

A STUDY OF METHODS FOR PREPARING CLAD 24S-T ALUMINUM-ALLOY SHEET SURFACES FOR ADHESIVE BONDING

**PART III. EFFECT OF CLEANING METHOD ON
RESISTANCE OF BONDED JOINTS TO SALT-WATER SPRAY**

December 1950

~~INFORMATION REVIEWED~~

~~AND REAFFIRMED~~

~~March 1956~~

INFORMATION REVIEWED

AND REAFFIRMED

1962



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

No. 1813-A

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

A STUDY OF METHODS FOR PREPARING CLAD 24S-T3 ALUMINUM-ALLOY

SHEET SURFACES FOR ADHESIVE BONDING¹

PART III. EFFECT OF CLEANING METHOD ON RESISTANCE OF
BONDED JOINTS TO SALT-WATER SPRAY²

By
H. W. EICKNER,
Chemical Engineer

Forest Products Laboratory³, Forest Service
United States Department of Agriculture

Summary

As a third part of the investigation of methods for preparing aluminum-alloy sheets for adhesive bonding that was reported in Forest Products Laboratory Report No. 1813, 0.064-inch 24S-T3 clad aluminum-alloy sheets having surfaces as normally received, silicate-stained surfaces, or oil-paraffin-treated surfaces, were cleaned by several representative cleaning methods, and then 1-inch lap-joint specimens were bonded by three adhesive bonding processes A, B, and C. The joint strengths of these bonds were then compared before and after a 30-day exposure to salt-water spray.

The use of a sodium dichromate-sulfuric acid cleaning method in preparing aluminum-alloy sheets having the three types of surface condition gave the best original bond strength with all three bonding processes, and these bonds also showed the highest strengths after a 30-day exposure to salt-water spray. Joints having moderate strength, both before and after a 30-day exposure to salt-water spray, were

¹This progress report is one of a series prepared and distributed by the Forest Products Laboratory under U. S. Navy Bureau of Aeronautics No. NBA-PO-NAer 00619, Amendment No. 2, and U. S. Air Force No. USAF-PO-(33-038)49-4696E.

²The first two parts of this study are reported in Forest Products Laboratory Report No. 1813, under the same title, dated May 1950.

³Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

obtained when the three bonding processes were used in bonding to aluminum alloy treated by washing with benzene and then abrading with aluminum wool. Even though moderately good initial bond strengths could be obtained to aluminum-alloy surfaces prepared by merely washing with benzene, by washing with benzene and immersing in sodium metasilicate solution, or by using the untreated surface, the bonds had poor corrosion resistance.

Introduction

The work described in this report included a study of the strength, before and after exposure to salt-water spray, of adhesive-bonded aluminum-to-aluminum lap-joint specimens tested in shear. Specimens were made from aluminum sheets that were either in the same condition as received or had oil-paraffin or sodium metasilicate stain contaminations on the surfaces. These surfaces were then cleaned by one of five methods before being bonded by each of three adhesive processes. The details of the procedures are as described in parts I and II of Forest Products Laboratory Report No. 1813.

Procedure

Preparation of Test Panels

Test panels were prepared from two 0.064-inch clad 24S-T3 aluminum-alloy sheets, 4 by 6 inches in size, by overlapping them for 1 inch along the 6-inch dimension. The aluminum-alloy sheets were selected or treated so as to have the three types of surface conditions described under part II; namely, surfaces as normally received, surfaces having a metasilicate stain, and surfaces coated with an oil-paraffin film.

The clad aluminum-alloy sheets (4 by 6 inches) having the three kinds of surface condition were then cleaned by a benzene wash and aluminum-wool abrasion (method B2), or a benzene wash and a sodium dichromate-sulfuric acid immersion (method E1). Aluminum-alloy sheets were also included for bonding where the surfaces as received were not given any treatment (method H), were merely washed with benzene (method A), or were washed with benzene and then immersed in sodium metasilicate solution (method D2). Method D2 was also used in preparing for bonding aluminum surfaces having an oil-paraffin film. Detailed descriptions of these various cleaning methods are given under part I of this study (Forest Products Laboratory Report No. 1813). These treated aluminum-alloy sheets were then bonded together by the same metal-bonding processes, A, B, and C, that were used in parts I and II of the study.

Three panels were prepared with each kind of surface condition and by each cleaning method and bonding process. One panel from each of the groups was selected for use as a control, and specimens from this panel were cut and tested as described in Part I. The other two panels were sent to the Air Materiel Command, where they were subjected to a 30-day exposure to salt-water spray.

Conditions of Exposure to Salt-water Spray

The Air Materiel Command at Wright-Patterson Air Force Base exposed the bonded lap-joint panels of 24S-T3 clad aluminum alloy to the standard 30-day exposure to salt-water spray specified in part 5, section IV of Federal Specification QQ-M-151a, "General Specifications for the Inspection of Metals." In this test a 20-percent-by-weight solution of sodium chloride was atomized by the use of compressed air and suitable nozzles so that every 80 square centimeters of the horizontal exposure zone of the test chamber received 0.5 to 3 milliliters of solution per hour. The chamber was maintained at a temperature of 95° F. The panels were hung vertically in the chamber with the 6-inch-wide edges of the adhesive joints in a horizontal position. The panels were also placed so that they were parallel to the horizontal flow of the fog through the chamber. After removal from the chamber, the panels were rinsed and dried and returned to the Forest Products Laboratory, where specimens were cut as they were for the control panels.

Testing

The lap-joint panels were each sawed into five 1-inch-wide test specimens. These specimens were loaded to failure in tension at a rate of 600 pounds per minute. The testing procedure was the same as used in parts I and II of this study. The failing load and estimated areas (expressed as percentages of total area of the joint) of adhesion failure, cohesion failure, and lack of contact were recorded.

Results

In table 7⁴ are given the shear strength results and types of failures obtained in the original control tests of part III, and also in the tests exposing 1-inch lap joints of 0.064-inch clad 24S-T3 aluminum alloy, bonded with three bonding processes, to a 30-day salt-water spray. Five types of cleaning are compared for preparing the clad aluminum-alloy sheets, with three types of surface conditions, for bonding into high-strength corrosion-resistant joints.

Generally, all three adhesive bonding processes gave similar performances in bonds exposed to salt-water spray when the surfaces of

⁴The table is numbered consecutively with those of Forest Products Laboratory Report No. 1813.

the aluminum were originally prepared in the same manner for each process. Exceptions were noted for adhesive A, which showed much lower strengths than B or C when aluminum surfaces contaminated with metasilicate stain were cleaned by washing with benzene and abrading with aluminum wool (method B2), or when aluminum surfaces contaminated with oil and paraffin were cleaned by washing with benzene and immersing in sodium metasilicate solution (method D2). It may be noted that sodium metasilicate solutions had been previously shown in parts I and II of this study to be less effective in cleaning aluminum for bonding with adhesive A than with adhesives B or C. This was also reflected in the original joint strength of specimens cleaned with sodium metasilicate and bonded with adhesive A in the present study. In the present study adhesive B also showed slightly lower strengths after exposure than either A or C when aluminum surfaces contaminated with oil and paraffin were cleaned by washing with benzene and abrading with aluminum wool (method B2), or when aluminum surfaces contaminated with metasilicate stain were cleaned by washing with benzene and immersing in sulfuric acid-sodium dichromate solution (method E1).

When aluminum surfaces as received were treated by washing with benzene (method A), were treated by washing with benzene and immersing in sodium metasilicate solution (method D2), or were not given any treatment (method H), the joints made with all three bonding processes had practically no strength after a 30-day exposure to salt-water spray. Some of these panels had original joint strengths, before exposure, as high as 2,044 to 2,920 pounds per square inch, but even these panels showed this poor resistance to exposure to salt-water spray.

When the aluminum surfaces as normally received were prepared for bonding by washing with benzene and abrading with aluminum wool (method B2), moderate initial joint strengths (2,196 to 2,444 p.s.i.) were obtained with the three bonding processes, and the resistance of these bonds to salt-water spray was also moderately good, as the joint strengths ranged from 1,286 to 1,440 pounds per square inch after the 30-day exposure.

The original quality of bonds made with the three bonding processes to the aluminum surfaces as normally received, which had been treated by washing with benzene and then immersing in sodium dichromate-sulfuric acid solution (method E1), was very good (2,848 to 3,264 p.s.i.); and after a 30-day exposure to salt-water spray these bonds retained 82 to 90 percent of their initial strength. Comparison of these results with those from cleaning by other methods indicated a definite increase in corrosion resistance of these joints as a result of the sulfuric acid-sodium dichromate cleaning process.

The initial-strength results obtained on surfaces having an oil-paraffin film that were cleaned prior to being bonded with the three adhesives, were found to be practically the same as those

obtained when surfaces as received were cleaned and bonded. After a 30-day exposure to salt-water spray, bonds made to the surfaces that originally had an oil-paraffin film were as strong as the bonds that had been made to surfaces as received, and in most instances the corrosion resistance of the joints made on oily surfaces was even better. Where such differences were noted, there was generally less evidence of excessive corrosion in the area of the bond on the specimens that had the oil-paraffin contamination than on the specimens made from aluminum as received. It is difficult to explain this increased corrosion resistance of the surfaces that were coated with oil and paraffin. It is possible that the various treatments used were successful in removing most of the oily layer and thus facilitated adequate bonding, but that the surfaces still retained a very thin film of oil that protected the metal from corrosion.

When silicate-stained surfaces were cleaned by washing with benzene and abrading with aluminum wool (method B2), the three bonding processes gave moderate original bond strengths (1,780 to 2,084 p.s.i.) that were somewhat lower than the strengths when aluminum surfaces as received were cleaned and bonded in the same manner (2,196 to 2,444 p.s.i.). After a 30-day exposure to salt-water spray, the bonds to the silicate-stained surfaces showed moderate joint strengths of 1,422 to 1,446 pounds per square inch for bonds made with bonding processes B and C, respectively, and weak joint strength (650 p.s.i.) for the bonds made with process A.

Treatment of the silicate-stained surfaces by washing with benzene and then immersing in sodium dichromate-sulfuric acid solution (method E1) resulted in high original bond strength with the three bonding processes (2,700 to 3,404 p.s.i.); and after a 30-day exposure to salt-water spray bonds made in this manner showed good joint strength with processes A and C (3,018 and 2,368 p.s.i.) and moderate joint strength with process B (1,818 p.s.i.).

Conclusions

- (1) The three adhesives studied generally showed similar performance in exposures to salt-water spray when the same types of aluminum surfaces were prepared for bonding in the same way.
- (2) Bonds prepared with aluminum surfaces as normally received and either not cleaned or cleaned only with a benzene wash showed no significant resistance by any adhesive to exposure to salt-water spray. However, a benzene wash followed by abrasion with aluminum wool significantly improved corrosion resistance of the joints.
- (3) From the standpoint of both initial dry strength and strength after exposure to salt-water spray, the sodium dichromate-sulfuric acid treatment gave the best results with all types of surfaces and with each of the adhesives investigated.

(4) Specimens bonded to surfaces coated with an oil-paraffin film often had greater corrosion resistance than those bonded to normal surfaces with the same adhesive and cleaned by the same methods.

(5) Treatment of aluminum surfaces having sodium metasilicate stain by washing with benzene and immersing in sodium dichromate-sulfuric acid was found to give much better initial bond strengths and also better bond strengths after exposures for 30 days to salt-water spray than cleaning with a benzene wash and abrading with aluminum wool.

(6) The corrosion resistance of aluminum lap specimens, in which the aluminum as normally received was cleaned with the sodium metasilicate process, was considerably lower than that when the sulfuric acid-sodium dichromate process was used, and it was inferior to the corrosion resistance when normal surfaces were cleaned by benzene washing followed by abrasion with aluminum wool.

Table 7. Effect of original metal surface condition, metal cleaning process, and adhesive bonding process on the strength and the salt-water spray resistance of bonded 1-inch lap joints of 0.005-inch thick clad aluminum alloy

Cleaning method ¹	Surface condition	Bonding process A						Bonding process B						Bonding process C											
		Control test			Test after 30-day salt-water spray exposure			Control test			Test after 30-day salt-water spray exposure			Control test			Test after 30-day salt-water spray exposure								
		Average shear strength ²	Type of failure ³	Ad. : Co. : N.C.	Average shear strength ²	Type of failure ³	Ad. : Co. : N.C.	Average shear strength ²	Type of failure ³	Ad. : Co. : N.C.	Average shear strength ²	Type of failure ³	Ad. : Co. : N.C.	Average shear strength ²	Type of failure ³	Ad. : Co. : N.C.	Average shear strength ²	Type of failure ³	Ad. : Co. : N.C.						
		P.s.i.	Per-cent	Per-cent	P.s.i.	Per-cent	Per-cent	P.s.i.	Per-cent	Per-cent	P.s.i.	Per-cent	Per-cent	P.s.i.	Per-cent	Per-cent	P.s.i.	Per-cent	Per-cent						
A1, benzene wash, no other treatment	As received ⁴	1,440	92	8	0	(7)	100	0	0	1,532	91	9	0	(7)	100	0	0	2,170	42	45	13	68	96	1	3
	As received ⁴	2,444	98	2	0	1,440	82	18	0	2,196	96	4	0	1,286	97	3	0	2,332	93	0	10	1,392	94	0	6
	Treated with oil-paraffin ⁵	2,206	92	8	0	2,000	82	18	0	2,034	98	2	0	1,240	97	3	0	2,166	90	0	10	1,692	91	0	9
	With silicone stain ⁶	1,780	100	0	0	650	87	13	0	1,900	94	6	0	1,422	88	12	0	2,084	81	10	9	1,446	84	1	15
D2, benzene wash and sodium metasilicate	As received ⁴	1,340	91	9	0	(7)	100	0	0	2,744	57	43	0	41	99	1	0	2,920	6	86	8	333	83	15	2
	Treated with oil-paraffin ⁵	1,136	93	7	0	(7)	100	0	0	2,685	66	34	0	1,124	46	14	0	2,734	0	86	14	1,326	46	48	6
E1, benzene wash and sulfuric acid-sodium dichromate	As received ⁴	2,848	44	56	0	2,578	38	62	0	3,264	43	57	0	2,632	41	59	0	2,874	1	88	11	2,434	1	83	16
	Treated with oil-paraffin ⁵	3,124	33	67	0	2,968	32	67	0	3,163	55	45	0	3,012	53	48	0	2,912	15	78	7	2,506	2	86	12
	With silicone stain ⁶	3,404	97	3	0	3,018	81	19	0	2,804	100	0	0	1,818	95	5	0	2,700	53	38	10	2,368	36	59	11
	As received ⁴	968	82	18	0	(7)	100	0	0	1,727	99	1	0	(7)	100	0	0	2,044	65	27	8	(7)	100	0	0
F1, no treatment																									

¹The solution temperatures and concentrations and the processes involved in each of the cleaning methods are described in the text of the report.

²Each value for the control tests is the average of 5 specimens cut from one 6-inch-wide lap-joint panel, and each value for the exposure tests is the average of 10 specimens cut from two 6-inch-wide lap-joint panels prepared at the same time and under the same conditions as the control panel.

³Joint failures were examined and estimates made of the percentages of adhesion-to-metal failure (Ad.), cohesion failure in the adhesive (Co.), and no or low contact between adhesive coatings on joining surfaces (N.C.). Where more than 25 percent of the area of the bond showed a readily visible corrosion of the metal, the adhesion-to-metal failure, under which this type of failure is included, are starred (*).

⁴The metal sheets used in preparing these specimens were sheets as normally received with red identification lettering on one side. With the exception of the specimens prepared without treatment, both surfaces were wiped with a cloth soaked in benzene prior to further treatment. The specimens prepared without treatment were bonded directly to the surface having the lettering.

⁵The metal sheets used in preparing these specimens were treated with an oil-paraffin solution to simulate the condition of surfaces that are unusually contaminated. The surfaces were prepared by wiping the identification lettering off with a cloth soaked in benzene, dipping the sheets in a solution of 50 parts carbon tetrachloride, 10 parts petroleum paraffin, and 25 parts S.A.E. No. 10 oil (all by weight), and then allowing the film to air dry.

⁶The metal sheets used in preparing these specimens were immersed in warm sodium metasilicate solution and allowed to air dry without rinsing to simulate the surface condition obtained when sodium metasilicate solution is not properly rinsed from sheets being cleaned with the solution.

⁷Panel fell apart during handling. No strength tests were made.