Designation: D 2651 – 01

Standard Guide for
Preparation of Metal Surfaces for Adhesive Bonding[1]

This standard is issued under the fixed designation D 2651; 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.

INTRODUCTION

It is impossible and impractical to present a single surface-preparation method for a metal group, such as aluminum alloys, stainless steels, magnesium alloys, etc. The wide variety of individual alloys and heat treatments under each group, the fact that certain adhesives exhibit specific compatibilities with surface preparation and the complexity and nature of parts being bonded preclude the use of an all-inclusive procedure for a metal group. Procedures for aluminum alloys are well standardized, possibly because more bonding has been done with these alloys. Preliminary tests should be conducted with the specific adhesive and the exact lot of metals to determine performance. This is especially true for stainless steel. The adhesive manufacturers' recommendation should also be considered.

1. Scope

1.1 This guide covers procedures that have proved satisfactory for preparing various wrought metal surfaces for adhesive bonding. It does not address the preparation of cast metals.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 Surface preparation methods are included for:

Metal	Section
Aluminum Alloys	7
Stainless Steel	8
Carbon Steel	9
Titanium Alloys	10
Magnesium Alloys	11
Copper and Copper Alloys	12

1.4 Procedures included herein are based on the commercial practice of numerous agencies and organizations. The methods may be revised or supplemented, as necessary, to include methods based on proven performance.

1.5 The surface preparation of metal systems used for qualification and quality control testing of the adhesive should be agreed upon by both manufacturer and user.

1.6 *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.*[2]

2. Referenced Documents

2.1 *ASTM Standards:*

D 907 Terminology Relating to Adhesives[3]

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water[4]

D 2674 Methods of Analysis of Sulfochromate Etch Solution Used in Surface Preparation of Aluminum[3]

D 3933 Guide for Preparation of Aluminum Surfaces for Structural Adhesives Bonding (Phosphoric Acid Anodizing)[3]

2.2 *ASM International Document:*[5]

Metals Handbook Volume V

2.3 *Military Specifications:*[6]

MIL-A-8625 Type II, Anodic Coatings for Aluminum and Aluminum Alloys

MIL-M-3171 Magnesium Alloy, Processes for Pretreatment and Prevention of Corrosion

MIL-M-45202 Magnesium Alloy, Anodic Treatment of

[1] This guide is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D14.80 on Metal Bonding Adhesives.

Current edition approved Oct. 10, 2001. Published December 2001. Originally published as D 2651 – 67. Last previous edition D 2651 – 90 (1995).

[2] A surfactant that has been found suitable for this purpose consists of a 50 % mixture of a cationic and a fluochemical surfactant such as Zonyl FSC manufactured by E.I. DuPont, Wilmington, DE 19898.

[3] *Annual Book of ASTM Standards*, Vol 15.06.

[4] *Annual Book of ASTM Standards*, Vol 11.01.

[5] Available from ASM International, Metals Park, OH 44073.

[6] Available from Standardization Documents, Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111, Attn: NPODS.

2.4 *SAE Standards:*[7]

Aerospace Material Specification 2470—Anodic Treatment of Aluminum Base Alloy, Chromic Acid Treatment

3. Terminology

3.1 *Definitions*—Many of the terms in this guide are defined in Terminology D 907.

4. Apparatus

4.1 *Trays or Pans*, suitable for preparation of laboratory specimens and small parts. Generally in production, a series of tanks containing the necessary solutions is arranged with overhead cranes to transport parts to be prepared. Tanks should be lined with suitable materials to resist the solutions, and methods for circulating the solutions and heating with temperature controls should be included if required.

4.2 *Hooks, Baskets, Clamps*, or other holding devices, may be used to convey parts being prepared. The use of a 300 Series stainless steel for such equipment is suggested. Other metals may be used if they are resistant to the solution used.

5. Conditions

5.1 *Handling*—All parts during and after processing and before bonding should be handled as little as possible and only with clean, lint-free cotton or nylon fabric gloves.

5.2 Water used for preparing solutions should be distilled, steam condensate, demineralized, deionized or otherwise treated, if necessary, to obtain a condition of not more than 50 ppm of solids and have a pH between 5.5 and 10, or not more than 50 ppm of dissolved solids and not over 30 micromhs conductance (see Test Methods D 1125). Rinsing may be done by spray or by dipping in a tank in which the water is circulated and constantly being renewed by an overflow method. After removing from any rinse the water-break test is commonly used. If the water film is discontinuous, it indicates that the process is unsatisfactory and must be repeated (see 5.5.4). Other methods, such as indicator paper and contact angle, have been used to determine the effectiveness of the process. Rinsing should be sufficiently long and vigorous to assure removal of soluble residue chemicals, or particles. Critical specific temperatures are specified for the rinse water in some processes. Where not specified, temperature is not critical. The number of times a process may be repeated depends upon the amount of metal removed in relation to the tolerance requirements for the part and whether clad or bare metal is used. Generally, not more than two repeat treatments are permitted.

NOTE 1—No entirely foolproof method exists to determine bondability of a metal surface after preparation. Trial bonding followed by testing and evaluation against mechanical strength standards should be conducted to determine initial and continuous effectiveness of a method.

5.3 *Solutions*—The life of solutions should be standardized. The useful life of solutions depends upon the number and size of the parts being prepared. Solutions should be sampled periodically and analyzed for materials pertinent to the particular treatment method, such as, titration for hexavalent chromium (CrO_3), iron, chlorides, aluminum, etc. A pH reading or acid content of trichloroethane vapor degreasing solution, or both, should be obtained. Standards should be established and records kept for such tests on each lot or vat of solution. Records should indicate the continuous conditions of solutions, such as amount of constituents added to bring solutions within limits, amount of contaminants present and date prepared (see Methods D 2674).

5.4 *Room Conditions* for surface preparation as well as other adhesive bonding operations should be controlled for temperatures of 18 to 24°C (65 to 75°F), relative humidity of 40 to 65 %, air-filtered to remove dust and pressure maintained at slightly higher than ambient.

5.4.1 The time and conditions between surface preparation and applying adhesive primer, if used, and bonding are critical. Usually, the time should not exceed 8 h and parts should be covered or wrapped in Kraft paper. Prepared surfaces can change their characteristics on standing and adhesives vary widely in their tolerance of adherend surface conditions. In production bonding of complicated or large parts, the time interval may exceed 8 h. Investigation should be made to determine the time limitation of the adhesive and the rate of change on the surface of the adherend. On the basis of the results of such an investigation, it may be possible to extend the length of time and establish standards for the interval between surface preparation and adhesive application to fit production schedules. Contaminating operations in the area should be avoided. Especially detrimental are paint or other spraying operations, processes using powdered materials, oil vapors from pumps and other machinery and spraying of mold release agents.

5.5 *Common Steps*:

5.5.1 Remove ink markings or stamped identifications from the metals. This can be accomplished by wiping with a cloth wetted with a variety of commercial solvents, such as acetone, methyl ethyl ketone, lacquer thinner and naphtha.

5.5.2 The preferred degreasing method is vapor degreasing with 1,1,1-trichloroethane. Commercial vapor degreasing equipment is usually used. This equipment has a deep tank with provision for heating a chlorinated solvent to 82 to 87°C (180 to 190°F). Parts are suspended in the vapor zone above the hot liquid and allowed to remain until there is condensation and run off (approximately 5 min). This step is repeated if necessary until all visible contamination is removed. The parts are raised above the tank and allowed to dry thoroughly. Previously bonded parts, such as honeycomb core materials, are usually not degreased due to difficulty in removing residue.

5.5.3 Following vapor degreasing, mechanical or chemical cleaning methods are usually employed.

5.5.4 A water-break test is a common method used to analyze surface cleanliness. This test depends on the observation that a clean surface (one that is chemically active or polar) will hold a continuous film of water, rather than a series of isolated droplets. This is known as a water-break-free condition. A break in the water film indicates a soiled or contaminated area. Distilled water should be used in the test, and a drainage time of about 30 s should be allowed. Any trace of residual cleaning solution should be removed or a false

[7] Available from the Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096.

conclusion may be made. If a water-break-free condition is not observed on the treated surface, it should not be used for bonding. The surface should be recleaned until the test is passed. If continuous failures occur, the treating process itself should be analyzed to determine the cause of the problem.

6. Report

6.1 The report for all methods consists of flow charts, record sheets or other methods that indicate all steps in the surface preparation used, with spaces for entries made upon completion of each step.

ALUMINUM ALLOYS

7. Procedure

7.1 Aluminum alloys may be pretreated with many different methods, using both acidic and basic cleaners.

7.2 A typical pretreatment would normally involve the following steps:

7.2.1 Vapor degrease or non-etching alkaline cleaner,

7.2.2 Rinse,

7.2.3 Acid or alkaline, and

7.2.4 Rinse.

7.3 After 7.2.4, the parts may be dried or they may be given an anodizing treatment.

7.4 Typical pretreatments are listed in 7.4.1. Refer to the appropriate literature for details on cleaning techniques and procedures.

7.4.1 Alkaline degreasing solutions may be used instead of, or in addition to, vapor degreasing. Commercially available proprietary solutions may be used. A common degreasing method is to immerse for 10 min at 70 to 82°C (160 to 180°F) in the following solution by weight: 3.0 parts sodium metasilicate, 1.5 parts sodium hydroxide, 0.5 parts sodium dodecylbenzene sulfonate, such as Nacconol 90G[8], to 133.0 parts water.

7.4.2 Acid etching solutions typically used are the following:

7.4.2.1 Sulfuric Acid/Sodium Dichromate (optimized FPL etch).

Material	Concentration
Sulfuric Acid (66°Be or Baume)	38 to 41.5 oz/gal (287.9 to 310.0 g/L)
Sodium Dichromate	4.1 to 9.0 oz/gal (28–67.3 g/L)
Aluminum Alloy–2024 bare	0.2 oz/gal (1.5 g/L), min
Temperature	66 to 71°C (150 to 160°F)
Immersion Time	12 to 15 min

7.4.2.2 P-2 Etch (Sulfuric Acid/Ferric Sulfate).

Material	Concentration
Sulfuric acid (6.5 to 9.5 N)	27 to 36 % by weight
Ferric sulfate	135 to 165 g/L (18 to 22 oz/gal)
Temperature	60 to 65°C (140 to 150°F)
Immersion Time	10 to 12 min

7.4.3 *Phosphoric Acid Anodizing* can be performed in accordance with Guide D 3933.

7.4.4 *Sulfuric Acid Anodizing*—A method found suitable for bare aluminum (nonclad), machined, or chemically milled parts which must be corrosion protected is to anodize with sulfuric acid. Do not seal the anodized parts with boiling water before bonding. Do not allow the time interval between treatment and bonding to exceed 72 h, and cover or wrap all parts during the interval with a non-contaminating vapor barrier material.

NOTE 2—Details of the process are contained in Specification AMS 2470E, or, Military Specification MIL-A8625.

7.4.5 *Proprietary Cleaners*—Some proprietary, commercially available metal surface preparations have been found suitable for preparing the surfaces for adhesive bonding. Full-scale property tests should be run with the metal surfaces so prepared with the adhesive in question before accepting the process.

7.4.6 *Secondary Bonding*—When secondary bonding (bonding of parts that contain previously bonded areas) or repair of a disbonded assembly in service is necessary, it is usually impossible and undesirable to immerse these parts in surface-preparation solutions without destroying or harming the original adhesive bond. Satisfactory surface preparations have been accomplished by using paste-type etchants. These paste systems can be made by adding fumed silica to the sulfuric-dichromic acid solutions. The application of these paste systems allows for their use in fairly controlled areas. The etching time is generally 10 to 20 min. Exercise extreme care to ensure removal of all traces of the etchant. A thorough rinsing with distilled water may be necessary to ensure complete removal. Several commercial products are available. Some commercial compounds under the classification of "wash primers" have been found to perform satisfactorily in place of the paste type systems. Bond strengths obtained using either of these surface preparations are somewhat lower than those from the immersion processes.

7.4.7 *Brush Plate Bond Etch (Stylus Method)*:

7.4.7.1 *General*—This method of electrochemical surface preparation, although not widely known, has been effectively demonstrated for small batch runs where tanks and other capital-intensive methods might have been used. Almost all of the solutions, "brushes" (or styli), and electrical power supplies are proprietary. When this method is used, the entire "family" of proprietary items should be used; mixed usage of items between vendors should not occur.

7.4.7.2 *General Precleaning*—Removal of grease, oil, and other organic soils can be accomplished with virtually any solvent, including trichlorotrifluoroethane and similar nonflammable types.

7.4.7.3 *Mechanical Preparation*—Surfaces which are scaled, corroded, or otherwise oxidized should be abraded using a nonmetallic abrasive. Aluminum oxide-impregnated nylon matting, glass-bead blasting and aluminum oxide cloth are typical acceptable abrasives. Abrasive and debris should be wiped or water rinsed from the surface.

7.4.7.4 *Electrochemical Cleaning*—This is accomplished by immersing the wrapped electrode in an alkaline electrocleaner-type solution and swabbing the surface while applying a cathodic current at approximately 5 to 15 V. (The surface is the cathode.) Thorough rinsing follows.

7.4.7.5 *Anodic Etching*—This step is recommended for most wrought alloys of aluminum. The surface is anodically

[8] Nacconol 90G, available from the Stephan Co., Northfield, IL 60093, has been found suitable for this purpose.