Designation: E866 - 23

# Standard Specification for Corrosion-Inhibiting Adhesive Primer for Aluminum Alloys to Be Adhesively Bonded in Honeycomb Shelter Panels<sup>1</sup>

This standard is issued under the fixed designation E866; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

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

#### 1. Scope

- 1.1 This specification covers sprayable, pigmented liquid primers for use on aluminum alloys that are to be adhesively bonded in the fabrication of honeycomb sandwich panels for tactical shelters. When applied to a properly cleaned surface of aluminum alloy, the primer imparts corrosion resistance and forms a surface suitable for structural bonding using adhesives complying with Specification E865 and for coating with shelter paint finishes.
- 1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.
- 1.3 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.
- 1.4 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

#### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

B117 Practice for Operating Salt Spray (Fog) Apparatus
B209 Specification for Aluminum and Aluminum-Alloy
Sheet and Plate (Metric) B0209\_B0209M

D522/D522M Test Methods for Mandrel Bend Test of Attached Organic Coatings

D1002 Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-to-Metal)

D3167 Test Method for Floating Roller Peel Resistance of Adhesives

E631 Terminology of Building Constructions

E864 Practice for Surface Preparation of Aluminum Alloys to Be Adhesively Bonded in Honeycomb Shelter Panels

E865 Specification for Structural Film Adhesives for Honeycomb Sandwich Panels

E1749 Terminology Relating to Rigid Wall Relocatable Shelters

2.2 Federal Specifications:<sup>3</sup>

QQ-A-250/8d Aluminum Alloy 5052H34, Plate and Sheet QQ-A-250/11d Aluminum Alloy 6061T6, Plate and Sheet

# 3. Terminology

3.1 *Definitions*—For definitions of general terms related to building construction used in this specification, refer to Terminology E631, and for general terms related to rigid wall relocatable shelters, refer to Terminology E1749.

# 4. Materials and Manufacture

4.1 The primer shall be a pigmented liquid composed of a modified epoxy resin system, compounded so that it can be spray-applied to produce a continuous uniform coating without addition of solvent.

### 5. Physical Requirements

- 5.1 The uncured primer must meet the requirements of Table 1. The cured primer must meet the requirements of Table 2 and Table 3. These requirements shall be verified by tests described in Section 6.
- 5.2 Properties of Cured Film—When applied and cured in accordance with 6.1.7, the cured film shall meet the requirements of Table 2 and Table 3 when tested as specified in 6.2 6.3.8.

<sup>&</sup>lt;sup>1</sup> This specification 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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<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Available from DLA Document Services, Building 4/D, 700 Robbins Ave., Philadelphia, PA 19111-5094, http://quicksearch.dla.mil.

**TABLE 1 Physical Properties of Uncured Liquid Primer** 

Test	Requirement	
Solids content, %	10 ± 1	
Inhibitor Content, <sup>A</sup> %	15 ± 3	
Color	easily visible film	
Sprayability	film is of uniform thickness and color	

A Based on mass of nonvolatile content.

**TABLE 2 Physical Properties of Cured Film on Primed Surfaces** 

Test	Requirement
Adhesion to metal	no cracking or loss of adhesion
Impact resistance	no cracking or loss of adhesion (on either side)
Pencil hardness	4H minimum
Water resistance	no blistering, cracking, softening, or loss of adhesion
Corrosion resistance	no blistering, cracking, or substrate degradation more than 3 mm from scribe mark
Humidity resistance	no blistering, cracking, or loss of adhesion
Heat resistance	no blistering, cracking, softening, or loss of adhesion
Low-temperature shock	no cracking or loss of adhesion

**TABLE 3 Physical Properties of Bonded Specimens** 

Test	Requirements (min)
Normal-temperature lap shear, 25 °C (77 °F), MPa (psi)	20.0 (2900)
Low-temperature lap shear, -55 °C (-67 °F), MPa (psi)	20.0 (2900)
High-temperature lap shear, 93 °C (200 °F), MPa (psi)	13.0 (1890)
Humidity-exposure lap shear, 93 °C (200 °F), MPa (psi)	5.0 (725)
Salt-spray exposure lap shear, 35 °C (95 °F), MPa (psi)	16.0 (2320)
Normal-temperature floating roller peel strength, 25 °C (77 °F), N/m (lbf/in.)	4400 (25.1)
Low-temperature floating roller peel strength, –55 °C (–67 °F), N/m (lbf/in.)	2625 (15.0)

- 5.3 Storage Stability—Primer stored in accordance with the manufacturer's instructions shall meet the requirements of Table 1 and when cured in accordance with 6.1.7 shall meet the requirements of Table 2 and Table 3 when tested as specified in 6.2 6.3.8.
- 5.4 Shelf Life—The shelf life of the primer at 25 °C  $\pm$  7 °C (77 °F  $\pm$  12 °F) shall be at least four days. After four days, when tested in accordance with 6.1.4, the primer shall be capable of meeting all of the requirements of this specification.
- 5.5 Film Thickness—The film thickness for all tests shall be between 0.003 mm and 0.007 mm (0.0001 in. and 0.0003 in.).

### 6. Test Methods

- 6.1 Uncured Primer:
- 6.1.1 Solids Content—Determine the solids content by heating a 5 g sample of thoroughly mixed primer in an aluminum weighing dish in a circulating air oven at  $105 \,^{\circ}\text{C} \pm 2 \,^{\circ}\text{C}$  (221 °F  $\pm$  3 °F) for not less than 3 h. Calculate the mass of solids remaining as a percentage of the initial sample mass. Weighings before and after heating shall be accurate to  $\pm 0.001 \, \text{g}$ .
- 6.1.2 *Inhibitor Content*—Determine the inhibitor content in accordance with the following procedure (burn out method):
  - 6.1.2.1 Tare four ignition loss crucibles and covers.

- 6.1.2.2 Pipette 2 mL portions of primer (1.75 g) from a well mixed sample into each crucible, add about 2 mL of MEK to each, replace the lids, and devolatize the primer at 121 °C  $\pm$  3 °C (250 °F  $\pm$  5 °F) for 60 min  $\pm$  5 min.
- 6.1.2.3 Cool to room temperature in a desiccator, weigh, and compute the mass of the nonvolatiles. The nonvolatile content requirement is 0.155 g to 0.195 g/2 mL (10 %  $\pm$  1 % by weight).
- 6.1.2.4 Place the crucibles with lids (and residue) in a furnace at 593 °C (1100 °F) for 60 min  $\pm$  5 min.
- 6.1.2.5 Cool to room temperature in a desiccator, weigh, and compute average mass of the inhibitor. The inhibitor content requirement is 0.025 g  $\pm$  0.003 g/2 mL (15 %  $\pm$  3 % by mass of nonvolatile content).
- 6.1.3 Storage Stability—Store a sample of the primer for six months from the date of shipment at -18 °C (0 °F), or two months at 5 °C (41 °F). Test the sample for ability to meet the requirements of Tables 1-3.
- 6.1.4 Shelf Life—Store a sample of the primer for four days at 25 °C  $\pm$  7 °C (77 °F  $\pm$  12 °F) and then test for ability to meet the requirements listed in Tables 1-3.
- 6.1.5 *Color*—Spray the primer onto an aluminum alloy panel, leaving a portion of the panel bare. Compare the coated and uncoated areas visually against the color requirements in Table 1.
- 6.1.6 Sprayability—The primer shall be capable of being readily applied in accordance with the manufacturer's instructions to all test panels of either 6061T6 or 5052H34 aluminum alloy (Fed. Spec. QQ-A-250/8d or QQ-A-250/11d, respectively, or Specification B209) and cleaned in accordance with Practice E864 for each of the tests described in 6.1.7 6.3.8. Inspect the coating for uniformity of thickness and color.
- 6.1.7 Curing Properties—Spray the primer onto a test panel 100 mm by 150 mm by 0.05 mm (4 in. by 6 in. by 0.020 in.) and air dry for at least 30 min at 25 °C  $\pm$  5 °C (77 °F  $\pm$  9 °F); then heat in air for 75 min to 90 min at 115 °C  $\pm$  5 °C (239 °F  $\pm$  9 °F). Test the cured film for ability to meet the requirements listed in Table 2.

## 6.2 Cured Film:

- 6.2.1 Adhesion to Metal—Test the adhesion of the primer to the metal by bending a primed metal sample over a conical mandrel in accordance with Test Methods D522/D522M, except use an aluminum alloy panel.
- 6.2.2 Impact Resistance—Determine the impact resistance of both the coated and uncoated side of a panel by subjecting the panel to the impact of a 0.45 kg (1 lb) mass dropped from a height of 1 m (40 in.) using a Gardner 160 in.·lbf capacity impact testing machine or equivalent. Use the 0.45 kg (1 lb) falling weight. Apply a strip of masking tape<sup>4</sup> over the most highly stressed area on the coated side of each impact spot. Remove the tape in one abrupt motion by pulling perpendicular to the panel.

<sup>&</sup>lt;sup>4</sup> The sole source of supply of No. 250 masking tape, which has been found satisfactory, known to the committee at this time is 3M Co., St. Paul, MN. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, <sup>1</sup> which you may attend.

- 6.2.3 *Pencil Hardness*—Determine the pencil hardness of the cured film using the following procedure:
- 6.2.3.1 Preparation of Pencils—Prepare a set of drawing pencils<sup>5</sup> ranging in hardness from 6B to 5H by stripping the wood away from the end approximately 10 mm (3/8 in.) without damaging the lead. Square the tip of the lead by holding the pencil in a vertical position and moving the lead back and forth over 400 grit or finer abrasive paper. Square the tip of the lead after each trial.
- 6.2.3.2 *Procedure*—Place the test panel with the cured primer applied in a horizontal position. Push pencils of increasing hardness into the coated surface of the panel at a  $45^{\circ}$  angle until one is found that will cut or scratch the coating. Use the number of this pencil to express the primer hardness.
- 6.2.4 Environmental Resistance Test—For each test described in 6.2.5 6.2.9, clean five test panels of approximately 75 mm by 125 mm (3 in. by 5 in.) of either 6061T6 or 5052H34 aluminum alloy (Fed. Spec. QQ-A-250/8d or QQ-A-250/11d, respectively, or Specification B209) in accordance with Practice E864. Apply the primer and cure in accordance with 6.1.7. These panels when tested in accordance with the procedures described in 6.2.5 6.2.9 shall meet the requirements of Table 2.
- 6.2.5 Water Resistance—Immerse the panels with the cured primer applied in distilled water for seven days at 24 °C  $\pm$  3 °C (75 °F  $\pm$  5 °F) and then expose to 100 % relative humidity at 50 °C  $\pm$  3 °C (121 °F  $\pm$  5 °F) for 30 days. After this exposure, inspect the film for blistering and cracking and test for loss of adhesion as described in 6.2.10 and for pencil hardness as in 6.2.3.
- 6.2.6 *Heat Resistance*—Heat the five test panels prepared as described in 6.2.4 at 120 °C  $\pm$  3 °C (248 °F  $\pm$  5 °F) for 70 h. After this exposure, inspect the film for blistering and cracking and test for loss of adhesion as described in 6.2.10 and for pencil hardness as in 6.2.3.
- 6.2.7 Low-Temperature Shock—Subject the five test panels prepared as in 6.2.4 to 24 cycles, each consisting of 25 min at 65 °C  $\pm$  3 °C (150 °F  $\pm$  5 °F) then, following transfer within 5 s, 5 min at –55 °C  $\pm$  3 °C (–66 °F  $\pm$  5 °F). On completion of the last cycle the panel shall remain in a cold box maintained at –55 °C  $\pm$  3 °C (–66 °F  $\pm$  5 °F) for 5 h. Bend the panel rapidly over a 100 mm (4 in.) diameter mandrel that has been conditioned at the same condition for a minimum of 45 min. Panel evaluation after exposure shall include inspection for cracking and the adhesion test in accordance with 6.2.10. When a normal work day will not allow completion of this test, the cycle shall be interrupted at the end of the low temperature exposure (store overnight at room temperature) and start again at the hot cycle.
- 6.2.8 Corrosion Resistance—Scribe the five test panels prepared in accordance with 6.2.4 through the coating to the base metal with two diagonal scribe marks extending from

corner to corner and exposed to 5 % salt spray in accordance with Practice B117 for 40 days.

6.2.9 Humidity Aging—Expose the five test panels prepared in accordance with 6.2.4 to a relative humidity of 100 % at  $50 \,^{\circ}\text{C} \pm 3 \,^{\circ}\text{C}$  (121  $^{\circ}\text{F} \pm 5 \,^{\circ}\text{F}$ ) for 30 days. After this exposure, inspect the film for blistering and cracking and test for loss of adhesion as described in 6.2.10.

6.2.10 Loss of Adhesion:

- 6.2.10.1 Thoroughly dry the panel by wiping with a clean dry cheese cloth. Conduct the test at 20 °C to 27 °C (68 °F to 80 °F).
- 6.2.10.2 Scribe two parallel scratches 25 mm (1 in.) apart, through the coating down to the metal, with a sharp-edge scribe.
- 6.2.10.3 Apply a strip of 25 mm (1 in.) wide masking tape (not more than five months old from date of manufacture) across the scratches at  $90^{\circ}$  in the panel area to be tested. Press the tape down using two passes of a 2 kg (4.5 lb) rubber-covered roller approximately 90 mm (3.5 in.) in diameter by 45 mm (1.75 in.) in width. The Durometer of the roller surface shall be 70 to 80 Shore A.
- 6.2.10.4 As an alternative to 6.2.10.3, the tape shall be pressed down by rolling the tape roll along the strip of applied tape using a firm hand pressure of approximately 2 kg (4 lb to 5 lb).
- 6.2.10.5 Remove the tape in one abrupt motion perpendicular to the panel. No primer shall be removed from the panel, other than that which was previously removed during the scribing procedure (see 6.2.10.2).
  - 6.3 Bonding Property Tests:
- 6.3.1 Preparation of Test Specimens—Prepare and test five test specimens, in accordance with Test Method D1002 and Practice E864, for each of the required lap-shear tests in Table 3. In addition, prepare five test specimens for each floating roller peel test in Table 3 in accordance with Test Method D3167 and Practice E864. The adherends shall be of either 6061T6 or 5052H34 aluminum alloy (Fed. Spec. QQ-A-250/8d or QQ-A-250/11d, respectively, or Specification B209). Cure the primer by air drying for not less than 30 min at 25 °C  $\pm$  5 °C (78 °F  $\pm$  9 °F); follow by heating at 115 °C  $\pm$  5 °C (239 °F  $\pm$  9 °F) for 75 min to 90 min. Bond the primed panel with an adhesive meeting Specification E865.
- 6.3.1.1 *Tests*—The primer shall be subjected to all the tests described in this section. Lap-shear tests (see 6.3.2 6.3.5) shall be performed in accordance with Test Method D1002.
- 6.3.2 Normal-Temperature Lap Shear—Subject each specimen to a lap shear test at 25 °C  $\pm$  3 °C (77 °F  $\pm$  5 °F).
- 6.3.3 Low-Temperature Lap Shear—Test each specimen for low-temperature lap shear at -55 °C  $\pm$  3 °C (-66 °F  $\pm$  5 °F). Bring the temperature of the specimen to -55 °C  $\pm$  3 °C (-66 °F  $\pm$  5 °F) as indicated by a thermocouple at the bond area and stabilize for 10 min just prior to test.
- 6.3.4 *High-Temperature Lap Shear*—Test each specimen for high-temperature lap shear at 93 °C  $\pm$  3 °C (200 °F  $\pm$  5 °F). Bring the temperature of the specimen to 93 °C  $\pm$  3 °C (200 °F  $\pm$  5 °F) as indicated by a thermocouple at the bond area and stabilize for 10 min just prior to test.

<sup>&</sup>lt;sup>5</sup> The sole source of supply of KOH-I-NOOR 1500 and Venus Drawing Pencil, which have been found satisfactory, known to the committee at this time is Faber-Castell USA, Inc., Cleveland, OH. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, <sup>1</sup> which you may attend.