



Standard Practice for Evaluating Intergranular Corrosion Resistance of Heat Treatable Aluminum Alloys by Immersion in Sodium Chloride + Hydrogen Peroxide Solution¹

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^{ε1} NOTE—Warning notes were editorially moved into main body text in October 2003.

1. Scope

1.1 This practice covers the procedures for immersion tests in sodium chloride + hydrogen peroxide solution. It is primarily for tests of wrought heat treatable aluminum alloys (2XXX and 7XXX) but may be used for other aluminum alloys, including castings. It sets forth the specimen preparation procedures and the environmental conditions of the test and the means for controlling them.

1.2 This practice is intended for evaluations during alloy development and for evaluating production where it may serve as a control test on the quality of successive lots of the same material (see MIL-H-6088 and U.S. Federal Test Method Std. 151b). Therefore strict test conditions are stipulated for maximum assurance that variations in results are attributable to lot-to-lot differences in the material being tested.

NOTE 1—This practice does not address sampling or interpretation or significance of results.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 3 Methods of Preparation of Metallographic Specimens³

E 407 Test Methods for Microetching Metals and Alloys³

G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens⁴

G 15 Terminology Relating to Corrosion and Corrosion Testing⁴

G 67 Test Method for Determining the Susceptibility to Intergranular Corrosion of 5XXX Series Aluminum Alloys by Mass Loss After Exposure to Nitric Acid (NAMLT Test)⁴

G 69 Practice for Measurement of Corrosion Potentials of Aluminum Alloys⁴

2.2 Other Documents:

U.S. Military Specification MIL-H-6088, Heat Treatment of Aluminum Alloys⁵

U.S. Federal Test Method, Standard No. 151b, Method 822.1, Intergranular Corrosion Test for Aluminum Alloys⁵

3. Summary of Practice

3.1 This practice consists of immersing etched test specimens in a sodium chloride + hydrogen peroxide solution for 6 or more hours. After immersion, metallographic sections are examined to determine the extent of intergranular corrosion (see Terminology G 15).

4. Significance and Use

4.1 This practice is especially useful for evaluating the adequacy of quenching when performed on material in the as-quenched condition. The practice may also be used to study the effect of subsequent thermal processes (for example, paint or bonding cures) or of actual precipitation treatments on the inherent type of corrosion. Intergranular corrosion resistance of heat treatable aluminum alloys is often directly related to the quenching conditions applied after solution heat treatment and to the subsequent aging treatment.⁶

¹ This practice is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 03.01.

⁴ *Annual Book of ASTM Standards*, Vol 03.02.

⁵ Available from Standardization Documents Order Desk, Building 4, Section D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.

⁶ Lifka, B. W., and Sprowls, D. O., "Significance of Intergranular Corrosion of High Strength Aluminum Alloy Products," *Localized Corrosion Cause of Metal Failure*, ASTM STP 516 (1972), pp. 120–144.

4.2 This practice is not well suited for non-heat treatable work hardening aluminum alloys, such as the 1XXX, 3XXX, and 5XXX series (see Test Method G 67).

4.3 This practice does not deal with the interpretation of resulting intergranular corrosion. The significance of the extent and depth of any intergranular corrosion resulting from this test is to be agreed upon between producer and user.

5. Reagents

5.1 Reagent grade chemicals [sodium chloride (NaCl), 70 % nitric acid (HNO₃), 48 % hydrofluoric acid (HF), 37 % hydrochloric acid (HCl), and 30 % hydrogen peroxide (H₂O₂)] shall be used for preparation of all solutions.

5.2 The solutions shall be prepared using distilled or deionized water conforming to the purity requirements for Specification D 1193, Type IV reagent water.

6. Solutions

6.1 Etching cleaner shall be prepared as follows: To 945 mL of reagent water add 50 mL of nitric acid (70 %) + 5 mL of hydrofluoric acid (48 %). (See 6.2).

6.2 Test solution shall be prepared as follows: 57 grams of sodium chloride + 10 mL of hydrogen peroxide (30 %—add just prior to initiation of exposure) diluted to 1.0 L with reagent water. (**Warning**—Care should be exercised in handling and mixing strong acids to avoid personal injury and damage to apparatus. Care should include use of personal protective equipment and use of appropriate apparatus and procedures for particular acids.)

NOTE 2—The concentration of the 30 % hydrogen peroxide may degrade; therefore, it should be verified before each use by the procedure stated in Practice G 69.

6.3 Metallographic etchant (Keller's etch) shall be prepared as follows: To 95 mL of reagent water add 2.5 mL of nitric acid (70 %) + 1.5 mL of hydrochloric acid (37 %) + 1.0 mL of hydrofluoric acid (48 %). A number of other etchants may be used, depending on the alloy and temper being examined (see Method E 407) (see 6.2).

7. Specimen Preparation

7.1 Place an identification mark on each specimen in an area that will not be subject to metallographic examination.

7.2 The entire surface of the specimen must be free of all foreign matter, including cladding, coatings, dirt, oils, and other residues.

7.3 Claddings (especially alcladding) shall be removed either mechanically or chemically to a depth of at least twice the nominal cladding thickness. Chemical removal can be accomplished by immersion in a 5 % sodium hydroxide solution at 140°F followed by immersion for 1 min in concentrated nitric acid. Examine typical specimen cross sections to be certain that cladding has been removed.

7.4 Organic materials, such as coatings, soil, oils, and other residues, should be removed with organic solvents, for example, acetone (see Practice G 1).

7.5 Prior to immersion in the test solution, immerse each specimen for 1 min in the etching cleaner (see 6.1) at 93°C. Rinse in reagent water. Immerse in concentrated nitric acid (70 %) for 1 min. Rinse in reagent water and air dry.

8. Test Setup

8.1 The test vessel should be of nonmetallic material of sufficient size to hold at least 5 mL of test solution per square cm of specimen surface area.

8.2 More than one specimen may be placed in a test vessel, provided specimens do not contact each other and provided condition outlined in 8.1 is observed.

8.3 Specimens shall be prevented from contacting the bottom of the test vessel by placing the specimens on glass rods or rubber stoppers. (Rubber stoppers may be deteriorated by hydrogen peroxide.)

8.4 The exposure shall be conducted at a temperature of 30 ± 3°C.

9. Procedure

9.1 Immerse the etched specimens in the test solution for a period of at least 6 h. Longer exposure periods of 24 hours or more may be used by agreement, especially in the case of corrosion resistant alloys and tempers (for example, 6XXX alloys). Shorter exposure periods of less than 6 h may be appropriate for very thin sheet.

9.2 After exposure, rinse each specimen with reagent water and allow to dry.

9.3 Examination of specimens.

9.3.1 Examine each exposed specimen at a magnification of 10 times or less to locate areas of corrosion attack.

9.3.2 Prepare at least one metallographic cross section approximately 20 mm in length from each specimen, preferably through a corroded area identified in 9.3.1. Prepare each section by mounting and metallographically polishing (see Method E 3).

9.3.3 Examine the unetched polished surface with a metallograph at magnifications of 100 to 500 times. If there is uncertainty as to the presence of intergranular corrosion, etch the polished surface to reveal the grain structure by immersion in the metallographic etchant solution for 6 to 20 s, rinse, dry, and reexamine at 500 times.

9.4 Compare or document the type, extent, and depth of intergranular corrosion in accordance with criteria established between producer and purchaser.

10. Documentation

10.1 The following essential information should be recorded for each specimen:

10.1.1 Identification of the heat or lot of the material,

10.1.2 Composition or standard alloy identification and temper,

10.1.3 Product form: sheet, plate, extrusion, forging, or casting,

10.1.4 Sampling location,

10.1.5 Duration of exposure,

10.1.6 Notation of any deviation in test procedure from that set forth in preceding paragraphs,

10.1.7 Disposition of specimen, and

10.1.8 Results of microscopic examination.

10.2 Other information that may be desirable:

10.2.1 Number of sections examined.

10.2.2 Was metallographic etching required?

10.2.3 Evaluation criteria utilized.

intergranular corrosion; intercrystalline corrosion

11. Keywords

11.1 2XXX aluminum alloys; 6XXX aluminum alloys;
7XXX aluminum alloys; heat treatable aluminum alloys;

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