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Anodize Optimization And Adhesive Evaluations For Repair Applications

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July 1978

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This technical report has been reviewed and is approved for publication.

A. Blandino

W. M. Scardino (AFML/MXE) Project Engineer

FOR THE COMMANDER

a. Oleviter

A. Olevitch, Chief Materials Engineering Branch Systems Support Division Air Force Materials Laboratory

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Following the anodizing variables investigation, bond verification tests were conducted to assess bondability of representative RT, 250°F, and 350°F cure adhesive systems. This work served as a basis for selecting the phosphoric acid non-tank anodize process parameter/conditions for Task II. The Task I work is reported in AFML-TR-78-7.

Task II was aimed at developing a data base to facilitate repair bonding. Surface preparation methods including optimized FPL etch, and two hand-clean procedures, phosphoric acid non-tank anodize (PANTA) and PasaJell 105, were evaluated. Bondability and environmental durability of current and on-coming adhesive systems from RT, 250°F, and 350°F cure categories were determined. Autoclave bonds versus vacuum bag bonds were compared.

FOREWORD

This final report summarizes the work accomplished in an "add-on" to Contract F33615-73-C-5171, "Adhesive Bonded Aerospace Standardized Repair Handbook," by the Boeing Commercial Airplane Company, P. O. Box 3707, Seattle, Washington.

Other phases of the basic contract have been reported as follows: Phase I report was a limited distribution document; Phase II-AFML-TR-75-158, AD-A017779; Phase III-AFML-TR-76-65, AD-A035601; Phase IV-AFML-TR-76-201/AFFDL-TR-76-131, AD-A042384; Completed Repair Handbook-AFML-TR-77-206/AFFDL-77-139; Task I of add-on-AFML-TR-78-7.

The add-on work was accomplished under the sponsorship of the Air Force Materials Laboratory (Project 7381/Task 06). Mr. W. Scardino, AFML/MXE, was the project engineer.

Mr. J. E. McCarty was the Boeing Program manager, with Mr. R. E. Horton as project engineer. Mr. M. C. Locke was the principal investigator for the add-on program.

The authors wish to acknowledge the efforts of Mr. R. Z. Mayberry and Mr. K. M. Harriman for the fabrication and testing work, and Mr. J. A. Curtis and Mr. E. A. Ledbury for the SEM analysis.

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SECTION I

INTRODUCTION AND SUMMARY

It was recognized during Phase II of the Repair Handbook Contract (F33615-73-C-5171) that additional studies were required to define and optimize the phosphoric acid non-tank anodize (PANTA) processing conditions. Although excellent durability results were obtained using PANTA as compared to other hand clean methods, the scope of the effort did not permit detailed investigation of critical anodizing parameters. As a result, this supplemental program was conducted to evaluate these variables. Parameters investigated included the effects of anodizing voltage, temperature, time, and rinse delay on bond durability. In addition, bondability of various adhesive system/surface preparation combinations was investigated. The data will form a data base for material selection and facilitate the repair process.

This program consists of two major tasks, as shown in figure 1. Task I was to investigate process variable effects on the phosphoric acid non-tank anodizing (PANTA) procedure. Bond verification tests were included. Task II focused on an evaluation of adhesive/surface preparation combinations, including vacuum bag curing to simulate repairs made under reduced temperature and pressure. The two tasks were performed over a 19-month period. The tasks and program schedule are shown in figure 2. The Task I program results were reported in AFML-TR-78-7 (ref. 1). A synopsis of the results is summarized in Section II.

The work accomplished in Task I consisted of the following:

- 1. Phosphoric acid non-tank anodize (PANTA) process variables investigation
- Determination of the effects of anodizing potential, temperature, and time
- Determination of the effects of rinse delay time, part size (to 24 x 24 in.), and anodizing mode, i.e., vertical, horizontal, etc.
- A scanning electron microscope (SEM) examination of oxide characteristics
- Evaluation of anodizing clad versus bare
- Anodizing with batteries
- Effect of anodizing over titanium and aluminum fasteners
- Identification of common errors in the application of PANTA
- 2. Bond verification tests
- Selection of optimum PANTA conditions
- Demonstration of compatibility with adhesive systems

For Task II, the work focused on an evaluation of surface preparation method/adhesive combinations to develop a data base to facilitate repair applications. Task II has been completed and the results are documented in this report. The following work was accomplished in this

task:

- 1. Surface preparation
- Optimized FPL etch-baseline
- Phosphoric acid non-tank anodize
- PasaJell 105 (non-tank)
- 2. Adhesive systems
- 250°F cure–AF 127-3, FM 73, EA 9628
- 350°F cure–AF 130, FM 400, FM 300, AF 143
- Room-temperature cure-EA 9320, EA 934
- 3. Cure conditions
- Autoclave bonds
- Vacuum bag bonds
- 4. Tests
- Bond strength-lap shear, L/t lap shear, metal-metal peel
- Durability-sustained stressed lap shear, wedge
- Environments-120°F/100% RH, salt spray (5% NaCl, 95°F), -67°F, 180°F, 350°F, room temperature (70-75°F)

No significant differences were noted among the three different surface preparations in their initial, unexposed bond strength. Surfaces prepared with PasaJell 105, however, resulted in reduced bond durability.

Bonds made under vacuum bag cure (reduced temperature and pressure) showed lower bond strength. Reductions in cycles to failure were also found in earlier work (ref. 3) in the vacuum bag bonds as compared to autoclave bonds. This points out an inherent problem in making vacuum bag bonded repairs—reduced strength and durability can be expected. This can be compensated for by keeping the stresses low in the splice bond. The adhesive systems that were tested showed that they are compatible with the evaluated surface preparations.

SECTION II

SYNOPSIS OF TASK I RESULTS*

The purpose of Task I was to investigate the process variables affecting the phosphoric acid non-tank anodize procedure. The effects of varying process parameters on bondability were assessed using the wedge crack extension test. Prior to bonding into wedge test assemblies, anodized surfaces were inspected for interference color, tape tested using 3M Company No. 250 tape, and examined for oxide characteristics with the scanning electron microscope (SEM).

Task I was conducted in two parts. The following processing parameters were investigated in Task I, Part A:

- Effect of voltage variation
- Effect of anodizing temperature
- Effect of anodizing time
- Effect of rinse delay
- Effect of part size
- Anodizing mode
- Dry cell battery anodizing
- Effect of anodizing over titanium and aluminum fasteners
- Identify common errors in PANTA application

Following the processing parameter study, bond verification tests were conducted as Part B of Task I using optimized PANTA surface preparation conditions. For the bond verification tests, both 2024-T3 clad and 7075-T6 bare aluminum adherends were used. Selected adhesive systems from room-temperature cure, 250°F cure, and 350°F cure materials were evaluated.

2.1 PROCESS VARIABLES INVESTIGATION

This part of Task I was aimed at an investigation of the processing parameters that affect the application and bond durability of PANTA-treated surfaces. Specific conclusions and results relating to individual processing parameters were reported in reference 1. Figure 3 shows the typical processing sequence for the Part A investigation. Table 1 shows a summary of the variables and processing conditions investigated. Based on the results of this investigation, including the SEM analysis of oxide characteristics, wedge test, tape test, and color inspection, recommended processing conditions are given. On the basis of the results, the following conditions were selected for the Part B bond verification tests:

- Voltage: 6 volts
- Temperature: 70-80°F

*Reported in AFML-TR-78-7 (ref. 1).

- Time: 10 minutes
- Rinse delay: less than 5 minutes
- Gelled phosphoric acid, 10-12% concentration: (PR 50 from Products Research)

A brief review of the more critical parameters affecting the PANTA process is given here:

2.1.1 VOLTAGE POTENTIAL

Applied voltages of 1, 2, 4 (baseline), 6, and 10 volts were investigated. Figures 4 and 5 compare the oxide characteristics on 7075-T6 bare and 2024-T3 clad as a function of voltage. It can be seen that oxide uniformity is increased as voltage increases. Little or very thin oxide was formed at 1 volt on 7075-T6 bare aluminum. These surfaces also produced wedge test failures. Figure 6 illustrates the effect of voltage potential on oxide thickness. These results provided the basis for increasing the voltage to 6 volts from the baseline 4 volts (ref. 1).

2.1.2 ANODIZING TEMPERATURE

Two other anodizing temperature conditions were evaluated in addition to ambient room temperature (70° F-80° F). Two temperature extremes (100° F and 40° F) were selected as conditions that may be encountered in repair situations. Wedge test failures were experienced on 100° F, 1- and 2-volt anodized surfaces (both bare and clad) and clad surfaces anodized at 40° F (ref. 1). Due to these sporadic failures, and SEM photomicrographs (see figs. 7-10) of oxides formed at these temperatures, it is recommended that the present process be limited to ambient room-temperature (70° F-80° F) conditions.

2.1.3 ANODIZING TIME

From the baseline 10 minutes, anodizing time was varied to 1, 5, 15, and 20 minutes to determine if shorter or longer times would affect oxide formation and bondability. Both bare and clad wedge specimens showed failure when anodized for 1 minute. Sporadic failures were noted on the clad, 5-minute anodized specimens. No apparent difference was observed on the 15- or 20-minute anodized specimens. The short time (1 to 5 minutes) is inadequate to form environmentally stable oxides (see figs. 11 and 12).

2.1.4 RINSE DELAY

The effects of rinse delay times of 2, 5, and 10 minutes were investigated and compared to nodelay cases. Except for wedge test failures on the 10-minute rinse delay specimens, no other failures were noted. The SEM photomicrographs (figs. 13 and 14) show increasing amounts of loosely adhering oxide to the substrate from no delay to 10 minutes delay. As reported by other investigators (ref. 2), phosphoric acid will dissolve the aluminum oxide formed if the acid is left on the surface after the current is turned off. For optimum processing conditions, no rinse delay or less than 5 minutes rinse delay is recommended.

2.1.5 SUMMARY OF PROCESS VARIABLES INVESTIGATION

Table 1 shows a summary of the variables and processing conditions investigated. Recommended processing limits are given. The results indicated that the phosphoric acid non-tank anodize (PANTA) process can be satisfactorily effected under varying conditions. These include variations in voltage potential (4 to 6 volts), temperature (70° F- 80° F), anodizing time (10-15 min), and rinse delay time (0-5 min); over large surfaces (2- x 2-ft); vertical or horizon-tal surface orientation; over titanium and aluminum fasteners; and using dry cell batteries.

2.2 BOND VERIFICATION TESTS

The purpose of Part B of Task I was to demonstrate process verification of the PANTA procedures and conditions selected from Part A. Table 2 shows the test matrix evaluated on representative room-temperature, 250°F, and 350°F cure adhesive systems. Tests were selected to provide bond strength data, e.g., lap shear, bond durability (sustained stressed lap shear), and compatibility (wedge and metal-metal peel). The comparative bondability performance of clad versus bare aluminum adherends was investigated.

Table 3 illustrates the data summary obtained on the different adhesive/primer systems. No apparent incompatibility in the bond strength or durability exposure on the non-tank anodized surfaces was noted. The optimum anodize conditions used produced environmentally durable bonds and reproducible bonding surfaces.

SECTION III EVALUATION OF ADHESIVE/SURFACE PREPARATION COMBINATIONS FOR ALUMINUM

This part of the program was aimed at an evaluation of the adhesive/surface preparation combinations for bonded repairs. The objective was to develop a data base on representative roomtemperature, 250°F and 350°F cure adhesive systems using three surface preparation methods. These included two hand-clean methods, phosphoric acid non-tank anodize and PasaJell 105, and the optimized FPL etch tank process. Special emphasis was placed on the non-tank anodize method. The other hand-clean method, PasaJell 105, was included because it is used in some Air Logistics Centers (ALC's). Optimized FPL etch (Boeing process specification BAC 5514) was chosen as the baseline on the basis of its current availability at the ALC's.

Adhesive systems evaluated include those in use currently as well as improved durability materials. The tests selected provide a balance of bond strength data and bond durability under environmental exposure.

3.1 SURFACE PREPARATION PROCEDURES

Three surface preparation procedures were selected for evaluation, including one tank immersion and two non-tank methods. The processes were selected on the basis of their current usage at the ALC's or with potential repair bonding applications. The following surface preparation processes were investigated.

- Optimized FPL etch (BAC 5514, tank immersion)
- Phosphoric acid non-tank anodize (PANTA)
- PasaJell 105 (non-tank procedure)

Detailed processing procedures are given in the following paragraphs.

3.1.1 OPTIMIZED FPL ETCH

- 1. Vapor degrease or solvent clean.
- 2. Alkaline clean 10 minutes.
- 3. Rinse 5 minutes in tap water, 110°F minimum.
- 4. Deoxidize 12-15 minutes at 150°F-160°F in: Na₂Cr₂O₇. 2H₂O - 4.1 to 12.0 oz/gal H₂SO₄ 66° BE' - 38.5 to 41.5 oz/gal Aluminum (2024 bare) - 0.20 oz/gal dissolved aluminum (minimum)
- 5. Rinse 5 minutes in tap water.
- 6. Dry at 140°F maximum.

3.1.2 PHOSPHORIC ACID NON-TANK ANODIZE (PANTA)

- 1. Solvent wipe with MEK, trichloroethane, or equivalent.
- 2. Abrade with nonwoven abrasive such as nylon abrasive pads or equivalent.
- 3. Dry wipe with clean gauze to remove dust and debris.
- 4. Apply a uniform coat of gelled 12% phosphoric acid or PR 50 (gelled phosphoric acid compound can be purchased from Products Research Corporation or made by adding Cab-O-Sil to acid until thickened) to aluminum surface.
- 5. Place two or three layers of gauze over top of coating; apply another coat of gelled phosphoric acid to completely saturate and wet out the gauze.
- 6. Secure a piece of stainless steel screen over the coating. Apply another coating of the gelled phosphoric acid.
 - NOTE: Be sure that the stainless steel screen DOES NOT CONTACT any part of aluminum surface being anodized.
- 7. Connect screen as cathode (-) and aluminum as anode (+) as shown in figure 16.
 - NOTE: Check this setup before proceeding: screen as cathode (-) and aluminum as anode (+).
- 8. Apply a DC potential of 6 volts for 10 minutes (4 to 6 volts for 10 to 12 minutes are satisfactory).
 - NOTE: A rectifier may be used to supply the voltage and current during anodizing. Current density should be in the range of 1 to 7 amps/sq ft. In an emergency, a fresh or fully charged dry or wet cell battery may be used to anodize small areas.
- 9. At the end of the anodizing time, open the circuit and remove the screen and gauze.
- 10. Moisten clean gauze with water. Lightly wipe off the gelled acid with moistened gauze without delay. The rinse delay time is limited to less than 5 minutes. Do not rub the anodized surface. Immersion or spray rinsing should be used if possible.
- 11. Air dry a minimum of 30 minutes at room temperature or force-air oven dry at 140°F to 160°F.
- 12. Check quality of prepared surface. A properly anodized surface will show an interference color when viewed through a polarizing filter rotated 90° at a low angle of incidence to fluorescent light or daylight. An inspection being accomplished is shown in figure 17.
- 13. If no color is observed, repeat steps 4 through 12.
 - NOTE: Machined surfaces or abraded surfaces sometimes are difficult to inspect for color. Rotation of the polarizing filter is required because some pale shades of yellow or green are so close to white that without a color-change inspection, they might be considered "no color," which would falsely indicate no anodic coating.

CAUTION: DO NOT TOUCH the dried anodized surface. DO NOT apply tape to the surface.

3.1.3 PASAJELL 105 METHOD

- CAUTION: PasaJell 105 can cause severe corrosion when it has been allowed to enter unsealed joints and recesses. Mask those areas prior to use of PasaJell 105. Completely flush the areas to remove all traces of PasaJell 105 after using.
- 1. Solvent clean with MEK, trichloroethane, or other approved solvent.
- 2. Abrade with a nylon abrasive pad or 400-grit aluminum oxide abrasive paper.
- 3. Dry wipe with clean gauze pads.
- 4. Apply PasaJell 105 to surface with a spatula, brush, or gauze.
 - WARNING: WEAR RUBBER GLOVES AND GOGGLES WHEN USING PASAJELL. IF SKIN OR EYES COME IN CONTACT WITH ACID, IMMEDIATELY FLUSH WITH WATER.
- 5. Leave PasaJell 105 on the surface for 10 to 15 minutes.
- 6. Wipe off PasaJell. Do not wipe PasaJell onto adjacent areas or crevices or allow it to enter honeycomb core.
- 7. Dampen a clean gauze pad with clean water and wipe the treated area. Repeat as necessary and check with litmus paper to be sure that all trace of acid has been removed.
- 8. Allow to air dry before applying primer or bonding.

3.2 ADHESIVE/PRIMER SYSTEMS

Adhesive/primer systems were selected for evaluation on the basis of their applicability for repairs and current or future utilization at the ALC's. Three categories of adhesive materials were evaluated:

- 250°F cure–AF 127-3, FM 73, EA 9628, all on BR 127 CIAP primer
- 350°F cure-AF 130/EC 2333, FM 400/BR 400, FM 300/BR 127, AF 143/EC 3917
- Room-temperature cure-EA 9320, EA 934, with BR 127 CIAP primer

These adhesive systems provide a representative spectrum of repair materials currently in use (e.g., AF 127-3 at San Antonio ALC, AF 130 at Sacramento ALC) and upcoming systems. The latter include high-durability 250°F cure adhesives such as FM 73 and EA 9628 or 350°F cure systems such as AF 143. The FM 300 material, a 350°F cure adhesive, is being considered to fill the gap for service temperatures between 180°F and 300°F.

3.3 CURE CONDITIONS

Both autoclave cure bonds and vacuum bag cure bonds were evaluated. The vacuum bag cures were made to simulate repair cure conditions under reduced temperature and pressure. The cure conditions shown in table 4 were investigated for the adhesive/primer systems studied.

3.4 250° CURE ADHESIVE SYSTEMS-AUTOCLAVE BONDS

Table 5 identifies the test matrix used to evaluate 250°F cure adhesive system, autoclave bonded panels. The adhesive systems include one presently used for repairs at the ALC's (AF 127-3) and two new, improved durability systems (FM 73 and EA 9628). In all cases, corrosioninhibiting adhesive primer (BR 127) was used.

Following surface preparation and priming on the aluminum adherends, the panels were bonded into wide-area (24- x 24-in.) lap shear, peel (14- x 17-in.), and wedge (6- x 6-in.) assemblies. Panel and specimen configurations for lap shear, peel, and wedge assemblies are shown in figures 18, 19, and 20.

Tables 6, 7, and 8 summarize the results obtained with AF 127-3, FM 73, and EA 9628. The effects of surface preparation are compared for each adhesive system.

No significant differences were found in the mechanical strength, i.e., stressed lap shear or peel tests, among the three different surface preparation methods. All three adhesive systems showed similar trends.

Test temperature had a significant effect on the bond strength properties. As shown in the figures 21, 22, and 23 L/t lap shear plots, tests at 180° F showed shear strength reductions in all cases. In comparison, a slight increase for the 1/2-in. overlap shear test was noted at -67° F. This trend was observed on all three adhesive systems and the three surface treatments. The characteristic behavior of modified epoxy materials to undergo visoelastic changes probably is responsible for the increased ultimate failure load of the 0.5-inch lap tests at -67° F. However, at the longer overlaps (1 and 2 in.), the influence of peel at the bond joint load-transfer edge prevents this trend from continuing. The -67° F values at these lap lengths therefore are nearly the same as the room-temperature values.

While the mechanical bond strengths were not significantly affected by the surface preparation methods, the bond durability under environmental conditions as evaluated by the wedge test was affected. Under an opening mode stress on the bondline, the influence of surface preparation is significantly demonstrated by the crack extension. The crack extension increased rapidly for PasaJell 105 bonds, particularly with FM 73 and EA 9628, as shown in tables 6, 7, and 8. It was less affected on the AF 127-3 system. Figure 24 illustrates the crack growth characteristics for the three adhesives and three surface treatment methods. Failure modes on the PasaJell 105 were 100% adhesive at the adherend interface. Cohesive failure or center-of-thebond failure modes were observed on the optimized FPL and PANTA bonded specimens.

Bonded specimen failure surfaces are shown in figures 25 through 33 for the three adhesive systems and three surface preparation methods. Comments regarding the mode of failure are as follows:

AF 127-3 System

Figures 25, 26, and 27 for optimized FPL etch, PANTA, and PasaJell 105, respectively. All show cohesive failures except for PasaJell 105 wedge crack specimens after exposure with 100% interfacial or adhesive failure.

FM 73 System

Figures 28, 29, and 30 for optimized FPL etch, PANTA, and PasaJell 105, respectively. All show cohesive failures except for PasaJell 105 wedge specimens with 100% adhesive failure after 4 hours exposure.

• EA 9628 System

Figures 31, 32, and 33 for optimized FPL etch, PANTA, and PasaJell 105, respectively. All show cohesive failures except for PasaJell 105 wedge specimens with 100% adhesive failure after 4 hours exposure.

The intent of the developed data is to serve as a basis for selecting material systems and surface preparation methods for repair bond with consideration for subsequent environmental durability. The latter is important because only certain tests shown environmental stability for bonds as influenced by processing variables. This was clearly demonstrated on the wedge tests with bonds made on PasaJell 105 treated surfaces.

As a further comparison to the wedge test results, metal-to-metal peel specimens (FM 73 adhesive) were tested in the dry/wet conditions using the "Bell" peel method. Figures 34, 35, and 36 are the test charts for these tests. Note that no difference was observed in the peel strengths on the optimized FPL etch and PANTA specimens tested dry and wet. However, on the PasaJell 105 specimen, the peel strength dropped to nearly zero when tested wet, with 100% adhesive, interfacial failure 'Similar failure modes were encountered on the wedge specimens, as shown in figures 28, 29, and 30. Although on certain adhesive systems, e.g., AF 127-3, the environmental durability results were less affected by the PasaJell 105 treatment, in general, the peel test and the wedge test demonstrated the lack of environmental durability on bonds made with PasaJell 105 treated surfaces.

3.5 250°F CURE ADHESIVE SYSTEMS-VACUUM BAG BONDS

The test matrix for this task is shown in table 9. L/t lap shear tests at room temperature, 180° F, and -67° F and stressed lap shear tests were run on the vacuum bag bonded specimens. It was felt that these tests would be adequate to provide a comparison to autoclave bonds, which were used as a baseline. The vacuum bag bonds were made under reduced temperature and pressure, as shown in section 3.3, to simulate repair situations. The 200°F cure temperature reflects repair practice to stay below the original bond cure temperature to avoid possible thermal degradation of the initial bond. The vacuum bag pressure (23-28 in. Hg) represents vacuum heat blanket capability.

The adhesive systems and surface preparations were the same as those used for the autoclave cured bonds. Tables 10, 11, and 12 summarize the test results, and the L/t versus test temperature data are plotted in figures 37, 38, and 39. A slight reduction in the bond strengths occured on the PasaJell 105 specimens for all adhesive systems. This reduction is also noted on the sustained stressed lap shear tests. The latter were residual strengths determined on specimens after the 60-day sustained stressed exposure. None of the specimens experienced any failure within the 60-day exposure time.

Failure surfaces are shown in figures 40 through 48 for the three adhesive systems and three surface treatments. Generally, failure modes were cohesive, center-of-the-bond type. Some instances of adhesive-primer failure were observed on the longer (2 in.) overlap specimens, particularly when tested at -67°F. This type of failure was more prominent on the PasaJell 105 specimens than on the FPL etched or PANTA specimens. This correlates to the marginal surface preparation results using PasaJell 105 on wedge and peel specimens.

A comparison was made between the vacuum bag bonds and the autoclave bonds. A reduction in shear strength was found in all cases on vacuum bag bonds. The reduced cure temperature and pressure resulted in a strength reduction ranging from 5% to 15%. This reduction was also noted in the PABST repair program (ref. 3), in which a significant reduction in cycles to failure was found on vacuum bag bonds versus autoclave bonds. The design of repair bonds made under vacuum bag conditions must consider this reduction. The effect of the quality of the surface treatment used also must be considered.

3.6 350°F CURE ADHESIVE SYSTEMS-AUTOCLAVE BONDS

The test matrix for this task is shown in table 13. Four 350°F cure systems were evaluated including AF 130, FM 400, AF 143, and FM 300. All systems are intended for elevated-temperatuer service to 350°F except for FM 300. The latter is a new adhesive being considered to fill the service temperature gap between 180°F and 300°F. The FM 300 system has improved toughness, i.e., higher peel strength, compared to the other 350°F cure systems evaluated.

As in earlier tasks with 250°F cure systems, three surface preparation methods were evaluated. Optimized FPL etch served as the baseline with PANTA and PasaJell 105 as the hand-clean methods. Mechanical strength tests and stressed durability tests were conducted.

Tables 14, 15, 16, and 17 summarize the results of this evaluation. Except for FM 300, all showed the characteristic low peel, low toughness typical of 350° F cure systems used for high-temperature applications. Further, all except FM 300 were unaffected by the elevated test temperature (350° F). Since the FM 300 was designed for a service temperature range of 180° F to 300° F, additional tests were run at 250° and 300° F to determine the effect of temperature. Also, since BR 127 CIAP primer (a 250° F curing system) was used with this system, tests were run with and without BR 127 to evaluate the degradation of the CIAP primer at high temperatures. These results are plotted in figures 49 and 50. The shear strength showed a significant dropoff from 250° F to 350° F. Lower values were obtained on the unprimed specimens. With both primed and unprimed specimens, higher values were obtained on the anodized surfaces than on the optimized FPL etch surfaces.

Failure surfaces of representative specimens are shown in figures 51 through 62 for the four adhesive systems and three surface treatments. General comments about the failure modes and comparative bond durability are given below.

AF 130/EC 2333 System

Figures 51, 52, and 53 show bonded surfaces for optimized FPL etch, PANTA, and Pasa-Jell 105 specimens, respectively. Mixed failure modes were observed on these specimens. Most specimens had cohesive failures, i.e., center of the bond or near the adhesive-primer interface. A few "slick" failures were noted on -67° F test specimens on all three surface preparations. Comparable results were obtained with all three surface treatments.

FM 400/BR 400 System

Typical failures modes of tested specimens are shown in figures 54, 55, and 56. Mixed failure modes were common with this adhesive system. Some "slick" failures had primeradhesive on the surface. Again, no significant differences in the test results were found among the three surface preparations, including the stressed lap shear and wedge tests. Large scatter in the data is evident from the large standard deviations shown in table 15.

FM 300/BR 127 System

Figures 57, 58, and 59 show typical failure surfaces for optimized FPL, PANTA, and Pasa-Jell 105 treated specimens. Failure modes were mostly cohesive, center of the bond, or at the primer-adhesive interface. Failure surfaces on specimens tested at 350° F showed apparent softening of the adhesive not observed on the other 350° F adhesive systems. The 350° F test results were extremely low. It was due to these results that additional tests were run at 250° F and 300° F (see figs. 49 and 50) to determine the effect of temperature. A definite reduction can be seen in figures 49 and 50 in the temperature range of 250° F to 350° F. Again, no significant difference was noted among the three surface preparations.

AF 143/EC 3917 System

Figures 60, 61, and 62 show the typical failure bond surface of specimens tested under various conditions for optimized FPL etch, PANTA, and PasaJell 105, respectively. As with the other adhesive systems, mixed failure modes were observed. Most failures were cohesive except for some "slick" type adhesive failures for the -67°F tests. In general, the test results showed less scatter than with the AF 130 or FM 400 systems. Some improvement in toughness is seen in the peel test values. Except for the peel values, results comparable to FM 400 were obtained.

3.7 350°F CURED ADHESIVE SYSTEMS-VACUUM BAG BONDS

The test matrix for this task is shown in table 18 with three adhesive systems evaluated under vacuum bag bond conditions. The cure temperatures and pressures are shown in table 4. The primary emphasis was to simulate repair conditions where vacuum heating blankets may be used and the cure is to be made at reduced temperatures. Test results are shown in tables 19, 20, and 21 for AF 130, FM 400, and AF 143, respectively.

A slight reduction in the bond strength was observed for the vacuum bag cured AF 130 and FM 400 specimens as compared to the autoclave bonds. No difference was noted on the AF 143 specimens. On the basis of these results, it appears that the 300°F cure was adequate to cure the bonds and vacuum bag pressure was satisfactory.

Typical bond failure surfaces are shown in figures 63 through 71. General comments regarding failure modes are noted below.

AF 130 System

Figures 63, 64, and 65 show the bond failure surfaces for optimized FPL etch, PANTA, and PasaJell 105, respectively. As seen previously in the autoclave bond specimens, mixed failure modes were observed on these specimens. The adhesive on the AF 130 seemed to separate from the carrier often. A few "slick" type failures were noted on the -67°F tests. Even these appeared to have polymer adhesive on the adherend surface.

FM 400 System

The bond failure surfaces are shown in figures 66, 67, and 68 for the three surface preparations. Generally, failure modes were similar to the autoclave bonds where mixed modes (cohesive and adhesive) were observed. Typically, the longer overlap length (1- and 2-in.) specimens showed larger amounts of adhesive-type failure, near the load transfer tips. No significant differences were found among the different surface preparations, although in some cases a little reduction was shown on PasaJell 105 specimens.

AF 143 System

Figures 69, 70, and 71 show the bond failure surfaces on the three surface preparations evaluated. Failure modes were similar to the autoclave bond specimens. Test results showed similar trends as for autoclave bonds when compared for each surface preparation (see tables 17 and 21). The 300°F cure appears to be satisfactory in fully curing the adhesive. The test results showed no significant difference in vacuum bag pressure bonded specimens.

3.8 ROOM-TEMPERATURE CURE ADHESIVE SYSTEMS

Table 22 identifies the test matrix used to evaluate two RT cure adhesive systems. EA 9320 is intended for lower service temperature (less than 180°F) applications, and EA 934 is used for higher temperature service. Both adhesive systems are used for repairs at various facilities.

Following surface preparation, BR 127 CIAP primer was applied to all bonding surfaces Widearea (24- x 24-in.) lap shear assemblies, metal-metal peel (14 x 17 in.) and wedge assemblies (6 x 6 in.) were fabricated. A positioning fabric, Monsanto Cerex 3603-23, was used to control bondline thickness. Curing was accomplished under vacuum bag pressure (approximately 20-27 in. Hg). Panel and specimen configurations are as shown in figures 18, 19, and 20.

Tables 23 and 24 summarize the results obtained with EA 9320 and EA 934, respectively. The effects of surface preparation, test temperature, and test environment are compared. Both ad-

hesive systems showed the characteristic reduction in mechanical bond strengths when tested at elevated temperature. Also, EA 934 showed its brittle behavior or lower toughness in the peel strengths and the initial crack lengths in the wedge specimens.

Due to the large amount of scatter in the data and the appearance of "adhesive-starved" bondlines, additional specimens were made and retested for comparison. As shown in figure 72, typical bondline thicknesses of the original specimens were measured to be 4-6 mils. The retest specimens had bondline thicknesses of approximately 10-12 mils. Generally, improved bond strengths were obtained on the thicker bondline specimens. It should be pointed out, however, that in fabricating the assemblies, much difficulty was encountered in an attempt to achieve uniform, void-free bonds. This is especially true for the larger panels such as the wide-area lap shear or metal-metal peel (24×24 in. or 14×17 in.).

Failure surfaces of typical specimens are shown in figures 73 through 78 for the three surface preparation methods and two adhesive systems. Some general comments on the failure modes are discussed below.

EA 9320 System

Figures 73, 74, and 75 show bonded surfaces of typical specimens after test from optimized FPL etch, PANTA, and PasaJell 105 treatments. Nearly all specimens had cohesive failure modes with a few specimens failing near the primer-adhesive interface on the -67°F tests. Some reduction in bond strength and durability on the PasaJell 105 specimens was observed. This is obvious in the peel and stressed lap shear residual strength tests (see table 23). Data on the optimized FPL etch and PANTA treated surfaces appeared similar.

EA 934 System

Typical bond failure surfaces are shown in figures 76, 77, and 78 for the three surface preparations evaluated. As with EA 9320, mixed failure modes were observed with most of the specimens exhibiting cohesive failures. Few primer-adhesive failures were observed on the -67° F tests and wedge specimens. No significant difference was found in the test results.

3.9 SUMMARY OF ADHESIVE/SURFACE PREPARATION COMBINATIONS

Based on the results of this task, the following general summary is concluded:

- Surface Preparation
 - Optimized FPL Etch
 - Phosphoric Acid Non-Tank Anodize
 - PasaJell 105

Satisfactory

Satisfactory

Reduced bond strength and durability when used with the 250°F cure adhesive systems

- Cure Conditions
 - Autoclave Bonds
 - Vacuum Bag Bonds
- Adhesive Systems
 - 250°F Cure
 - 350°F Cure
 - RT Cure

Satisfactory Reduced bond strength

All compatible with surface preparation and cure conditions

SECTION IV

CONCLUSIONS

Work accomplished in this program resulted in the following general conclusions. Specific conclusion relating to individual tasks have been presented in sections II and III and in the Interim Report (ref. 1).

- The phosphoric acid non-tank anodize (PANTA) process was evaluated over varying conditions of voltage potential, temperature range, anodizing time, and rinse delay time. Extreme high or low anodizing temperatures, long rinse delay time, short anodizing time, or low voltage potential produced sporadic bond failures.
- The PANTA surface treatment produced durable bonds on clad or bare aluminum alloys; on surfaces oriented in various positions, e.g., vertical or overhead; using dry cell batteries; and over large surfaces (2 x 2 ft.).
- Of the three surface treatments evaluated, phosphoric acid non-tank anodize (PANTA) showed overall better performance than PasaJell 105, and comparable results to optimized FPL etch (tank process) when used with the 250°F cure adhesive systems. Comparable results were obtained with all three surface treatments when they were used with the R.T. and the 350°F cure adhesives.
- No incompatibility was observed with the adhesive systems evaluated on the three surface treatments and cure conditions.
- Vacuum bag bonds exhibited reduced bond strength and durability as compared to autoclave bonds.
- SEM analysis of oxide characteristics showed that optimum non-tank anodizing conditions produced environmentally stable oxide with good bond durability.





Figure 1.-Add-On Program Tasks

Figure 2.—Program Schedule



Figure 3.-Typical Processing Sequence

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Figure 4.-SEM Photomicrograph of Oxide as a Function of Voltage-7075-76 Bare

Phosphoric acid non-tank anodize 2024-T3 Clad 5000X



Figure 5.-SEM Photomicrograph of Oxide as a Function of Voltage-2024-T3 Clad













Figure 11.-SEM Photomicrograph-Anodize Time Variation-7075-T6 Bare


Figure 12.-SEM Photomicrograph-Anodize Time Variation-2024-T3 Clad





Tank Processes, Aluminum Surface Preparation



Nontank Aluminum Surface Preparation



Figure 15.--Surface Preparation Processing Steps

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Figure 16.-Cross Section of Non-Tank Anodize Set Up-Typical



Figure 17.-Polarized Light Test-Verification of Anodic Oxide Film



All coded areas are 1×7 inches minimum. Typical cut widths should be 1.06 inch minimum to provide excess for milling the edges.



Figure 18.-Lap Shear Panel/Specimen Configuration

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Figure 19.—Metal-Metal Peel Panel/Specimen Configuration



Figure 20.-Wedge Panel/Specimen Configuration



Figure 21.-L/t Lap Shear-AF 127-3/BR 127, Autoclave Bonds



Figure 22.-L/t Lap Shear-FM 73/BR 127, Autoclave Bonds



Figure 23.-L/t Lap Shear-EA 9628/BR 127, Autoclave Bonds



Figure 24.-Wedge Test Comparison-250°F Cure Adhesive Systems



Figure 25.-Specimen Failure Surfaces-AF 127-3, Optimized FPL Etch, Autoclave Bonds



Figure 26.-Specimen Failure Surfaces-AF 127-3, Non-Tank Anodized, Autoclave Bonds



Figure 27.-Specimen Failure Surfaces-AF 127-3, PasaJell 105, Autoclave Bonds



Figure 28.-Specimen Failure Surfaces-FM 73, Optimized FPL Etch, Autoclave Bonds

- Andrew Providence





Figure 30.-Specimen Failure Surfaces-FM 73, PasaJell 105, Autoclave Bonds





Figure 32.-Specimen Failure Surfaces-EA 9628, Non-Tank Anodized, Autoclave Bonds



Figure 33.-Specimen Failure Surfaces-EA 9628, PasaJell 105, Autoclave Bonds

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Figure 34.-Dry/Wet Peel-FM 73, Optimized FPL Etch



Figure 35.-Dry/Wet Peel-FM 73, Non-Tank Anodized



Figure 36.-Dry/Wet Peel-FM 73, PasaJell 105



Figure 37.-L/t Lap Shear-AF 127-3/BR 127 Vacuum Bag Bonds



Figure 38.-L/t Lap Shear-FM 73/BR 127, Vacuum Bag Bonds



Figure 39.-L/t Lap Shear-EA 9628/BR 127, Vacuum Bag Bonds



Figure 40.-Specimen Failure Surfaces-AF 127-3, Optimized FPL Etch, Vacuum Bag Bonds

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Figure 49.-Effect of Temperature on Shear Strength-With Primer, FM 300/BR 127



Figure 50.-Effect of Temperature on Shear Strength-No Primer, FM 300



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Figure 51.-Specimen Failure Surfaces-AF 130, Optimized FPL Etch, Autoclave Bonds








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Figure 56.-Specimen Failure Surfaces-FM 400, PasaJell 105, Autoclave Bonds







Figure 58.-Specimen Failure Surfaces-FM 300, Non-Tank Anodized, Autoclave Bonds



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Figure 63.-Specimen Failure Surfaces-AF 130, Optimized FPL Etch, Vacuum Bag Bonds







Figure 65.-Specimen Failure Surfaces-AF 130, PasaJell 105, Vacuum Bag Bonds









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Figure 69.-Specimen Failure Surfaces-AF 143, Optimized FPL Etch, Vacuum Bag Bonds











Figure 73.-Specimen Failure Surfaces-EA 9320, Optimized FPL Etch, RT Bonds

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Figure 76.-Specimen Failure Surfaces-EA 934, Optimized FPL Etch, RT Bonds





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Calendary Press

Process variable	Conditions investigation	Recommended processing conditions
Voltage	1, 2, 4, 6, 10 volts	4 - 6 volts
Temperature	40, RT(70-80), 100°F	70 - 80°F
Anodizing time	1, 5, 10, 15, 20 min	10 - 15 min
Rinse delay	0, 2, 5, 10 min	less than 5 min
Part size	6" × 6", 6" × 12", 24" × 24"	All satisfactory
Anodize mode	Vertical	Satisfactory
	Horizontal	Satisfactory
	Both surfaces	
영양 전 영양 이 방송 같은 모양 전에	Bottom	한 명령 성격 전 영국 것이 없는 것
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Battery anodizing	2, 4, 6, 12 volts	6 and 12 volts
Anodizing over	Ti and Al	Satisfactory
fasteners		
Bare vs clad	7076-T6 bare and	Satisfactory
	2024-T3 clad	
Common error	4 conditions identified	Corrective actions provided

Table 1.-Summary of Variables and Processing Conditions Investigated

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0.5-inch lap shear Wedge M/M peel	T 180°F 1200 psi 120°F/100% RH RT _67°F Salt spray	x x x x x	X X X X X	X X X X X	(350°F) X X X X X X	x x x x x	x x x x x	X X X X X X	X X X X X	(350°F) X X X X X X	x x x x x
Wedg	1200 psi 120° F/ 10 stress	×	× ×	×	×	××	×	×	××	×	×
.5-inch lap shear	180° F	×	×	×	(350°F)	×	×	×	×	(350°F)	×
0	RT	×	×	×	×	×	×	×	×	×	×
	Adhesive	AF 127-3	EA 9601	FM 73	AF 130	EA 9320	AF 127-3	EA 9601	FM 73	AF 130	EA 9320
	Alloy		2024-T3	Clad				7075-TG	Bare		

Table 2.-Bond Verification Test Matrix

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🕑 EA 9320 –

250°F cure 1 AF 127-3 EA 9601 FM 73

350° cure I AF 130

120 F/100% RH exposure, 4 specimens each all others are 5 specimens. 600 psi for EA 9320 A

Table 3.-Summary Bond Verification Tests

													-
		30 day	0.13	0.14	0.40	0.42	0.25	0.30	0	0	0.65	0.50	
% RH	th, in.	14 day	0.10	0.11	0.28	0.32	0.22	0.24	0	0	0.50	0.29	COO aci
€/100	ck grow	24 hr	0.07	0.04	0.18	0.20	0.15	0.15	0	0	0.13	0.12	6
test -120	Cra	4 hr	0.05	0.01	0.18	0.20	0.10	0.11	0	0	0.06	0.12	EO°E
Wedge 1		1 hr	0.01	0.01	0.16	0.18	0.08	0.11	0	0	0.06	0.12	act at 3
	Initial	inches	1.38	1.48	1.33	1.46	1.59	1.69	2.52	2.85	1.34	1.52	4
peel	14 day salt	spray	64	52	74	28	4	45	œ	œ	57	47	0°F
tal-metal p iń./in.	1910	1./0-	20	42	33	9	80	9	12	6	7	4	min 70-8
PE We	ł	2	99	51	79	60	56	39	ব	ю	57	48	volte 10
si	1200 psi	stress	4400	4900	3634	3991	4810	5275	3040	3244	3545	3630	PANTA 6
p shear, p	19/05		3560	3818	2705	2870	3528	3664	2074	1817	775	554	each test
e1	μ		4670	5080	4452	4932	4744	5340	1668	1646	3496	3608	specimens
Athenia	primer		FM 73-/ BR 127	FM 73/ BR 127	AF 127-3/ BR 127	AF 127-3/ BR 127	EA 9601/ BR 127	EA 9601/ BR 127	AF 130/ EC 2333	AF 130/ EC 2333	EA 9320/ BR 127	EA 9320/ BR 127	Average of 5
	Adherend		2024T3 clad	7075T6 bare	2024T3 clad	7075T6 bare	2024T3 clad	7075T6 bare	2024T3 clad	7075T6 bare	2024T3 clad	7075T6 bare	NOTE:

Table 4.—Cure Conditions	or Adhesive/Primer Systems
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Cure Method	Materials	Cure	e Conditions
Autoclave Bonds	Adhesives AF 127-3 FM 73 EA 9628	Temperature rise: Cure: Pressure:	4-6°F/min 225-250°F for 90 min 35-50 psi
	AF 130 FM 400 FM 300 AF 143	Temperature rise: Cure: Pressure:	4-6°F/min 340-360°F for 60 min 35-50 psi
Vacuum Bag Bonds	Adhesives AF 127-3 FM 73 EA 9628	Temperature rise: Cure: Pressure:	4-6°F/min 200°F for 2 hrs 10-14 psi vacuum bag pressure only
	AF 130 FM 400 AF 143	Temperature rise: Cure: Pressure:	4-6°F/min 300°F for 2 hrs 10-14 psi vacuum bag pressure only
Oven Bake	Primers BR 127	Application: Cure: Thickness:	Spray Air dry 1/2 hr, then cure at 250°F for 1 hr 0.1 to 0.4 mil
	EC 2333	Application: Cure: Thickness:	Spray Air dry, then bake at 160°F for 30 min 0.1 to 0.4 mil
	BR 400	Application: Cure: Thickness:	Spray Air dry, then bake at 200°F for 30 min 0.1 to 0.2 mil
	EC 3917	Application: Cure: Thickness:	Spray Air dry, then bake at 250°F for 1 hr 0.1-0.4 mil
RT Cure	Adhesives EA 9320 EA 934	Temperature: Cure: Pressure:	Ambient RT (70-75°F) 7 days 10-14 psi vacuum bag pressure only

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		0.1	5-in Lap) Shear	L/t L -67°F	ap She	ar 180°F	Wedge	Metal-N	Metal Peel
Surface Preparation Methods	Adhesive System/ Primer	RT	180°F	60 Days 1200 psi Stress, 120°F/ 100% RH	0.5	1.0	2.0	120°F/ 100% RH	RT	Salt Spray, 14 Days
o Optimized FPL Etch (Tank)	AF 127-3/ BR 127	15	15	12	45	45	45	15	15	15
o Phosphoric Acid Anodize (Non-Tank) o PasaJell 105	FM 73/ BR 127	15	15	12	45	45	45	15	15	15
	EA 9628/ BR 127	15	15	12	45	45	45	15	15	15
Total Specimens = 666		45	45	36	135	135	135	45	45	45

NOTE: 5 specimens each test except 4 for sustained stressed lap shear.

Table 6.-Data Summary-AF 127, Autoclave Bonds

		30 days	0.50			0.53			0.87		
inch	Ŧ	14 days	0.50			0.43			0.79		
tension,	F/100% R	24 hours	0.28			0.33			0.59		
Crack Ex	120°	4 hours	0.15			0.20			0.29		
Wedge		hour	0.04			0.07			0.07		
		Initial	1.45			1.46			1.42		
eel. ./in.	Salt Spray 14 days		57.9 2.0			54.0 1.5			56.1 2.3		
M-M P 1b-in		RT	54 1.2			53.8 3.6			51.8		
ear, 1b 180°F	2.0 in.		8176 255	5452 215	6754 622	8012 58	5578 61	6554 737	7768 423	5500 169	4438 610
	1.0 in.		4322 21	2632 48	4020 65	4384 81	2948 94	4454 320	4350 61	2824 102	3514 138
Lap Sh °F, RT,	0.5 in.		2482 43	1624 22	3268 272	2522 37	1626 27	3456 131	2538 30	1620 52	3160 231
L/t -67		Test Temp.	RT	180°F	-67°F	RT	180°F	-67°F	RT	180°F	-67°F
hear, psi	1200 bei	Stress, 60 days	4395 154			4267 234			4055 209		
in Lap SI		180°F	3048 135			3300 108			3320 135		
0.5-		RT	4948 48			5064			5044 138		
	Surface					Phosphoric acid non-	cally allouize		PasaJell 105		
		Adhesive/ Primer	AF 127-3/ BR 127								

NOTE:

o Average of 5 specimens each test except 4 for stressed lap shear.

o 7075-76 bare.

o Sub values in the table indicate standard deviation.

Table 7.-Data Summary-FM 73/BR 127, Autoclave Bonds

								_				
		30 days	0.12			0.07			2.24			
inch	н	14 days	0.05			0.04			2.17			
tension,	F/100% R	24 hours	0			0			2.09			
Crack Ext	120°1	4 hours	0			0			1.10			
Wedge (hour	0			0	0			0.15		
		Initial	1.49			1.52			1.44			
eel, ./in.	+Leo	Spray 14 days	44.7 2.9			43.5			45.6 0.8			
M-M Pe lb-in		RT	45.0 2.1			40.0	40.0			45.6 1.3		
ar, 1b 180°F	2.0 in.		7274 327	5654 229	7278 339	7238 212	5666 158	7250	6970 217	5602 221	6706 165	
	1.0 in.		4368 87	3124 85	4614 205	4306	308 4 50	4494 179	4268 67	3046 105	4536 47	
Lap She F, RT,	0.5 in.		2538 58	1992 36	3422 96	2410 31	1830 60	3268 108	2452 53	1924 49	3280 110	
L/t -67	Test Temp.		RT	180°F	-67°F	RT	180°F	-67°F	RT	180°F	-67°F	
ear, psi	1200 Det	Stress, 60 days	4615 209			4695 128			4590 189			
n Lap Sh		180°F	3948 21			3796 33			3748 27			
0.5-i		RT	5036 57			4884 163			5000 158			
	Surface Preparation R		Optimized FPL etch			Phosphoric acid non-	Callk anouize		PasaJell 105			
		Adhesive/ Primer	FM 73/ BR 127									

NOTE:

o Average of 5 specimens each test except 4 for stressed lap shear.

o 7075-T6 bare.

o Sub values in the table indicate standard deviation.

Autoclave Bonds
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Table .

								+				
		30 days	0.56			0.15			2.25			
inch	Ŧ	14 days	0.48			0.11			2.18			
tension,	-/100% R	24 hours	0.23			0.09			1.75			
Crack Ext	120°1	4 hours	0.15			0.02			0.83			
Wedge		1 hour	0.03			0			0.07			
		Initial	1.47			1.52			1.51			
eel. ./in.	Salt Spray 14 days		55.7 1.4			54.9 2.3	54.9 2.3					
M-M P lb-in	M-M F 1b-ir		58.0			52.8 2.9			55.8 2.4			
	2.0 in.		7828 138	6094 62	6852 290	7808 273	6164 98	7170	7188 245	6284 65	3970 422	
ear, 1b 180°F	1.0 in.		4824 31	3350 61	4810 32	4692 27	3300 10	4676 149	4536 116	3276 39	3120 672	
°F, RT,	0.5 in.		2774 27	2040 37	3682 37	2722 34	1984 9	3652 26	2666 36	1896 31	2906 323	
L/t -67	Test Temp.		RT	180°F	-67°F	RT	180°F	-67°F	RT	180°F	-67°F	
ear, psi	1200 psi Stress, 60 days		5070 279	5070 279			5000			4 750 285		
n Lap Sh		180°F	4024 93			4056 70	4056 70			3696 234		
0.5-i		RT	5516 41			5432 67			5252 119			
		Surface Preparation	Optimized FPL etch			Phosphoric acid non-	Lank anou 26		Pasajell 105			
		Adhesive/ Primer	EA 9628/ BR 127									

NOTE:

o Average of 5 specimens each test except 4 for stressed lap shear.

o 7075-T6 bare.

o Sub values in the table indicate standard deviation.
		L/t L -67°F	ap She , RT,	ar 180°F	0.5-in Lap Shear
Surface Preparation Methods	Adhesive System/ Primer	0.5	1.0	2.0	60 Days, 1200 psi Stress 120°F/100% RH
o Optimized FPL Etch (Tank)	AF 127-3/ BR 127	75	45	45	12
o Phosphoric Acid Anodize (Non-Tank) o PasaJell 105	FM 73/ BR 127	75	45	45	12
	EA 9628/ BR 127	75	45	45	12
Total Specimens = 531		225	135	135	36

Table 9.-Test Matrix, 250° F Cure Adhesive Systems-Vacuum Bag Bonds

NOTE: 5 specimens each test except 10 for RT and 180°F 1/2-in. overlap and 4 each for sustained stressed lap shear.

			L/t La	ap Shea	r - 1b	60-day, 1200
Adhesive/ Primer	Surface Preparation	Test Temp.	0.5 in.	1.0 in.	2.0 in.	lap shear, 120°F/100% RH
AF 127-3/ BR 127	Optimized FPL etch	RT	2467 96	3960 376	7376 446	4390 252
		180°F	1558 56	2644 296	5264 176	
		-67°F	3178 42	4198 402	6896 747	
	Phosphoric acid non-	RT	2390 244	4194 75	7372 532	4450 129
		180°F	1553 67	2770 90	4964 667	
		-67°F	3178 127	3932 341	5880 772	
	PasaJell 105	RT	2194 266	3632 331	6724 428	3745 654
		180°F	1449 201	2688 487	5604 453	
		-67°F	2360 260	2914 285	3658 318	

Table 10.-Data Summary-AF 127-3/BR 127, Vacuum Bag Bonds

NOTE:

• Average of 10 specimens each test for RT and 180°F, 1/2-in. overlap; others are 5 specimens each except 4 for stressed lap shear.

o 7075-T6 bare.

			L/t La	ap Shea	r - 1b	60-day, 1200
Adhesive/ Primer	Surface Preparation	Test Temp.	0.5 in.	1.0 in.	2.0 in.	lap shear, 120°F/100% RH
FM 73/ BR 127	Optimized FPL etch	RT	2400 51	3964 100	6542 282	4380 375
		180°F	1786 81	2788 97	5118 191	
		-67°F	3144 84	3908 50	5938 321	
	Phosphoric acid non-	RT	2274 45	3658 106	6080 147	4560 347
		180°F	1681 47	2604 21	4902 33	
		-67°F	3074 42	3860 105	5658 366	
	PasaJell 105	RT	2290 113	3478 301	5602 193	4150 640
		180°F	1593 171	2576 264	5096 405	
		-67°F	2760 167	3286 213	4632 643	

Table 11.-Data Summary-FM 73/BR 127, Vacuum Bag Bonds

NOTE:

• Average of 10 specimens each test for RT and 180°F, 1/2-in. overlap; others are 5 specimens each except 4 for stressed lap shear.

o 7075-T6 bare.

			L/t La	ap Shear	r - 1b	60-day, 1200
Adhesive/ Primer	Surface Preparation	Test Temp.	0.5 in.	1.0 in.	2.0 in.	lap shear, 120°F/100% RH
EA 9628/ BR 127	Optimized FPL etch	RT	2249 124	3616 197	5852 511	4155 240
		180°F	1526 120	2506 72	5158 72	
		-67°F	3028 164	3670 219	5260 62	
	Phosphoric acid non-	RT	2144 169	3960 115	6374 420	4300 82
		180°F	1508 93	2594 210	5120 210	
		-67°F	2968 168	4130 345	6028 107	
	PasaJell 105	RT	2133 175	3640 197	5652 389	4045 306
		180°F	1491 121	2702 181	5076 373	
		-67°F	2634 92	2872 254	3422 327	

Table 12.-Data Summary-EA 9628/BR 127, Vacuum Bag Bonds

NOTE:

• Average of 10 specimens each test for RT and 180°F, 1/2-in. overlap; others are 5 specimens each except 4 for stressed lap shear.

o 7075-T6 bare.

Table 13.-Test Matrix, 350° F Cure Adhesive Systems-Autoclave Bonds

		0.	5-in. Lap	Shear	L/t L -67°F	ap She , RT,	ar 350°F	Wedge	Metal-N	letal Peel	
Surface Preparation Methods	Adhesive System/ Primer	RT	350°F	60 Days 1200 psi Stress, 120°F/ 100% RH	0.5	1.0	2.0	120°F/ 100% RH	RT	Salt Spray, 14 Days	
o Optimized FPL Etch (Tank)	AF 130/ EC 2333	15	15	12	45	45	45	15	15	15	
o Phosphoric Acid Anodize (Non-Tank) o PasaJell 105	FM 400/ BR 400	15	15	12	45	45	45	15	15	15	
	FM 300/ BR 127	15	15	12	45	45	45	15	15	15	
	AF 143/ EC 3917	15	15	12	45	45	45	15	15	15	
Total Specimens = 888		60	60	48	180	180	180	60	60	60	
NOTE. 5 specimens as	ch tact avra	nt 4	for suctai	nad ctraccad	lan choa						

5 specimens each test except 4 for sustained stressed lap shear. NUIE:

Additional tests at 300°F, 250°F with/without primer on optimized FPL etch and PANTA -- 40 specimens.

Table 14.-Data Summary-AF 130/EC 2333, Autoclave Bonds

		30 days	0.03			0.04			0.06		
inch	т	14 days	0.03			0			0.02		
tension,	F/100% R	24 hours	0.03			0			10.0		
Crack Ex	120°	4 hours	0.03			0			10.0		
Wedge		1 hour	0.03			0			0.01		
		Initial	2.56			2.76			2.39		
eel. ./in.	Call	Spray 14 days	3.0			3.1 0.2			4.8 0.7		
M-M P 1b-in		RT	3.0			3.3 0.3			3.0 0		
		2.0 in.	1478 94	2204 31	1390	1550	2328 94	1292 89	1354 138	2012 90	1306
ar, 1b 180°F		1.0 in.	1277 75	1622 133	1254 73	1406 94	1688 95	1400 93	1260 74	1478 41	1138 84
Lap She F. RT.		0.5 in.	1340 57	1166 25	1186 87	1240 76	1227	1132 121	1250 87	1150	1112
L/t -67		Test Temp.	RT	350°F	-67°F	RT	350°F	-67°F	RT	350°F	-67°F
lear, psi	1200 pri	Stress, 60 days	4025 229			3880 285			3885 193		
n Lap St		350°F	2400 91			2504 38			2352 50		
0.5-i		RT	2680 190			2475 145			2656 257		
		Surface Preparation	Optimized FPL etch			Phosphoric acid non-	Cally anouse		PasaJell 105		
		Adhesive/ Primer	AF 130/ EC 2333								

NOTE:

o Average of 5 specimens each test except 4 for stressed lap shear.

o 7075-T6 bare.

Table 15.-Data Summary-FM 400/BR 400, Autoclave Bonds

		0.5-i	n Lap Sh	ear, psi	L/t -67	Lap She F. RT.	ear, lb 180°F		M-M P lb-in	eel, ./in.		Wedge	Crack Ex	tension,	inch	
				1000						-1-3			120°	F/100% R	Ŧ	
Adhesive/ Primer	Surface Preparation	RT	350°F	Stress, 60 days	Test Temp.	0.5 in.	1.0 in.	2.0 in.	RT	Spray 14 days	Initial	1 hour	4 hours	24 hours	14 days	30 days
FM 400/ BR 400	Optimized FPL etch	3770	3210 152	3900 134	RT	1876 304	1876 172	2080 227	3.0	3.0 0	1.72	0.04	0.07	0.07	0.12	0.14
					350°F	1655 60	1970 145	2588 271								
					-67°F	1803 263	2080 262	2052 223								
	Phosphoric acid non-	3948 367	2920 228	3480 198	RT	1962 256	1808 198	2062 432	3.0	3.5 0	1.79	0.10	0.13	0.15	0.21	0.23
	rank anouse				350°F	1468	1749 187	2526 192								
					-67°F	1745 276	1928 197	2033								
	PasaJell 105	3364 305	2892 137	3480 210	RT	1794 124	1798 202	1918 212	3.0	3.0 0	1.75	0	0.05	60.0	0.12	0.13
					350°F	1477 58	1766 134	2474 292								
					-67°F	1784 237	1770 270	1822 189								

NOTE:

o Average of 5 specimens each test except 4 for stressed lap shear.

o 7075-76 bare.

Table 16,-Data Summary-FM 300/BR 127, Autoclave Bonds

	0.5-i	n Lap Sh	ear, psi	L/t -67°	Lap She F, RT,	lar, lb		M-M P 1b-in	eel, ./in.		Wedge	Crack Ex	tension,	inch	
			1200 nei						tes)			120°	F/100% RI	Ŧ	
face	RT	350°F	Stress, 60 days	Test Temp.	0.5 in.	1.0 in.	2.0 in.	RT	Spray 14 days	Initial	1 hour	4 hours	24 hours	14 days	30 days
timized etch	5460 153	840 285	5300 277	RT	2740 63	3340 28	6066 227	18.8	18.3 0.7	1.68	0	0	0	0	0.01
				350°F	423 60	763 41	1218 60								
				-67°F	1854 93	2026 78	2612 163								
osphoric id non-	4944 135	832 107	5270 96	RT	2496 43	3958 108	6062 106	14.4	14.1 2.1	1.66	0	0	0.01	10.0	0.01
at Inoup All				350°F	397 69	748 36	1307 106								
				-67°F	1930 407	2016 156	2336 201								
saJell 105	5436 121	676 110	5070 234	RT	2634 70	3678 304	5514 645	19.2 4.5	15.8 3.1	1.77	0.17	0.29	0.29	0.46	0.46
				350°F	372 29	575 51	1120 65								
				-67°F	1646 65	1820 258	1914 174								

NOTE:

o Average of 5 specimens each test except 4 for stressed lap shear.

o 7075-T6 bare.

o Sub values in the table indicate standard deviation.

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Table 17.-Data Summary-AF 143/EC 3917, Autoclave Bonds

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		30 days	0.03			0.09			0.02		
inch	H	14 days	0.03			0.03			0		
tension,	F/100% R	24 hours	0.03			0.02			0		
Crack Ex	120°	4 hours	0.03			10.0			0		
Wedge		hour	0.03			10.0			0		
		Initial	1.71			1.68			1.73		
eel. ./in.	Calt	Spray 14 days	7.8 0.7			7.5 0.4			6.6 0.8		
M-M P lb-in		RT	7.2 0.7			6.7 0.4			6.0		
		2.0 in.	2968 116	3434 84	1834 97	3350 125	3254 138	1826 232	2544 284	3354 110	1594 63
ar, 1b 180°F		1.0 in.	2234 152	1888 13	1566 75	2532 84	1804 65	1632 124	1932 70	1796 26	1434 137
Lap She °F, RT,		0.5 in.	2000	1052 34	1478 64	2076 88	1060 31	1344 74	1922 118	1128 31	1350 61
L/t -67		Test Temp.	RT	350°F	-67°F	RT	350°F	-67°F	RT	350°F	-67°F
ear, psi	1200 nei	Stress, 60 days	4630 151			4300 40	one spec. failed	days	4590 160		
n Lap Sh		350°F	2050			2194 162			2088 134		
0.5-i		RT	3970 201			4116			3836 143		
		Surface Preparation	Optimized FPL etch			Phosphoric acid non-	Lank anouse		PasaJell 105		
		Adhesive/ Primer	AF 143/ EC 3917								

NOTE:

o Average of 5 specimens each test except 4 for stressed lap shear.

o 7075-T6 bare.

		L/t L -67°F	ap She , RT,	ar 350°F	0.5-inch Lap Shear
Surface Preparation Methods	Adhesive System/ Primer	0.5	1.0	2.0	60 Days, 1200 psi Stress 120°F/100% RH
o Optimized FPL Etch (Tank)	AF 130/ EC 2333	75	45	45	12
o Phosphoric Acid Anodize (Non-Tank) o PasaJell 105	FM 400/ BR 400	75	45	45	12
	AF 143/ EC 3917	75	45	45	12
Total Specimens = 531		225	135	135	36

Table 18.-Test Matrix, 350° F Cure Adhesive Systems-Vacuum Bag Bonds

NOTE: 5 specimens each test except 10 for RT and 350°F, 1/2-in. overlap and 4 each for sustained stressed lap shear.

			L/t La	ap Shear	r - 1b	60-day, 1200
Adhesive/ Primer	Surface Preparation	Test Temp.	0.5 in.	1.0 in.	2.0 in.	lap shear, 120°F/100% RH
AF 130/ EC 2333	Optimized FPL etch	RT	1303 87	1211 89	1388 154	3535 192
		350°F	1083 45	1447 42	2064 50	
		-67°F	1252 187	1057 91	1188 129	
	Phosphoric acid non-	RT	1285 147	1266 107	1486 120	3685 194
		350°F	1104 57	1492 55	2225 85	
		-67°F	1194 145	1236 112	1548 281	
	PasaJell 105	RT	1333 178	1201 119	1471 171	3695 266
		350°F	1078 73	1308 233	1951 159	
		-67°F	1107 72	1280 181	1165 111	

Table 19.-Data Summary-AF 130/EC 2333, Vacuum Bag Bonds

NOTE:

- Average of 10 specimens each test for RT and 350°F, 1/2-in. overlap;
 others are 5 specimens each except 4 for stressed lap shear.
- o 7075-T6 bare.
- o Sub values in the table indicate standard deviation.

			L/t La	ap Shear	- 1b	60-day, 1200
Adhesive/ Primer	Surface Preparation	Test Temp.	0.5 in.	1.0 in.	2.0 in.	lap shear, 120°F/100% RH
FM 400/ BR 400	Optimized FPL etch	RT	1786 254	1801 250	1927 196	3205 637
		350°F	1304 177	1686 185	2422 236	
		-67°F	1608 263	1700 213	1906 317	
	Phosphoric acid non- tank anodize	RT	1825 238	1782 194	2010 356	3765 351
		350°F	1321 105	1573 227	2242 358	
		-67°F	1802 215	1786 166	2218 164	
	PasaJe11 105	RT	1539 251	1858 34	1960 239	2800 327
		350°F	1255 155	1606 201	2179 434	
		-67°F	1440 308	1751 221	1607 142	

Table 20.-Data Summary-FM 400/BR 400, Vacuum Bag Bonds

NOTE:

• Average of 10 specimens each test for RT and 350°F, 1/2-in. overlap; others are 5 specimens each except 4 for stressed lap shear.

- o 7075-T6 bare.
- o Sub values in the table indicate standard deviation.

			L/t La	ap Shea	r - 1b	60-day, 1200	
Adhesive/ Primer	Surface Preparation	Test Temp.	0.5 in.	1.0 in.	2.0 in.	lap shear, 120°F/100% RH	
AF 143/ EC 3917	Optimized FPL etch	RT	2053 62	2472 58	3308 78	4615 357	
		350°F	954 74	1632 69	2890 187		
		-67°F	1572 155	1752 95	1862 176		
	Phosphoric acid non-	RT	1985 59	2264 59	3046 118	4710 244	
		350°F	887 127	1476 152	2810 221		
		-67°F	1568 85	1682 98	1890 64		
	PasaJell 105	RT	1860 142	2187 96	3010 110	4726 31	
		350°F	933 125	1734 50	2950 475	One specimen failed during	
		-67°F	1355 139	1406 115	1776 236	loading	

Table 21.-Data Summary-AF 143/EC 3917, Vacuum Bag Bonds

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NOTE:

- Average of 10 specimens each test for RT and 350°F, 1/2-in. overlap;
 others are 5 specimens each except 4 for stressed lap shear.
- o 7075-T6 bare.
- o Sub values in the table indicate standard deviation.

Table 22.-Test Matrix, Room Temperature Cure Adhesive Systems

		0.	5-in. L	ap Shear	L/t -67°	Lap Sh F, RT,	lear 180°F	Wedge	Metal	-Metal Peel
Surface Preparation Methods	Adhesive System/ Primer	RT	180°F	60 Days 1200 psi Stress, 120°F/ 100% RH	0.5	1.0	2.0	120°F/ 100% RH	RT	Salt Spray, 14 Days
o Optimized FPL Etch (Tank)	EA 9320/ BR 127	15	15	12	45	45	45	15	15	15
o Phosphoric Acid Anodize (Non-Tank) o PasaJell 105	EA 934/ BR 127	15	15	12	45	45	45	15	15	15
Total Specimens = 444		30	30	24	06	90	06	30	30	30

NOTE: 5 specimens each test except 4 for sustained stressed lap shear.

Retest of 90 specimens.

Table 23.-Data Summary-EA 9320/BR 127, RT Cure Bonds

		30 days	1.29			1.37			1.12		
xtension, inch	Ŧ	14 days	1.17			1.30			1.00		
	F/100% R	24 hours	0.92			1.1			0.76		
Crack Ex	120°	4 hours	0.69			16.0			0.53		
Wedge		hour	0.44			0.70			0.30		
		Initial	1.59			1.44			1.45		
eel. ./in.	+1-3	Spray 14 days	54.9 5.5			477 1.6			40.2 3.1		
M-M P lb-in		RT	48.2 10.7			48.6 3.3			40.5 2.4		
	2.0 in.		3060 402	383 85	3972 767	4864 414	590 21	4534 387	4764 797	687 153	3354 331
ear, 1b 180°F	1.0 in.		2068 260	206 40	2320 616	2458 217	321 18	3308 317	2430 600	379 44	2656 394
Lap She °F, RT,		0.5 in.	886 263	119 29	2162 462	1302 223	176 33	2558 209	1506 450	214 46	2422 460
L/t -67	Test Temp.		RT	180°F	-67°F	RT	180°F	-67°F	RT	180°F	-67°F
ear, psi	600 psi Stress, 60 days 3767		3767 110			3680 257			2353 50 one spec. failed 28 days		
n Lap Sh	180°F		250 82	593 80 retest		380 39	693 50 retest		349 146 486 46 retest		retest
0.5-iı		RT 1584 244 3660 563 retest		retest	2636 311	3793 141 retest		2316 748 3410 551 retest		retest	
	urface treparation		Optimized FPL etch			Phosphoric acid non-	רמווג מווטטו לפ		PasaJell 105		
dhesive/			EA 9320/ BR 127								

NOTE:

Average of 5 specimens each test except 4 for stressed lap shear. 0

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Table 24:-Data Summary-EA 934/BR 127, RT Cure Bonds

Wedge Crack Extension, inch		30 days	0.35			0.53			0.57		
	Ŧ	14 days	0.23			0.44			0.43		
	F/100% F	24 hours	0.19			0.23			0.18		
	120	4 hours	0.16			0.15			0.15		
		1 hour	0.08			01.0			0.13		
		Initial	2.64			2.21			2.13		
eel, ./in.	+[*)	Spray 14 days	13.8 0.1			15.6 1.7			14.7 1.3		
M-M P lb-in		RT	10.5 2.4			1.5			3.2		
	2.0 in.		2928 173	1281 163	1950 241	2850 370	1289 132	1990 342	3230	1519 264	2096 193
t Lap Shear, lb 7°F, RT, 180°F	1.0 in.		1948 154	882 97	1678 85	1846 465	702 103	1436 161	2124 197	993 81	1808 58
	0.5 in.		1232 66	424 86	1646 96	1118	436 84	1324 270	1302 234	657 46	1829 156
L/t -67		Test Temp.	RT 180°F -67°F			RT	RT 180°F -67°F			180°F	-67°F
ear, psi	600 psi Stress, 60 days		4090 137			4125			3810 674		
ı Lap Sh	180°F		1436 60	1882 423 retest		878 123	893 197 retest		1292 108	813 266 retest	
0.5-ir	RT		2284 267	3080 482 retest		2860 414	3272 375 retest		2840 234	234 2440 105 retest	
iurface Treparation		Optimized FPL etch			Phosphoric acid non-	רמווע מווחתו לב		PasaJell 105			
		Adhesive/ Primer	EA 934/ BR 127								

NOTE:

o Average of 5 specimens each test except 4 for stressed lap shear.

o 7075-T6 bare.

o Sub values in the table indicate standard deviation.

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