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Standard Practice for Surface Preparation of Aluminum Alloys to Be Adhesively Bonded in Honeycomb Shelter Panels[1]

This standard is issued under the fixed designation E 864; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This practice covers the preparation of clean uniform surfaces of aluminum alloys suitable for formation of durable adhesive bonds to nonmetallic honeycomb materials in the manufacture of sandwich panels for tactical shelters.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* ~~For specific precautionary statements, see Note 1 of 5.2~~For a specific warning statement, see 5.2.1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 2674~~Test Method for~~ Methods of Analysis of Sulfochromate Etch Solution Used in Surface Preparation of Aluminum

D 3167 Test Method for Floating Roller Peel Resistance of Adhesives

E 865 Specification for Structural Film Adhesives for Honeycomb Sandwich Panels

E 866 Specification for Corrosion-Inhibiting Adhesive Primer for Aluminum Alloys to Be Adhesively Bonded in Honeycomb Shelter Panels

2.2 *APHA Standard:*

~~Sections 402, 403, and 408 of Standard Methods for the Examination of Water and Waste Water (15th Edition, 1980)~~ [3]

APHA Standard Methods for the Examination of Water and Waste Water (15th Edition, 1980), Sections 402, 403, and 408

3. Significance and Use

3.1 Durable adhesive bonds to aluminum alloys can be obtained reliably only through proper selection and careful control of the materials used and the steps in the bonding process. The preparation of the aluminum alloys to obtain clean, uniform surfaces with appropriate characteristics is a critical step. This practice describes how such surfaces can be obtained.

4. Apparatus

4.1 *General Processing*:

4.1.1 All heated tanks shall be equipped with automatic temperature controls and shall have means for agitation to prevent local overheating of the solution. Solutions may be heated by any internal or external means that do not change their compositions. Steam shall not be introduced into any solution. Compressed air introduced into any solution or equipment shall have been filtered to remove oil and moisture.

4.1.2 Tanks shall be made from, or lined with, materials that have no adverse effects on the solutions used or the parts being treated. All tanks shall be of sufficient size to allow complete immersion of the largest part or assembly to be treated.

4.2 *Rinse Tanks*—Immersion rinse tanks shall be equipped with a means for skimming or overflowing or both to remove surface contamination. The tanks shall be equipped with a means for flushing hollow sections.

4.3 *Rinses*—~~Rinses, other than final rinses, shall be maintained in such a manner to prevent carryover of materials that would adversely affect the next solution.~~ —Rinses, other than final rinses, shall be maintained in such a manner to prevent carryover of

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[1] This practice is under the jurisdiction of ASTM Committee E06 on Performance of Buildings and is the direct responsibility of Subcommittee E06.53 on Materials and Processes for Durable Rigidwall Relocatable Structures.

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[2] Available from the American Public Health Assn., 1015 15th St. N.W., Washington, DC 20005.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from the American Public Health Association (APHA), 800 I Street, NW, Washington, DC 20001-3710, http://www.apha.org.

materials that would adversely affect the next solution (for example, using a fog water rinse as the aluminum part/assembly is being withdrawn from the rinse tank).

5. Materials

5.1 *Water*—Water used for makeup of processing solutions and final rinsing shall be deionized water or shall meet the requirement of Table 1. Analyses shall be performed as often as necessary to assure that the water meets the requirements. Samples for analysis shall be collected at the processing tanks.

5.2 *Etch Solution*:

5.2.1 *Method I, Sulfo-Chromate Etch (FPL)*—The chemical analysis of the etch solution shall be maintained at approximately 30 parts by mass of water, ten parts by mass of sulfuric acid (sp gr 1.84), and one to four parts by mass of sodium dichromate. Prior to use, a minimum of 0.06 part by mass of dissolved 2024 aluminum shall be added.

~~NOTE 1—It~~ (**Warning—**It is recognized that chromates present a hazard to health. Use and disposal procedures are governed by Federal and Local EPA and DER limitations.

5.2.2 *Method II, Sulfo-Ferric Etch (P_2)*—The chemical analysis of the etch solution shall be maintained at approximately 27 to 36 % by weight of sulfuric acid (sp gr 1.84) and 2.9 to 4.7 oz/gal of ferric iron or 18 to 22 oz/gal of ferric sulfate. This is the equivalent of 2 gal of concentrated sulfuric acid and 12.5 lb of ferric sulfate in every 10 gal of solution. Two gallons of a 50 % ferric sulfate solution may be used in place of the 12.5 lb of the powdered ferric sulfate.

NOTE ~~2—Only~~ 1—Only virgin ferric sulfate solution shall be used in this process. Impurities in reclaimed ferric sulfate will cause unwanted reactions when the aluminum is treated.

5.3 *Alkaline Cleaning Solution*—Nonetch, alkaline cleaning solution shall be prepared in accordance with the manufacturer's recommendations, or as indicated in 7.1.2. When the aluminum being cleaned is immersed in the alkaline cleaner for the time and at the temperature used for processing, there shall be no evidence of gas evolution, etching, or metal removal. The alkaline solution shall not contain silicates.

5.4 *Quality Assurance Adhesive*—The adhesive system used for the quality assurance tests of 8.6 shall meet Specification E 865. The adhesive shall be changed only when a batch is almost exhausted so the results of any particular day's testing can be directly compared to the results of previous tests. When a change is made in the batch or lot of adhesive used, an additional set of tests shall be made to compare the old batch or lot with the new one to establish a basis for comparison between the results obtained with each.

6. Test Methods

6.1 *Chemical Analyses*—Perform chemical analyses of the water and solutions as indicated in 6.2 and 6.3. Analyze as often as necessary to maintain the required concentrations at a minimum of every day of operation. If a process is not in use during the normal analysis period, note this on the analysis record and analyze the solution prior to further use.

6.2 *Water Analyses*—Analyze as described in APHA Standard Methods for the Examination of Water and Waste Water.

6.3 *Etch Solution Analysis*:

6.3.1 *Sulfo-Chromate Etch*—Perform the analyses in accordance with ~~Test Method~~Methods D 2674.

6.3.2 *Sulfo-Ferric Etch*—Perform the analyses in accordance with 6.3.2.1 and 6.3.2.2.

6.3.2.1 *Sulfuric Acid*—Pipet a 1-mL (0.03-oz) sample into a 250-mL (8.5-oz) Erlenmeyer flask containing 100 mL (3.4 oz) of distilled water. Add 1 g (0.03 oz) of tribasic sodium citrate and 1 g (0.03 oz) of sodium fluoride. Add 2 to 3 drops of phenolphthalein indicator. Titrate with 0.4 N sodium hydroxide to clear or faint pink. Calculate the normality of sulfuric acid in accordance with Table 2.

6.3.2.2 *Ferric Ion*—Pipet a 2-mL (0.06-oz) sample into a 250-mL (8.5-oz) Erlenmeyer flask containing 100 mL (3.4 oz) of distilled water. Add 10 mL (0.3 oz) of concentrated hydrochloric acid. Add 1 to 2 g (0.03 to 0.06 oz) of potassium iodide titrate with 0.10 N sodium thiosulfate to a greenish color. Add about 2 mL of starch solution. Continue titration to a clear green end point. Calculate ferric sulfate as follows:

$$\text{mL sodium thiosulfate used} \times 2.79 = \text{g/L ferric ion} \tag{1}$$

6.4 *Visual Inspection*—Inspect parts visually for stains and water break.

TABLE 1 Requirements for Water to Be Used in Solutions and Rinses

	Requirements	
	Min	Max
pH	6.0	8.0
Total solids, ppm	...	200
Total alkalinity as $CaCO_3$, ppm	...	125
Chloride content, ppm	...	15