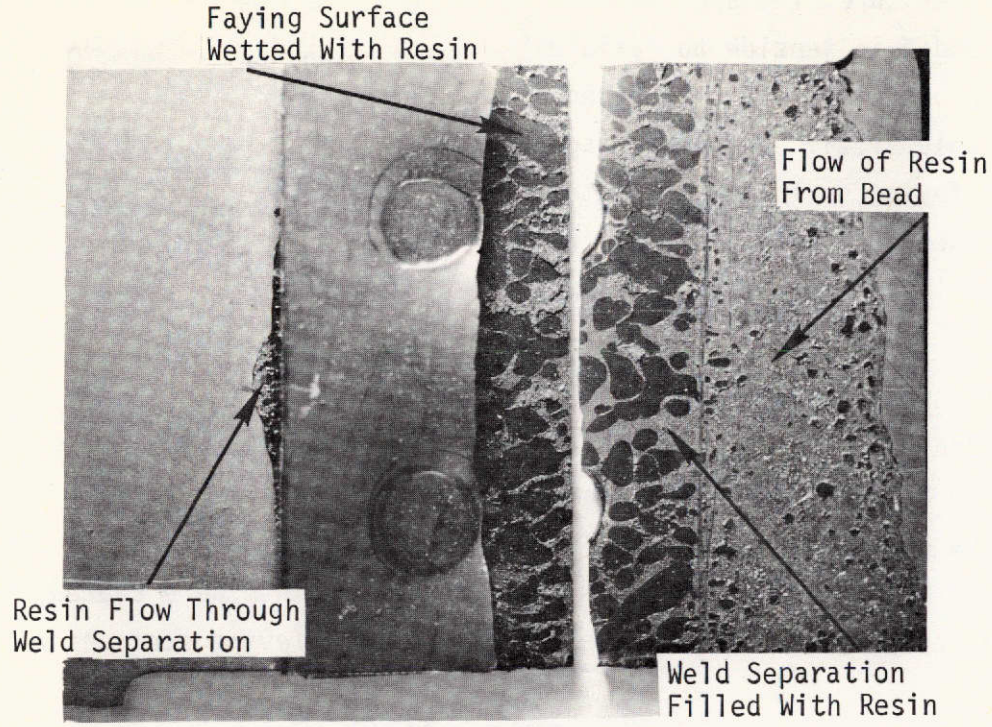


Figure 8. Capillary-Flow Joint

TABLE VII.

PRELIMINARY PROCESS AND ADHESIVE FORMULATION SCREENING MATRIX

Weld Schedule I[b] Cure Cycle[c]	Adhesive Formulation[a] A	B	C	D
16/R.T. 2/533(500)	90	30	30	100[d]
16/R.T. 2/422(300) 2/533(500)	90	20	20	100
Weld Schedule II[b] Cure Cycle				
16/R.T. 2/533(500)	90	80	60	100
16/R.T. 2/422(300) 2/533(500)	70	20	80	100

[a] A = NA/TDA/BSDA 50% w/w DMF
B = NA/TDA/BSDA 50% w/w NMP
C = NA/TDA/BSDA 50% w/w MEK
D = NA/TDA/BSDA 50 pbw, AI-1137 50 pbw, DMF 80 PBW

[b] See Table VI.

[c] Cure cycle in air hrs/°K (°F).

[d] Percent of joint surface area coated with adhesive.

Lap-shear joints were prepared by spot welding the test panels in accordance with the two welding schedules defined in Table VI. Adhesive formulations then were prepared in accordance with Table VII and a small bead of adhesive was splined along one edge of the lap joint (see Figure 9). These specimens then were allowed to dry overnight (*i.e.*, 16 hours at 294 K (70°F) in 50% R.H. atmosphere) after which they were cured in an air circulating oven for either 2 hours at 422 K (300°F) plus 2 hours at 533 K (500°F) or for two hours at 533 K (500°F) only (see Table VII).

After cure, the specimens were loaded in tension to failure and the breaking load determined (see Table VIII). The percentage of the faying surface area coated with adhesive then was calculated and tabulated (see Tables VII and VIII).

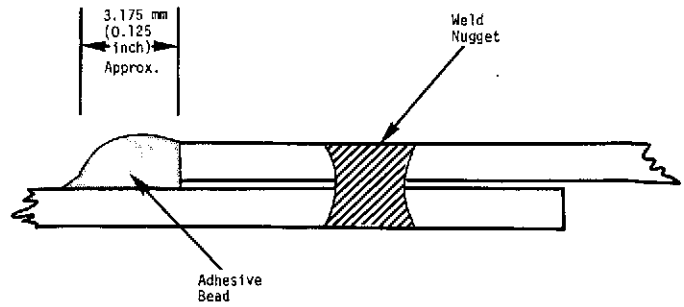


Figure 9. Application of Adhesive For Capillary-Flow Process

The results of this study indicated that the DMF containing adhesive formulations A and D and the fast heat-up rate cure cycle provided the most promising flow characteristics. Although 100% of the faying surfaces in these joints were coated with the adhesive, complete filling of the gap between the faying surfaces was not achieved.

TABLE VIII.

PRELIMINARY EVALUATION OF WELD SCHEDULE, ADHESIVE FORMULATION AND CURE CYCLE

Weld [a] Schedule	Cure Cycle,[b] Hrs/K (°F)	Adhesive [c] Formulation	Adhesive [d] Flow, %	Breaking Load,[e] N (lbs)
I	16/R.T.	A	90	26243(5900)
	2/422(300)	B	20	25798(5800)
	2/533(500)	C	20	25798(5800)
		D	100	24464(5500)
II	16/R.T.	A	70	26688(6000)
	2/422(300)	B	20	27578(6200)
	2/533(500)	C	80	27578(6200)
		D	100	28467(6400)
I	16/R.T.	A	90	25798(5800)
		B	30	24909(5600)
		C	30	26688(6000)
		D	100	23574(5300)
II	16/R.T.	A	90	26688(6000)
	2/533(500)	B	80	26243(5900)
		C	60	28022(6300)
		D	100	27578(6200)

[a] See Table VII.

[b] Cure in air circulating oven

[c] A = NA/TDA/BSDA 50% w/w DMF
B = NA/TDA/BSDA 50% w/w NMP
C = NA/TDA/BSDA 50% w/w MEK
D = NA/TDA/BSDA 50 pbw, AI-1137 50 pbw, DMF 80 pbw

[d] Percent of joint surface area coated with adhesive.

[e] Lap-shear joint 50.8 mm (2-inch) wide, 25.4 mm (1-inch) overlap, 2 weld nuggets per joint, tested in tension.

It was estimated that 30% of the joint area consisted of a complete bondline, *i.e.*, gap-fill, and the remainder consisted of adhesive coatings on each faying surface. Consequently, it was decided to evaluate the effect of a filler in the adhesive formulation.

In order to assess the potential of incorporating a filler in the adhesive formulation to provide good gap-filling, an experiment was performed using aluminum alloy powder, Grade 101. Lap-shear specimens were welded using the weld schedules described earlier. An adhesive formulation was prepared consisting of:

NA/TDA/BSDA	-	50 pbw
AI-1137	-	50 pbw
Aluminum Alloy Powder, Grade 101	-	50 pbw
DMF	-	80 pbw

A bead consisting of this adhesive formulation was splined along the edge of the welded joint which then was cured in an air circulating oven for 16 hours at R.T. plus 2 hours at 533 K (500°F). The resultant weld bonded joints contained approximately 60% of the total surface coated with adhesive with approximately 50% of the coated surface providing a complete gap-fill. These results indicated high promise of achieving good capillary-flow in combination with good gap-fill by the use of fillers in the above adhesive formulation. Consequently, an adhesive formulation/cure cycle screening matrix was designed for investigation (see Table IX).

All of the adhesive compound formulations evaluated during this study contained equal proportions by weight of TRW A-type polyimide prepolymer, NA/TDA/BSDA and Amoco AI-1137 amide-imide resin. Various amounts of a thixotropic agent were evaluated in order to determine whether a thixotropic agent would control the capillary-flow of the adhesive through the weld separation and produce the desired gap-fill. Similarly, a standard adhesive compound filler, aluminum alloy powder Grade 101, also was evaluated for the same purpose. Details of the formulations investigated are provided in Table X.

TABLE IX.

ADHESIVE FORMULATION/CURE CYCLE SCREENING MATRIX

Adhesive Formulation[b]	Cure Cycle[a] 16/R.T. 2/422(300) 2/561(550)	16/R.T. 2/477(400) 2/561(550)	16/R.T. 2/505(450) 2/561(550)	16/R.T. 2/533(500) 2/561(550)	
50/50/50/0	--	--	--	25798 (5800) 60 30	Breaking Load, N (lbs) Joint Area Covered, % Joint Area Filled, %
50/50/100/0	25798 (5800) 60 5	27578 (6200) 60 10	26688 (6000) 50 10	23574 (5300) 60 10	Breaking Load, N (lbs) Joint Area Covered, % Joint Area Filled, %
50/50/50/5	27133 (6100) 50 5	25798 (5800) 60 5	27578 (6200) 40 5	24464 (5500) 5 0	Breaking Load, N (lbs) Joint Area Covered, % Joint Area Filled, %
50/50/0/5	29802 (6700) 10 0	28912 (6500) 10 0	29357 (6600) 10 0	24464 (5500) 60 10	Breaking Load, N (lbs) Joint Area Covered, % Joint Area Filled, %
50/50/100/5	26688 (6000) 60 5	26243 (5900) 60 5	25798 (5800) 50 10	25354 (5700) 10 0	Breaking Load, N (lbs) Joint Area Covered, % Joint Area Filled, %

[a] Cure in air, hrs/K (°F)

[b] a/b/c/d where: a = AI-1137, pbw
b = NA/TDA/BSDA, pbw
c = Aluminum alloy powder, Grade 101, pbw
d = Cab-O-Sil, pbw

Titanium alloy 6Al4V lap-shear test panels were welded in accordance with Schedule II of Table VI, and a bead of the appropriate adhesive formulation then was applied along one edge of the joint (see Figure 9). The adhesive beads on these specimens were allowed to dry overnight at room temperature, *i.e.*, 16 hours at ∿294 K (∿70°F) before cure. Specimens then were placed into a preheated air circulating oven (see Table IX) for two hours to promote capillary-flow and then cured for two hours at 561 K (550°F).

TABLE X.

ADHESIVE COMPOUND FORMULATIONS FOR THIXOTROPIC AND FILLER STUDIES

Constituent	Quantity, pbw				
Constituent Proportions (see Table IX).	50/50/50/0	50/50/100/0	50/50/50/5	50/50/100/5	50/50/0/5
Amoco AI-1137 amide-imide resin, solids of amide acid.	50	50	50	50	50
NA/TDA/BSDA polyimide resin, solids of polyimide.	50	50	50	50	50
DMF	80	80	80	80	80
Aluminum alloy powder, Grade 101	50	100	50	100	0
Cab-O-Sil thixotropic agent, silica.	0	0	5	5	5

The resultant test specimens were loaded in tension and the load at failure was recorded (see Table IX). Measurements were taken of the area coated with the adhesive and of the adhesive joint which was filled with adhesive. These results (see Table IX) indicated that:

- The thixotropic agent decreased capillary-flow and did not produce the desired viscous flow required to provide gap-fill.
- Lower amounts of aluminum alloy powder (~50 pbw) provided the most promising gap-fill.

Evaluation of variations in the loading temperature (*i.e.*, flow temperature) did not provide definitive differences in results because the adhesive formulation variations provided overwhelming differences. Consequently, adhesive formulation variation studies were continued employing fillers only instead of thixotropic agents. Adhesive formulations were prepared in accordance with Table XI which included aluminum alloy powder and flake, clay, alumina (aluminum oxide) and titanium dioxide. Results from this study (see Table XII) did not indicate significant improvement in the adhesive's flow and gap-filling characteristics from use of alternative filler materials over aluminum alloy powder.

In order to investigate the effects of different adhesive resin solvent systems on resin flow it was necessary to obtain a dry amide-imide, amide-acid powder. Because this material was not available locally from Amoco Chemical Corporation, a small amount of resin was precipitated out of the

TABLE XI.

ADHESIVE FORMULATIONS FOR FILLER EVALUATIONS

Constituents	Quantity, pbw										
Formulation Code	TH	TL	AF5H	AF5L	AF6H	AF6L	APH	APL	DCH	DCL	AOH
Amoco AI-1137 amide-imide resin, solids of amide-acid	50	50	50	50	50	50	50	50	50	50	50
NA/TDA/BSDA polyimide resin, solids of polyimide	50	50	50	50	50	50	50	50	50	50	50
DMF	80	80	80	80	80	80	80	80	80	80	80
Titanium dioxide	50	25	--	--	--	--	--	--	--	--	--
Aluminum alloy flake, grade 552	--	--	50	25	--	--	--	--	--	--	--
Aluminum alloy flake, grade 606	--	--	--	--	50	25	--	--	--	--	--
Aluminum alloy atomized powder	--	--	--	--	--	--	50	25	--	--	--
Dixie clay	--	--	--	--	--	--	--	--	50	25	--
Alumina (aluminum oxide)	--	--	--	--	--	--	--	--	--	--	50

TABLE XII.

EVALUATION OF ADHESIVE FILLER MATERIALS

Adhesive Formulation [a]	TH	TL	AF5H	AF5L	AF6H	AF6L	APH	APL	DCH	DCL	AOH
Cure Cycle, hrs/°K [b]	16/R.T. 2/533 2/561	16/R.T. 2/533 2/561	16/366 2/533 2/561	16/366 2/533 2/561	16/366 2/533 2/561	16/366 2/533 2/561	16/R.T. 2/533 2/561	16/R.T. 2/533 2/561	16/R.T. 2/533 2/561	16/R.T. 2/533 2/561	16/R.T. 2/533 2/550
Breaking Load, N (lbs)	28022 (6300)	26688 (6000)	28467 (6400)	28467 (6400)	26688 (6000)	26688 (6000)	25798 (5800)	25798 (5800)	25798 (5800)	24464 (5500)	25798 (5800)
Joint Area Covered, %	60	70	50	85	50	85	100	95	40	90	60
Joint Area Filled, %	10	20	25	40	30	50	50	30	15	40	20

[a] See Table XI.

[b] 366 K = 200°F
533 K = 500°F
561 K = 550°F

AI-1137 varnish. This was accomplished by adding AI-1137 varnish dropwise to acetone and permitting the amide-acid to precipate in the bottom of a beaker. The solvents then were decanted out of the beaker and the amide-acid precipitate was washed three times with acetone. This amide-acid resin then was dried in an air circulating oven at 311 K (100°F).

Lap-shear test panels then were prepared in accordance with Table XIII using adhesive formulations that provided one solvent-based system (acetone)

TABLE XIII.

ADHESIVE SOLVENT/REACTIVE
DILUENT FORMULATIONS

Constituents	Quantity, pbw	
	Solvent System	Reactive Diluent System
Amoco AI-1137 amide-imide, amide-acid powder	50	50
NA/TDA/BSDA polyimide resin, solids of polyimide	50	50
HYSTL C-1000, carboxy terminated polybutadiene, 90% 1,2 vinyl content	--	0.5
Acetone	50	--

and one reactive diluent system (carboxy terminated polybutadiene). Satisfactory adhesive flow did not occur using either of these approaches suggesting that small amounts of DMF may aid in promoting resin flow.

2.2.2 Evaluation of BFBI/BMPM Resin

Because studies under Contract NAS3-15834 identified a resin consisting of *bis*(furfuryl) benzophenone tetracarboxylic imide (BFBI) and *bis* (4-maleimidophenyl) methane (BMPM) (see Appendix A), that provides excellent flow characteristics, a trial experiment was performed to assess the potential of this resin for capillary-flow weld-bonding use. Results from this experiment showed that excellent wetting of both surfaces in the welded joint occurred. Consequently, adhesive formulations containing the BFBI/BMPM resin were screened together with the NA/TDA/BSDA resin (see Table XIV). Included in this study was an alternative approach to the use of variations in adhesive formulation for changing the contact angle of the adhesive resin while undergoing capillary-flow. This approach investigated variations in the surface roughness of the metal adherends in the joints. Previous studies performed by R. E. Johnson, Jr. and R. H. Dettre (Reference 2) have shown that surface roughness, although not the only contributing factor is a major factor affecting the contact angles of liquids. The study (see Table XIV) was designed to define the correct surface roughness for obtaining a contact angle which provided a solid resin fill of the weld separation. Subsequently, lap-shear panels were spot-welded using the surface preparation procedures defined in Table XIV. The adhesive systems were prepared in accordance with Table IX and applied to the spot-welded specimens.

TABLE XIV.
EXPERIMENTAL SCREENING MATRIX

Constituent or Specimen Condition[a]		Quantity/Value[b]											
BFBI/BMPM Resin		50	75	100	50	75	100	--	--	--	--	--	--
NA/TDA/BSDA Resin		--	--	--	--	--	--	50	75	100	50	75	100
AI-1137		50	25	--	50	25	--	50	25	--	50	25	--
Aluminum Alloy Powder		25	25	25	--	--	--	25	25	25	--	--	--
Sand-Blasted Adherend Surface	c)	0	0	100	40	0	100	10	25	100	25	75	100
	d)	0	0	65	20	0	45	--	--	40	--	10	40
	e)	22685 (5100)	22685 (5100)	21350 (4800)	20461 (4600)	23130 (5200)	23130 (5200)	25354 (5700)	25798 (5800)	23130 (5200)	28912 (6500)	27133 (6100)	26243 (5900)
Pasa-Jel 107 Surface	c)	0	0	100	100	100	100	0	0	100	0	5	100
	d)	0	0	15		50	40	0	0	35	0	--	60
	e)	26243 (5900)	27133 (6100)	27578 (6200)	23574 (5300)	24019 (5400)	24019 (5400)	25354 (5700)	25798 (5800)	24019 (5400)	23574 (5300)	23574 (5300)	23130 (5200)

[a] BFBI/BMPM - poly(Diels Alder) polyimide resin developed under Contract NAS3-15834
NA/TDA/BSDA - A type polyimide resin similar to P11BA

[b] All adhesive formulation varnishes were ~65-70% solids content in DMF. Quantities are percent by weight. Adhesive cure cycle was: 16/284 (70), 2/533 (500), 2/561 (550) hrs/°K (°F).

[c] Joint area covered, %.

[d] Joint area filled, %.

[e] Breaking load, N (lbs).

These specimens were dried at room temperature for 16 hours, then cured in an air circulating oven for 2 hours at 533 K (500°F) plus 2 hours at 561 K (550°F).

The resultant specimens were broken by applying a tensile load until the weld-bonded joints failed in shear. A post-failure examination of the weld bonded joints was made in order to estimate the percent of surface area covered and percent of weld separation filled. These results are provided in Table XIV and indicate the following:

- Sand-blasted adherend surfaces retard the adhesive's flow
- Both polyimide resin systems provide adequate capillary-flow but the amide-imide resin retards resin flow without improving the adhesive's gap-filling characteristics
- Filler in the adhesive system retards flow and does not improve gap-fill

Another key factor which was introduced into the selection of a capillary-flow weld bond adhesive resin resulted directly from OSHA identifying TDA as a potential carcinogenic material. This material immediately became unavailable commercially because Southern Dyestuff Company (the sole producer) halted all production activities.

Therefore, the unfilled BFBI/BMPM resin system and the following process were selected for use in preparing capillary-flow weld bonded specimens throughout the remainder of the program:

> Prepare the faying surfaces of the adherends by degreasing with methyl ethyl ketone (MEK) then immerse the specimens in Pasa-Jel 107 for 15 minutes at room temperature. Rinse in distilled water and dry in an air circulating oven at 339 K (150°F). Spot-weld the specimens within four hours and immediately apply a bead of BFBI/BMPM adhesive resin paste (30% w/w DMF). Air dry for 16 hours at room temperature then cure for two hours at 533 K (500°F) plus 2 hours at 561 K (550°F).

3. WELD BONDING PROCESS EVALUATION

Weld bonded joints prepared by both procedures, *i.e.*, weld-through and capillary-flow, were evaluated statically and dynamically (fatigue tests). Joints bonded with both adhesive systems, *i.e.*, P4/A5F and BFBI/BMPM as well as welded-only joints were tested and provided baseline data for comparison with the weld bonding procedures. These tests demonstrated that weld bonding provides a significant improvement in fatigue life over welded-only joints. They also showed that although the BFBI/BMPM resin system provides excellent capillary-flow processing characteristics, this resin requires optimization in order to obtain high adhesive properties.

3.1 EVALUATION OF WELD-THROUGH PROCESS

Titanium alloy lap-shear test specimens prepared by the weld-through weld bonding process were evaluated in order to determine the advantages of this new process over conventional spot welding. Because aluminum alloy weld bonded joints containing epoxy adhesives have demonstrated superior fatigue strength over conventionally welded aluminum alloy joints, fatigue tests were performed during this study as well as conventional static tests. Also, because adhesive bonded joints are vulnerable to decay in certain extreme environmental conditions, both iso-thermal aging at 561 K (550°F) and thermal cycling tests over a temperature range from 219 K (-65°F) to 561 K (+550°F) were performed. These tests were designed to define the effects of thermo-oxidative degradation and of any cycle stresses caused by thermal expansion mismatch between the adherends and adhesives.

3.1.1 Fabrication Procedure

The titanium alloy 6Al4V faying surfaces of the lap-shear finger test panels (see Figure 10) were vapor degreased, grit blasted with 50 micron alumina and water rinsed. They then were immersed in Pasa-Jel 107 for 15 minutes at 294 K (70°F), water rinsed and dried at 339 K (150°F). Paper masks then were applied for the spot-weld areas as described in Section 2.1. Adhesive primer was applied by brush, dried in an air circulating oven for 15 minutes at 403 K (275°F) and imidized by thermal treatment for 5 minutes at 450 K (350°F). Paste adhesive was splined onto the primed surface to provide a total thickness (primer plus adhesive paste) of 0.25 mm (0.010 inch) then dried and imidized by the same process as used for the primer.

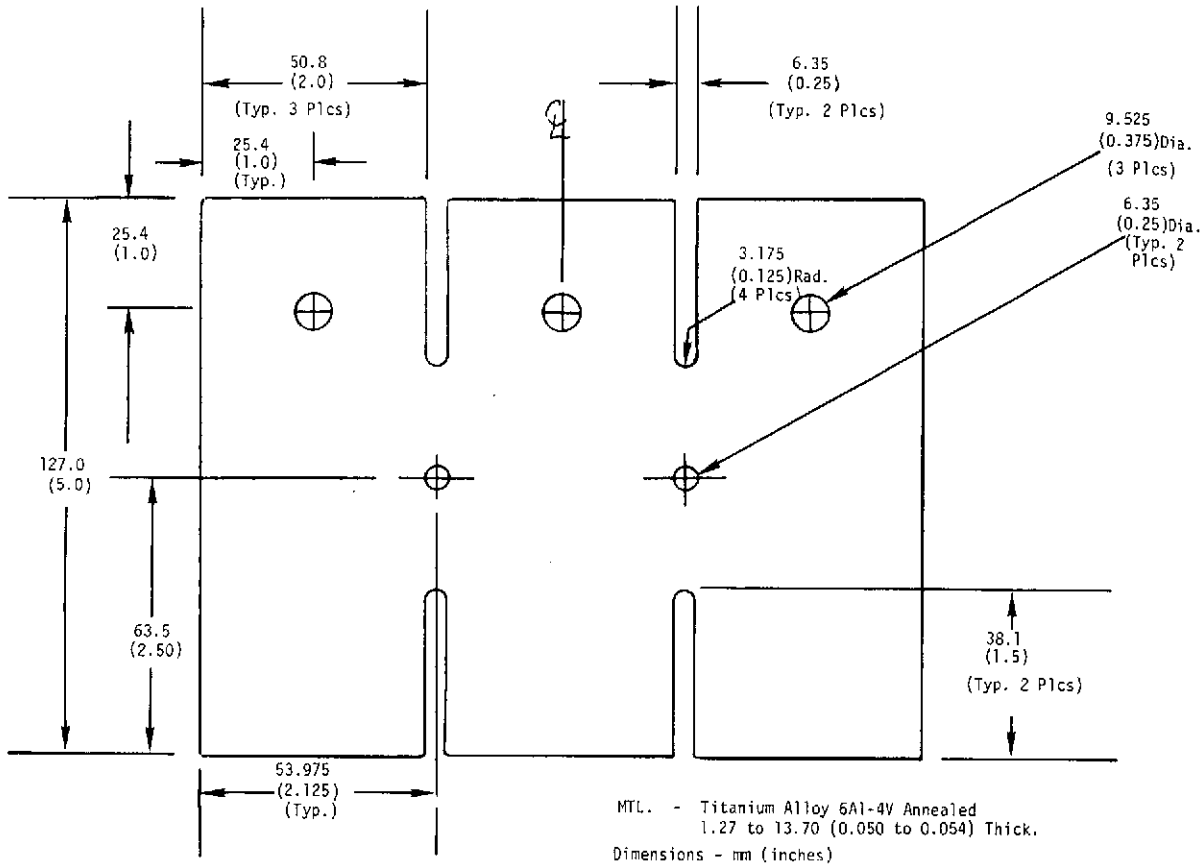


Figure 10. Weld Bond Shear Specimen Blank

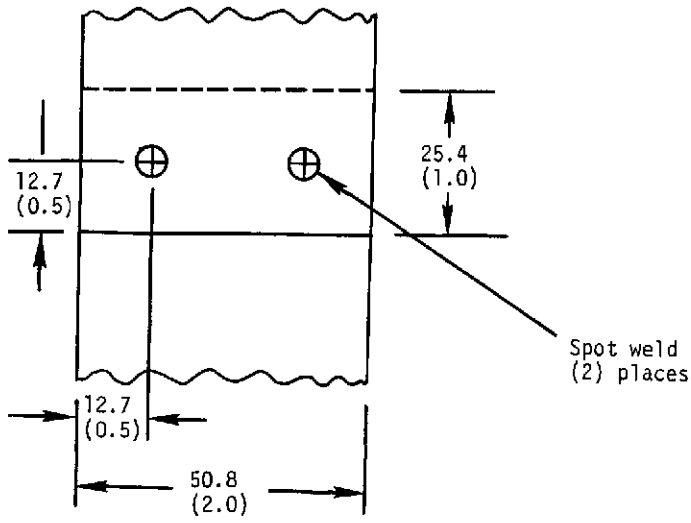


Figure 11. Weld Bond Lap-Shear Joint

These specimens were assembled, spot-welded (see Figure 11) and then oven cured by heating to 589 K (600°F) at 5.56 K (10°F) per minute followed by a 16-hour cure at 589 K (600°F). The welded-only specimens were prepared identically to the weld bonded specimens except the adhesive paste was omitted. Bonded-only panels were prepared using P4 primer and A5F adhesive film by press-bonding in accordance with Appendix D.4

(Reference 1), *i.e.*, 0.7 MN/m^2 (100 psig) pressure at 589 K (600°F) for 60 minutes cure plus 16 hours at 589 K (600°F) postcure.

Titanium alloy 6Al4V doublers were fabricated and spot-welded onto the ends of specimens in accordance with Figure 12. This was necessary because preliminary lap-shear tests at 219 K (-65°F) and 561 K (+550°F) resulted in elongation of the loading pin hole prior to joint failure.

3.1.2 Static Tests

Lap-shear test specimens, as described in 3.1.1, were tested in triplicate at room temperature using pin and clevis type test grips and a loading rate of 6227 N (1400 lbs) per minute. For the low temperature tests, specimen temperature was stabilized at 219°K (-65°F) by preconditioning the specimens for ten minutes in a test chamber cooled to 219 K (-65°F) by liquid nitrogen refrigeration. Similarly, specimen temperature was stabilized at 561 K (550°F) for elevated temperature tests by preconditioning the specimens for ten minutes in a test chamber heated to 561 K (550°F) with circulating air. Additional specimens were submitted to thermal aging and thermal cycling prior to performing the above tests.

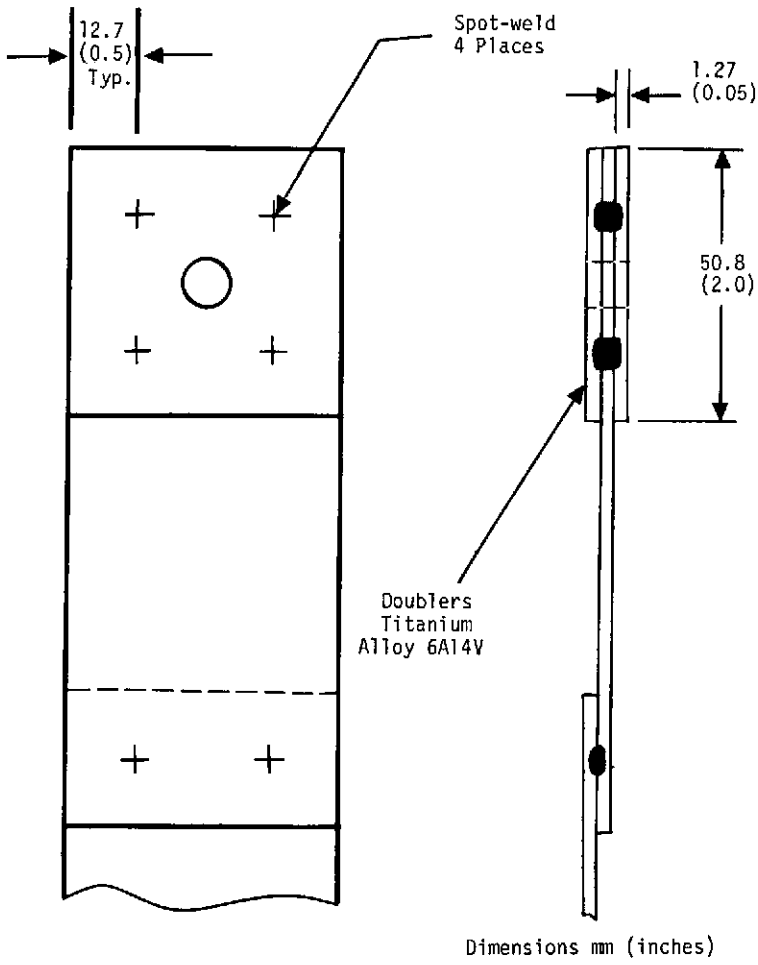


Figure 12. Doublers For Lap-Shear Test Specimens

Thermal cycling was performed by loading the specimens into a Conrad-Missimer test chamber cooled with refrigerated circulating air at 219 K (-65°F) for 30 minutes

after which they were removed and immediately loaded into a 561 K (550°F) air circulating oven with a horizontal air velocity of 127 cm/sec (250 feet/minute) and an air change rate of 0.19 m^3/sec (400 cubic feet/minute). After 30 minutes the specimens were removed and then recycled 100 times. Isothermal aging of specimens was performed in a similar 561 K (550°F) air circulating oven. Results of these tests are provided in Tables XV, XVI and XVII and examples of the observed failure modes are depicted in Figures 13 and 14.

TABLE XV.

STATIC TESTS AT 219 K (-65°F) FOR WELD-THROUGH PROCESS

Specimen Description	Specimen Preconditioning	Load at Failure, N (lbs)	Type of Failure [a]
Bonded Only	As Prepared	12632(2840)	Cohesive (3)
	500 hrs/561 K (550°F)	7295(1640)	Cohesive (3)
	1000 hrs/561 K (550°F)	3114(700)	Adhesive (3)
	Thermal Cycling	11031(2480)	Cohesive (3)
Welded Only	As Prepared	29134(6550)	Pulled Nuggets (3)
	500 hrs/561 K (550°F)	29268(6580)	Sheet Section Failure (3)
	1000 hrs/561 K (550°F)	27310(6140)	Pulled Nuggets (3)
	Thermal Cycling	33404(7510)	Pulled Nuggets (3)
Weld Bonded	As Prepared	28200(6340)	Pulled Nuggets (2) Sheet Section Failure (1)
	500 hrs/561 K (550°F)	31269(7030)	Pulled Nuggets (2) Sheet Section Failure (1)
	1000 hrs/561 K (550°F)	29045(6530)	Pulled Nuggets (3)
	Thermal Cycling	29624(6660)	Pulled Nuggets (3)

[a] See Figures 13 and 14 for illustration of failure modes, ( ) shows number of specimens.

TABLE XVI.

STATIC TESTS AT ROOM TEMPERATURE FOR WELD-THROUGH PROCESS

Specimen Description	Specimen Preconditioning	Load at Failure, N (lbs)	Type of Failure [a]
Bonded Only	As Prepared	10720(2410)	Cohesive (3)
	500 hrs/561 K (550°F)	-- --	
	1000 hrs/561 K (550°F)	3114(700)	Cohesive (3)
	Thermal Cycling	10942(2460)	Cohesive (3)
Welded Only	As Prepared	30469(6850)	Sheet Section Failure (3)
	500 hrs/561 K (550°F)	27355(6150)	Sheet Section Failure (1) Pulled Nuggets (2)
	1000 hrs/561 K (550°F)	28200(6340)	Sheet Section Failure (3)
	Thermal Cycling	31225(7020)	Pulled Nuggets (3)
Weld Bonded	As Prepared	26377(5930)	Pulled Nuggets (3)
	500 hrs/561 K (550°F)	32026(7200)	Pulled Nuggets (3)
	1000 hrs/561 K (550°F)	31492(7080)	Pulled Nuggets (3)
	Thermal Cycling	28779(6470)	Pulled Nuggets (3)

[a] See Figures 13 and 14 for illustration of failure modes, ( ) shows number of specimens.

TABLE XVII.

STATIC TESTS AT 561 K (550°F) FOR WELD-THROUGH PROCESS

Specimen Description	Specimen Preconditioning	Load at Failure N (lbs)	Type of Failure
Bonded Only	As Prepared	10008(2250)	Cohesive (3)
	500 hrs/561 K (550°F)	6094(1370)	Cohesive (3)
	1000 hrs/561 K (550°F)	2758(620)	Cohesive (2) Adhesive (1)
	Thermal Cycling	17436(3920)	Cohesive (3)
Welded Only	As Prepared	31047(6980)	Pulled Nuggets (3)
	500 hrs/561 K (550°F)	31403(7060)	Sheet Section Failure (3)
	1000 hrs/561 K (550°F)	27756(6240)	Pulled Nuggets (3)
	Thermal Cycling	31714(7130)	Pulled Nuggets (3)
Weld Bonded	As Prepared	32470(7300)	Pulled Nuggets (3)
	500 hrs/561 K (550°F)	29401(6610)	Sheet Section Failure (2) Pulled Nuggets (1)
	1000 hrs/561 K (550°F)	32292(7260)	Pulled Nuggets (3)
	Thermal Cycling	30513(6860)	Pulled Nuggets (3)

a) See Figures 13 and 14 for illustration of failure modes, ( ) shows number of specimens.

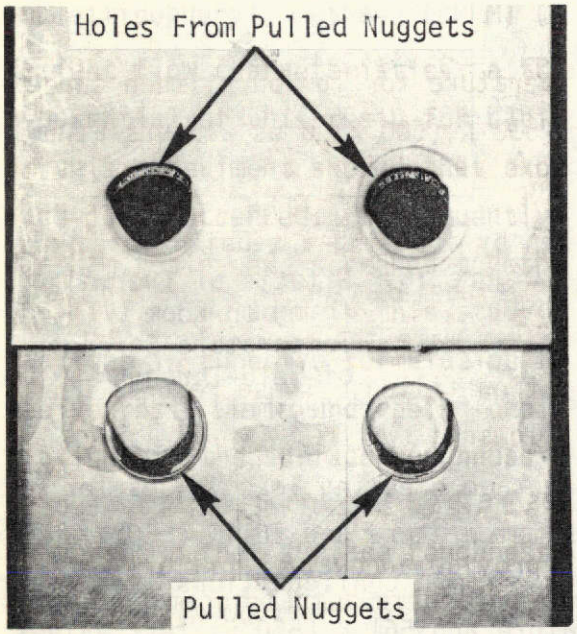


Figure 13. Pulled Nuggets Failure

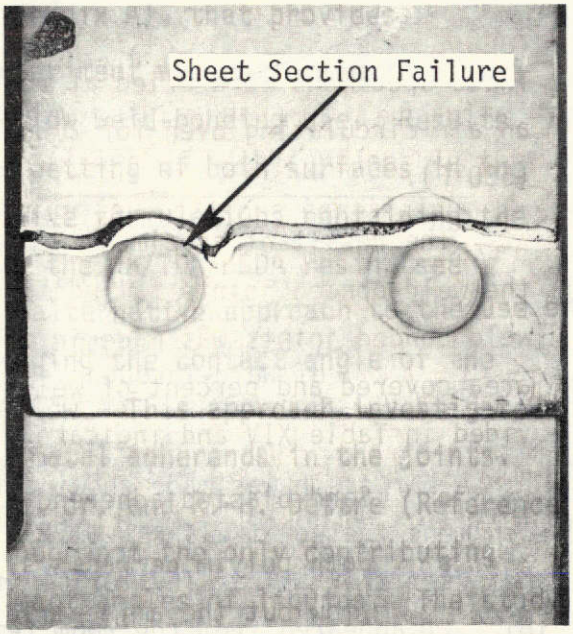


Figure 14. Sheet Section Failure

The average breaking loads at 21 K (-65°F), R.T., and 561 K (+550°F) of the three specimens of each configuration were plotted as a function of aging duration (see Figures 15, 16 and 17). There appeared to be no significant differences between the plot for the welded and weld bonded specimens and neither indicated any property degradation trends except that the values were fifteen percent lower for the 561 K (550°F) tests. As expected, the bonded-only specimens plot defined a significant strength degradation trend which was not reflected in the weld bonded specimen plot.

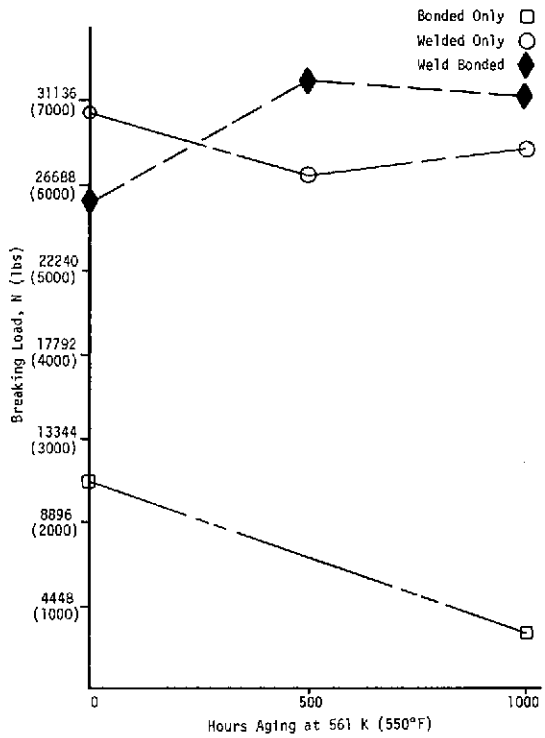


Figure 15. Effects of Thermal Aging on 219 K (-65°F) Strength of Weld-Through Specimens

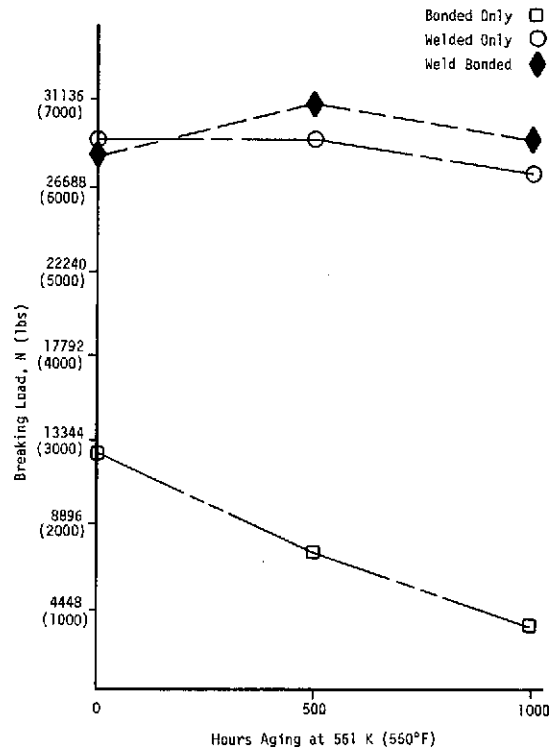


Figure 16. Effects of Thermal Aging on Room Temperature Strength of Weld-Through Specimens

A bar graph was made showing the average breaking load values for specimens tested at 219 K (-65°F), room temperature and 561 K (550°F) before and after thermal cycling for all three specimen configurations (see Figure 18). It appeared from the room temperature and 561 K (550°F) test values for the bonded-only specimens that thermal cycling had degraded the breaking-load values by approximately twenty percent. However, the welded-only and weld bonded specimen plots indicated opposite trends, *i.e.*, the breaking loads increased by approximately eight percent after thermal cycling.

Breaking load *vs* test temperature plots were made for specimens of all three configurations after being subjected to thermal cycling. These plots indicated that the breaking load values for the welded-only and weld bonded specimens were lower than the room temperature values at both 219 K (-65°F) and 561 K (+550°F). A plot of strengths for "as-fabricated" specimens (see Figure 19) showed higher values at 219 K (-65°F) for the welded-only specimens. Lower values at 219 K (-65°F) and higher values at 561 K (+550°F) were shown for the weld-bonded specimens. The plot for the bonded-only specimens showed a lower strength only at 561 K (550°F) although a plot of strengths for "as-fabricated" specimens also showed lower values at 219 K (-65°F) (see Figure 20).

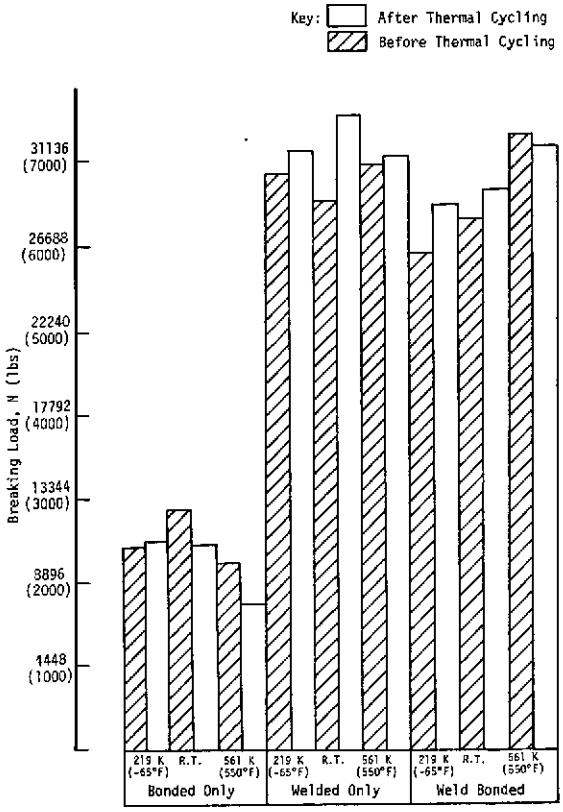


Figure 18. Effects of Thermal Cycling on Strength of Weld-Through Specimens

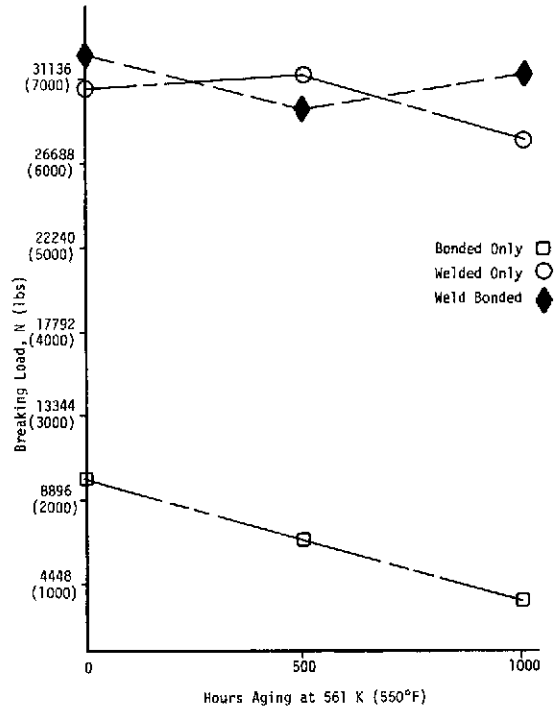


Figure 17. Effects of Thermal Aging on 561 K (550°F) Strength of Weld-Through Specimens

3.1.3 Fatigue Tests

Lap-shear test panels of the same configuration used for the static tests were prepared for fatigue tests. The fatigue tests were performed on a Sonntag fixed frequency, fixed waveform, variable load fatigue tester (see Figures 21 and 22) in accordance with the test schedule shown in Table XVIII. All of the specimens were loaded in clevis-pin grips

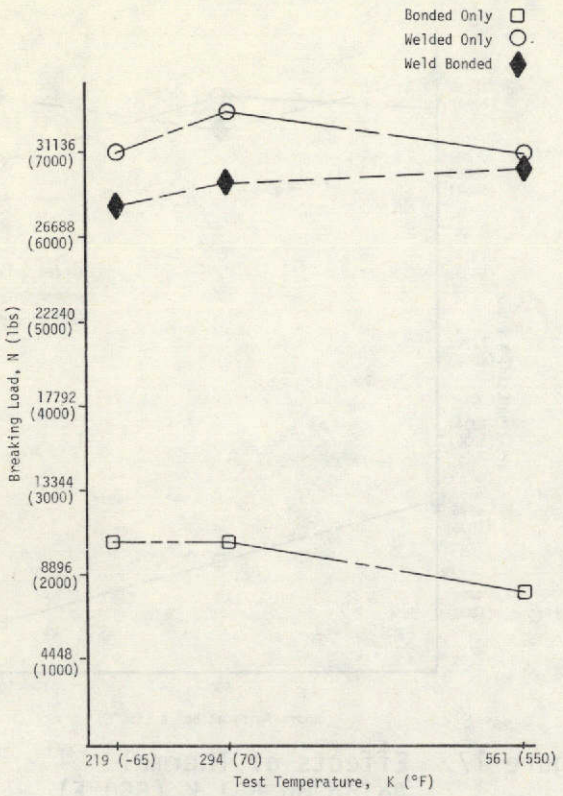


Figure 19. Effects of Test Temperature on Strength of Thermally Cycled Weld-Through Specimens

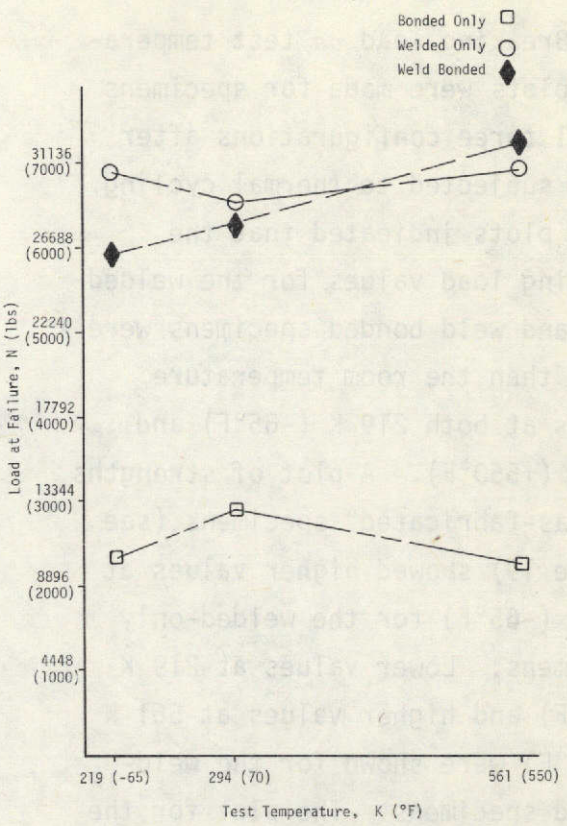


Figure 20. Effects of Test Temperature on Strength of "As-Fabricated" Weld-Through Specimens

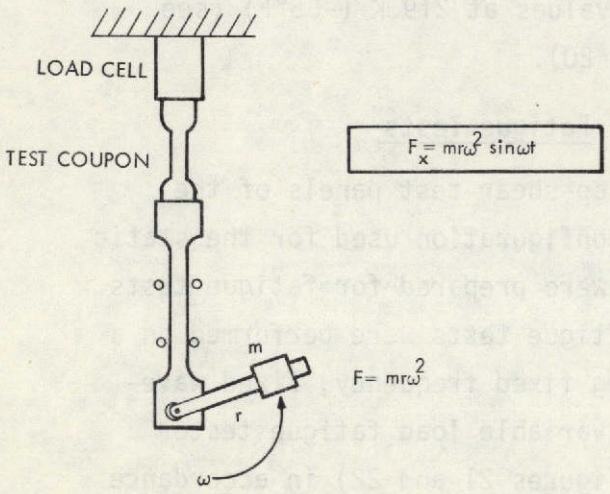


Figure 21. Schematic of Fixed Frequency, Fixed Waveform, Variable Load Fatigue Tester

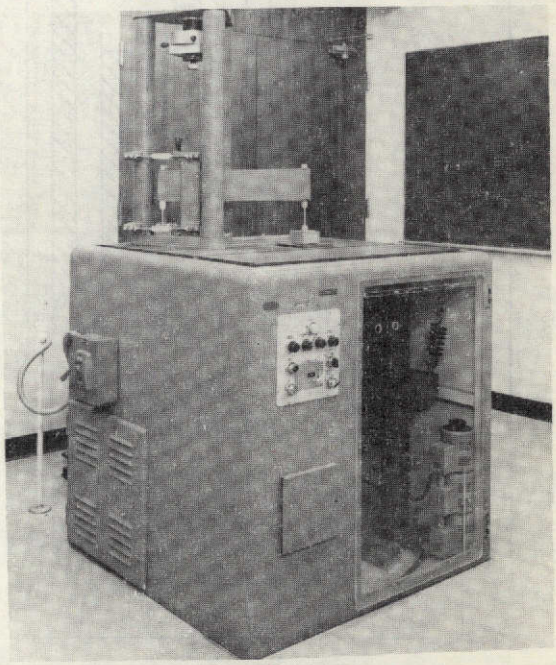


Figure 22. Fixed Frequency, Fixed Waveform, Variable Load Fatigue Tester

(see Figure 23) and cyclic loaded at 1800 cycles per minute (30 Hz) at a load ratio (R) of 0.1.

TABLE XVIII.

FATIGUE TEST SCHEDULE

Load Level, [a] %	Type of Specimen		
	Bonded-Only	Welded-Only	Weld-Bonded
60	3[b]	3	3
40	3	3	3
20	3	3	3

[a] Percent of failure load at room temperatures from static tests.

[b] Number of specimens at each test condition.

Results from these tests are provided in Table XIX and Figure 24 which indicate a significant improvement in fatigue strength of the weld bonded specimens over the weld-only specimens. The bonded-only specimens as plotted (percent failure load *vs* cycles to failure) appear better than the weld bonded specimens. However, when actual loads are compared (see Figure 25) it is obvious that the weld bonded specimens possess higher actual fatigue strength than either the welded or bonded-only specimens.

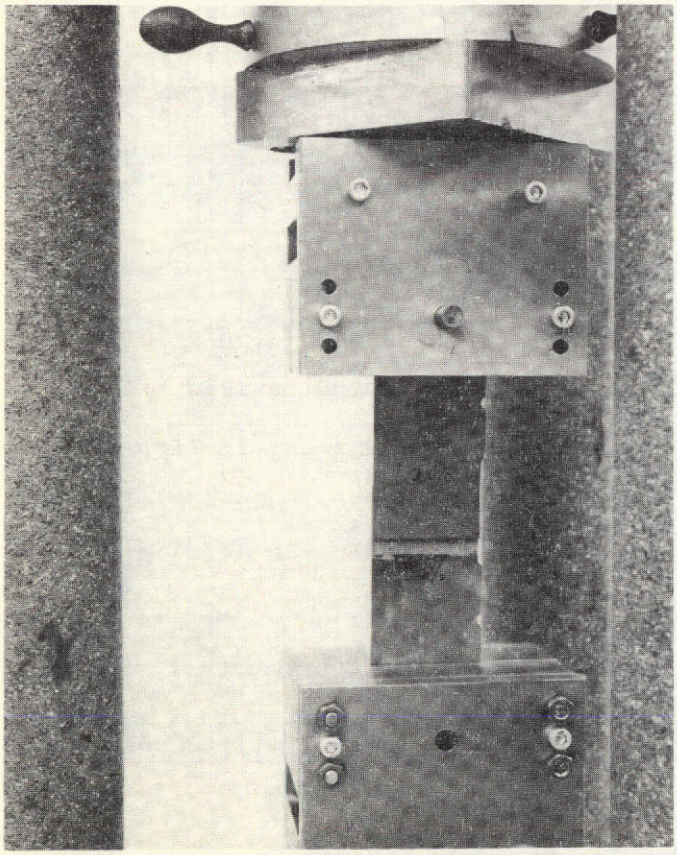


Figure 23. Lap Specimen and Fatigue Grips in Tensile Fatigue Machine

3.2 EVALUATION OF CAPILLARY-FLOW PROCESS

A similar evaluation was performed of titanium alloy lap-shear test specimens prepared by the capillary-flow weld bonding process to that performed for the weld-through weld bonding process. As discussed in Section 2.2, the BFBI/BMPM resin system was used as an adhesive although it must be remembered that this resin is not a developed adhesive system. Consequently, as anticipated, breaking load values of bonded-only specimens evaluated during this effort were lower than those obtained with the P4/A5F adhesive system. However, the values obtained were higher than those obtained originally with the P13N or P11BA resin systems (Reference 3).

TABLE XIX.

FATIGUE TEST RESULTS FOR WELD-THROUGH PROCESS

Load Level,[a] %	Cycles At Failure (X 10^3)		
	Welded-Only	Bonded-Only	Weld-Bonded
60	1.0 1.8 1.3	1.0 11.0 0.5	5.5 2.5 3.0
40	5.5 6.0 5.0	60.0 81.0 723.0	14.0 14.0 16.0
20	30.0 33.0 31.0	(b)	227.0 216.0 186.0

a) Percent of failure load at room temperature from static tests.

b) Run-out, *i.e.*, no failure before completion of 1 million cycles.

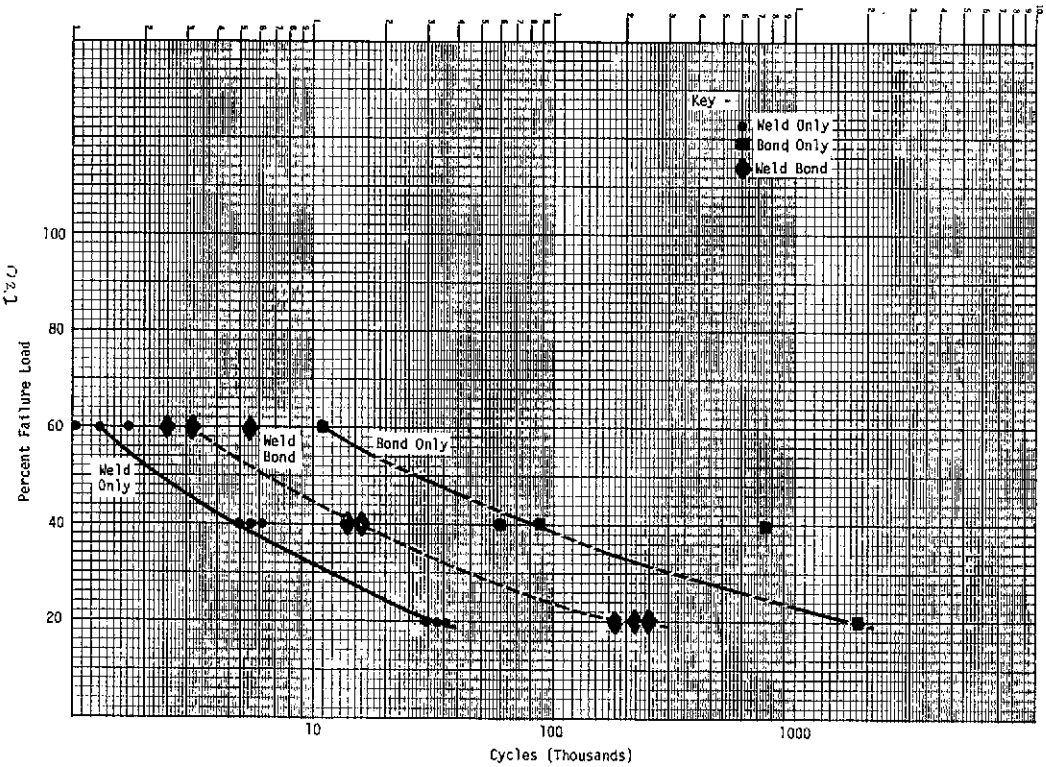


Figure 24. Fatigue Test Results For Weld-Through Specimens

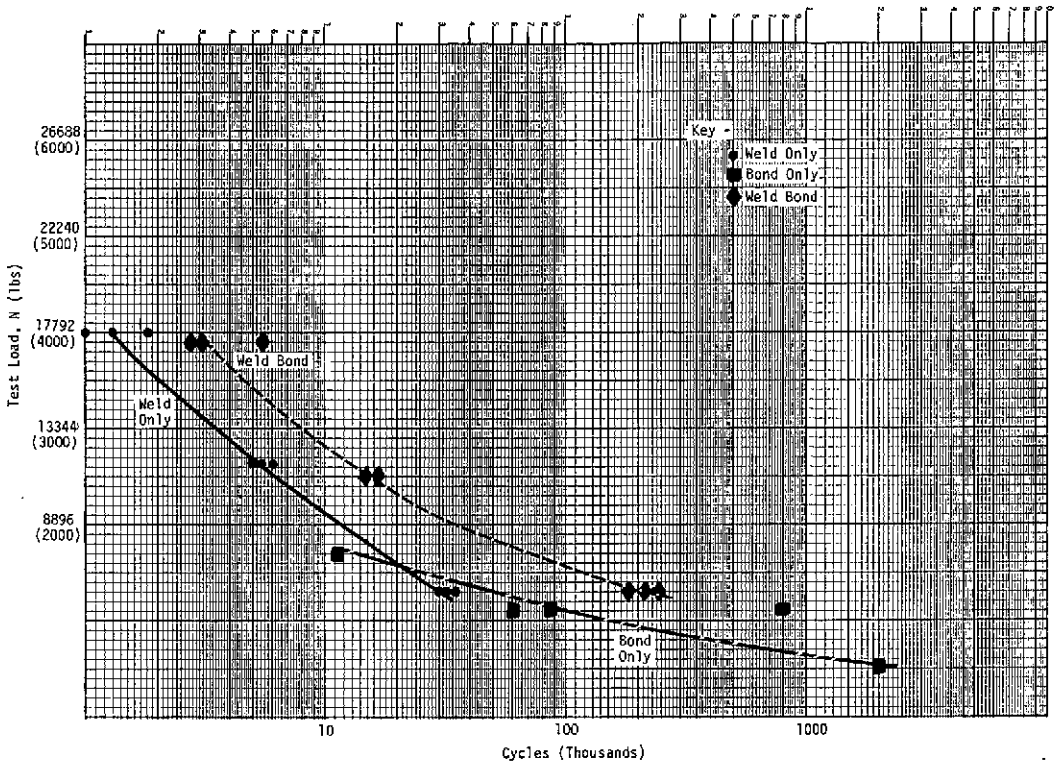


Figure 25. Comparison of Fatigue Test Results For Weld-Through Specimens

3.2.1 Fabrication Procedure

The titanium alloy 6Al4V faying surfaces of the lap-shear finger test panels (see Figure 10) were prepared by the same surface treatment procedure as described in Section 3.1.1 for the welded-only specimens. The specimens were spot-welded by the same weld schedule (see Section 3.1.1) within four hours and a bead of BFBI/BMPM resin paste (30% w/w DMF) was applied. The specimens were air dried for 16 hours at room temperature then cured for two hours at 533 K (500°F) plus 2 hours at 561 K (550°F).

Bonded-only specimens were prepared by treating the adherend surfaces by the same procedure as above and then coating them with a BFBI/BMPM adhesive paste. This paste contained aluminum powder (150 parts by weight per hundred of resin) to control the resin flow during bonding. The adhesive coating was air dried for 30 minutes at room temperature plus 5 minutes at 389 K (240°F). After drying, the faying surfaces were mated and assembled in a bonding jig. This assembly was placed into a cold press and 0.7 MN/m^2 (100 psig) pressure was applied. The press platen temperature was raised to 477 K (400°F) at 5.56 K (10°F)/minute and the adhesive was cured for two hours at 477 K (400°F).

Pressure was released after cooling the assembly to <422 K (300°F) and the bonded joints then were postcured in an air circulating oven:

30 minutes/450 K (350°F)
15 minutes/477 K (400°F)
15 minutes/505 K (450°F)
15 minutes/533 K (500°F)
2 hours/561 K (550°F)
4 hours/589 K (600°F)

3.2.2 Static Tests

Identical static tests were performed on specimens prepared by the capillary-flow weld bonding process as were performed on those prepared by the weld-through weld bonding process (see Section 3.1.2). The static lap-shear breaking loads of all of the adhesive bonded specimens (see Tables XX, XXI and XXII) were significantly lower than those of the P4/A5F system (see Tables XV, XVI and XVII). However, this was expected because the BFBI/BMPM resin used for bonding these specimens has not been developed as an adhesive resin. It is expected that further development work directed towards obtaining an adhesive resin similar to the BFBI/BMPM resin, *i.e.*, another poly(Diels Alder) (PDA) resin, plus adhesive compounding studies could provide an equivalent adhesive system to the P4/A5F adhesive system.

TABLE XX.
STATIC TESTS AT 219 K (-65°F) FOR CAPILLARY-FLOW PROCESS

TABLE XX.
STATIC TESTS AT 219 K (-65°F) FOR CAPILLARY-FLOW PROCESS

Specimen Description	Specimen Preconditioning	Load at Failure, N (lbs)	Type of[a] Failure
Bonded Only	As Prepared	8642 (1943)	Cohesive (3)
	500 hrs/561 K (550°F)	5738 (1290)	Cohesive (3)
	1000 hrs/561 K (550°F)	[b]	
	Thermal Cycling	467 (105)	Cohesive (3)
Welded Only	As Prepared	30469 (6850)	Sheet Section Failure (3)
	500 hrs/561 K (550°F)	27355 (6150)	Sheet Section Failure (1) Pulled Nuggets (2)
	1000 hrs/561 K (550°F)	28200 (6340)	Sheet Section Failure (3)
	Thermal Cycling	31225 (7020)	Pulled Nuggets (3)
Weld Bonded	As Prepared	29103 (6543)	Pulled Nuggets (3)
	500 hrs/561 K (550°F)	30638 (6888)	Pulled Nuggets (3)
	1000 hrs/561 K (550°F)	28067 (6310)	Pulled Nuggets (3)
	Thermal Cycling	29806 (6701)	Pulled Nuggets (3)

[a] See Figures 26 and 27 for illustration of failure modes, ( ) shows number of specimens.
b) Not tested.

TABLE XXI.

STATIC TESTS AT ROOM TEMPERATURE FOR CAPILLARY-FLOW PROCESS

Specimen Description	Specimen Preconditioning	Load at Failure, N (lbs)	Type of[a] Failure
Bonded Only	As Prepared	9786 (2200)	Cohesive (3)
	500 hrs/561 K (550°F)	4715 (1060)	Cohesive (3)
	1000 hrs/561 K (550°F)	[b]	
	Thermal Cycling	8647 (1944)	Cohesive (3)
Welded Only	As Prepared	29134 (6550)	Pulled Nuggets (3)
	500 hrs/561 K (550°F)	29268 (6580)	Sheet Section Failure(3)
	1000 hrs/561 K (550°F)	27310 (6140)	Pulled Nuggets (3)
	Thermal Cycling	33404 (7510)	Pulled Nuggets (3)
Weld Bonded	As Prepared	32439 (7293)	Pulled Nuggets (3)
	500 hrs/561 K (550°F)	28080 (6313)	Pulled Nuggets (3)
	1000 hrs/561 K (550°F)	29223 (6570)	Pulled Nuggets (3)
	Thermal Cycling	29370 (6603)	Pulled Nuggets (3)

[a] See Figures 26 and 27 for illustration of failure modes, ( ) shows number of specimens.
b) Not tested.

TABLE XXII.

STATIC TESTS AT 561 K (550°F) FOR CAPILLARY-FLOW PROCESS

Specimen Description	Specimen Preconditioning	Load at Failure N (lbs)	Type of [a] Failure
Bonded Only	As Prepared	7753	Cohesive (3)
	500 hrs/561°K (550°F)	5115 (1150)	Cohesive (3)
	1000 hrs/561°K (550°F)	6005 (1350)	Cohesive (3)
	Thermal Cycling	6298 (1416)	Cohesive (3)
Welded Only	As Prepared	31047 (6980)	Pulled Nuggets (3)
	500 hrs/561°K (550°F)	31403 (7060)	Sheet Section Failure (3)
	1000 hrs/561°K (550°F)	27756 (6240)	Pulled Nuggets (3)
	Thermal Cycling	31714 (7130)	Pulled Nuggets (3)
Weld Bonded	As Prepared	31447 (7070)	Pulled Nuggets (3)
	500 hrs/561°K (550°F)	26065 (5860)	Pulled Nuggets (3)
	1000 hrs/561°K (550°F)	27244 (6125)	Pulled Nuggets (3)
	Thermal Cycling	26746 (6013)	Pulled Nuggets (3)

[a] See Figures 26 and 27 for illustration of failure modes, ( ) shows number of specimens.

The static tests performed on the capillary-flow weld bonded specimens (see Tables XX, XXI and XXII) did not show any significant difference in breaking loads for those obtained with specimens bonded by the weld-through process (see Tables XV, XVI and XVII). However, this also was expected because the spot-welds carry most of the static loads in these lap joints.

3.2.3 Fatigue Tests

Fatigue tests were performed on lap-shear test specimens prepared by the capillary-flow process in exactly the same manner as were performed on those prepared by the weld-through process (see Section 3.1.3). Evaluation of the weld bonded specimens provided cycles to failure values (see Table XXIII) lower than those obtained with the specimens prepared by the weld-through weld bonding process (see Table XIX). Similarly, the values obtained for the bonded-only specimens also were lower than those obtained with the P4/A5F adhesive system. Plots were made depicting percent of ultimate breaking load *vs* cycles to failure (see Figure 26) and test load *vs* cycles to failure (see Figure 27). As discussed in Section 3.2.2, it is expected that adhesive development work with the BFBI/BMPM or similar resin system would provide equivalent values to those obtained with the P4/A5F adhesive system used for the weld-through weld bonding process.

TABLE XXIII.

FATIGUE TEST RESULTS FOR CAPILLARY-FLOW PROCESS

Load Level,[a] %	Cycles at Failure (X 10^3)		
	Welded-Only	Bonded-Only	Weld-Bonded
60	1.0 1.8 1.3	108 80 3	2.0 2.0 2.0
40	5.5 6.0 5.0	95 426 178	6.0 6.0 7.0
20	30.0 33.0 31.0	(b) (b) (b)	30.5 30.0 40.0

[a] Percent of failure load at room temperature from static tests.

[b] Run-out, *i.e.*, no failure before completion of 1 million cycles.

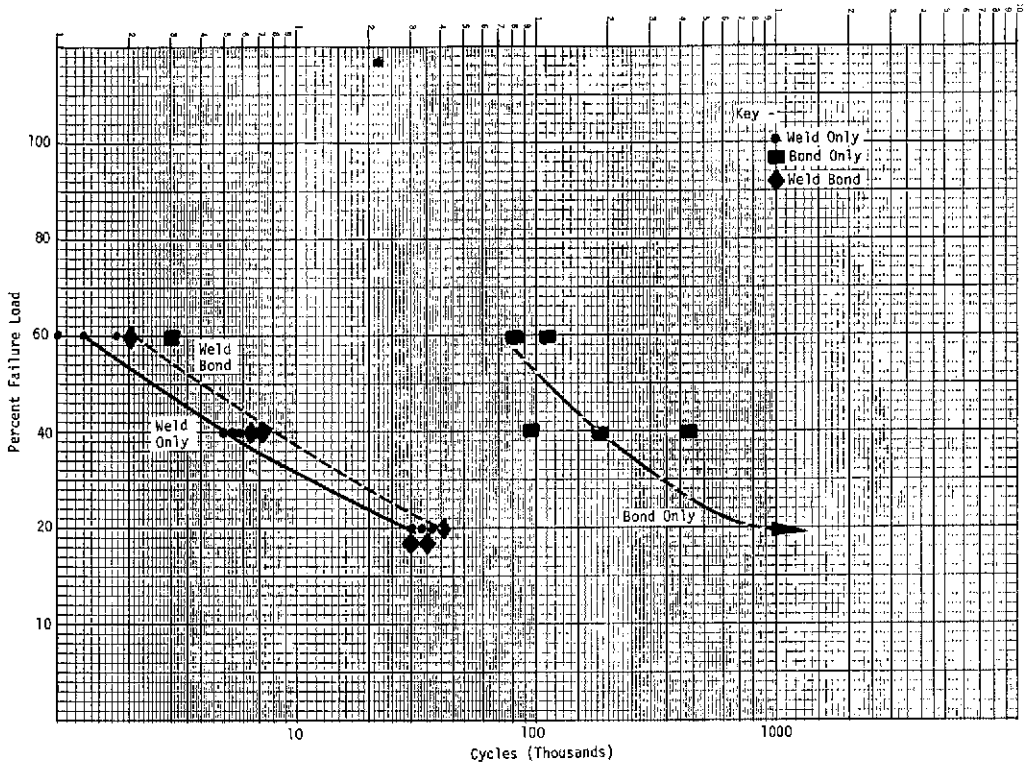


Figure 26. Fatigue Test Results For Capillary-Flow Specimens

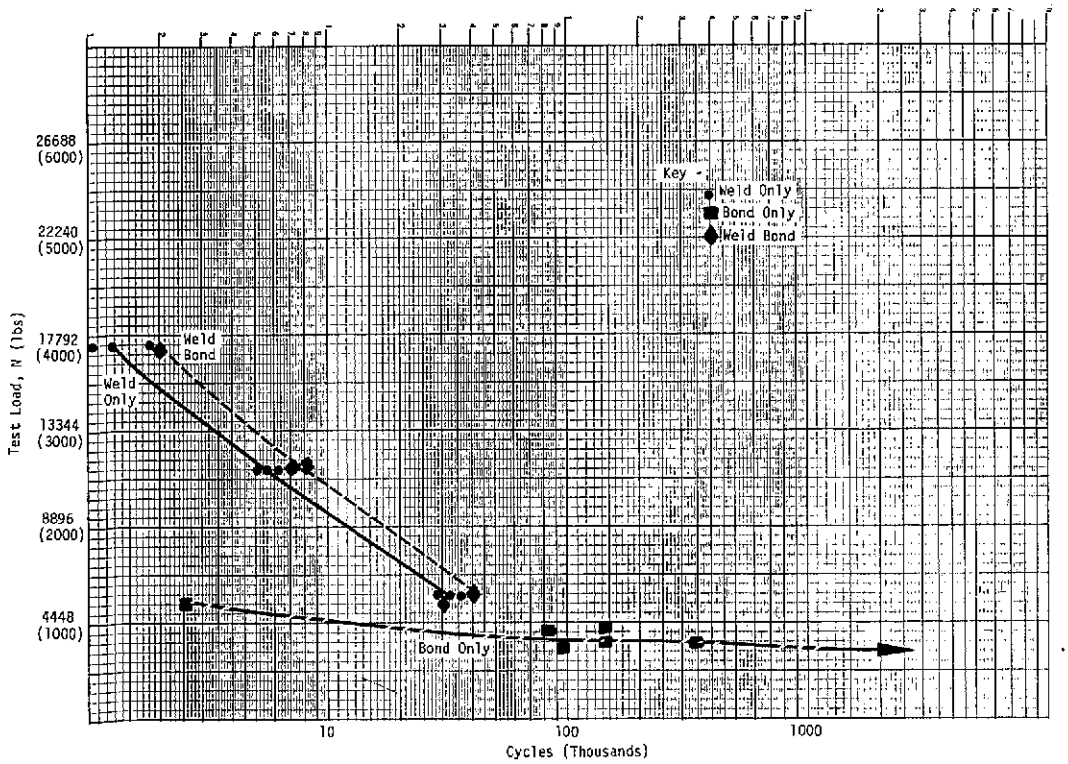


Figure 27. Comparison of Fatigue Test Loads For Capillary-Flow Specimens

4. STRUCTURAL PANEL FABRICATION AND EVALUATION

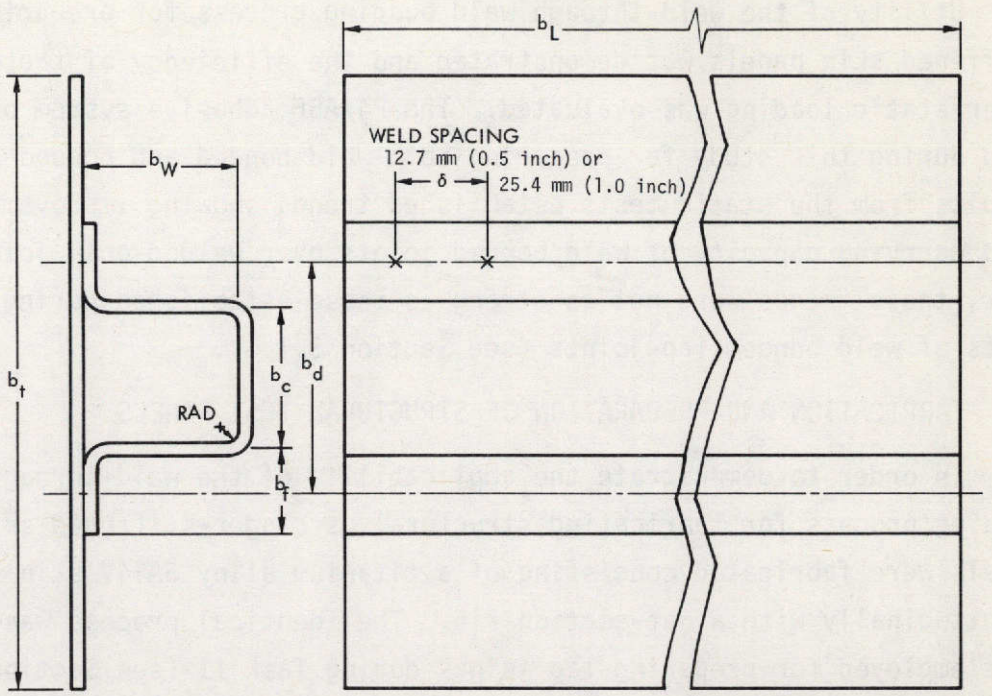
Utility of the weld-through weld bonding process for preparing stringer stiffened skin panels was demonstrated and the efficiency of these joints under static loading was evaluated. The P4/A5F adhesive system only was used during this study for preparing both weld bonded and bonded-only joints. Results from the static tests established trends showing improvement in the load carrying capacity of weld bonded joints over welded-only joints. However, these trends were not as strong as those established during the fatigue tests of weld bonded lap-joints (see Section 3.1.3).

4.1 FABRICATION AND PREPARATION OF STRUCTURAL TEST PANELS

In order to demonstrate the applicability of the weld-through weld bonding process for fabricating structural, stringer-stiffened skins, test panels were fabricated consisting of a titanium alloy 6Al4V skin stiffened longitudinally with a hat-section rib. The identical process was used to that employed for preparing lap joints during Task II (see Section 3.1). Welded and bonded-only panels also were prepared in order to provide comparative data. Spot-weld pitches for both welded-only and weld bonded panels were varied to demonstrate whether this affected the efficiency of the adhesive in the weld bonded joint. Strain gauges then were installed on the completed test panels in preparation for the structural panel tests.

4.1.1 Fabrication of Panels

Components for the structural test panels (see Figure 28) were fabricated from titanium alloy 6Al4V sheet. These components were prepared for welding and bonding by the Pasa-Jel process described in Section 3.1.1. Adhesive paste then was applied using the same masking procedure as used for the lap-shear test specimens (see Section 3.1.1). A photograph of the adhesive coated components for the 25.4 mm (1 inch) pitch weld bonded specimens is provided in Figure 29. The components then were spot-welded using a weld jig in accordance with Figure 30. Equal quantities of panels were welded using 12.7 mm (0.5 inch) and 25.4 mm (1.0 inch) weld spacings. Bonded-only structural test panels were prepared using P4 adhesive primer and A5F adhesive film by the process described in Appendix A. All test panels were bonded in a single vacuum bag as one autoclave load.



Panel Dimensions, mm (inches)

b_t-120.65(4.75)	b_c-25.4(1.00)
b_d-44.45(1.75)	b_L-254.0(10.00)
b_w-31.75(1.25)	RAD-0.52(0.20)
b_f- 1.59(0.62)	

Figure 28. Structural Test Panel

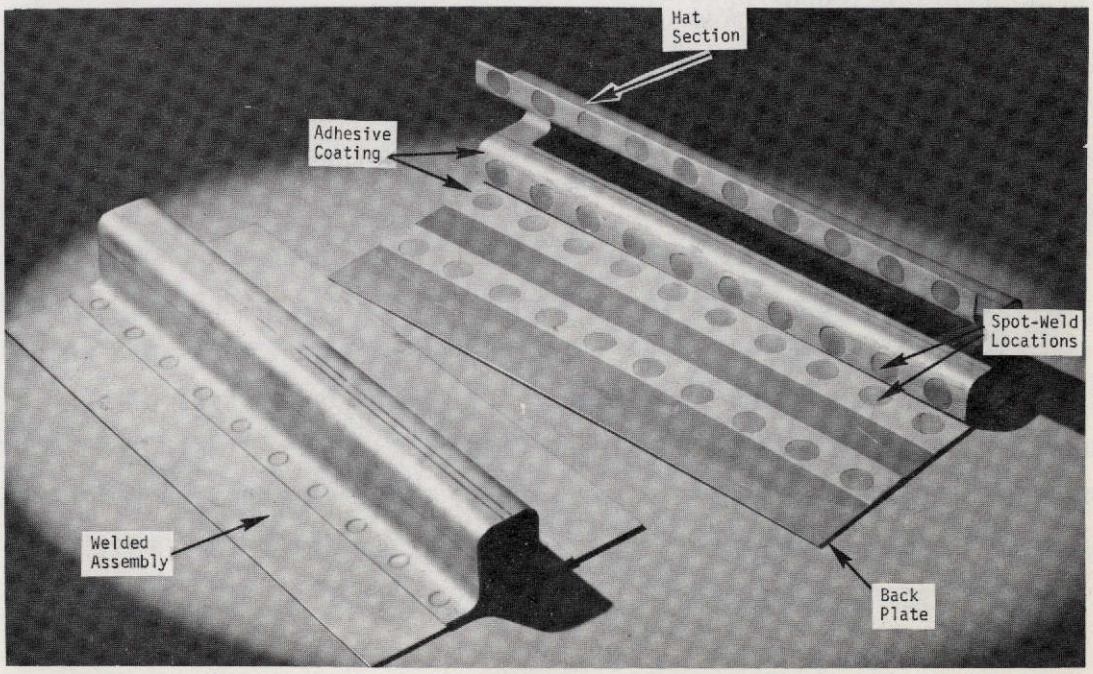


Figure 29. Structure Test Panel Details

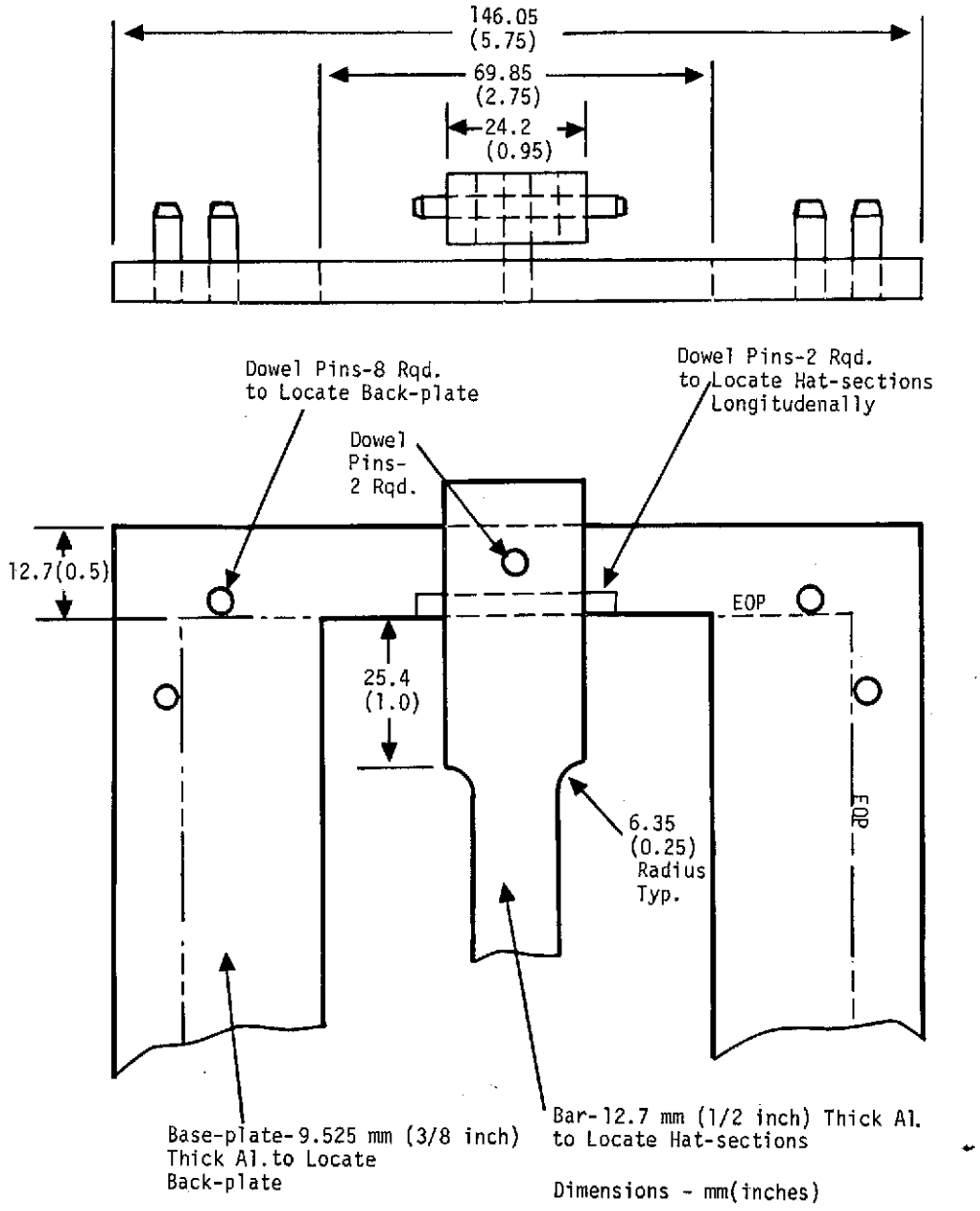
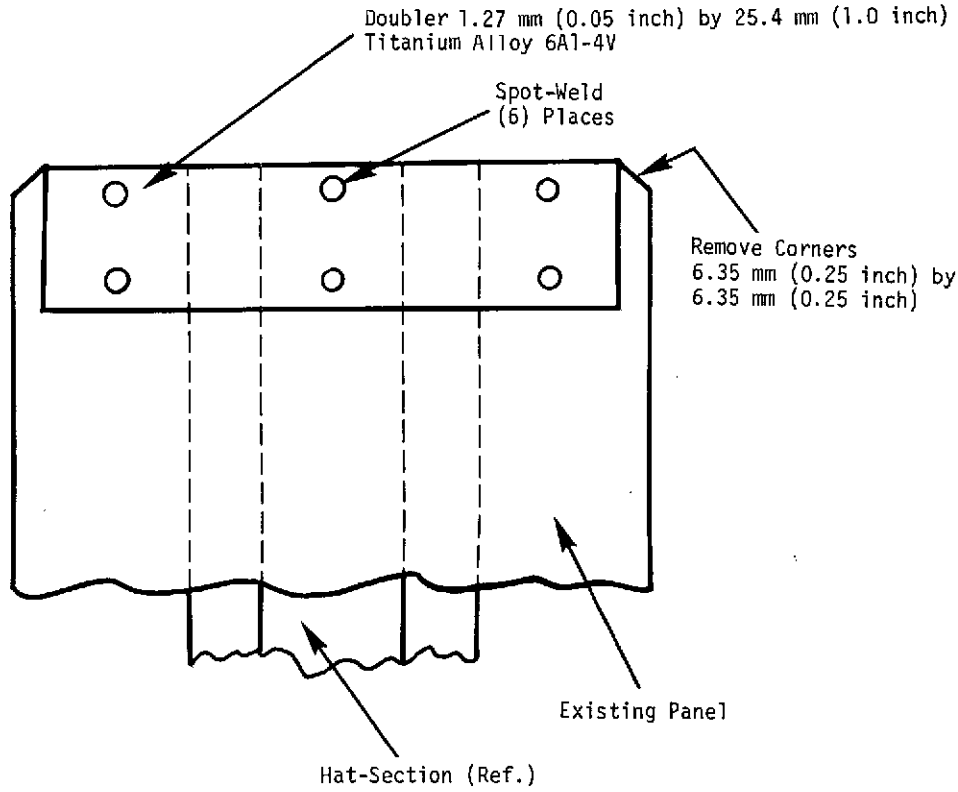


Figure 30. Welding Jig For Structural Test Panels

4.1.2 Preparation of Panels

The original panel configuration did not have a doubler along the top and bottom loaded edges. A preliminary test of a panel produced premature crippling along its upper edge. Consequently, the remaining test panels were reworked and localized doublers were attached along the top and bottom edges as shown in Figure 31. The edge was reground to ensure a flat and parallel loading surface.



NOTE: Typical Both Ends

Figure 31. Structural Test Panel Reinforcement

Table XXIV indicates the type of panels, number of panels and test environment used in the test program. Procedures for thermal cycling were identical to those employed for the lap-shear test specimens (see Section 3.1.2). Those panels that were subjected to a thermal cycling, were instrumented after the thermal cycling.

Five strain gauges were installed on each panel in accordance with Figure 32. The titanium alloy surfaces were prepared for bonding by the Pasa-Jel treatment (see Appendix A). The strain gauges used were Micro-Measurements WK-05-125BT-350 which were nickel-chromium alloy on epoxy-phenolic glass fabric backing and self-temperature compensating. They were bonded onto the test panels using M-Bond 600 adhesive together with CTF-60D Bondable Printed Circuit Terminals. All solder joints were made with 570-28R high lead content, resin flux core solder. The strain gauge installation then

was coated with Budd GW-1 coating and subjected to a two-stage cure process: 394 K (250°F) for 2 hours plus 533 K (500°F) for 2 hours. For those panels that were tested at elevated temperature, the strain gauges also were protected with Bean Gagekote #1 plastic coating.

TABLE XXIV.
STRUCTURAL PANEL TEST SCHEDULE

Type of Panel	Test Condition		
	294 K(70°F)	294 K(70°F) After Thermal Cycling a)	561 K(550°F)
Welded Only b)	4	4	4
Adhesive Bonded	2	2	2
Weld Bonded b)	4	4	4

a) 100 Cycles of 30 minutes at 219 K (-65°F) and 561 K (550°F).
b) Two tests each 12.7mm (0.5 inch) and 25.4 mm (1.0 inch) weld-pitch.

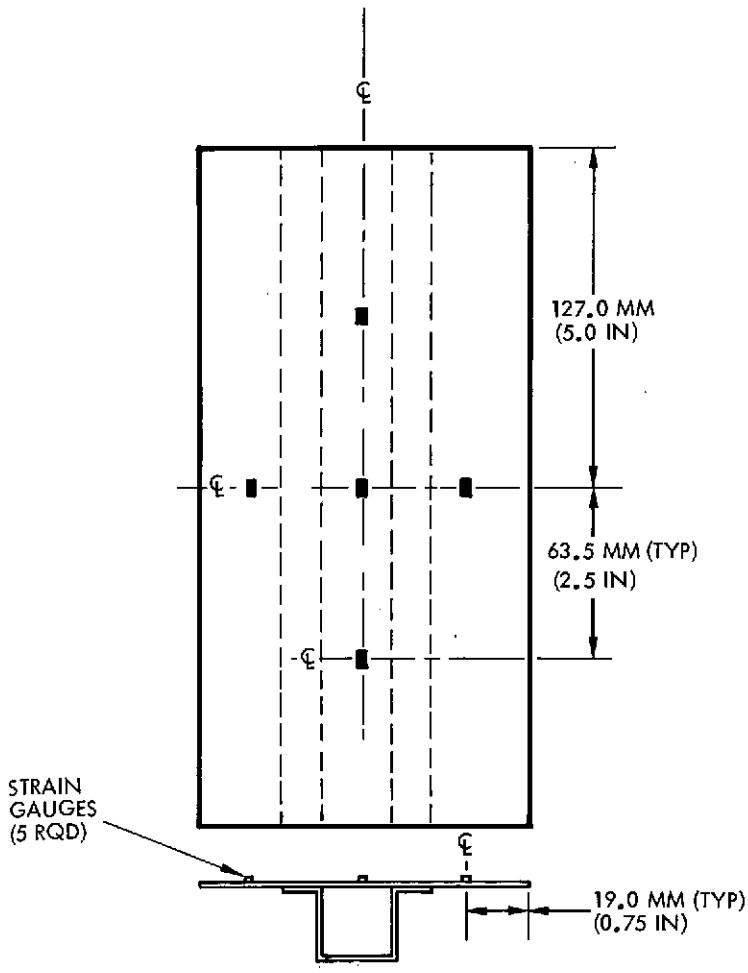


Figure 32. Strain Gauge Location For Structural Test Panels

Three thermocouples (T/C) were used to monitor the panel temperatures for the elevated temperature tests. They were located as illustrated in Figure 33. A fourth thermocouple (#4) was located near T/C #2 and used to control the heat input to the panel. Chromel-Alumel, 28 gauge thermocouples were spot-welded to the test specimen and coated with Omega CC High Temperature Cement.

4.2 TEST DESCRIPTION AND EVALUATION OF RESULTS

Structural panel tests were performed in order to determine whether weld bonding

improved the static structural performance of joints over welded-only joints. The stringer stiffened flat test panels (see Section 4.1) were tested statically in compression. Both room temperature and elevated temperature tests were performed on these panels.

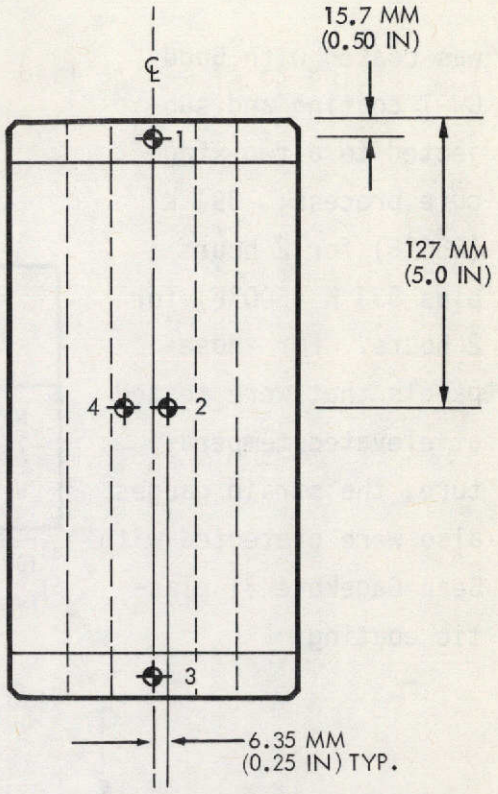


Figure 33. Thermocouple Location For Structural Test Panels

4.2.1 Test Procedures and Set-up

Figure 34 shows an overall view of the room temperature test set-up. Compression tests were conducted with a Baldwin Tate-Emery hydraulic testing machine, whose full load capacity is 533,784 N (120,000 lbs). Ultimate loads during this test program were in the range of 88,964 N (20,000 lbs) to 222,410 N (50,000 lbs). Load *vs* strain and load *vs* end-shortening (or end deflection) were monitored on a bank of six Moseley *xy* recorders. End-shortening was measured with a G. L. Collins linear velocity displacement transducer (LVDT) located between the heads of the loading machine.

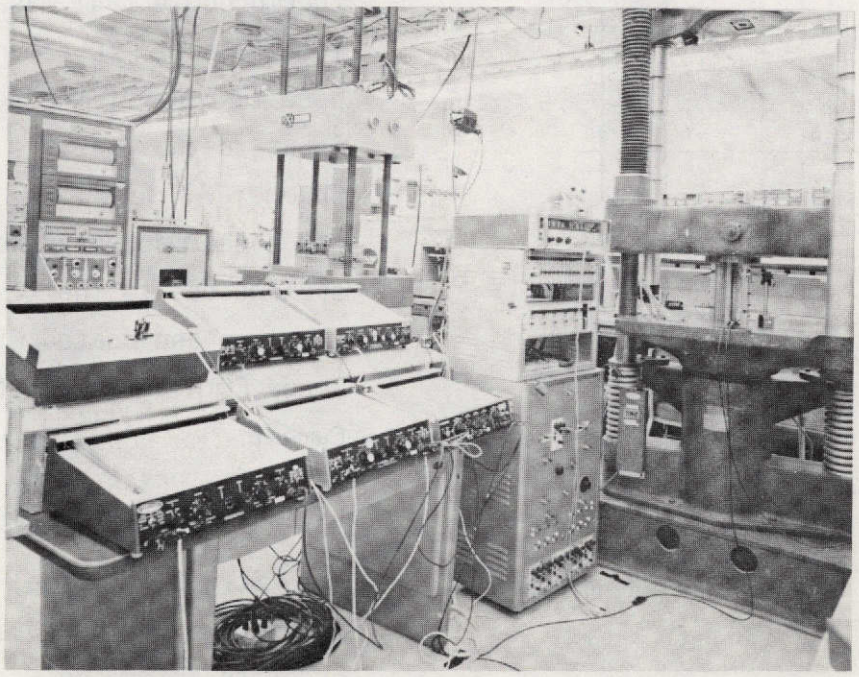


Figure 34. Structural Panel Test Facility (Room Temperature Test Panel Shown)

Figure 35 shows a detailed view of the test specimen positioned on the loading machine, along with the strain gauges, LVDT and edge supports. A hemispherical swivel head was used

for loading to eliminate load concentrations due to any deviation in parallelism between the loaded ends. The longitudinal edge supports are shown in Figure 36. They were steel construction and essentially provided a knife-edge support along the length of the panel. Thus, only longitudinal rotation is permitted along the unloaded edges of the panel. The clamps were used to help ensure that the edge support mechanism did not pry open when local or general panel buckling occurred. No longitudinal load was applied to the edge supports because of the clearance between the face of the swivel loading head and the top end of the edge support mechanism as shown in Figure 37.

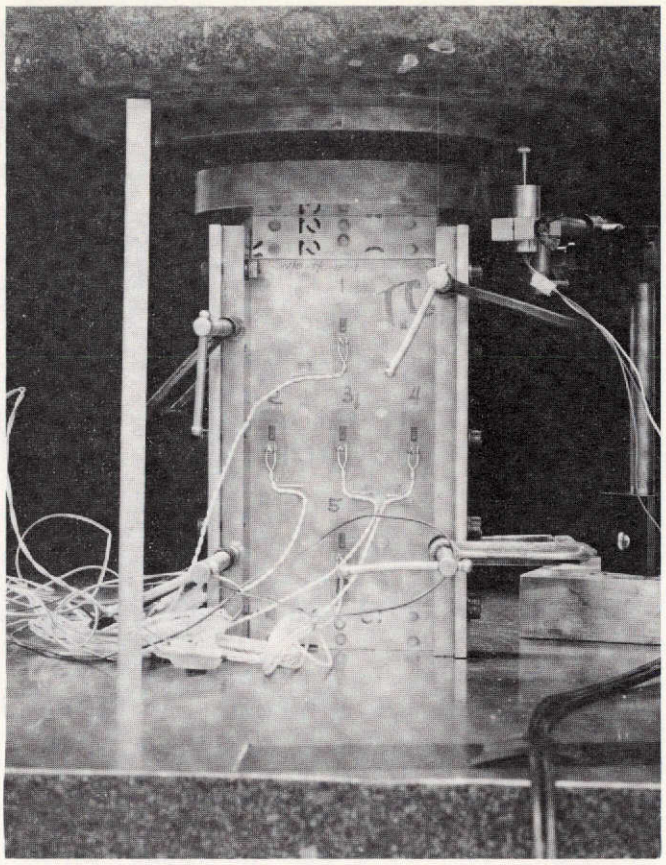


Figure 35. Detail View of Room Temperature Test Panel Set-up

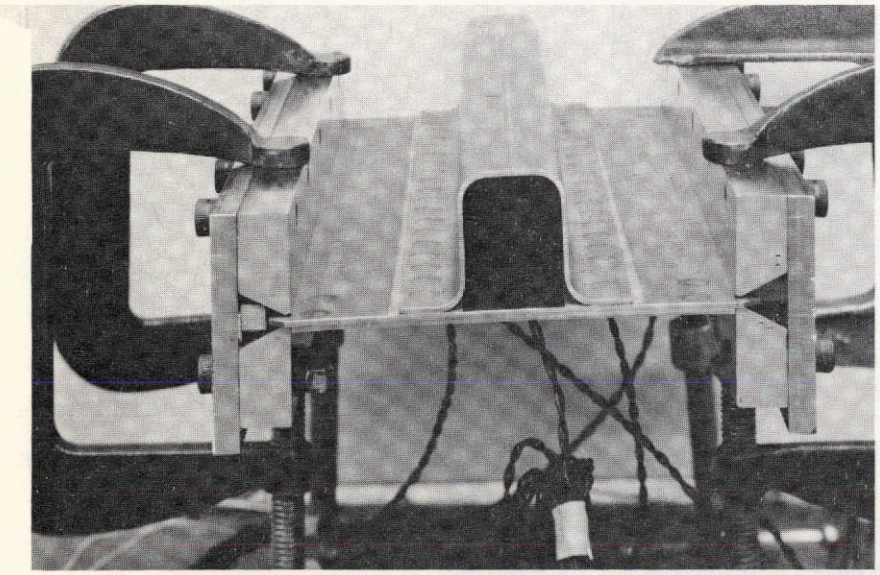


Figure 36. Panel Longitudinal Edge Supports

The primary difference in the elevated temperature test set-up and procedures from that used in the room temperature tests centered around trying to achieve a uniform temperature distribution on the panel. The initial panel was instrumented with ten thermocouples, with a control thermocouple attached to the center of the panel. The specimen was heated by

a bank of four high intensity tungsten/ quartz lamps with a total potential output of 32,000 watts. A typical set-up is shown in Figure 38. Power to the lamps was controlled *via* the center thermocouple by a Thermac power control system.

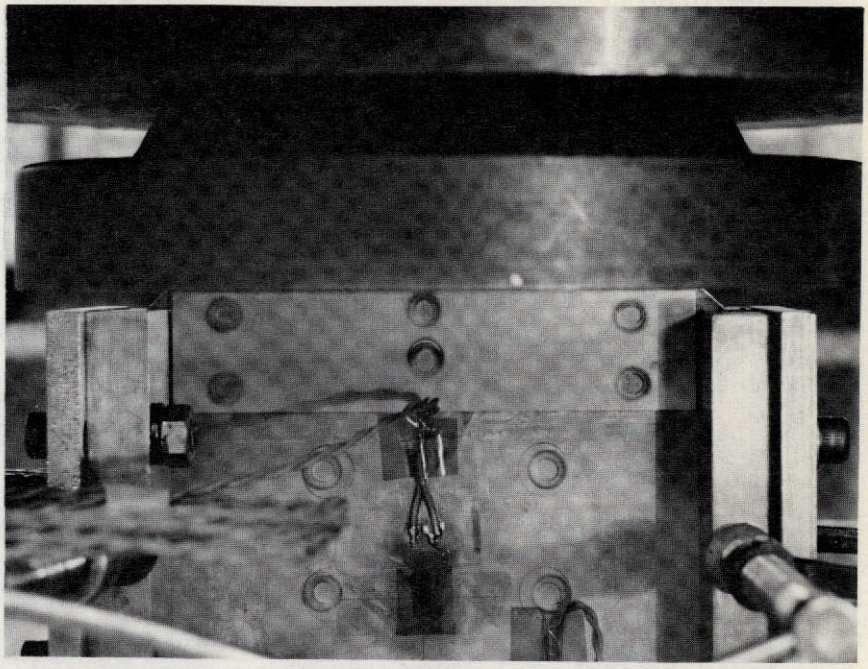


Figure 37. Clearance Between Loading Head And Edge Support Fixture

Much trial and error occurred in trying to get the entire panel up to the proper elevated temperature and achieve a uniformity of temperature over the panel. This required a combination of things. First, the monitoring thermocouple connected to the power supply had to be shaded to prevent it from achieving its prescribed temperature too quickly. Shading was achieved with a piece of cement-asbestos plate interposed between the lamps and the thermocouple (see Figure 39). Second, to achieve some uniformity of temperature, the entire test specimen had to be insulated from the loading machine. This was because the loading surfaces of the test machine acted as heat sinks. Layers of cement-asbestos sheeting were used as insulation. The sheeting,

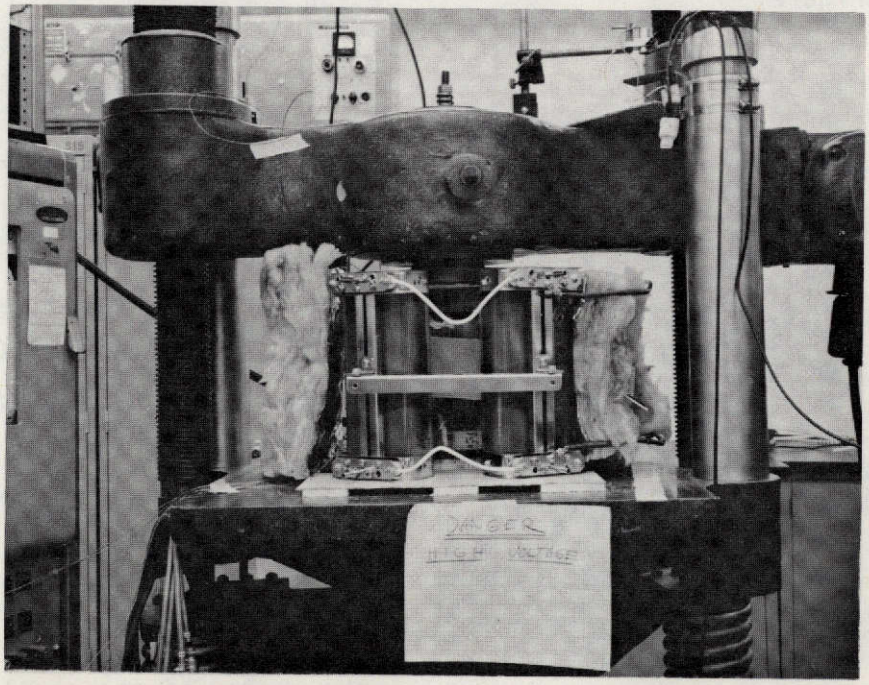


Figure 38. Test Set-up For Elevated Temperature Testing of Structural Panels

however, proved not to be sufficiently strong to resist the compressive loads when placed in direct contact with the ends of the panel. Thus, a metal plate was placed between the insulation sheeting and the test specimen. This tended to degrade, somewhat, the attempts to achieve a uniform surface temperature due to the heat sink behavior of the metal plate. Finally, with some additional adjusting of the test set-up, temperature levels along the bond or weld line over 80 percent of the length were held to 561 K±14 K (550°F±25°F).

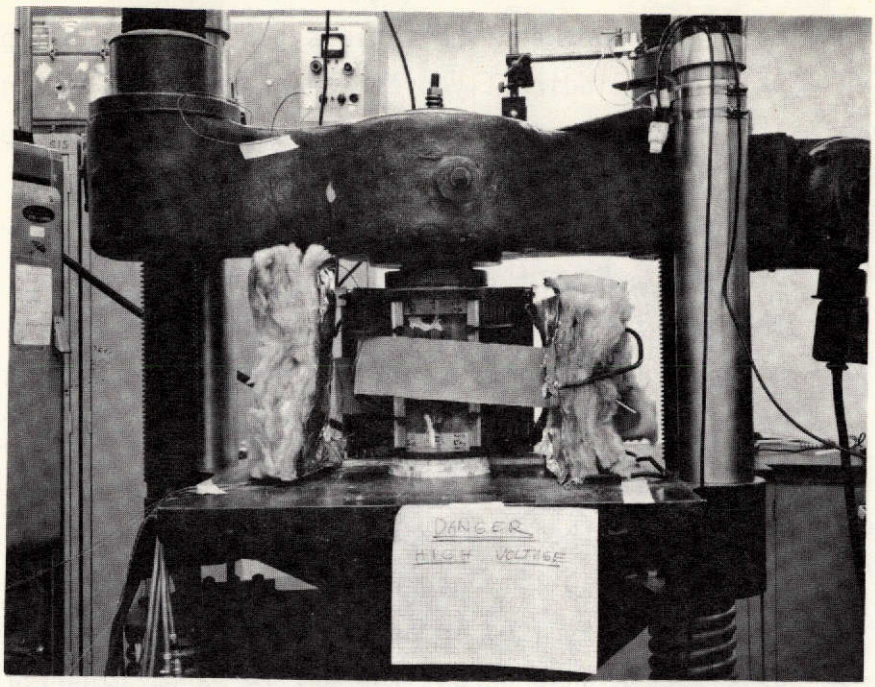


Figure 39. Detail of Elevated Temperature Test Set-up (Front two lamps are removed)

Subsequent panels used only four thermocouples, instead of ten, to monitor temperature. Figure 40 indicates a sample time-temperature history. Once the panel was brought up to the proper temperature, which took 30 to 40 minutes, the compressive loading commenced. Loading lasted 4 to 8 minutes depending on the ultimate capability of the panel.

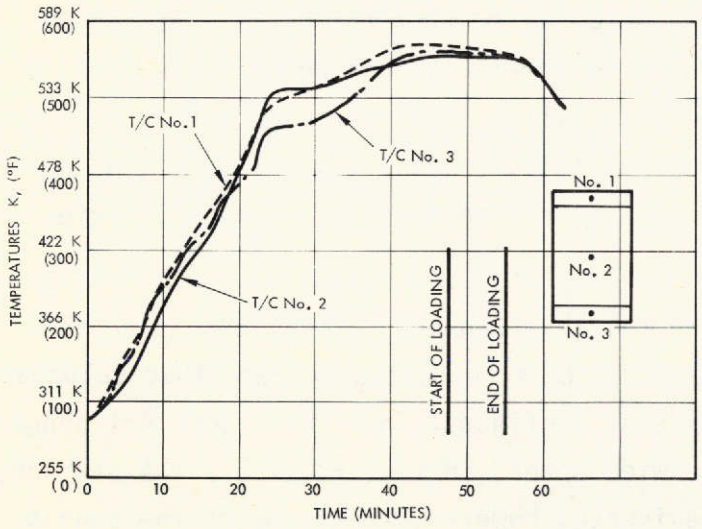


Figure 40. Sample Temperature-Time History For Elevated Temperature Tests

4.2.2 Test Results

The nature of the panel tests and the small number of test panels available limit the ability to obtain a definitive and conclusive

comparison between the various types of joining processes. The compression panel only indirectly provided a means of testing the joining process since the joint between the hat-section stiffener and plate was not loaded directly. Results from the other tests performed on the program such as lap-shear and fatigue are probably more definitive and incisive concerning relative joint behavior. Nevertheless, as will be indicated by the panel test results, the data do provide gross trends concerning the joining processes under consideration, and the experience gained does demonstrate the applicability of the weld-through weld bonding technique for fabrication purposes.

Data available for evaluation included load *vs* end-shortening and load *vs* strain. The former data were used to establish the load level where general instability occurred or the ultimate load carrying capacity. This load level resulted in crippling of the hat-section stiffener or generation of a buckle wave across the entire panel (identified at the point where there was a load drop-off). The strain data were used primarily to identify the load level where localized elastic buckling occurred (indicated by a strain reversal) which lead to eventual failure of the panel. The strain data were not used to monitor the stress distribution across the panel due to the limited number of strain gauges. Table XXV summarizes the loading capabilities for all the panels tested.

Figures 41 and 42 show the typical extremes in panel behavior. For the bonded-only panel, failure was sudden and catastrophic, with the hat-section stiffener almost completely delaminated from the plate. As can be seen there was little or no strain reversal, indicating neglible local buckling and load drop-off once the bond failed. For the panels that were spot-welded (with or without a bond) failure was a gradual event, with significant localized buckling preceeding general instability or overall crippling of the hat-section stiffener.

Figure 43 presents the relative rankings of the panels tested at room temperature. Included are the data spread, the average load value and number of panels tested for each joint configuration. There does not appear to be any obvious reason for the wide spread in test results for a particular joint configuration. There is evidence, however, that some of the panels having bonded-only or weld bonded joints did sustain damage during installation of the doublers in the form of hairline cracks in the bond.

TABLE XXV.

STRUCTURAL TEST PANEL DATA SUMMARY

Specimen (Joint Process)	Room Temperature	Temperature Cycled	Elevated Temperature	Local Crippling Load, $NX10^3$ (lbsX10^3)	Ultimate Panel Load $NX10^3$ (lbsX10^3)
Bond Only -1	X			122.3(27.5)	130.8(29.4)
Bond Only -2	X			124.6(28.0)	142.3(32.0)
Bond Only -1[a]		X		48.9(11.0)	75.6(17.0)
Bond Only -2		X		151.2(34.0)	160.1(36.0)
Bond Only -1			X	95.6(21.5)	97.9(22.0)
Bond Only -2			X	108.9(24.5)	118.8(26.7)
Spotweld, 1.57P-1	X			184.6(41.5)	196.6(44.2)
Spotweld, 1.57P-2	X			140.1(31.5)	177.9(40.0)
Spotweld, 1.57P-1		X		146.8(33.0)	173.5(39.0)
Spotweld, 1.57P-2[b]		X		-- (--)	-- (--)
Spotweld, 1.57P-1			X	129.0(29.0)	144.6(32.5)
Spotweld, 1.57P-2			X	129.0(29.0)	144.1(32.4)
Spotweld, 2.54P-1	X			133.4(30.0)	151.2(34.0)
Spotweld, 2.54P-2	X			120.1(27.0)	137.9(31.0)
Spotweld, 2.54P-1		X		106.8(24.0)	124.6(28.0)
Spotweld, 2.54P-2		X		175.7(39.5)	193.5(43.5)
Spotweld, 2.54P-1[c]			X	-- (--)	-- (--)
Spotweld, 2.54P-2			X	120.1(27.0)	141.0(31.7)
Bond/Weld, 1.57P-1	X			146.8(33.0)	184.6(41.5)
Bond/Weld, 1.57P-2	X			184.6(41.5)	210.4(47.3)
Bond/Weld, 1.57P-1		X		200.2(45.0)	217.9(49.0)
Bond/Weld, 1.57P-2		X		209.1(47.0)	220.2(49.5)
Bond/Weld, 1.57P-1			X	140.1(31.5)	143.7(32.3)
Bond/Weld, 1.57P-2			X	133.4(30.0)	157.0(35.3)
Bond/Weld, 2.54-1	X			129.0(29.0)	199.3(44.8)
Bond/Weld, 2.54-2	X			133.4(30.0)	193.5(43.5)
Bond/Weld, 2.54-1		X		135.7(30.5)	194.8(43.8)
Bond/Weld, 2.54-2		X		108.9(24.5)	203.7(45.8)
Bond/Weld, 2.54-1			X	120.1(27.0)	129.0(29.0)
Bond/Weld, 2.54-2			X	102.3(23.0)	140.6(31.6)

[a] Damaged bond due to doubler re-work.

[b] Premature failure due to lack of doublers.

[c] Cement-asbestos insulation sheets broke under load.

Nevertheless, the results do indicate some noticeable trends. From the standpoint of local buckling, the 12.7 mm (one-half inch) pitch joints are superior, whether or not bonding is present. This result is not surprising. However, for ultimate capability, the presence of bonding in connection with welding improves the situation and is attributed to an increased panel stiffness resulting from the continuous nature of the joint between the stiffener and plate as compared to the point attachment inherent in the spotweld joint. For a given spotweld pitch, the presence of the bond improves the overall joint efficiency. Likewise, the presence of the spotwelds inhibits the catastropic failure of the bond-only joint.

There were no consistent trends concerning the effect of thermal cycling on panel strength. This is attributed to the fact that all panels were subject to some thermal cycling when the strain gauges were installed (see Section 4.1.2). Thus, in reality, there were only two kinds of environmental test conditions: 1) room temperature tests on thermally cycled panels and 2) elevated temperature tests on thermally cycled panels.

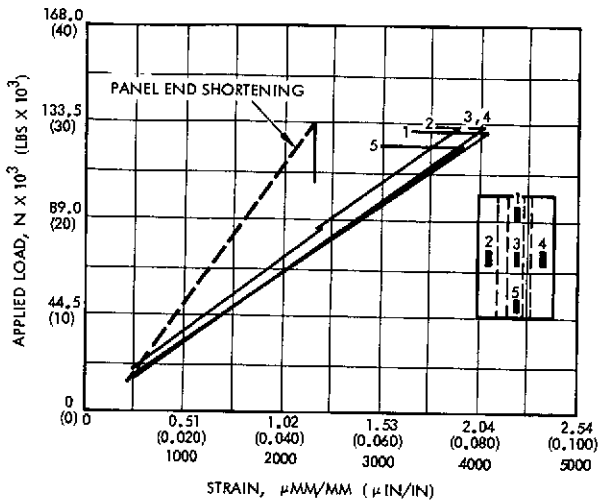


Figure 41. Typical Strain and End-Shortening Data (bond-only, room temperature test panel)

Figure 44 shows the comparative rankings for the elevated temperature tests. Because of the limited number of test panels, individual panel data has been superimposed on the results of Figure 41. As expected, the data fall below that for the room temperature tests. At least 20% of the degradation can be attributed to thermal reduction on the elastic modulus of the titanium material, which directly affects buckling. However, in some instances the degradation was more severe. Other possible factors contributing to degradation of panel capability are strength degradation of the bond material and loading eccentricities introduced into the test set-up due to the presence of the cement-asbestos insulating plates. However, no detailed investigation was conducted to evaluate the other factors. Nevertheless, as illustrated in Figure 42, the presence of the bond in conjunction with the spotweld does improve the capability of the panel.

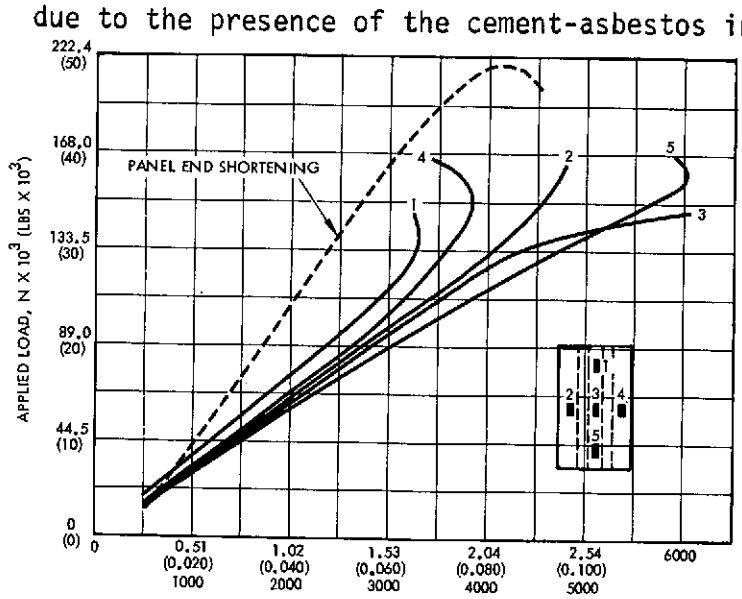


Figure 42. Typical Strain and End-Shortening Data (12.7 mm pitch, weld-only, room temperature test panel)

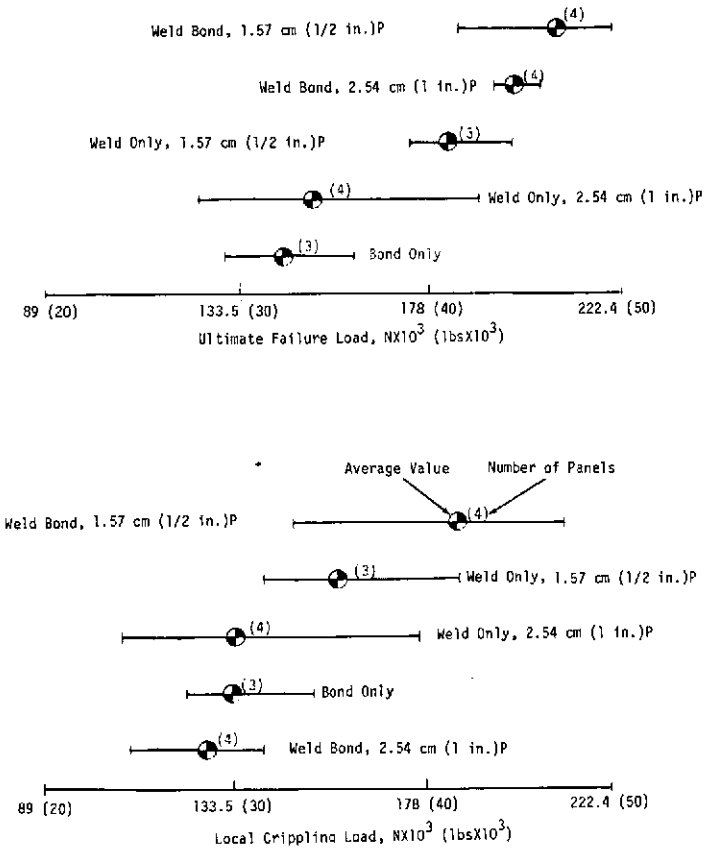


Figure 43. Relative Ranking, Room Temperature Structural Panel Tests

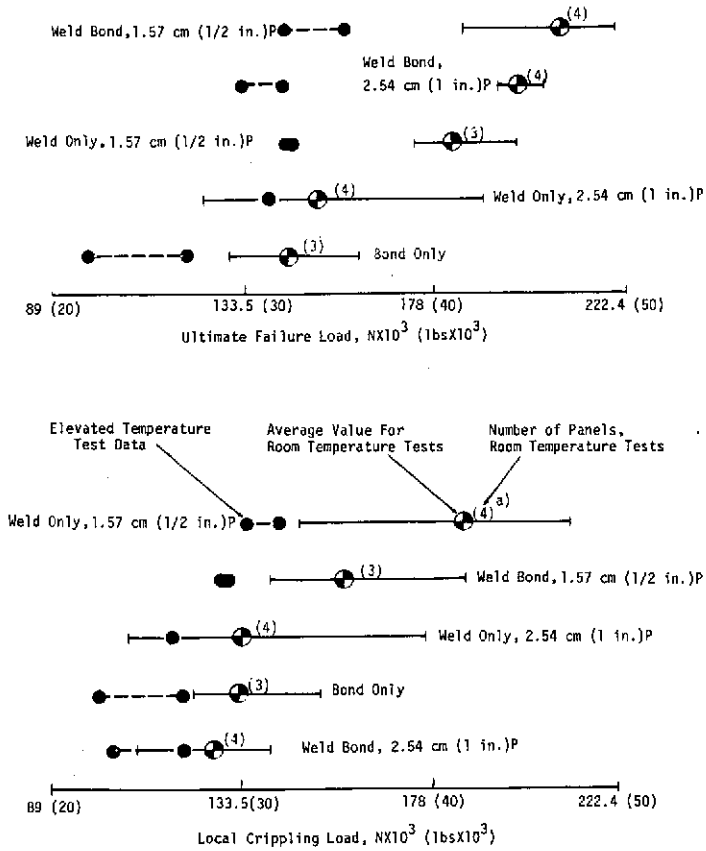


Figure 44. Relative Ranking, Elevated Temperature Structural Panel Tests Superimposed on Room Temperature Rankings

5. CONCLUSIONS AND RECOMMENDATIONS

Summarized below are the conclusions reached during this effort to assess the potential of the application of resistance spot-welding - polyimide adhesive bonding to structural panel fabrication. Based on these findings, recommendations are given for further developmental activities.

5.1 CONCLUSIONS

1. A weld-through weld bonding process was developed which uses an adhesive system similar to the P4/A5F adhesive system developed under Contract NAS1-9532. It was demonstrated that this process is suitable for preparing structural panels and is ameniable to production operations.

2. A capillary-flow weld bonding process was developed which uses a PDA polyimide resin developed under Contract NAS3-15834. However, it was demonstrated that further work is necessary in order to tailor this laminating resin for adhesive bonding applications.

3. The results from fatigue testing demonstrated that weld bonding provides superior joints to conventional spot-welding. The static loading carrying capacity of weld bonded joints also is improved over that of spot-welded joints.

4. Thermal aging at 561 K (550°F) and thermal cycling over a temperature range from 219 K (-65°F) to 561 K (+550°F) does not degrade the weld bonded joints.

5.2 RECOMMENDATIONS

1. Studies to develop a high temperature structural adhesive system with the BFBI/BMPM resin system are warranted in order to improve the efficiency of weld bonded joints produced by the capillary-flow process. These studies should include both polymer optimization studies and adhesive compounding studies similar to those performed under Contract NAS1-9532.

2. Studies to apply a BFBI/BMPM adhesive system to the weld-through weld bonding process are warranted because:

    a) The BFBI/BMPM resin cures at a lower temperature than the P4/A5F adhesive, *i.e.*, 450 K (350°F) *vs* 561 K (550°F, and

    b) TDA, a key ingredient of the P4/A5F system is not available commercially. These studies should include similar evaluation of joints to those performed during this program.

3. Application of the weld bonding process to rivet-bonding is required for certain assemblies in aircraft manufacture. Therefore, developmental studies are warranted for demonstrating the suitability of the technology developed under this contract to rivet-bonding.

4. A more detailed determination of weld bonded joints will be required before this technique can be applied to flight hardware. An example of the work that should be performed is as follows:

    a) Determine properties of stressed and unstressed weld bonded joints after long-term exposure (>5000 hours) at 505 K (450°F) to 561 K (550°F).

    b) Determine properties of weld bonded joints after exposure to high humidity environments.

    c) Determine the peel strength of weld bonded joints.

    d) Determine efficiency of weld bonded joints for serving as crack-stoppers, *e.g.*, holographic/fracture-toughness studies.

    e) Determine fatigue strength at temperature extremes, *e.g.*, 100 K (-100°F) to 561 K (550°F).

f) Determine effects of corrosive environments on weld bonded joints.

g) Determine strength of adhesive in weld bonded joints.

6. NEW TECHNOLOGY

Methods of preparing structural joints with titanium alloy by weld bonding processes were developed. Techniques for fabricating weld bonded joints by pre-application of the adhesive prior to welding (weld-through) and post-application of the adhesive after welding (capillary-flow) were defined. These concepts have been described in New Technology Disclosures submitted to the TRW Patent Office. The subject matter of these disclosures is listed below:

Docket Number	Title
71-153	Process For Preparing Low Void Content, High Temperature, Weld Bonded Joints
73-164	Capillary-Flow Resin And Process For Fabrication Of High Temperature Weld Bonded Joints

APPENDIX A.
MATERIALS PREPARATION PROCEDURES

A.1 PREPARATION OF ADHESIVE RESIN P11BA

The following quantity of ingredients were mixed as described below to produce a 40% solids varnish:

meta-Phenylenediamine (MPD)	170.00 g
4,4'-diaminodiphenyl sulfide (TDA)	67.93 g
Nadic anhydride (NA)	233.40 g
Benzophenone tetracarboxylic acid dianhydride (BTDA)	378.67 g
	850.00 g solids
Dimethyl formamide (DMF)	1275 g

The MPD and TDA were dissolved in 400 ml of DMF in a round bottomed flask fitted with a stirring apparatus and a nitrogen gas bleed. A slurry of NA in 252 ml of DMF was added slowly (with stirring) to the diamine mixture while controlling the solution temperature between 293 K (68°F) and 298 K (77°F). After the last addition of the NA slurry, stirring was continued for ten minutes, then a slurry of BTDA in 700 ml of DMF was added until all ingredients were combined in a temperature of 293 K (68°F) to 298 K (77°F). The material was stirred for two hours and then allowed to stand for 30 minutes under a nitrogen purge.

A.2 P4/A5F SERIES OF FORMULATIONS

The A-type polyimide and Amoco AI-1137 amide-acid varnishes first were blended together (see Table A.2-1). Aluminum powder then was added and blended together with Cab-O-Sil for adhesive pastes and the arsenic compounds where applicable. The primer formulations then were diluted with DMF. Adhesive film was prepared by immersing Style 104 A1100 glass scrim

in the adhesive paste and drawing the resultant film through wiper bars with a 0.45 mm gap. The resultant films then were air dried for 30 minutes and then dried in an air circulating oven for 15 minutes at 408 K (275°F) plus 5 minutes at 450 K (350°F).

TABLE A.2-1

PRIMER/ADHESIVE FORMULATIONS

Constituents	Parts by Weight of Constituents	
P11BA (Resin Solids)[a]	50	50
AI-1137 (Resin Solids)[b]	50	50
Aluminum Powder Grade 101[c]	100	175
Cab-O-Sil[d]	--	5
DMF[e]	400	150
Glass Scrim[f]	--	--

a) See Appendix A-1.
b) Amoco Corporation
c) Aluminum Corp. of America
d) Cabot Corporation
e) Baker Reagent Grade
f) Style 104 glass scrim, A1100 amino silane coupling agent.

A.3 SYNTHESIS OF *BIS*(2-FURFURYL) PYROMELLITIMIDE (BFPI)

To a mixture of 87.2 g (0.4 moles) of pyromellitic dianhydride and 300 ml of dimethylformamide was added 77.6 g (0.8 moles) of furfurylamine dropwise over a 30 minute period. The mixture was stirred an additional 20 minutes and then 500 ml of xylene was added. The reactions mixture was heated to reflux and heating was continued for 16 hours during which time the water from the imidization reaction was collected in a Dean-Stark trap. The mixture was cooled to 273 K (32°F) and the resulting preciptate was collected by filtration. Recrystallization from acetone afforded 114 g (76%) of *bis*-imide; mp 495-497 K (430-437°F).

A.4 SYNTHESIS OF *BIS*(4-MALEIMIDOPHENYL) METHANE (BMPM)

To a solution of 158 g (0.8 mole) of methylenedianiline in 480 ml of dimethyl formamide was added a solution of 157 g (1.6 moles) of maleic

anhydride in 240 ml of dimethyl formamide at such a rate as to keep the temperatures below 343 K (158°F). After stirring the mixture for an additional 15 minutes, it was cooled to room temperature and 204 g (2 moles) of acetic anhydride followed by 16 g (0.2 moles) of sodium acetate was added. The resulting mixture was heated to 323 K (122°F) and maintained there for 3 hours. The crude product was precipitated by pouring the reaction mixture into 4000 ml portions of water. The precipitate was collected by filtration, washed twice with 4000 ml portions of water and dried. Crystallization from methanol afforded 203 g (71%) of *bis*-imide, mp 429-432 K (312-319°F).

A.5 PREPARATION OF TITANIUM ALLOY 6Al4V FOR BONDING

Step 1. Degrease the titanium alloy faying surfaces with MEK.

Step 2. Grit blast the faying surfaces with 50 micron alumina and water rinse.

Step 3. Immerse grit blasted surfaces in Pasa-Jel 107 for 15 minutes at 294 K (70°F).

Step 4. Rinse in distilled water and air dry at 339 K (150°F).

Step 5. Apply primer thinly by brush within two hours of Pasa-Jel treatment.

Step 6. Thermally treat primed surfaces in an air circulating oven for 15 minutes at 403 K (275°F) and 5 minutes at 450 K (350°F).

A.6 PREPARATION OF PRESS BONDED LAP-SHEAR SPECIMENS

Step 1. Prepare titanium alloy 6Al4V lap-shear panels for bonding, cleaning and priming with Adhesive Primer in accordance with A.5.

Step 2. Lay adhesive film onto one of the mating surfaces.

Step 3. Thermally treat adhesive film and primed surfaces in an air circulating oven for 15 minutes at 403 K (275°F) plus 5 minutes at 450 K (350°F).

Step 4. Assemble panels in a bonding jig.

Step 5. Load into press preheated to 589 K (600°F) and apply 7 MN/m^2 (100 psig) pressure.

Step 6. Cure for 60 minutes under pressure.

Step 7. Postcure specimens in an air circulating oven for 16 hours at 561 K (550°F).

A.7 PREPARATION OF AUTOCLAVE BONDED SPECIMENS

Step 1. Prepare titanium alloy 6Al4V panels for bonding, cleaning and priming with adhesive primer in accordance with A.5.

Step 2. Lay adhesive film onto one of the mating surfaces.

Step 3. Thermally treat adhesive film and primed surfaces in an air circulating oven for 15 minutes at 403 K (275°F) plus 5 minutes at 450 K (350°F).

Step 4. Assemble panels in a bonding jig.

Step 5. Prepare vacuum bag assembly.

Step 6. Install assembly in an autoclave.

Step 7. Evacuate air out of the vacuum bag.

Step 8. Apply 7 MN/m^2 (100 psig) nitrogen gas pressure.

Step 9. Heat assembly to 575 K (575°F) at the rate of 5.56 K (10°F) per minute.

Step 10. Cure for 60 minutes under pressure.

Step 11. Release pressure and cool the assembly down to room temperature in the vacuum bag.

Step 12. Remove lap-shear specimens and postcure in an air circulating oven for 16 hours at 561 K (550°F).

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2. Rulon, E. Johnson, Jr. and Robert H. Dettre, "Contact Angle Hysterisis", Contact Angle, Wettability and Adhesion Symposium, Division of Colloid and Surface Chemistry, ACS, Los Angeles, April 1963.

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NASA CR-132434
National Aeronautics and Space Administration
POLYIMIDE WELD BONDING FOR TITANIUM ALLOY JOINTS. R. W. Vaughan and R. M. Kurland, June 1974, 63 pp.

Two weld bonding processes were developed for joining titanium alloy; one process utilizes a weld-through technique and the other a capillary-flow technique. The adhesive used for the weld-through process is similar to the P4/A5F system. A new polyimide laminating resin, BFBI/BMPM, was used in the capillary-flow process. Static property information was generated for weld-bonded joints over the temperature range of 219 K (-65°F) to 561 K (+550°F) and fatigue strength information was generated at room temperature. Significant improvement in fatigue strength was demonstrated for weld-bonded joints over spot-welded joints. A demonstration was made of the applicability of the weld-through weld-bonding process for fabricating stringer stiffened skin panels.

I. Vaughan, R. W., Kurland, R. M.

II. NASA CR-132434

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