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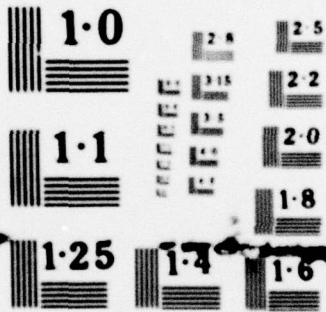
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PART III

ENVIRONMENTAL DURABILITY TESTING OF STRUCTURAL ADHESIVES  
PART III, RELIABOND 500/RELIABOND 398

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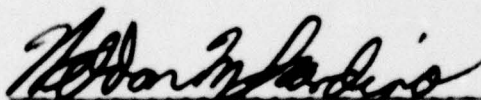
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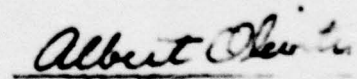
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20 ABSTRACT (Continue on reverse side if necessary and identify by block number) → A program has been conducted to investigate the durability of a 350°F (121°C) curing modified epoxy structural adhesive in an elevated temperature, high humidity environment while under stress. Durability was assessed with three different types of environmental exposure. Unstressed lap shear specimens were exposed to both a 140°F (60°C)/95-100% R.H. environment and a 140°F (60°C) →			

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20. Abstract (Concluded)

water soak and residual strength tests were conducted after various aging intervals. Stressed lap shear specimens were exposed to a 140°F (60°C)/95-100% R.H. environment and time-to-failure recorded.

The unstressed agings produced a progressive loss of residual strength and a progressive increase in the amount of adhesive (interfacial) failure with increased aging time.

The stressed agings produced times-to-failure at least as long as those previously obtained (AFML-TR-78-35, Part I) for the PL-729-3/PL-728 and AF-143-2/EC-3917 adhesive/primer systems on the same substrates and surface preparations. Had exposure times been extended beyond 2400 hours, the R398/R500 system may have proven more durable than the two previously tested systems.

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PREFACE

This report covers work performed during the period from June 1978 to December 1978 under Air Force Contract F33615-78-C-5002, Project 7381. This is the third report to be published in a series dealing with the same general theme. Part I describes the environmental durability testing of AF-143/EC-3917 and PL-729-3/PL-728, both 350°F (177°C) curing adhesive systems. Part II describes the environmental durability testing of FM-123/BR-123, a 250°F (121°C) curing adhesive system. This report, Part III, covers the environmental durability testing of Reliabond 398/Reliabond 500, a 350°F (177°C) curing adhesive system. The work was administered under the direction of the Systems Support Division of the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio. Mr. Weldon Scardino (AFML/MXE) acted as the Project Engineer.

The Principal Investigator on this investigation was D. Robert Askins. The major portion of the laboratory work was conducted by Messrs. David Klosterman and David Maxwell, research technicians.

This report was submitted by the author in November 1979. The contractor's report number is UDR-TR-79-24.

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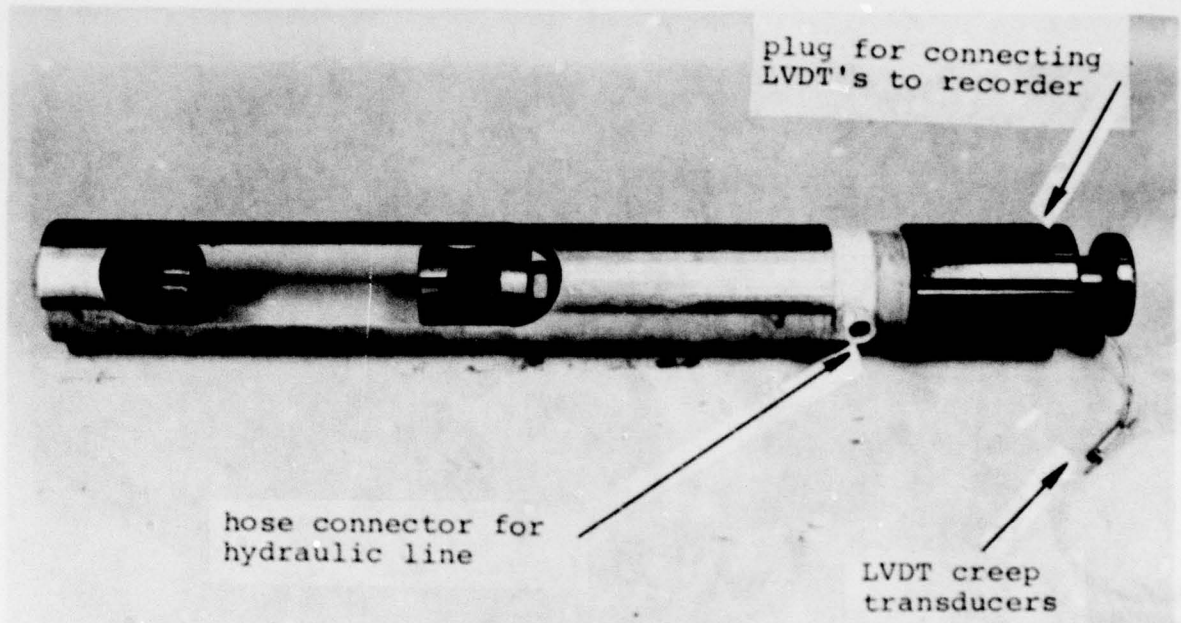
SECTION I  
INTRODUCTION

The University of Dayton Research Institute has been conducting investigations into the durability of adhesives and adhesive bonded structures for several years. Much of this work in recent years has involved the use of environmental stress-rupture testing of lap shear joints. The test apparatus which permits the measurement of the durability of bonded joints while exposed to elevated temperature and humidity under a controlled stress level was designed and constructed by the University of Dayton and has been in service for several years. It permits time-to-failure measurements on stressed adhesive bonds in adverse environments and also has the capability of measuring joint deformation as a function of exposure time. AFML-TR-78-35, Parts I and II, described investigations of the durability of two 350°F (177°C) curing adhesive systems and one 250°F (121°C) curing adhesive system on acid etched and anodized adherend surfaces.

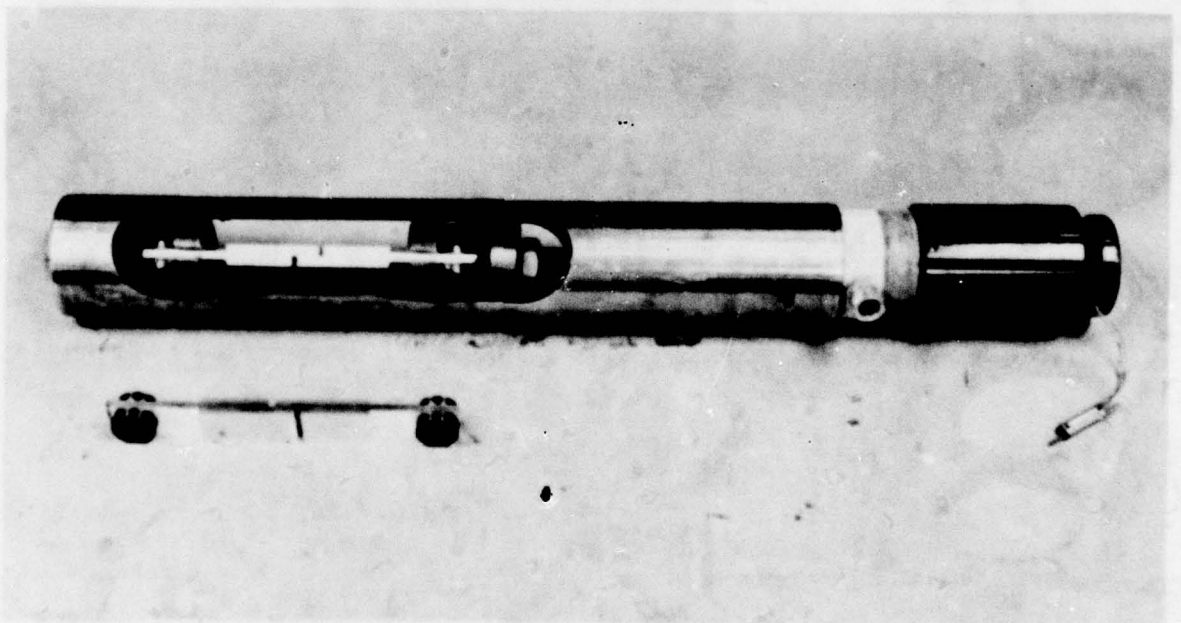
In the investigation reported here, another 350°F (177°C) curing adhesive system (Reliabond 398) has been investigated on an optimized Forest Products Laboratory (OFPL) acid etched adherend surface (2024T3 bare aluminum). Durability tests using stressed lap shear joints in the environmental stress-rupture apparatus have been conducted at 140°F (60°C). A second type of durability test in which unstressed lap shear joints are soaked in water at 140°F (60°C) has also been conducted to provide a comparison of results obtained from the two different test procedures.

SECTION II  
DURABILITY TEST APPARATUS

The durability test apparatus provides the capability of conducting environmental exposures on specimens subjected to a constant tensile load during the exposure period. The environment can be controlled between 95°F (35°C) and 200°F (93°C) and between 40% and 95% R.H. Loads are applied hydraulically and can be controlled to within  $\pm 5$  lbs ( $\pm 22$  N) over a range from 0 to 2500 lbs (0 to 11,125 N). Figures 1 to 3 illustrate the test apparatus and specimen mounting cells. An adhesive lap shear specimen of the type used in this program is shown mounted and also lying beside the test cell in Figure 1b. The tester can accommodate 12 specimens simultaneously. Although all 12 are exposed to the same temperature and humidity conditions, the load on each can be independently controlled. The exposure cabinet is a standard Blue M humidity cabinet, model AC-7502HA-1, which has 12 holes cut through the door for insertion of the test cells. Each test cell permits free access of the environment to the test specimen. Small Linear-Variable-Differential-Transformers (LVDT) transducers are mounted in the hydraulic loading heads of each cell. These transducers permit continuous recording of specimen creep deformation during exposure. The creep measurement capability was not utilized in this investigation, however; only time-to-rupture was recorded.



(a) Empty cell



(b) Cell with mounted specimen

Figure 1. Specimen Mounting Cells For the Durability Test Apparatus.

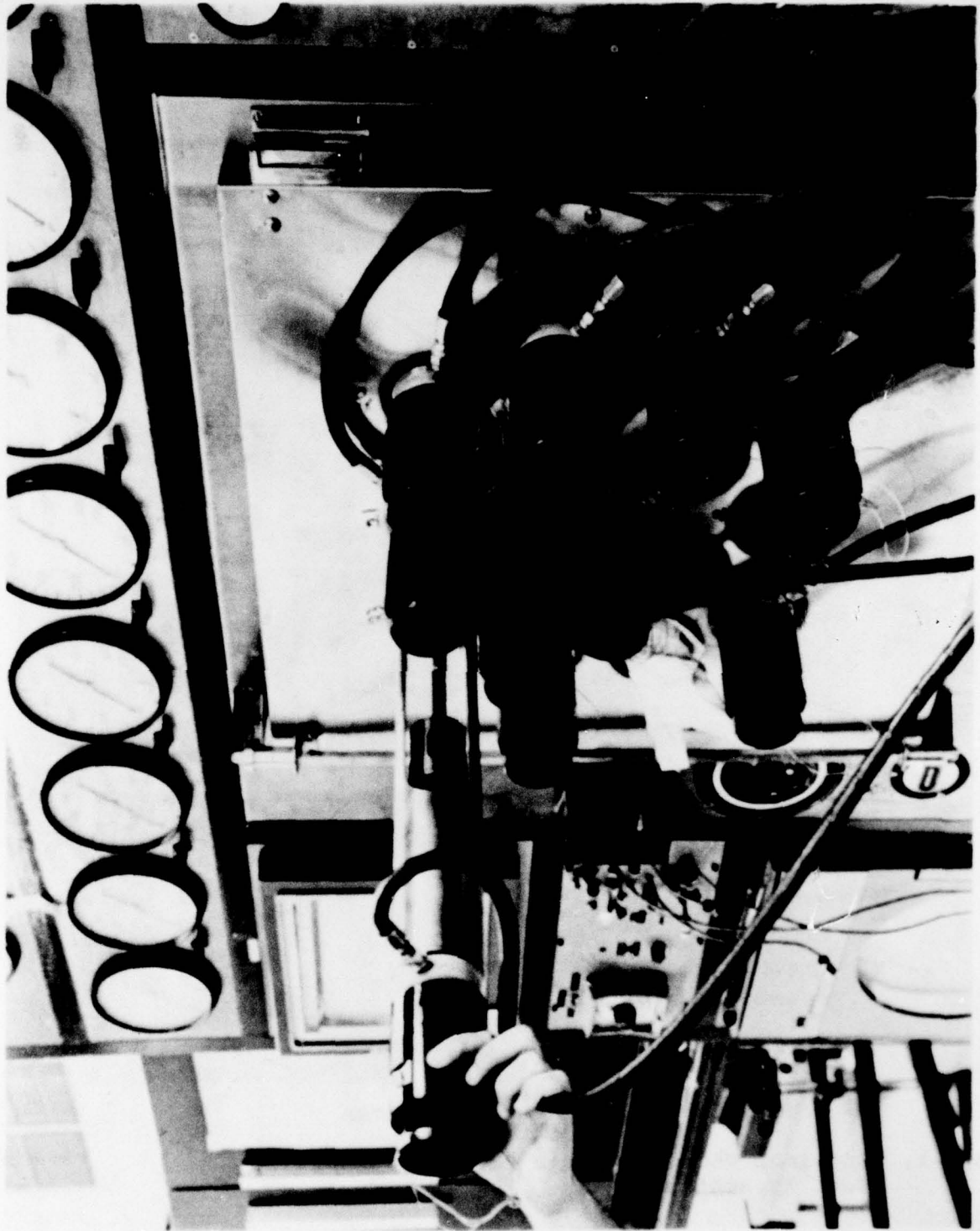


Figure 2. Specimen Mounting Cell Being Inserted Into Humidity Cabinet.

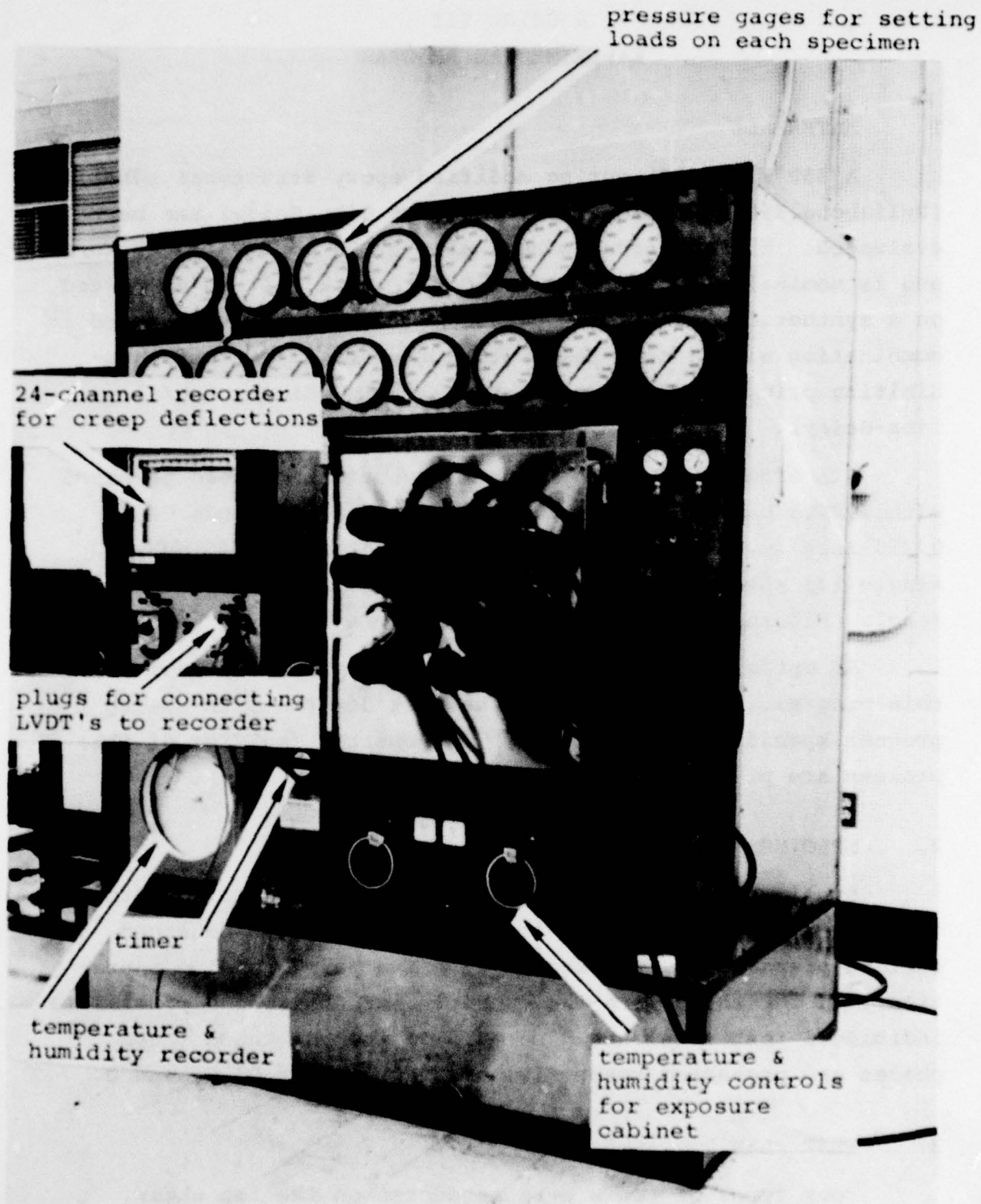


Figure 3. Overall View of Durability Test Apparatus.

SECTION III  
EXPERIMENTAL PROGRAM

1. MATERIALS

A 350°F (177°C) curing modified epoxy structural adhesive (Reliabond 398 by Reliable, division of Ciba-Geigy) has been evaluated. The adhesive is nominally 0.090 lb/ft<sup>2</sup> (0.439 Kg/m<sup>2</sup>) and is nominally 0.012 inch (0.030 cm) thick. It is supported on a synthetic woven fabric carrier. This adhesive was used in combination with a high temperature resistant, corrosion inhibiting primer (Reliabond 500 by Reliable, division of Ciba-Geigy).

All of the specimens in this investigation were prepared with 2024T3 bare aluminum adherends. These adherends were 0.250 inch (0.64 cm) thick and were used to prepare machined single lap shear specimens (also known as blister shear specimens). Figure 4 illustrates this specimen.

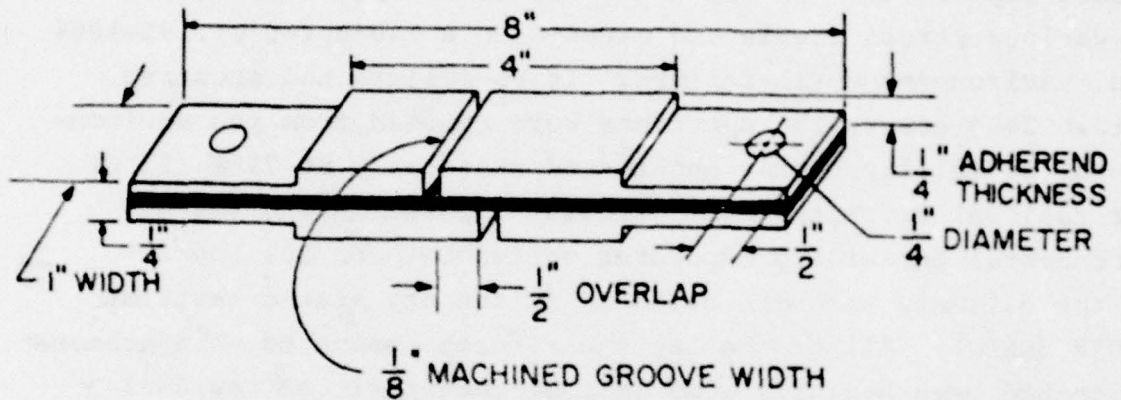
An optimized FPL etch surface preparation was used for this program. This is the same as that described in Boeing process specification BAC 5514. The central features of this process are presented in Appendix B.

2. SPECIMEN FABRICATION

Specimen fabrication procedures can be separated into three general phases. The first phase deals with adherend surface preparation, the second with the panel bonding operation, and the third with the machining of the bonded panel into individual test specimens. Details of each of these three phases are presented respectively in Appendices B, C, and D.

3. TEST PLAN

Four types of tests were conducted on the lap shear specimens in this investigation. The first was a simple static



0.250 inch (0.64cm), thick adherend, machined single lap shear specimen

Figure 4. Single Lap Shear Adhesive Specimen

test on the as-fabricated, dry specimens at three different temperatures; 72°F (22°C), 140°F (60°C), and 250°F (121°C). The second type was also a simple static test at 72°F (22°C) on specimens which had been exposed to elevated temperature, high-humidity aging [140°F (60°C) and 95-100% R.H.] for 30 and 100 days prior to testing. The third type of test was also a simple static test at 140°F (60°C) on specimens which had been soaked in water at 140°F (60°C) for 100 and 1000 hours prior to testing. The fourth type of test was an environmental stress-rupture test in which the lap shear specimens were loaded to various stress levels and exposed to a 140°F (60°C), 95-100% R.H. environment until failure. If no failure had occurred within 2400 hours, the specimens were removed from the environmental durability tester and tested statically at 72°F (22°C) for residual strength. The stresses imposed during the environmental durability exposures varied between 20% and 60% of the ultimate strength obtained in the dry static tests at 140°F (60°C). All of the lap shear tests conducted on specimens which had been humidity aged (either the static or residual strength tests) or water soaked were completed within 30 minutes after the specimen was removed from the environment. Additionally, each of these specimens was wrapped with a wet cloth to prevent dryout during this period.

SECTION IV  
DISCUSSION OF RESULTS

Tables 1 and 2 present the test results obtained during this investigation. Complete tabulations of all the individual test data, including computed standard deviations are presented in Appendix A. In addition to these tabulations, the environmental stress-rupture durability data are graphically illustrated in Figure 5.

1. STATIC LAP SHEAR TEST RESULTS

As can be seen from the data in Table 1, the static lap shear strength and failure mode of dry specimens is little affected by temperature from 72°F (22°C) to 250°F (121°C). In fact, the strength of the bonds made with the R398/R500 system in this investigation retained a considerably higher fraction of their room temperature value than those previously reported in AFML-TR-78-35, Part I, for two other 350°F (177°C) curing adhesive systems (PL-729-3/PL-728 and AF-143-2/EC-3917). Failure modes for all three of these adhesive systems on OFPL etched surfaces and 2024T3 bare aluminum adherends was predominately cohesive for the dry static tests.

2. ENVIRONMENTAL EXPOSURE TEST RESULTS

The unstressed specimens aged in a humidity cabinet at 140°F (60°C) and 95-100% R.H. exhibited a residual strength reduction of about 12% after 30 days and 22% after 100 days (based on original 72°F (22°C) dry static strength). The failure mode of these specimens becomes progressively more adhesive (interfacial) in nature as aging time increases.

The unstressed specimens aged in a water bath at 140°F (60°C) exhibited a residual strength reduction of about 8% after 100 hours and 25% after 1000 hours (again based on original 72°F (22°C) dry static strength). The failure mode of these

**TABLE 1**  
**SINGLE LAP SHEAR STRENGTH OF ADHESIVE JOINTS**

Adherends: 2024T3 bare aluminum  
 Surface Preparation: Optimized FPL etch  
 Surface Primer: Reliabond 500  
 Adhesive: Reliabond 398

Test Temperature (°F) (°C)		Pre-Test Conditioning	Ultimate Strength (psi) (MPa)		Failure Mode (% Coh)	No. of Specimens Represented
72	22	None	6430	44.3	85	5
140	60	None	6160	42.5	98	5
250	121	None	5610	38.7	75	5
72	22	30 days at 140°F (60°C) and 95-100% R.H. - unstressed	5630	38.8	65	5
72	22	100 days at 140°F (60°C) and 95-100% R.H. - unstressed	5040	34.7	40	5
140	60	100 hr. soak in water at 140°F (60°C)-unstressed	5920	40.8	85	5
		1000 hr. soak in water at 140°F (60°C)-unstressed	4790	33.0	40	5

TABLE 2  
 ENVIRONMENTAL STRESS-RUPTURE LAP SHEAR  
 BEHAVIOR OF ADHESIVE JOINTS

Adherends: 2024T3 bare aluminum  
 Surface Preparation: Optimized FPL etch  
 Surface Primer: Reliabond 500  
 Adhesive: Reliabond 398  
 Exposure Environment: 140°F(60°C) and 95-100% R.H.

Joint Shear Stress During Exposure (% of 140°F <sup>1</sup> dry ultimate)			Time to Failure (hrs)	Residual Lap Shear Strength (psi) (MPa)		Failure Mode (% Coh)	No. of Specimens Represented
3700	25.5	60	560	---	---	40	3
2460	16.9	40	2010 <sup>1</sup>	5950	41.0	60	3
1850	12.7	30	2400 <sup>2</sup>	5280	36.4	55	3
1230	8.5	20	2400 <sup>2</sup>	5960	41.1	75	3

<sup>1</sup>Two specimens survived 2400-hour exposure period.

<sup>2</sup>All specimens survived 2400-hour exposure period.

<sup>3</sup>Base strength at 140°F - 6160 psi (42.4 MPa).

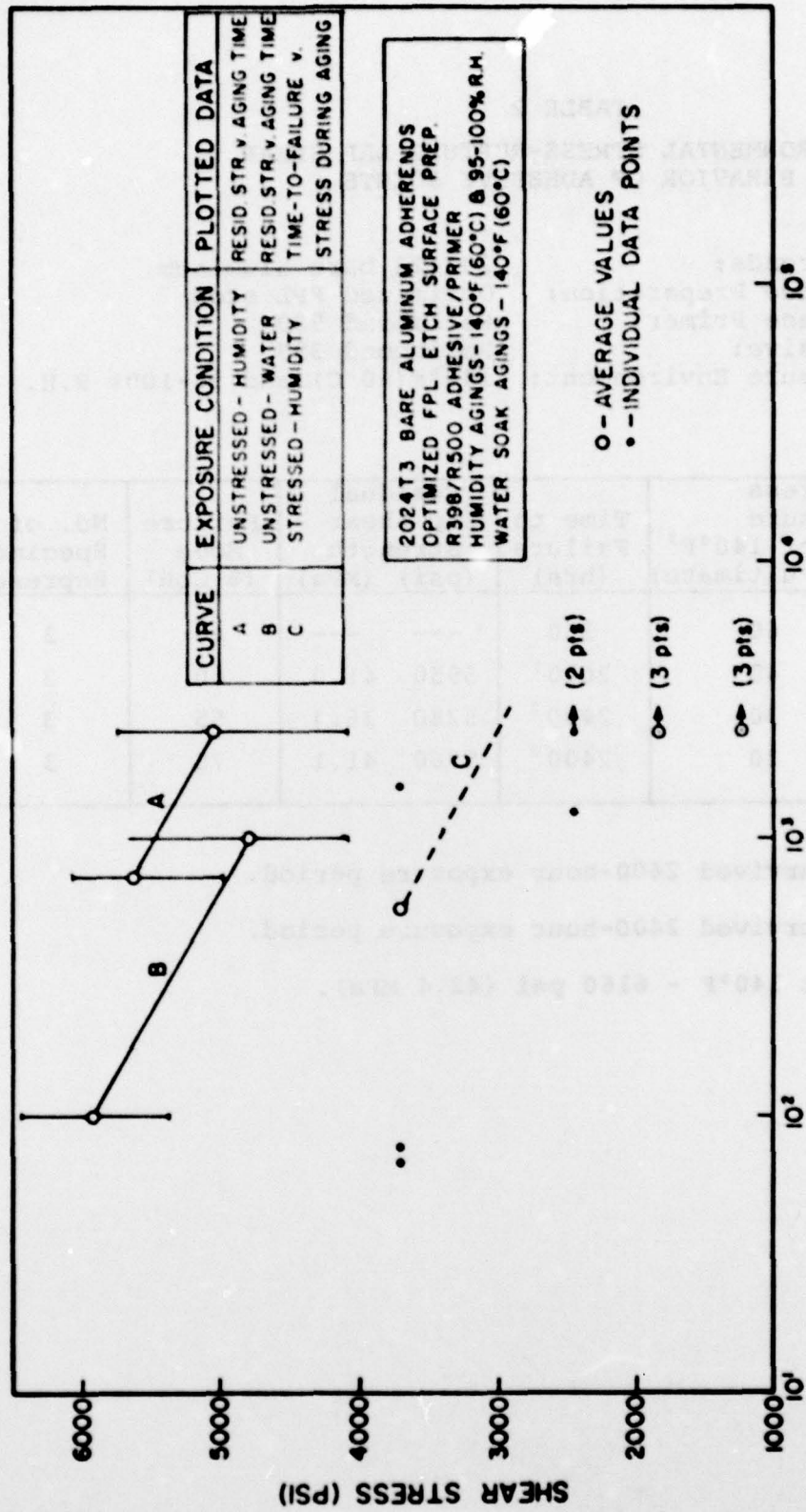


Figure 5. Environmental Degradation Behavior of Machined Single Lap Shear Adhesive Joints.

specimens also becomes progressively more adhesive in nature as aging time increases.

An interesting point to note in comparing the residual strength characteristics of unstressed specimens aged in the humidity cabinet and in a water bath is that the slope of the residual strength versus aging time curves plotted in Figure 5 have the same slope. The relative location of these two curves (A and B) is probably the result of two factors. First, the residual strengths of the water soaked specimens (curve B) were obtained at a 140°F (60°C) test temperature while those for the humidity aged specimens (curve A) were obtained at a 72°F (22°C) test temperature. This difference in test temperature caused about a 5 percent decrease in strength on the dry unaged specimens (Table 1). Since the difference in the positions of curves A and B in Figure 5 represents a difference of about 10 percent in strength for equivalent aging time, it would appear that the difference in aging conditions may also be contributing to this difference. The progressive decrease in the amount of cohesive failure (alternatively, the progressive increase in the amount of adhesive or interfacial failure) with increasing exposure time indeed indicates a progressive degradation of the interfacial bond, leading to reduced strengths for longer exposures. Since it is reasonable that water would diffuse into the bond-line more rapidly in a water immersion exposure than in a high humidity exposure, it is also reasonable that the differences in exposure conditions between curves A and B would indeed contribute to a shift in their relative positions.

The specimens which were exposed to 140°F (60°C) and 95 to 100 percent relative humidity under stress exhibited no failures at all for aging times of up to 2400 hours at stresses of 1850 psi (12.7 MPa) and below. Two of the three specimens subjected to a 2460 psi (16.9 MPa) stress level also survived for 2400 hours without failure. Since this only provides one good data point for purposes of plotting a stress versus time-to-failure curve in Figure 5, a broken curve, C, has been plotted

through the single real average value with a slope arbitrarily equal to curves A and B. Wegman, et al.<sup>(1)</sup> have developed evidence that, for some adhesives, this procedure provides a reasonably valid method of prediction of time-to-failure for stressed joints in environmental aging. There is insufficient evidence to establish what the true slope of curve C should be. To do this, substantially more time would have had to be expended in carrying the exposures completely out to joint failure.

The levels of durability observed are very comparable to those reported for the AF-143-2/EC-3917 and PL-729-3/PL-728 systems in AFML-TR-78-35, Part I, particularly below 2800 psi (19.3 MPa) where the stress is low enough that the adherend surface oxide layer should not fracture (see pp. 27-30 of AFML-TR-78-35, Part I). A comparison of the environmental stress-rupture time-to-failure behavior of these two systems with the R398/R500 system, tested here, is presented in Figure 6. Here both the broken curve plotted in Figure 5 as well as the relevant data points for the R398/R500 system are presented for comparison with the AF-143-2 and PL-729-3 curves. At worst it is evident that the R398 system is at least equal to the two previously tested. If the broken curve, obtained as described above, is indeed a reasonable estimate of the R398 behavior, then this system would appear to be more durable than the previous two for this type of test condition.

While the residual strength of the aged unstressed specimens consistently decreases with increasing aging times, as would be expected, the residual strength behavior of the specimens aged under stress is more difficult to explain. While one might normally expect that the residual strength of those specimens which survived the entire 2400-hour aging period would be higher for the specimens which were at a lower stress and lower for

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(1) Wegman, R. F., Bodnar, M. J., and Ross, M. C., "A New Technique for Assessing the Durability of Structural Adhesives," SAMPE Journal, Vol. 14, No. 1, January/February 1978.

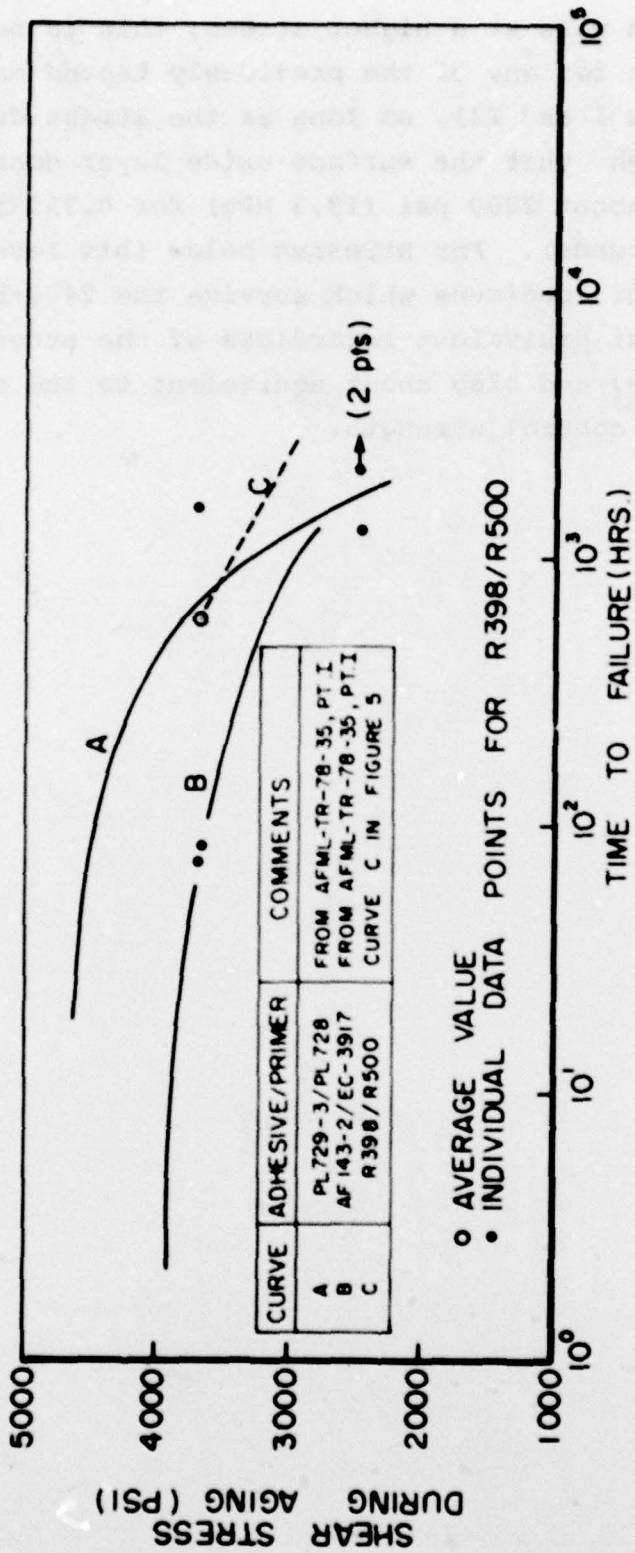


Figure 6. Comparative Environmental Stress-Rupture Time-to-Failure Behavior of Three 350°F Curing Adhesive Systems at 140°F (60-C) and 95-100% Relative Humidity.

those specimens which were at a higher stress, this is not the case, nor has it been for any of the previously tested materials (AFML-TR-78-35, Parts I and II), so long as the stress during exposure is low enough that the surface oxide layer does not fracture (less than about 2800 psi (19.3 MPa) for 0.250 inch (0.64 cm) thick adherends). For stresses below this level, the residual stress of specimens which survive the 2400-hour exposure are all about equivalent regardless of the stress level during exposure, and also about equivalent to the original dry room temperature control strength.

SECTION V  
CONCLUSIONS

1. Unstressed aging of lap shear adhesive joints made with the Reliabond 398/Reliabond 500 (R398/R500) adhesive/primer system on optimized FPL etched 2024T3 bare aluminum adherends in both a 140°F (60°C)/95-100% R.H. environment and a 140°F (60°C) water soak results in a progressive loss of residual strength with increased aging time and a progressive increase in the amount of adhesive (interfacial) failure with increased aging time.
2. Unstressed agings in the 140°F (60°C) water soak produce about a 10% lower residual strength, for equivalent aging time, than unstressed agings in a 140°F (60°C)/95-100% R.H. environment.
3. Specimens made with the R398/R500 adhesive/primer system and subjected to shear stress during aging in a 140°F (60°C)/95-100% R.H. environment produced times-to-failure comparable to those obtained previously (AFML-TR-78-35, Part I) for the PL-729-3/PL-728 and AF-143-2/EC-3917 adhesive/primer systems on the same substrates. It is possible, had aging times been carried out beyond 2400 hours, that the R398/R500 system would have produced longer times-to-failure than either of the other two systems for shear stress levels during exposure of less than about 2800 psi (19.3 MPa). This is the approximate level at which oxide fracture occurs on this type specimen (see AFML-TR-78-35, Part I-Appendix D).
4. The R398/R500 system loses less strength during a 250°F (121°C) test on dry unaged specimens than either the PL-729-3/PL-728 or AF-143-2/EC-3917 systems tested earlier.



TABLE A.1

## SINGLE LAP SHEAR STRENGTH OF DRY ADHESIVE JOINTS

Adherends: 0.250 inch (0.64 cm) thick 2024T3  
bare aluminum  
Surface Preparation: Optimized FPL Etch  
Surface Primer: Reliabond 500  
Adhesive: Reliabond 398

Test Temperature (°F) (°C)		Ultimate Strength (psi) (MPa)		Failure Mode (% Coh. Failure)
72	22	7150	49.3	50
72	22	7000	48.3	80
72	22	6090	42.0	95
72	22	5790	39.9	95
72	22	6100	42.1	100
Average		6430	44.3	85
Std. Dev.		610	4.2	20
140	60	6850	47.2	95
140	60	6550	45.2	95
140	60	5500	37.9	100
140	60	5730	39.5	100
140	60	6150	42.4	100
Average		6160	42.5	98
Std. Dev.		560	3.9	3
250	121	5800	40.0	80
250	121	5890	40.6	95
250	121	5800	40.0	90
250	121	5320	36.7	70
250	121	5250	36.2	50
Average		5610	38.7	75
Std. Dev.		300	2.1	20

TABLE A.2  
LAP SHEAR STRENGTH OF ENVIRONMENTALLY  
AGED ADHESIVE JOINTS

Adherends: 0.250 inch (0.64 cm) thick 2024T3  
bare aluminum  
Surface Preparation: Optimized FPL Etch  
Surface Primer: Reliabond 500  
Adhesive: Reliabond 398

Test Temperature (°F) (°C)		Pre-Test Conditioning	Ultimate Strength (psi) (MPa)		Failure Mode (% Coh. Failure)
72	22	30 days at 140°F (60°C) and 95-100% R.H. ↓	5300	36.5	60
72	22		5950	41.0	95
72	22		5760	39.7	60
72	22		4970	34.3	50
72	22		6180	42.6	70
Average				5630	38.8
Std. Dev.			490	3.4	15
72	22	100 days at 140°F (60°C) and 95-100% R.H. ↓	5560	38.3	50
72	22		4080	28.1	10
72	22		4550	31.4	5
72	22		5740	39.6	75
72	22		5260	36.3	50
Average				5040	34.7
Std. Dev.			700	4.8	30
140	60	100 hr. soak in water at 140°F (60°C) ↓	6430	44.3	90
140	60		5900	40.7	50
140	60		6440	44.4	95
140	60		5460	37.6	100
140	60		5350	36.9	100
Average				5920	40.8
Std. Dev.			520	3.6	20
140	60	1000 hr. soak in water at 140°F (60°C) ↓	5670	39.1	90
140	60		4110	28.3	10
140	60		5020	34.6	30
140	60		4080	28.1	20
140	60		5050	34.8	50
Average				4790	33.0
Std. Dev.			680	4.7	30

TABLE A.3  
ENVIRONMENTAL STRESS-RUPTURE LAP SHEAR  
BEHAVIOR OF ADHESIVE JOINTS

Adherend: 0.250 inch (0.64 cm) thick 2024T3 bare aluminum  
 Surface Preparation: Optimized FPL Etch  
 Surface Primer: Reliabond 500  
 Adhesive: Reliabond 398  
 Exposure Environment: 140°F(60°C) and 95-100% R.H.

Joint Shear Stress During Exposure [% of 140°F (60°C) dry ultimate]			Time to Failure (hrs)	Residual Lap Shear Strength <sup>2</sup>		Failure Mode (% Coh.)
(psi)	(MPa)			(psi)	(MPa)	
3700	25.5	60	1544	---	---	30
3700	25.5	60	77	---	---	50
3700	25.5	60	68	---	---	50
Average			560	---	---	40
Std. Dev.			850	---	---	10
2460	16.9	40	1240	---	---	10
2460	16.9	40	2400 <sup>1</sup>	5950	41.0	95
2460	16.9	40	2400 <sup>1</sup>	5940	41.0	80
Average			2010	5950	41.0	60
Std. Dev.			670	~0	~0	45
1850	12.7	30	2400 <sup>1</sup>	4770	32.9	10
1850	12.7	30	2400 <sup>1</sup>	5030	34.7	60
1850	12.7	30	2400 <sup>1</sup>	6030	41.6	90
Average			2400	5280	36.4	55
Std. Dev.			0	670	4.6	40
1230	8.5	20	2400 <sup>1</sup>	5540	38.2	50
1230	8.5	20	2400 <sup>1</sup>	6470	44.6	80
1230	8.5	20	2400 <sup>1</sup>	5860	40.4	95
Average			2400	5960	41.1	75
Std. Dev.			0	470	3.2	25

<sup>1</sup>Joints did not fail during 2400-hour exposure period and were removed for residual strength testing.

<sup>2</sup>All residual strengths obtained at 72°F(22°C) (Section III.3).

APPENDIX B  
SURFACE PREPARATION PROCEDURES

The specimens prepared and tested in this investigation were made with either an optimized FPL etch or with a phosphoric acid anodized surface preparation. Each of these are described below. The referenced BAC numbers refer to processing specifications developed by the Boeing Aircraft Company.

1. OPTIMIZED FPL ETCH

The stepwise procedure used for this surface is:

- (1) Wipe adherend surface with MEK and dry.
- (2) Vapor degrease in trichloroethylene according to BAC 5408.
- (3) Acid etch with the solutions and procedures contained in BAC 5514 for optimized FPL etch.
- (4) Rinse immediately in continuously flowing tap water for ten minutes and dry with an air heat gun.
- (5) Apply primer within 1/2 hour.

APPENDIX C  
PANEL BONDING PROCEDURES

The stepwise procedure used to cure the panels from which individual specimens were subsequently machined is:

- (1) Layup primed panels and adhesive film into assembly required for final specimens.
- (2) Place layup assembly in autoclave at room temperature.
- (3) Apply  $40 \pm 10$  psi ( $276 \pm 69$  KPa) over the bladder and then release the vacuum.
- (4) Heat the autoclave to  $350^{\circ}\text{F}$  ( $177^{\circ}\text{C}$ ) in less than four hours.
- (5) Hold at  $350^{\circ}\text{F}$  ( $177^{\circ}\text{C}$ ) for 60 minutes.
- (6) Cool the autoclave to below  $200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ), maintaining the  $45 \pm 5$  psi ( $310 \pm 34$  Pa) over the bladder.
- (7) Release pressure and remove the panel from the autoclave.

APPENDIX D  
SPECIMEN PREPARATION PROCEDURES

The as-fabricated panels are 16 inches (40.6 cm) wide when bonded and are first cut into 13 individual specimens on a bandsaw. These rough-cut specimens are then finish milled down to their final one inch (2.54 cm) wide by seven inches (17.8 cm) long dimension. Holes are drilled through the ends for mounting in the test grips as well as for specimen location in a machining fixture when the specimens are slotted across their width. The slots are cut across the specimens to provide the lap joint. These slots are machined down to, but not through, the adhesive layer. Finally, the ends of the specimen are machined down to a 0.250 inch (0.64 cm) thickness to fit into the test grips on the environmental stress-rupture durability tester.