ADHESIVE BONDING PROPERTIES OF VARIOUS METALS AS AFFECTED BY CHEMICAL AND ANODIZING TREATMENTS OF THE SURFACES

Revised April 1954

(Addendum Added)

Supplement available with revisions. No. 1842-A

This Report is One of a Series
Issued in Cooperation with
AIR FORCE-NAVY-CIVIL SUBCOMMITTEE
on
AIRCRAFT DESIGN CRITERIA
Under the Supervision of the
AIRCRAFT COMMITTEE
of the
MUNITIONS BOARD

No. 1842

UNITED STATES DEPARTMENT OF AGRICULTURE
FOREST SERVICE
FOREST PRODUCTS LABORATORY
Madison 5, Wisconsin
In Cooperation with the University of Wisconsin

ADHESIVE BONDING PROPERTIES OF VARIOUS METALS AS AFFECTED BY CHEMICAL AND ANODIZING TREATMENTS OF THE SURFACES[1]

By

H. W. EICKNER, Chemical Engineer

Forest Products Laboratory,[2] Forest Service
U. S. Department of Agriculture

Summary

Investigations were made with four commercial metal-bonding adhesives of the bonding properties of magnesium, bare and clad aluminum, stainless steel, chrome-molybdenum steel, and titanium metal sheets given various chemical and anodizing treatments.

The bare and clad aluminum alloys generally showed the best bonding properties. A sulfuric acid-dichromate etch solution gave optimum results in preparing the bare and clad aluminum alloys. Moderately good bonds were obtained to sulfuric acid-anodized aluminum alloys, but bonding obtained to the chromic acid-anodized aluminum was poor.

Moderately good bonds were obtained with all four adhesives to magnesium degreased in sodium metasilicate-pyrophosphate solution or given a hydrofluoric acid etch-dichromate seal (Military Specification MIL-M-3171, Type III) protective treatment. Two of the four adhesives also gave this quality of bond to magnesium given a Manodyze treatment. Only one adhesive gave moderately good bonds to a zinc chromate prime coat when that prime coat was applied over a Manodyzed magnesium surface.

Alkaline degreasing of stainless steel resulted in bonds as good as those obtained with an acid etch treatment. Two of the adhesives were definitely better than the other two for bonding to stainless steel.

---

[1] This progress report is one of a series prepared and distributed by the Forest Products Laboratory under U. S. Navy Bureau of Aeronautics Order No. NAer 01319 and U. S. Air Force No. USAF-18(600)-70. Results here reported are preliminary and may be revised as additional data become available.

[2] Maintained at Madison, Wisconsin, in cooperation with the University of Wisconsin.

The two adhesives that produced best bonding to the stainless steel also produced the best bonding to the chrome-molybdenum steel alloy.

The titanium metal was found difficult to bond, and good bonding was obtained with only one of four adhesives investigated (one of the two adhesives that gave good bonds to the steel alloys) when the metal was prepared with a nitric-hydrofluoric acid solution.

In the main part of this study, only initial dry tests were made. Therefore, ratings of the gluability of the different types of surfaces in this part have been based solely on results of such dry tests. Subsequently, additional test specimens were subjected to a salt-spray exposure since such tests had proved to be of added value in rating various surface treatments in an earlier investigation[3] on the bonding of clad 24S-T3 aluminum alloy. Test data and a summary of results for salt-water spray tests made for some of the surface treatments of the main study have been included in the addendum at the end of this report.

Introduction

Metal parts of aircraft structures are now being bonded together with adhesives, and it is anticipated that this method will find wider use as aircraft fabrication continues.

The principal work on adhesive bonding of metals was done at first with clad 24S-T3 aluminum alloy. The various adhesive manufacturers and aircraft fabricators noted that, in bonding this metal, the type and thoroughness of the cleaning method were important in obtaining the highest-quality bonds. In previous work at this Laboratory,[3] the use of a sulfuric acid-sodium dichromate solution for cleaning the clad 24S-T3 aluminum alloy resulted in bonds of more consistently high quality, both in original dry strength tests and after salt-water spray tests, than did the use of abrasive, solvent, or alkaline cleaning methods.

In more recent work on adhesive bonding of metals in aircraft fabrication, it has seemed desirable to bond other metals in addition to clad 24S-T3 aluminum alloy, including bare 24S-T3 aluminum, bare and clad 75S-T6 aluminum magnesium, titanium, stainless steel, and chrome-molybdenum steel alloys. The purpose of the present study was to determine the bond strength values that can be obtained with the presently available metal-bonding adhesives to these various types of metals when the metal surfaces are treated using some of the more commonly known degreasing, etching, anodizing, and sealing treatments. These treatments for each of the metals were selected from a number suggested by different authorities in the field, and are believed to include methods currently used in preparing each of the metals for further processing or use.

---

[3]Eickner, H. W., and Schowalter, W. E. A Study of Methods for Preparing Clad 24S-T3 Aluminum-Alloy Sheet Surfaces for Adhesive Bonding. Forest Products Laboratory Reports Nos. 1813 and 1813A, 1950.

Procedure

Type and number of test specimens

Small 4- by 5-1/2-inch test panels of the type shown in figure 1 were prepared by bonding two 3- by 4-inch pieces of metal with a 0.5-inch overlap of the 4-inch edges. Metals of the following types and thicknesses were used in preparing these test panels:

1. Clad 24S-T3 aluminum alloy (Army-Navy Aeronautical Specification AN-A-13) - 0.064 inch

2. Clad 75S-T6 aluminum alloy (Army-Navy Aeronautical Specification AN-A-10) - 0.064 inch

3. Bare 24S-T3 aluminum alloy (Army-Navy Aeronautical Specification AN-A-12) - 0.064 inch

4. Bare 75S-T6 aluminum alloy (Army-Navy Aeronautical Specification AN-A-9a) - 0.064 inch

5. FS1-H24 magnesium alloy, plain and oiled surface (Federal Specification QQ-M-44) - 0.064 inch

6. Type 302 18-8 stainless steel, 2B finish, annealed (Military Specification MIL-S-5059, type G) - 0.064 inch

7. SAE 4130 chrome-molybdenum steel alloy, annealed (Army-Navy Aeronautical Specification AN-QQ-S-685) - 0.064 inch

8. RC-70 titanium alloy, 1/2 hard, (-------) - 0.032 inch

The small metal pieces were cut from sheets so that the roll direction of the metal would coincide with the 5-1/2-inch lengthwise direction of the test panels. Any cutting burrs on the edges of the pieces were removed with a hand file before the surfaces were prepared for bonding.

Six test panels were prepared for each bonding variable investigated. Three test specimens, each 1 inch wide, were cut from three of the six panels with a milling machine cut-off saw for the titanium and a metal-cutting bandsaw for the other metals. Nine specimens were thus available for evaluating the effect of each bonding variable. The other three test panels prepared for each of the bonding variables have been saved for future exposure tests.

Preparation of metal surfaces

The metal surfaces, as received, were wiped with a cloth saturated with lacquer thinner[4] to remove part of the protective oil and grease coatings and identification markings from the metal surfaces. The metal surfaces were then given the following additional treatments before being bonded:

For all treatments where water solutions were used, the solutions were, unless otherwise stated, rinsed from the metal surfaces with running cold water before the metal pieces were placed in another solution. After the metal was removed from the final water solution, the chemicals were rinsed from the metal pieces with cold and hot running water and the water was force-dried with a fan without air filters from the metal surfaces. During rinsing, the metal was watched to see if the film of water broke; a break indicated that the surface was not free of grease or waxes. No water rinse or water-break test was made after the non-water treatments. All chemical solutions were constantly agitated to maintain uniform temperatures and concentrations throughout the solution. These solutions were changed frequently to avoid contamination.

Clad aluminum alloys, 24S-T3 and 75S-T6.--The clad aluminum-alloy surfaces were prepared for bonding with one of four processes.

CA-1, Vapor degrease process.-The metal surfaces were degreased by placing them for approximately 3 minutes in a small commercial vapor-degreasing machine containing hot vapors of stabilized trichloroethylene (Army-Navy Aeronautical Specification AN-T-37a).

CA-2, Sulfuric acid-dichromate etch process.-The metal surfaces were etched by immersing them for 10 minutes at 140° F. in a solution (pH of less than 0.1) of the composition:

45.0 ounces concentrated sulfuric acid (Specific Gravity 1.84)
4.5 ounces sodium dichromate, crystalline
1.0 gallon water

CA-3, Sodium metasilicate degrease process.-The metal surfaces were degreased by immersing them for 5 minutes at 170° to 190° F. in an alkaline solution (pH of 12.2) of the composition:

5.0 ounces sodium metasilicate
0.5 ounce Nacconal NR
1.0 gallon water

[4] The lacquer thinner (Military Specification MIL-T-6094a) was prepared by mixing together the following solvents: 25 percent by weight of normal butyl acetate, 22 percent of ethyl acetate, 10 percent of normal butyl alcohol, 22 percent of toluene, and 21 percent of aliphatic petroleum naphtha.

CA-4, Sodium metasilicate degrease process with chromic acid rinse.—The metal surfaces were degreased by immersing them in sodium metasilicate solution as in treatment CA-3, rinsed in hot water, and then immersed for 3 minutes at 140° to 160° F. in a solution (pH of 0.6 to 0.8) of the composition:

7.0 ounces chromic acid
1.0 gallon water

Bare aluminum alloys, 24S-T3 and 75S-T6.—The bare aluminum alloy surfaces were prepared for bonding with one of three processes.

BA-1, Chromic acid anodize process.[5] -The metal surfaces were degreased by immersing them in sodium metasilicate solution as in treatment CA-3 for clad aluminum alloys and then, after being rinsed in hot and cold running water, the aluminum pieces were anodically treated while immersed at 90° to 100° F. in a solution (pH of 0.1 to 0.3) of the composition:

14.0 ounces chromic acid
1.0 gallon water

This anodic treatment consisted of applying a small direct current potential (3 to 5 volts) between the aluminum pieces as anodes suspended in the chromic acid solution and the cathode, which was the metal container for the solution. This voltage was gradually increased to 40 volts during the next 8 to 10 minutes. This maximum voltage was then applied for 40 minutes. The maximum current density used was 8 amperes per square foot, and at the end of the anodizing period the current density was usually 2.5 to 3.0 amperes per square foot. The anodizing tank was a lead-lined steel tank and the electrical clips to the aluminum pieces were made of aluminum.

After removal from the anodizing solution, the metal pieces were rinsed in cold water, and the coating was stabilized by heating the pieces in distilled water for 1 hour at a temperature of 180° F.

BA-2, Sulfuric acid anodize process.-The metal surfaces were degreased, as in treatment CA-3 for clad aluminum alloy, by immersing them in sodium metasilicate solution. After being rinsed in hot and cold running water, the pieces were anodized in sulfuric acid solution and then sealed, using Aluminum Company of America Alumilite Process No. 205.

BA-3, Sulfuric acid-dichromate etch.-The metal surfaces were etched in a sulfuric acid-dichromate solution as in treatment CA-2 for clad aluminum alloy.

---

[5] The chromic acid anodizing was done essentially as outlined by Navy Aeronautical Specification PT-19 to meet the requirements of Army-Navy Aeronautical Specification AN-QQ-A-696.

FS1-H24 magnesium alloy, plain surface finish and oiled.--The magnesium alloy sheets were prepared for bonding with one of five treatments.

M-1, Sodium metasilicate-pyrophosphate degrease process.-The metal surfaces were degreased by immersing them for 10 minutes at 160° to 190° F. in a solution (pH of 12.65) of the composition:

3.0 ounces sodium metasilicate
1.5 ounces tetra sodium pyrophosphate
1.5 ounces sodium hydroxide
0.5 ounce Nacconal NR
1.0 gallon water

M-2, Specification MIL-M-3171, Type III, corrosion-protective treatment.-The metal surfaces were given an initial vapor degreasing in trichloroethylene, as in treatment CA-1 for clad aluminum alloy, and then surface oxidation was removed by immersing them for 10 minutes at 140° F. in a solution (pH of less than 0.1) of the composition:

24.0 ounces chromic acid
1.0 gallon water

After the chromic acid was rinsed from the metal pieces with cold and hot running water, they were given a final alkaline cleaning for 10 minutes at 160° to 190° F. in a sodium metasilicate-pyrophosphate solution of the type used in treatment M-1. The alkaline solution was then rinsed from the metal pieces in hot running water and they were etched by immersing them for 5 minutes at 70° to 90° F. in a solution of the composition:

1 part by volume hydrofluoric acid (50 percent)
2 parts by volume water

The hydrofluoric acid solution was then rinsed from the metal pieces in cold running water and they were sealed by immersing them for 30 minutes in a boiling solution (pH of 5.19) of the composition:

32.0 ounces sodium dichromate
0.4 ounce calcium fluoride
1.0 gallon water

M-3, Specification MIL-M-3171, Type III, treatment and zinc chromate primer.-The metal surfaces were first treated as in treatment M-2, then spray coats of zinc chromate primer (Military Specification MIL-P-6889A), 1 part by volume of primer to 2 parts toluene, were applied. These spray coats were dried at room conditions for 30 minutes between coats and for 48 hours after the final coat before adhesive was applied. The total thickness of the zinc chromate prime coat was 0.0005 to 0.0010 inch.

M-4, Manodyze[7] treatment.-The metal pieces were given an initial vapor degreasing in trichloroethylene, as in treatment CA-1 for clad aluminum alloy. The surfaces were then cleaned by a cathodic treatment for 1 to 3 minutes at 180° F. in a solution (pH of 12.1) of the composition:

12.0 to 16.0 ounces sodium hydroxide
0.12 ounce Nacconal NR
1.0 gallon water

The current density used was 30 to 50 amperes per square foot of cathode surface (magnesium alloy) with a direct current voltage of 4.0 to 6.0 volts. A stainless steel tank was the anode.

After being rinsed in cold running water, the metal pieces were immersed for 2 to 3 seconds at 70° to 90° F. in a solution (pH of 0.3) of the composition:

4.3 ounces concentrated sulfuric acid (Specific Gravity 1.84)
2.8 ounces concentrated nitric acid (Specific Gravity 1.42)
1.0 gallon water

This solution was then rinsed from the surfaces with cold running water before the pieces were given an electrolytic treatment for 5 to 7 minutes at 180° to 200° F. in a solution (pH of 10.9) of the composition:

40.0 ounces sodium hydroxide
0.5 ounce phenol
5.2 ounces sodium silicate, 41° Baume.
Water to make 1 gallon

A 4.0- to 6.6-volt alternating current at 40 amperes per square foot of magnesium surface was applied between the magnesium pieces and a stainless steel tank, while the metal was in the solution. Magnesium clips were used to attach the leads to the magnesium pieces.

After removal from the electrolytic solution, the metal pieces were rinsed in hot water for 5 to 15 minutes at 180° to 200° F. and then neutralized for 1 to 1-1/2 minutes at 135° to 145° F. in a solution (pH of 2.6) of the composition:

0.08 ounce chromic acid
1.0 gallon water

Immediately after removal from the neutralizing solution and without any further rinse, the metal pieces were force-dried in hot air.

---

[7] The Manodyze process for magnesium is a proprietary process. Further information can be obtained from Hanson-Van Winkle-Munning Co., Matawan, N.J., Agents.

M-5, Manodyze treatment and zinc chromate primer.-The metal surfaces were first treated as in treatment M-4, then a zinc chromate primer was applied as in treatment M-3.

Type 302, 18-8 annealed stainless steel, 2B finish.--The stainless steel sheets were prepared with one of three treatments.

SS-1. Sodium metasilicate-pyrophosphate degrease process.-The metal surfaces were degreased with sodium metasilicate-pyrophosphate solution, as in treatment M-1 for magnesium alloy.

SS-2. Sulfuric-hydrochloric and nitric-hydrofluoric acid etch.-The metal surfaces were degreased with sodium metasilicate-pyrophosphate solution, as in treatment M-1, and then, after a cold water rinse, the pieces were immersed for 10 minutes at 140° F. in a solution (pH of less than 0.1) of the composition:

7.0 percent by volume concentrated sulfuric acid (Specific Gravity 1.84)
3.0 percent by volume concentrated hydrochloric acid (Specific Gravity 1.18)
90.0 percent by volume water

This solution was rinsed from the surfaces, and the metal pieces were immersed for 5 to 10 minutes at 70° to 75° F. in a solution of the composition:

43.0 ounces concentrated nitric acid (Specific Gravity 1.42)
3.5 ounces hydrofluoric acid (50%)
Water to make 1.0 gallon

SS-3. Sodium metasilicate-pyrophosphate degrease and chromic acid rinse.-The metal surfaces were degreased with sodium metasilicate-pyrophosphate solution, as in M-1, and then, after a hot water rinse, the pieces were immersed for 3 minutes at 140° to 160° F. in a solution (pH of 0.6 to 0.8) of the composition:

7.0 ounces chromic acid
1.0 gallon water

SAE 4130 annealed chrome-molybdenum steel alloy.--The steel alloy sheets were prepared for bonding with one of four treatments.

SA-1. Trichloroethylene vapor degrease process.-The metal surfaces were vapor degreased in trichloroethylene as in treatment CA-1.

SA-2. Sulfuric acid-dichromate etch.-The metal surfaces were etched with sulfuric acid-dichromate solution, as in treatment CA-2.

SA-3. Sodium metasilicate-pyrophosphate degrease process.-The metal surfaces were degreased in sodium metasilicate-pyrophosphate solution, as in treatment M-1.

SA-4. Sodium metasilicate-pyrophosphate degrease and chromic acid rinse.-The metal surfaces were degreased in sodium metasilicate-pyrophosphate solution, rinsed in hot water, and then immersed in chromic acid, as in treatment SS-3 for stainless steel alloy.

RC-70-1/2 Hard Titanium.--The titanium sheets were prepared for bonding using one of four treatments.

T-1. Sodium metasilicate-pyrophosphate degrease process.-The metal surfaces were degreased in sodium metasilicate-pyrophosphate solution, as in treatment M-1 for magnesium alloy.

T-2. Sodium metasilicate-pyrophosphate degrease and chromic acid rinse.-The metal surfaces were degreased in sodium metasilicate-pyrophosphate solution, rinsed in hot water, and immersed in chromic acid, as in treatment SS-3 for stainless steel alloy.

T-3. Nitric-hydrofluoric acid etch.-The metal pieces were etched by immersing them for 15 minutes at room temperatures in a nitric-hydrofluoric acid solution of the same composition as used in treatment SS-2 for stainless steel alloy.

T-4. Nitric-hydrofluoric acid etch and chromic acid rinse.-The metal pieces were etched in nitric-hydrofluoric acid solution, as in treatment T-3, rinsed in cold water, and then immersed for 3 minutes at 140° to 160° F. in a chromic acid solution of the same composition as used in treatment SS-3 for stainless steel alloy.

Adhesive bonding processes

The following four adhesive bonding processes were used in bonding the six lap-joint panels prepared with each metal and surface condition.

Adhesive 33.-A high-temperature-setting formulation of the vinyl-phenolic type.

Adhesive 34.-A high-temperature-setting, two-component formulation of a phenol resin solution and a vinyl polymer powder.

Adhesive 38.-A high-temperature-setting adhesive formulation of neoprene, nylon, and phenol resins supported as a film on nylon-fabric tape.

Adhesive 45.-A high-temperature-setting formulation of acrylonitrile-butadiene rubber and phenol resin in the form of an unsupported tape.

It was not possible to complete the treatment of the various metals and do the bonding with one adhesive for the entire study in a single bonding run. Therefore, as the several bonding runs were made with each adhesive, a set of panels (three panels of three specimens in each) of clad 24S-T3 aluminum alloy, cleaned by treatment CA-2, was also made to be a control for the quality of bonding with the adhesive for each bonding run.

The conditions of bonding with each of the adhesives were as follows:

Adhesive 33.--Five spray coats of adhesive, thinned with 1-1/2 parts by volume of adhesive solvent to 1 part of adhesive, were applied to the metal with a 30-minute air-drying period between coats, and overnight air drying after the final coat. The adhesive film was then precured for 1 hour at 150° F. in an oven. Following the precure, the joint was assembled and placed in a hot press, where it was preheated without pressure for 5 minutes at 335° F. before it was given the final cure for 25 minutes at 335° F. and 200 pounds of pressure per square inch.

Adhesive 34.--One medium coat of the liquid component of the adhesive was brushed on the metal, and the powdered component of the adhesive was sprinkled immediately into the wet spread of adhesive. Any excess powder was brushed from the surface. The adhesive film was air dried overnight, and the joint was assembled and pressed for 20 minutes at a temperature of 300° F. under a pressure of 200 pounds per square inch.

Adhesive 38.--A sufficient number (two to four) of spray coats of the priming component were applied to the metal pieces to result in a 0.001 to 0.002 inch film of adhesive. The adhesive film was dried for 20 minutes between coats and 3 to 5 hours after the final coat. The joint was then assembled with single layer of the tape adhesive and pressed for 38 minutes at a pressure of 50 pounds per square inch. The temperature of the press platen was adjusted to approximately 300° F. at the start of the pressing period, and increased so that the bond temperature was 335° F. during the final 30 minutes of pressing.

Adhesive 45.--A single film of the tape adhesive was assembled in the joint. The assembly was cured for 35 minutes at a bond temperature of 330° F. and a pressure of 50 pounds per square inch.

Testing

Three of the lap-joint panels prepared with each bonding process, metal, and surface treatment were sawn into individual 1-inch wide specimens of the type shown in figure 1. Cutting was done with metal-cutting saws, using a slow rate of feed and a holding jig to minimize any mechanical damage or overheating of joint. The specimens were tested by loading them to failure in tension at a rate of 300 pounds per minute. The ends of the specimens were held in 1-inch wide Templin-type grips that extended down from the ends of the specimens to within 1 inch of the edge of the lap. Testing was done at a temperature of 72° to 76° F. The failing load, calculated as a unit stress, was recorded along with estimated areas (expressed as percentages of the total area) of adhesive failure, adhesion and cohesion, and metal primer failure.

In addition to the tests on adhesive-bonded joints, standard strength tests as outlined in Federal Specification QQ-M-151 for tensile strength, yield strength at 0.2 percent strain, and elongation in 2 inches were made with each of the metals.

Test Results

The test results obtained with the four metal-bonding adhesives in lap-joint specimens of the different metals prepared for bonding by several surface treatments are given in tables 1 through 8.

These data were analyzed for each of the metals bonded, to obtain bonding characteristics for the metal, adhesive, and surface treatments.

Clad 24S-T3 and 75S-T6 aluminum alloys

Clad 24S-T3 and 75S-T6 aluminum alloy showed similar adhesive bonding properties (tables 1 and 2) and generally appeared to give more consistently good results in bonding with the variety of adhesives and cleaning procedures used than did any of the other metals in this study. The sulfuric acid-dichromate solution (treatment CA-2) usually gave results equal to or better than those obtained by the use of any of the three other treatments. With the two lower-strength adhesives (38 and 45) the three other surface treatments (CA-1, CA-3, and CA-4) gave bonds that had strength values of 83 percent or more of the strength values of bonds for control specimens (specimens bonded at the same time to clad 24S-T3 aluminum cleaned in sulfuric acid-dichromate solution). Vapor degreasing (treatment CA-1) was found to give good results in bonding with the high-strength adhesive 34, but comparatively poor bonding with adhesive 33.

Inconsistencies in the quality of bonding were obtained with adhesives 33 and 34 to aluminum that was cleaned by the two metasilicate treatments, CA-3 and CA-4. It appeared that good-quality bonds can sometimes be obtained to aluminum cleaned with metasilicate solutions, but that some variable in the use of this treatment is critical. This critical variable might be the time elapsed between removal of the metal pieces from the hot metasilicate solution and the rinsing of the metal. The metasilicate was difficult to rinse from the sheets if the solution cooled or dried on the metal prior to the hot-water rinse. In these tests the period between removal from the metasilicate solution and the beginning of the hot-water rinse was 10 to 15 seconds. The use of a chromic acid rinse following the metasilicate degrease has normally been considered good practice. However, the results of the tests with clad aluminum alloy (treatments CA-3 and CA-4) showed an improvement in bonding in only three of the eight tests when the chromic acid rinse was used, and in these instances the bonding to the metal cleaned with metasilicate solution alone (treatment CA-3) were of unusually low quality.

Bare 24S-T3 and 75S-T6 aluminum alloys

Bare 24S-T3 and 75S-T6 aluminum alloys showed similar bonding properties (tables 3 and 4). With the sulfuric acid-dichromate treatment, the bond strength values obtained with the bare aluminum alloys were 94 percent or more of the control tests made to the clad 24S-T3 aluminum etched with this treatment. With adhesives 33 and 34, the bonds made to the bare 75S-T aluminum prepared with the sulfuric acid-dichromate solution had unusually high bond strength values, exceeding the strength values of all other bonds made in this study.

The general level of joint strength values was the same for all four adhesives used in bonding the bare 24S-T3 and 75S-T6 aluminum alloys anodized with sulfuric acid and sealed by the Alumilite Process No. 205. However, with the two high-strength adhesives, 33 and 34, this level of strength was lower than obtained with the bare aluminum alloys etched with sulfuric acid-dichromate solution.

The bare 24S-T3 and 75S-T6 aluminum alloys anodized with chromic acid and sealed by boiling in water showed rather poor adhesive bonding properties. Adhesive 38 gave the best bonding to this type of surface, and the strength values with this adhesive were about 75 percent of the strength values obtained when bonding to the bare aluminum alloys treated with sulfuric acid-dichromate solution. It is possible that better adhesive bonding properties might be obtained to thinner chromic acid-anodized coatings, as better-quality bonds were obtained in exploratory tests for this study when the coatings appeared to be thinner. The type of sealing treatments used with the anodized surfaces might also greatly influence the bonding characteristics of these surfaces.

FS1-H24 oiled, plain finish magnesium alloy

No high-strength bonds were obtained on FS1-H24 oiled, plain-finish magnesium alloy as compared to aluminum alloys (table 5). Most consistent results were obtained with all four adhesives to the magnesium alloy degreased in sodium metasilicate-pyrophosphate solution (treatment M-1) or given the MIL-M-3171, type III, corrosion protective treatment (treatment M-2). With these treatments moderately good-quality bonds having strength values of over 1,000 pounds per square inch were noted. Bonding to the magnesium surface given the Manodyze treatment (M-4) was also moderately good with adhesives 34 and 38, but poor with the other two adhesives.

Bonding to the magnesium surfaces primed with a zinc chromate primer (treatments M-3 and M-5) generally gave poor results, with the exception of adhesive 38 to the Manodyzed and zinc chromated surface (treatment M-5), which had bond strength values of over 1,000 pounds per square inch. This result was, however, somewhat lower than when the Manodyze surface without primer was bonded with the same adhesive. Treatment M-5 is essentially the same process, except that the primer was applied by two spray coats instead of a single thin dip coat, as used by some aircraft manufacturers to result in bond strength values as high as 1,600 pounds per square inch. The thickness and method of applying this primer may be an important factor in determining the strength of bonds that can be obtained to the surfaces. Generally high percentages of cohesive failure were noted in the zinc chromate primer film.

Type 302, 18-8 stainless steel, 2B finish

Of the four adhesives evaluated in bonding to type 302, 18-8 stainless steel with 2B finish, adhesive 34 gave the best results (table 6), regardless of which of the three surface treatments was used, and adhesive 33 was next.

Merely degreasing in sodium metasilicate-pyrophosphate solution (treatment SS-1) gave bonds that were equal to or better than those obtained with the more involved treatments of degreasing and acid etching (treatment SS-2) or degreasing and neutralizing with chromic acid (treatment SS-3).

The lower strengths with adhesives 33 and 45 to stainless steel with method SS-3, using a chromic acid rinse following alkaline degreasing, were not consistent with the general advantages of such an acid rinse when using these adhesives on other metals.

SAE 4130 annealed chrome-molybdenum steel

The best surface preparation of the SAE 4130 annealed chrome-molybdenum steel, from the standpoint of producing moderately good bonds with all four adhesives, was with treatment SA-4, an alkaline degrease and chromic acid rinse (table 7). Highest bond strength with adhesives 33 and 45, however, was obtained to the steel prepared by vapor degreasing (treatment SA-1), and with adhesive 38 to the steel prepared by an alkaline degrease with acid rinse (treatment SA-3). Adhesives 33 and 34 gave bonds to the surfaces prepared under optimum conditions of more than 4,000 pounds per square inch.

The annealed SAE 4130 steel, as received, was heavily coated with oil and a dark-colored substance. This substance, which was removed only by treatment SA-2, might be responsible for some of the inconsistent bonding. When this substance was removed by method SA-2 an oxide coating was formed during the drying after the treatment.

RC-70, 1/2 Hard Titanium alloy

Of the several cleaning methods investigated in preparing RC-70, 1/2 hard titanium alloy for adhesive bonding, the use of the nitric-hydrofluoric acid etch solution (treatments T-3 and T-4) generally resulted in the best bonding (table 8). Adhesive 34 produced bonds to titanium treated by method T-3 that had an average strength of 3,354 pounds per square inch. Of the four adhesives investigated, adhesive 34 generally gave the best bonds to titanium treated by the other methods.

Degreasing in sodium metasilicate-pyrophosphate solutions (treatments T-1 and T-2) generally resulted in inferior bonds for the adhesives investigated; some specimens failed when they were sawed into samples.

It should be noted that the two acid treatments (T3 and T4) for titanium were at room temperature and were not preceded by a degreasing, other than wiping them with a solvent. It may be that much better results would be obtained with these acid treatments if they were preceded by a more efficient degreasing procedure or conducted at a somewhat elevated temperature.

Tensile properties

Table 9 presents the tensile properties obtained in tests on the sheets of metal used in this work. These tests indicate that the sheets used meet the existing specification requirements for the metals.

Table 1.--Test results obtained on adhesive bonds made to 0.064-inch clad 24S-T3 aluminum alloy sheet (Specification AN-A-13)

Surface treatment for metals[1]	Adhesive	Shear strength: Average[2]	Shear strength: Maximum[2]	Shear strength: Minimum[2]	Shear strength: Average control[3]	Shear strength: Percent of control[4]	Failure: Cohesion[2]	Failure: Adhesion to metal[2]
		P.s.i.	P.s.i.	P.s.i.	P.s.i.		Percent	Percent
CA-1. Vapor degrease in trichloroethylene (No etch)	33	2,134	2,780	1,320	4,101	52	4	96
	34	3,602	3,890	3,098	4,325	83	34	66
	38	2,416	2,674	1,970	[5]2,830	85	61	39
	45	3,040	3,302	2,656	3,322	91	84	16
CA-2. Etch in sulfuric acid-dichromate solution (Etched, good water-film test)	33	4,133	4,770	2,640	....	....	28	72
	34	4,843	5,000	4,569	....	....	95	5
	38	2,583	2,971	2,235	....	....	71	29
	45	3,192	3,394	2,970	....	....	70	30
CA-3. Degrease in sodium metasilicate solution (No etch, good water-film test)	33	2,955	3,280	2,560	4,133	71	15	85
	34	3,699	3,939	3,500	4,325	86	18	82
	38	2,596	2,793	2,327	2,679	97	84	16
	45	3,309	3,490	3,140	3,160	105	69	31
CA-4. Degrease in sodium metasilicate solution, rinse in chromic acid. (No etch, good water-film test)	33	3,492	4,088	2,963	4,098	85	18	82
	34	3,362	4,030	2,670	4,607	73	24	76
	38	2,440	2,659	2,163	2,472	99	69	31
	45	3,165	3,323	3,000	3,177	100	72	28

[1] The appearance of the surfaces following each treatment as to any noticeable etch or surface coatings and the results of the water-film tests are given in parentheses.

[2] Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1 inch wide, three from each of three bonded panels. Percentages of failure are averages for these groups of nine specimens.

[3] Average strength for nine control specimens of a 0.5-inch lap joint of 0.064-inch clad 24S-T3 aluminum alloy cleaned with sulfuric acid-dichromate solution and bonded in the same bonding run and under the same conditions as the test specimens.

[4] The percent of control strength is based on the ratio of the average strength for the group of test specimens to the average strength of the control specimens.

[5] Test result obtained on six 0.5-inch lap-joint specimens, 1 inch wide, three from each of two bonded panels.

Table 2.—Test results obtained on adhesive bonds made to 0.064-inch clad 75S-T6 aluminum alloy sheet (Specification AN-A-10)

Surface treatment for metals[1]	Adhesive	Shear strength: Average[2]	Shear strength: Maximum[2]	Shear strength: Minimum[2]	Shear strength: Average control[3]	Shear strength: Percent of control[4]	Failure: Cohesion[2]	Failure: Adhesion to metal[2]
		P.s.i.	P.s.i.	P.s.i.	P.s.i.		Percent	Percent
CA-1. Vapor degrease in trichloroethylene (No etch)	33	2,296	2,530	1,820	4,101	56	8	92
	34	4,052	4,520	3,570	[5]4,325	94	29	71
	38	2,393	2,880	1,850	[5]2,830	85	53	47
	45	3,057	3,163	2,530	3,322	92	83	17
CA-2. Etch in sulfuric acid-dichromate solution (Etched, good water-film test)	33	4,531	4,760	4,275	4,098	111	23	77
	34	5,040	5,240	4,588	[5]4,843	104	92	8
	38	2,480	2,800	2,151	[5]2,738	91	65	35
	45	2,998	3,160	2,680	3,192	94	52	48
CA-3. Degrease in sodium metasilicate solution (No etch, good-water-film test)	33	4,189	5,200	2,610	4,133	101	7	93
	34	2,780	3,521	1,694	[5]4,325	64	10	90
	38	2,137	2,430	1,910	[5]2,586	83	31	69
	45	3,260	3,490	2,930	3,160	103	64	36
CA-4. Degrease in sodium metasilicate solution, rinse in chromic acid. (No etch good water-film test)	33	3,802	4,126	3,208	4,098	93	15	85
	34	4,370	4,630	3,827	[5]4,607	95	86	14
	38	2,551	2,712	2,280	[5]2,480	103	76	24
	45	3,250	3,438	3,020	3,231	101	69	31

[1]The appearance of the surfaces following each treatment as to any noticeable etch or surface coatings and the results of the water-film tests are given in parentheses.

[2]Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1 inch wide, three from each of three bonded panels. Percentages of failure are averages for these groups of nine specimens.

[3]Average strength for nine control specimens of a 0.5-inch lap-joint of 0.064-inch clad 24S-T3 aluminum alloy cleaned with sulfuric acid-dichromate solution and bonded in the same bonding run and under the same conditions as the test specimens.

[4]The percent of control strength is based on the ratio of the average strength for the group of test specimens to the average strength of the control specimens.

[5]Test results obtained on six 0.5-inch lap-joint specimens, 1 inch wide, three from each of two bonded panels.

Table 3.--Test results obtained on adhesive bonds made to 0.064-inch bare 24S-T3 aluminum alloy sheet (Specification AN-A-12)

Surface treatment for metals[1]	Adhesive	Shear strength: Average[2]	Shear strength: Maximum[2]	Shear strength: Minimum[2]	Shear strength: Average control[3]	Shear strength: Percent of control[4]	Failure: Cohesion[2]	Failure: Adhesion to metal or coating[2]	Failure: Coating[2]
		P.s.i.	P.s.i.	P.s.i.	P.s.i.		Percent	Percent	Percent
BA-1. Chromic acid anodize (Light gray film, good water-film test)	33	448[5]	540	50[5]	4,435	10	0	100	0
	34	1,055	1,150	990	4,409	24	0	100	0
	38	2,115	2,460	1,806	2,824	75	20	80	0
	45	912	1,100	720	3,283	28	0	100	0
BA-2. Sulfuric acid anodize (Light green film, good water-film test)	33	2,551	3,220	2,020	4,435	57	31	69	0
	34	2,936	3,280	2,290	4,182	70	0	0	100
	38	3,087	3,150	3,060	2,758	112	85	15	0
	45	2,893	3,530	2,210	3,283	88	64	18	18
BA-3. Etch in sulfuric acid-dichromate solution (Etched, good water-film test)	33	4,128	4,960	2,950	4,101	101	97	2	1
	34	4,807	5,020	4,654	4,843	99	98	2	0
	38	2,467	2,837	1,780	2,583	96	60	40	0
	45	3,010	3,183	2,900	3,192	94	90	10	0

[1]The appearance of the surfaces following each treatment as to any noticeable etch or surface coatings and the results of the water-film tests are given in parentheses.

[2]Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1 inch wide, three from each of three bonded panels. Percentages of failure are averages for these groups of nine specimens.

[3]Average strength for nine control specimens of a 0.5-inch lap-joint of 0.064-inch clad 24S-T3 aluminum alloy cleaned with sulfuric acid-dichromate solution and bonded in the same bonding run and under the same conditions as the test specimens.

[4]The percent of control strength is based on the ratio of the average strength for the group of test specimens to the average strength of the control specimens.

[5]Several of the specimens failed during sawing, but they were disregarded in computing average strength.

Table 4.--Test results obtained on adhesive bonds made to 0.064-inch bare 75S-T6 aluminum alloy sheet (Specification AN-A-9a)

Surface treatment for metals[1]	Adhesive	Shear strength: Average[2]	Shear strength: Maximum[2]	Shear strength: Minimum[2]	Shear strength: Average control[3]	Shear strength: Percent of control[4]	Failure: Cohesion[2]	Failure: Adhesion to metal or coating[2]	Failure: Coating[2]
		P.s.i.	P.s.i.	P.s.i.	P.s.i.		Percent	Percent	Percent
BA-1. Chromic acid anodize (Light gray film, good water-film test)	33	[5]480	480	[5]0	4,435	11	0	100	0
	34	1,386	1,700	970	4,367	32	0	100	0
	38	2,310	2,410	2,170	2,977	78	25	75	0
	45	873	1,060	600	3,283	27	0	100	0
BA-2. Sulfuric acid anodize (Light green film, good water-film test)	33	2,885	3,680	2,551	4,435	65	16	72	12
	34	3,231	3,520	2,900	4,182	77	0	0	100
	38	2,812	2,900	2,640	2,811	100	96	4	0
	45	3,361	3,570	2,840	3,283	102	89	11	0
BA-3. Etch in sulfuric acid-dichromate solution (Etched, good water-film test)	33	5,518	5,900	4,840	4,101	134	99	1	0
	34	5,553	5,817	5,088	4,843	115	100	0	0
	38	2,591	2,727	2,361	[6]2,738	95	68	32	0
	45	3,060	3,210	2,860	3,177	97	62	38	0

[1] The appearance of the surfaces following each treatment as to any noticeable etch or surface coatings and the results of the water-film tests are given in parentheses.

[2] Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1 inch wide, three from each of three bonded panels. Percentages of failure are averages for these groups of nine specimens.

[3] Average strength for nine control specimens of a 0.5-inch lap-joint of 0.064-inch clad 24S-T3 aluminum alloy cleaned with sulfuric acid-dichromate solution and bonded in the same bonding run and under the same conditions as the test specimens.

[4] The percent of control strength is based on the ratio of the average strength for the group of test specimens to the average strength of the control specimens.

[5] Several of the specimens failed during sawing, but they were disregarded in computing average strengths.

[6] Test result obtained on six 0.5-inch lap-joint specimens, 1 inch wide, three from each of two bonded panels.

Table 5.--Test results obtained on adhesive bonds made to 0.064-inch, oiled, plain-finish, FS1-H24 magnesium alloy sheets (Specification QQ-M-44

Surface treatment for metals[1]	Adhesive	Shear strength: Average[2]	Maximum[2]	Minimum[2]	Average control[3]	Percent of control[4]	Failure: Cohesion[2]	Adhesion to metal or coating[2]	Coating[2]
		P.s.i.	P.s.i.	P.s.i.	P.s.i.		Percent	Percent	Percent
M-1. Degrease in sodium metasilicate pyrophosphate solution (No etch, good water-film test)	33	1,366	1,800	1,090	4,990	27	97	3	0
	34	1,331	1,550	1,170	4,349	31	0	100	0
	38	1,558	1,710	1,420	3,024	44	81	19	0
	45	1,133	1,260	920	3,290	34	91	9	0
M-2. Type III, MIL-M-3171 treatment (Dark brown film, good water-film test)	33	1,066	1,396	510	4,559	23	20	75	5
	34	1,567	1,775	1,255	4,349	36	0	0	100
	38	1,821	2,000	1,640	2,844	64	9	91	0
	45	1,100	1,268	928	3,502	31	12	86	2
M-3. Type III, MIL-M-3171 treatment and two coats of zinc chromate primer	33	527	900	200	4,559	12	0	80	20
	34	290	360	180	4,399	7	0	1	99
	38	379	483	262	2,597	15	0	17	83
	45	834	968	574	3,502	24	0	0	100
M-4. Manodyze treatment (Light greenish-brown film, good water-film test)	33	484	775	302	4,280	11	0	100	0
	34	1,092	1,320	840	3,900	28	4	96	0
	38	1,373	2,000	710	3,192	43	37	62	0
	45	430	650	210	3,201	13	0	100	0

Table 5.—Test results obtained on adhesive bonds made to 0.064-inch, oiled, plain-finish, FS1-H24 magnesium alloy sheets (Specification QQ-M-44) (continued)

Surface treatment for metals[1]	Adhesive	Shear strength					Failure		
		Average[2]	Maximum[2]	Minimum[2]	Average control[3]	Percent of control[4]	Cohesion[2]	Adhesion to metal or coating[2]	Coating[2]
		P.s.i.	P.s.i.	P.s.i.	P.s.i.		Percent	Percent	Percent
M-5. Manodyze treatment and two coats of zinc chromate primer	33	666	940	529	4,280	16	0	0	100
	34	363	450	[5] 0	3,900	9	1	0	99
	38	1,091	1,320	770	3,192	34	4	0	96
	45	375	480	260	3,201	12	0	0	100

[1]The appearance of the surfaces following each treatment as to any noticeable etch or surface coatings and the results of the water-film tests are given in parentheses.

[2]Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1 inch wide, three from each of three bonded panels. Percentages of failure are averages for these groups of nine specimens.

[3]Average strength for nine control specimens of a 0.5-inch lap-joint of 0.064-inch clad 24S-T3 aluminum alloy cleaned with sulfuric acid-dichromate solution and bonded in the same bonding run and under the same conditions as the test specimens.

[4]The percent of control strength is based on the ratio of the average strength for the group of test specimens to the average strength of the control specimens.

[5]Several of the specimens failed during sawing, but they were disregarded in computing average strength.

Table 6.--Test results obtained on adhesive bonds made to 0.064-inch, 2B finish, type 302, 18-8 stainless steel sheets (Specificiation MIL-S-5059)

Surface treatment of metal[1]	Adhesive	Shear strength: Average[2]	Shear strength: Maximum[2]	Shear strength: Minimum[2]	Shear strength: Average control[3]	Shear strength: Percent of control[4]	Failure: Cohesion[2]	Failure: Adhesion to metal[2]
		P.s.i.	P.s.i.	P.s.i.	P.s.i.		Percent	Percent
SS-1. Degrease in sodium metasilicate-pyrophosphate solution (No etch, good water-film test)	33	3,265	4,050	2,080	4,287	76	37	63
	34	4,408	4,489	4,303	4,687	95	6	94
	38	1,061	1,250	980	2,744	39	0	100
	45	2,388	2,910	1,900	3,286	73	13	87
SS-2. Degrease in sodium metasilicate pyrophosphate solution etch in sulfuric-hydrochloric and nitric-hydrofluoric acids. (No appreciable etch, good water-film test)	33	3,317	3,910	2,780	4,287	77	72	28
	34	4,388	4,649	4,052	4,606	95	35	65
	38	1,456	1,740	1,310	2,977	49	5	95
	45	1,191	1,990	520	3,012	40	12	88
SS-3. Degrease in sodium metasilicate pyrophosphate solution, rinse in chromic acid. (No etch, good water-film test)	33	2,565	3,290	2,200	4,287	60	31	69
	34	4,571	4,771	4,240	4,606	99	19	81
	38	1,033	1,177	960	2,899	36	0	100
	45	961	1,240	734	3,012	32	4	96

[1]The appearance of the surfaces following each treatment as to any noticeable etch or surface coatings and the results of the water-film tests are given in parentheses.

[2]Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1 inch wide, three from each of three bonded panels. Percentages of failure are averages for these groups of nine specimens.

[3]Average strength for nine control specimens of a 0.5-inch lap joint of 0.064-inch clad 24S-T3 aluminum alloy cleaned with sulfuric acid-dichromate solution and bonded in the same bonding run and under the same conditions as the test specimens.

[4]The percent of control strength is based on the ratio of the average strength for the group of test specimens to the average strength of the control specimens.

Table 7.—Test results obtained on adhesive bonds made to 0.064-inch, annealed, SAE 4130 chrome-molybdenum steel alloy sheets

Surface treatment of metal[1]	Adhesive	Shear strength: Average[2]	Shear strength: Maximum[2]	Shear strength: Minimum[2]	Shear strength: Average control[3]	Shear strength: Percent of control[4]	Failure: Cohesion[2]	Failure: Adhesion to metal	Failure: Coating[2]
		P.s.i.	P.s.i.	P.s.i.	P.s.i.		Percent	Percent	Percent
SA-1. Vapor degrease in trichloroethylene (No etch)	33	4,206	4,500	3,620	4,287	98	8	92	0
	34	3,318	3,700	2,580	4,030	82	7	93	0
	38	1,124	1,320	890	2,958	38	0	58	42
	45	3,045	3,260	2,615	3,793	80	31	69	0
SA-2. Etch in sulfuric acid-dichromate solution (Etched oxide film, good water-film test)	33	2,629	3,100	2,320	4,287	61	11	89	0
	34	4,277	4,440	4,150	4,030	106	19	81	0
	38	1,439	1,940	1,030	2,958	49	0	0	100
	45	1,970	2,300	1,460	3,437	57	0	100	0
SA-3. Degrease in sodium metasilicate-pyrophosphate solution (No etch, good water-film test)	33	2,870	4,520	1,400	4,287	67	50	50	0
	34	3,951	4,640	3,520	4,687	85	1	99	0
	38	1,951	2,240	1,600	3,049	64	5	94	1
	45	2,131	2,518	1,679	3,043	70	6	88	6
SA-4. Degrease in sodium metasilicate-pyrophosphate solution, and rinse in chromic acid. (No etch, good water-film test	33	3,610	4,480	2,800	4,287	84	54	46	0
	34	4,324	4,440	4,150	4,379	99	29	79	0
	38	1,712	1,920	1,440	3,049	56	2	96	2
	45	2,533	3,180	2,180	3,415	74	2	97	1

[1]The appearance of the surfaces following each treatment as to any noticeable etch or surface coatings and the results of the water-film tests are given in parentheses.

[2]Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1 inch wide, three from each of three bonded panels. Percentages of failure are averages for these groups of nine specimens.

[3]Average strength for nine control specimens of a 0.5-inch lap joint of 0.064-inch clad 24S-T3 aluminum alloy cleaned with sulfuric acid-dichromate solution and bonded in the same bonding run and under the same conditions as the test specimens.

[4]The percent of control strength is based on the ratio of the average strength for the group of test specimens to the average strength of the control specimens.

Table 8.--Test results obtained on adhesive bonds made to 0.032-inch, RC-70-1/2 hard titanium alloy sheets

Surface treatment of metal[1]	Adhesive	Shear strength: Average[2]	Shear strength: Maximum[2]	Shear strength: Minimum[2]	Shear strength: Average control[3]	Shear strength: Percent of control[4]	Failure: Cohesion[2]	Failure: Adhesion to metal[2]
		P.s.i.	P.s.i.	P.s.i.	P.s.i.		Percent	Percent
T-1. Degrease in sodium metasilicate-pyrophosphate solution (No etch, good water-film test)	33	[5]1,402	2,619	[5]0	4,287	33	0	100
	34	2,043	2,803	1,261	4,687	44	0	100
	38	821	880	700	2,744	30	0	100
	45	797	1,176	408	2,785	29	5	95
T-2. Degrease in sodium metasilicate-pyrophosphate and rinse in chromic acid solution (No etch, good water-film test)	33	[5]400	400	[5]0	4,287	9	0	100
	34	1,800	3,287	969	4,606	39	1	99
	38	418	530	418	3,134	13	0	100
	45	[5]213	240	[5]0	3,437	6	0	100
T-3. Etch in nitric-hydrofluoric acid solution (No noticeable etch, poor water-film test)	33	1,161	1,495	955	4,287	27	0	100
	34	3,354	4,053	1,989	4,606	73	21	79
	38	855	1,080	670	3,134	27	1	99
	45	843	1,240	618	3,328	25	1	99
T-4. Etch in nitric-hydrofluoric acid solution and rinse in chromic acid (No noticeable etch, poor water-film test)	33	2,143	2,380	1,772	4,287	50	0	100
	34	2,011	2,570	1,150	4,606	44	0	100
	38	941	1,180	650	3,134	30	0	100
	45	690	1,009	362	2,695	26	1	99

[1]The appearance of the surfaces following each treatment as to any noticeable etch or surface coatings and the results of the water-film tests are given in parentheses.

[2]Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1 inch wide, three from each of three bonded panels. Percentages of failure are averages for these groups of nine specimens.

[3]Average strength for nine control specimens of a 0.5-inch lap joint of 0.064-inch clad 24S-T3 aluminum alloy cleaned with sulfuric acid-dichromate solution and bonded in the same bonding run and under the same conditions as the test specimens.

[4]The percent of control strength is based on the ratio of the average strength for the group of test specimens to the average strength of the control specimens.

[5]Several of the specimens failed during sawing, but they were disregarded in computing average strength.

Table 9.--Tensile properties[1] of metal sheets[2] used in adhesive bonding tests

Metal	Specification	Tensile strength	Yield stress at 0.2 percent strain	Proportional limit stress	Modulus of elasticity	Elongation in 2 inches
		P.s.i.	P.s.i.	P.s.i.	1,000 p.s.i.	Percent
24S-T3 clad aluminum	AN-A-13	68,750 [3](62,000)	51,100 (40,000)	45,800	10,100	20.3 (13.0)
24S-T3 bare aluminum	AN-A-12	67,800 (64,000)	49,400 (42,000)	41,500	10,200	16.4 (15.0)
75S-T6 clad aluminum	AN-A-10b	79,650 (72,000)	72,100 (62,000)	52,100	9,600	12.6 (8.0)
75S-T6 bare aluminum	AN-A-9a	80,700 (77,000)	73,500 (66,000)	62,100	9,900	13.2 (8.0)
FS-1-H24 magnesium	QQ-M-44	41,650 (39,000)	29,900 (29,000)	15,800	6,100	7.2 (4.0)
Type 302, 18-8 stainless steel, annealed	M-S-5059	95,200 (75,000)	47,000	20,500	27,800	71.0 (50.0)
SAE 4130 steel, annealed	AN-QQ-S685	70,200 (85,000 max.)	41,600	18,600	27,900	26.8
RC-70 titanium, 1/2 hard	....	[4]130,000	[4]119,000	....	....	[4]10.0

[1]Properties obtained following the general methods of Federal Specification QQ-M-151, "Metals, General Specification for Inspection Of." Values given are for two or three tests on each metal made parallel to the roll direction of the sheet.

[2]The metal sheets were approximately 0.064-inch thick, except for the titanium metal, which was 0.032-inch thick.

[3]Values in parentheses are minimums required by the Specification listed.

[4]Tensile properties supplied by the manufacturer.

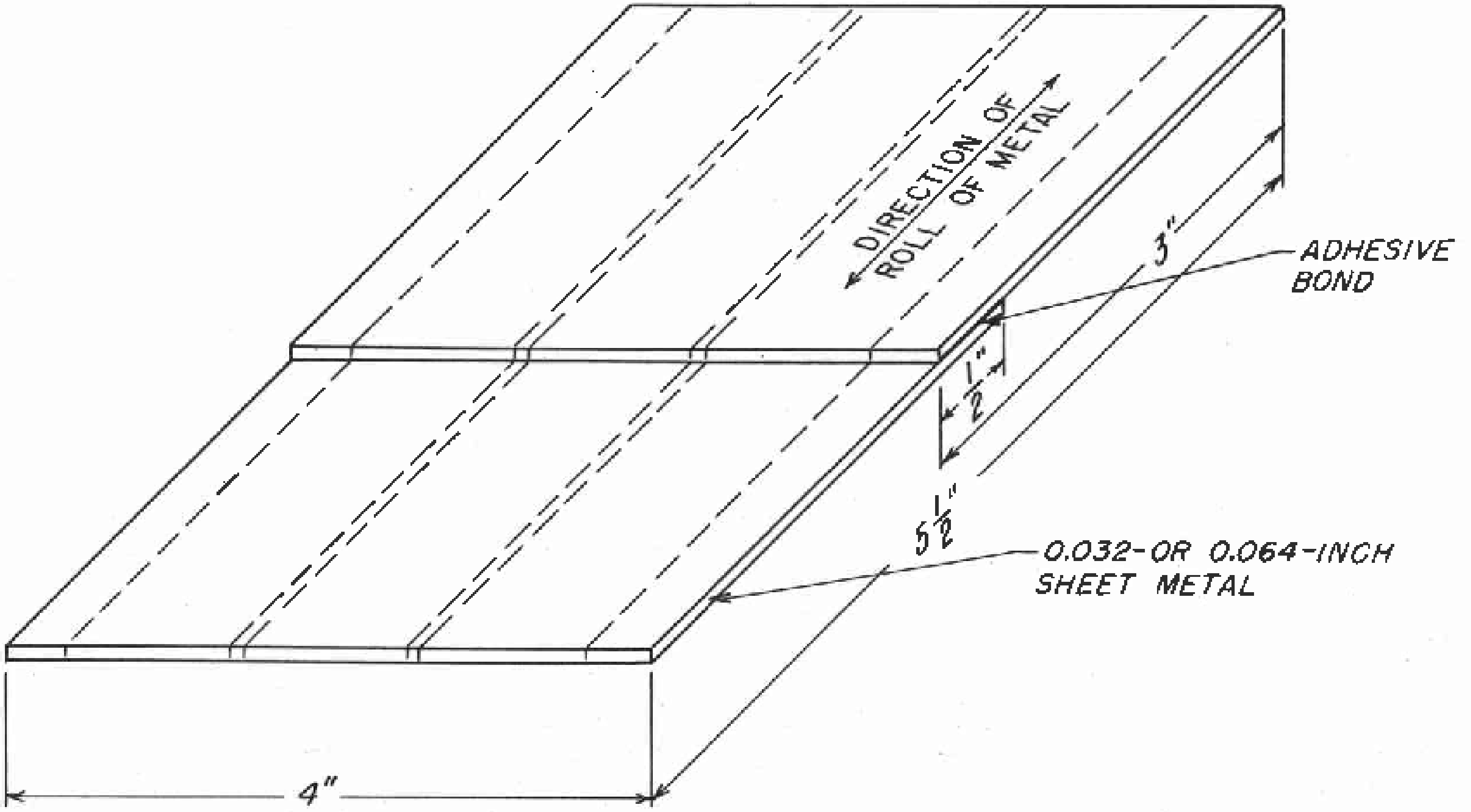

Figure 1.--Lap-joint metal test panels prepared for the investigation of the adhesive bonding properties of metal sheets given various chemical and anodizing treatments. The approximate positions of the 1-inch wide test specimens are indicated by dotted outlines.

ZM 92375 F

Addendum Salt-Water Spray Tests

ADHESIVE BONDING PROPERTIES OF VARIOUS METALS AS AFFECTED BY CHEMICAL AND ANODIZING TREATMENTS OF THE SURFACE

Procedure

The investigation reported in Forest Products Laboratory Report No. 1842 included original dry tests made at 72° to 76° F. on lap-joint specimens of magnesium, stainless steel, chrome-molybdenum steel, titanium, and bare and clad aluminum. Three to 5 surface treatments were included with each metal, and 4 commercial metal-bonding adhesives were investigated. Six lap-joint panels, 4 by 5-1/2 inches, having 1/2-inch overlaps, were prepared to represent each metal-treatment-adhesive variable. The bonded panels of aluminum and magnesium were given further treatment with two coats of zinc chromate primer prior to exposure or testing. In the original investigation, only 3 of the 6 panels representing each bonding variable were cut into individual specimens and tested.

If initial dry strengths were satisfactory, the remaining 3 test panels with these same bonding variables were subsequently given a 30-day salt-water spray exposure (specimens exposed by Wright Air Development Center according to Federal Specification QQ-M-151). After exposure, the panels were cut into individual 1-inch-wide test specimens (3 specimens per panel) on a metal-cutting bandsaw, and then tested dry at 72° to 76° F., as in the initial dry tests, by tensile loading at a rate of 600 pounds per square inch per minute. No complete salt-water spray tests were run on specimens of the chrome-molybdenum steel alloy because of excessive corrosion of the metal.

Results

The results of these tests after salt-water spray exposure are given in tables 10 through 16.

Clad 24S-T3 and 75S-T6 Aluminum (Tables 10 and 11)

Adhesive 38 generally showed the lowest resistance to the salt-water spray, retaining only 31 to 52 percent of the original bond strength for 24S-T3 alloy and 55 to 77 percent for the 75S-T6 alloy. The original bond strengths with this adhesive were lower (2,416 to 2,596 pounds per square inch) than for the other 3 adhesives.

The other adhesives, 33, 34, and 45, generally showed good resistance to the salt-water spray exposure, even with cleaning treatments other than sulfuric acid-dichromate (treatment CA-2). Exceptions to this good performance of adhesives 33, 34, and 45 were vapor degreasing (CA-1) for bonding with adhesive 33, and an inconsistent value on clad 75S-T6 alloy treated by method CA-3 and bonded with adhesive 34. It may be noted that in each of these three low strength values after salt-water spray, the original dry strengths of the joints were also low.

Bare 24S-T3 and 75S-T6 Aluminum (Tables 12 and 13)

With one exception, the high-strength adhesives 33, 34, and 45 showed good salt-water spray resistance in bonds given sulfuric acid anodize (BA-2) or sulfuric acid-dichromate etch (BA-3) treatments. Bond strengths exceeded 2,500 pounds per square inch, and 83 percent of the original control strengths. With adhesives 33 and 34 to the bare aluminum alloys treated with sulfuric acid-dichromate (BA-3), the bond strengths after exposure were more than 4,500 pounds per square inch. On bare 24S-T3 aluminum prepared by the sulfuric acid anodizing treatment (BA-2), adhesive 33 retained, after salt-water spray exposure, only 69 percent of its initial dry strength, but it retained all the initial dry strength on the bare 75S-T6 aluminum prepared by the same process.

Adhesive 38 did not, in general, give high-quality bonds to the bare aluminum alloys after the salt-water spray exposure. This adhesive had shown much better initial dry strengths (tables 3 and 4 of Forest Products Laboratory Report No. 1842) on bare aluminum prepared by the chromic acid anodize process (BA-1) than did the other 3 adhesives, and was the only one evaluated after this process under salt spray conditions.

FS-1H24 Magnesium (Table 14)

Bond strengths to the magnesium metal were generally low after 30 days of salt-water spray exposure. Only the use of the type III MIL-M-3171 treatment (M-2) with adhesives 34 and 38 and Manodyze treatment (M-4) with adhesive 38 gave bond strengths of more than 1,000 pounds per square inch.

Stainless Steel (Table 15)

Adhesive 34 still showed good bonding to stainless steel after 30 days' salt-water spray exposure for the 3 surface treatments (SS-1, SS-2, and SS-3). The bonds with adhesive 33 were inconsistent, with best strength and uniformity being obtained with the alkaline degreasing process (SS-1).

Titanium (Table 16)

The highest strengths in titanium-to-titanium bonds in the original dry tests (table 8 of Forest Products Laboratory Report No. 1842) were obtained with treatment T-3 (nitric-hydrofluoric acid etch) and adhesive 34. This combination still had 78 percent of the original bond strengths after the salt-water spray exposure and had an average bond strength of 2,600 pounds per square inch. None of the other adhesives gave sufficiently high initial dry strengths on titanium to justify exposure to salt-water spray.

Table 10.--Test results obtained after 30-day salt-water spray exposure of bonds made to 0.064-inch clad 24S-T3 aluminum alloy sheet

Surface treatment for metal[1]	Adhesive[1]	Shear strength[2]				Failure[2]	
		Average	Maximum	Minimum	Percent unexposed controls[3]	Cohesion	Adhesion to metal or coating
		P.s.i.	P.s.i.	P.s.i.		Percent	Percent
CA-1. Vapor degrease in trichloroethylene	33	1,696	2,180	1,370	80	0	100
	34	3,591	3,890	3,340	99	23	77
	38	802	1,240	450	33	2	98
	45	3,275	3,564	2,979	108	48	52
CA-2. Etch in sulfuric acid-dichromate solution	33	4,500	4,878	3,990	109	17	83
	34	4,768	4,950	4,607	99	91	9
	38	1,351	1,947	918	52	44	56
	45	3,500	3,660	3,410	110	63	37
CA-3. Degrease in sodium metasilicate solution	33	4,706	4,885	4,470	160	17	83
	34	3,186	3,640	1,653	86	16	84
	38	811	1,420	600	31	18	82
	45	3,293	3,690	1,990	99	69	31
CA-4. Degrease in sodium metasilicate and rinse in chromic acid	33	3,537	3,867	3,220	101	7	93
	34	3,170	3,937	2,317	93	22	78
	38	766	1,770	250	31	15	85
	45	3,279	3,500	2,630	104	77	23

[1] The surface treatments and adhesive processes are described in detail in the section on procedures of Forest Products Laboratory Report No. 1842.

[2] Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1-inch wide, 3 cut from each of 3 bonded panels after exposure. Percentages of failure are averages for these groups of 9 specimens.

[3] The values for the percentage of shear strength of the unexposed controls are based on the ratios of the average shear strengths for the specimens after exposure to the average shear strength of the 9 specimens prepared under the same conditions (Forest Products Laboratory Report No. 1842) as the exposed specimens, but tested without being exposed.

Table 11.--Test results obtained after 30-day salt-water spray exposure of bonds made to 0.064-inch clad 75S-T6 aluminum alloy sheet

Surface treatment for metal[1]	Adhesive[1]	Shear strength[2]				Failure[2]	
		Average	Maximum	Minimum	Percent unexposed controls[3]	Cohesion	Adhesion to metal or coating
		P.s.i.	P.s.i.	P.s.i.		Percent	Percent
CA-1. Vapor degrease in trichloroethylene	33	2,053	2,418	1,928	89	0	100
	34	4,037	4,330	3,600	99	54	46
	38	1,314	1,530	1,104	55	9	91
	45	3,447	3,660	2,980	113	70	30
CA-2. Etch in sulfuric acid-dichromate solution	33	4,684	4,980	4,200	103	15	85
	34	4,584	4,950	4,150	90	92	8
	38	1,714	1,878	1,520	69	36	64
	45	3,355	3,740	2,606	112	70	30
CA-3. Degrease in sodium metasilicate solution	33	4,744	5,200	4,260	113	10	90
	34	1,984	2,448	1,550	71	3	97
	38	1,650	1,938	1,490	77	8	92
	45	3,441	3,851	3,092	106	71	29
CA-4. Degrease in sodium metasilicate and rinse in chromic acid	33	3,273	4,080	1,960	86	12	88
	34	4,586	4,720	4,423	105	84	16
	38	1,591	1,860	1,340	62	24	76
	45	3,340	3,640	3,082	103	81	19

[1] The surface treatments and adhesive processes are described in detail in the section on procedures of Forest Products Laboratory Report No. 1842.

[2] Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1-inch wide, 3 cut from each of 3 bonded panels after exposure. Percentages of failure are averages for these groups of 9 specimens.

[3] The values for the percentage of shear strength of the unexposed controls are based on the ratios of the average shear strengths for the specimens after exposure to the average shear strength of the 9 specimens prepared under the same conditions (Forest Products Laboratory Report No. 1842) as the exposed specimens, but tested without being exposed.

Table 12.--Test results obtained after 30-day salt-water spray exposure of bonds made to 0.064-inch bare 24S-T3 aluminum alloy sheet

Surface treatment for metal[1]	Adhesive[1]	Shear strength[2]				Failure[2]	
		Average	Maximum	Minimum	Percent unexposed controls[3]	Cohesion	Adhesion to metal or coating
		P.s.i.	P.s.i.	P.s.i.		Percent	Percent
BA-1. Chromic acid anodize	38	1,130	1,250	802	53	5	95
BA-2. Sulfuric acid anodize	33	1,764	2,190	1,530	69	100	0
	34	2,606	3,377	2,060	89	3	97
	38	2,297	2,580	1,960	74	64	36
	45	3,037	3,610	2,210	105	98	2
BA-3. Etch in sulfuric acid-dichromate solution	33	4,863	5,000	4,500	118	55	45
	34	4,868	5,220	4,113	101	0	100
	38	1,400	1,790	894	57	30	70
	45	2,773	3,020	2,390	92	43	57

[1]The surface treatments and adhesive processes are described in detail in the section on procedures of Forest Products Laboratory Report No. 1842.

[2]Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1-inch wide, 3 cut from each of 3 bonded panels after exposure. Percentages of failure are averages for these groups of 9 specimens.

[3]The values for the percentage of shear strength of the unexposed controls are based on the ratios of the average shear strengths for the specimens after exposure to the average shear strength of the 9 specimens prepared under the same conditions (Forest Products Laboratory Report No. 1842) as the exposed specimens, but tested without being exposed.

Table 13.--Test results obtained after 30-day salt-water spray exposure of bonds made to 0.064-inch bare 75S-T6 aluminum alloy sheet

Surface treatment for metal[1]	Adhesive[1]	Shear strength[2]				Failure[2]	
		Average	Maximum	Minimum	Percent unexposed controls[3]	Cohesion	Adhesion to metal or coating
		P.s.i.	P.s.i.	P.s.i.		Percent	Percent
BA-1. Chromic acid anodize	38	1,816	1,873	1,770	85	18	82
BA-2. Sulfuric acid anodize	33	3,309	3,930	2,290	130	64	36
	34	3,739	4,340	3,430	127	85	15
	38	1,701	1,920	1,410	55	45	55
	45	3,438	3,920	2,765	119	100	0
BA-3. Etch in sulfuric acid-dichromate solution	33	4,558	5,440	3,400	83	98	2
	34	5,680	5,940	5,260	102	100	0
	38	1,480	1,690	1,290	57	38	62
	45	3,405	3,655	3,265	111	64	36

[1] The surface treatments and adhesive processes are described in detail in the section on procedures of Forest Products Laboratory Report No. 1842.

[2] Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1-inch wide, 3 cut from each of 3 bonded panels after exposure. Percentages of failure are averages for these groups of 9 specimens.

[3] The values for the percentage of shear strength of the unexposed controls are based on the ratios of the average shear strengths for the specimens after exposure to the average shear strength of the 9 specimens prepared under the same conditions (Forest Products Laboratory Report No. 1842) as the exposed specimens, but tested without being exposed.

Table 14.--Test results obtained after 30-day salt-water spray exposure of bonds made to 0.064-inch FS-1H24 magnesium alloy sheet

Surface treatment for metal[1]	Adhesive[1]	Shear strength[2]				Failure[2]		
		Average	Maximum	Minimum	Percent of unexposed control[3]	Cohesion	Adhesion to metal or coating	Coating
		P.s.i.	P.s.i.	P.s.i.		Percent	Percent	Percent
M-1. Degrease in sodium metasilicate-pyrophosphate solution	34	608	730	522	46	0	100	0
	38	887	1,130	640	57	41	59	0
M-2. Type III, MIL-M-3171 treatment	34	1,386	1,553	1,095	89	0	0	100
	38	1,407	1,540	1,200	77	2	98	0
M-4. Manodyze treatment	34	685	1,020	310	62	0	100	0
	38	1,047	1,250	740	76	9	91	0
M-5. Manodyze treatment and 2 coats zinc chromate primer	38	774	1,470	120	71	6	0	94

[1]The surface treatments and adhesive processes are described in detail in the section on procedures of Forest Products Laboratory Report No. 1842.

[2]Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1-inch wide, 3 cut from each of 3 bonded panels after exposure. Percentages of failure are averages for these groups of 9 specimens.

[3]The values for the percentage of shear strength of the unexposed controls are based on the ratios of the average shear strengths for the specimens after exposure to the average shear strength of the 9 specimens prepared under the same conditions (Forest Products Laboratory Report No. 1842) as the exposed specimens, but tested without being exposed.

Table 15.--Test results obtained after 30-day salt-water spray exposure of bonds made to 0.064-inch, 2B finish, type 302 annealed stainless-steel sheet

Surface treatment for metal[1]	Adhesive[1]	Shear strength[2]				Failure[2]	
		Average	Maximum	Minimum	Percent unexposed controls[3]	Cohesion	Adhesion to metal or coating
		P.s.i.	P.s.i.	P.s.i.		Percent	Percent
SS-1. Degrease in sodium metasilicate-pyrophosphate solution	33	3,311	4,570	1,596	101	54	46
	34	3,597	4,340	2,489	82	19	81
SS-2. Degrease in sodium metasilicate-pyrophosphate solution and etch in sulfuric-hydrochloric and nitric-hydrofluoric acids	33	2,336	3,186	250	70	59	41
	34	4,014	4,122	3,969	92	44	56
SS-3. Degrease in sodium metasilicate-pyrophosphate solution and rinse in chromic acid	33	2,237	3,540	900	87	23	77
	34	4,092	4,337	3,878	89	15	85

[1] The surface treatments and adhesive processes are described in detail in the section on procedures of Forest Products Laboratory Report No. 1842.

[2] Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1-inch wide, 3 cut from each of 3 bonded panels after exposure. Percentages of failure are averages for these groups of 9 specimens.

[3] The values for the percentage of shear strength of the unexposed controls are based on the ratios of the average shear strengths for the specimens after exposure to the average shear strength of the 9 specimens prepared under the same conditions (Forest Products Laboratory Report No. 1842) as the exposed specimens, but tested without being exposed.

Table 16.--Test results obtained after 30-day salt-water spray exposure of bonds made to 0.032-inch RC-70, 1/2 hard titanium alloy sheet

Surface treatment for metal[1]	Adhesive[1]	Shear strength[2]				Failure[2]	
		Average	Maximum	Minimum	Percent unexposed controls[3]	Cohesion	Adhesion to metal or coating
		P.s.i.	P.s.i.	P.s.i.		Percent	Percent
T-1. Degrease in sodium metasilicate-pyrophosphate solution	34	894	1,136	703	44	0	100
T-2. Degrease in sodium metasilicate-pyrophosphate solution and rinse in chromic acid solution	34	1,862	2,244	1,429	103	0	100
T-3. Etch in nitric-hydrofluoric acid solution	34	2,601	3,040	2,032	78	17	83
T-4. Etch in nitric-hydrofluoric acid solution and rinse in chromic acid	34	1,439	2,221	593	72	0	100

[1]The surface treatments and adhesive processes are described in detail in the section on procedures of Forest Products Laboratory Report No. 1842.

[2]Average, maximum, and minimum test results obtained on groups of nine 0.5-inch lap-joint specimens, 1-inch wide, 3 cut from each of 3 bonded panels after exposure. Percentages of failure are averages for these groups of 9 specimens.

[3]The values for the percentage of shear strength of the unexposed controls are based on the ratios of the average shear strengths for the specimens after exposure to the average shear strength of the 9 specimens prepared under the same conditions (Forest Products Laboratory Report No. 1842) as the exposed specimens, but tested without being exposed.