



Designation: G66 – 99 (Reapproved 2018)

Standard Test Method for Visual Assessment of Exfoliation Corrosion Susceptibility of 5XXX Series Aluminum Alloys (ASSET Test)¹

This standard is issued under the fixed designation G66; 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.

1. Scope

1.1 This test method covers a procedure for continuous immersion exfoliation corrosion testing of 5XXX series aluminum-magnesium alloys containing 2.0 % or more magnesium.

1.2 This test method applies only to wrought products.

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

1.4 *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.*

1.5 *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:*²

D1193 Specification for Reagent Water

G15 Terminology Relating to Corrosion and Corrosion Testing (Withdrawn 2010)³

2.2 *ASTM Adjuncts:*

ASSET Tested Specimens (Glossy Prints)⁴

¹ This test method 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. This method was developed by a joint task group with The Aluminum Assoc., Inc.

Current edition approved Oct. 1, 2018. Published November 2018. Originally approved in 1980. Last previous edition approved in 2013 as G66 – 99 (2013). DOI: 10.1520/G0066-99R18.

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 The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from ASTM International Headquarters. Order Adjunct No. ADJG0066. Original adjunct produced in 1987.

3. Terminology

3.1 *Definitions:*

3.1.1 *exfoliation*—corrosion that proceeds laterally from the sites of initiation along planes parallel to the surface, generally at grain boundaries, forming corrosion products that force metal away from the body of the material, giving rise to a layered appearance (see Terminology G15).

4. Summary of Test Method

4.1 Specimens are immersed for 24 h at $65 \pm 1^\circ\text{C}$ ($150 \pm 2^\circ\text{F}$) in a solution containing ammonium chloride, ammonium nitrate, ammonium tartrate, and hydrogen peroxide. The susceptibility to exfoliation is determined by visual examination using performance ratings established by reference to standard photographs.

5. Significance and Use

5.1 This test method provides a reliable prediction of the exfoliation corrosion behavior of Al-Mg alloys in marine environments.^{5,6,7} The test is useful for alloy development studies and quality control of mill products such as sheet and plate.

6. Apparatus

6.1 Any suitable glass or plastic container can be used to contain the solution and specimens during the test period. Depending upon the shape and size of the specimens, rods or racks of glass, plastic, or other inert substance shall be used to support the specimens above the bottom of the container. The container should be fitted with a removable cover to reduce evaporation.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests.

⁵ Aluminum Association Technical Report T1, "Exfoliation Corrosion Testing of Aluminum Alloys 5086 and 5456".

⁶ Sprowls, D. O., Walsh, J. D. and Shumaker, M. B., "Simplified Exfoliation Testing of Aluminum Alloys", *Localized Corrosion—Cause of Metal Failure, ASTM STP 516*, ASTM, 1972, pp 38–65.

⁷ Summerson T. J., Interim Report, Aluminum Association Task Group on Exfoliation and Stress Corrosion Cracking of Aluminum Alloys for Boat Stock; Proceedings Tri-Service Corrosion Military Equipment Conference, October 29–31, 1974; Technical Report AFML-TR-75-42, Vol. II, p. 193–221, February 1, 1975.

7.2 *Purity of Water*—Distilled or deionized water conforming to Specification D1193. Type IV shall be used to prepare the test solution except chloride ion sodium limits can be disregarded.

8. Test Solution

8.1 Preparation of Test Solution:

8.1.1 The test solution shall have the following composition:

NH ₄ Cl	(1.0 M)
NH ₄ NO ₃	(0.25 M)
(NH ₄) ₂ C ₄ H ₄ O ₆	(0.01 M)
H ₂ O ₂	(0.09 M)

8.1.2 Dissolve 53.5 g ammonium chloride (NH₄Cl), 20.0 g ammonium nitrate (NH₄NO₃), 1.8 g ammonium tartrate ((NH₄)₂C₄H₄O₆), and 10 mL of 30 % stock solution hydrogen peroxide (H₂O₂) in a small amount of water. After dissolving, mix the components together thoroughly and adjust the final dilution to 1 L.

NOTE 1—If a stock solution of the above chemicals is to be stored, the hydrogen peroxide should not be added until the solution is heated for the test.

8.2 The solution will have a typical pH of 5.2 to 5.4.

8.3 The solution shall be used in sufficient quantity to provide a volume-to-exposed specimen surface area ratio of at least 100 L/m² (65 mL/in.²).

8.4 The temperature of the solution shall be maintained at 65 ± 1°C (150 ± 2°F).

9. Sampling

9.1 The procedure for sampling mill products is covered in product specifications, or otherwise, and is considered outside the scope of this standard.

10. Test Specimen

10.1 While this test method can be used with any form of specimen or part that can be immersed in the test solution, it is preferred that specimens be at least 40 by 100 mm (1.5 by 4.0 in.) with the metal working direction in the 40-mm (1.5-in.) dimension.

10.2 The specimens should be sawed or machined to minimize introducing residual stresses in edges during preparation of the specimens (sheared edges are allowed only if the edges are sufficiently dressed or filed down a distance equal to the thickness of the specimen in order to remove metal deformed by shearing).

11. Standardization

11.1 To provide an indication when some inadvertent deviation from the correct test conditions occurs, it is necessary to expose to the test at regular intervals a control specimen of a material of known susceptibility. This control should exhibit the same degree of exfoliation each time it is included in the test.

11.2 The control may be any material of the Al-Mg series that has a well-documented susceptibility to exfoliation corrosion, preferably one with an intermediate susceptibility.

12. Procedure

12.1 Degrease the specimens with a suitable solvent. After degreasing, prepare specimens as follows: Etch 1 min in 5 % by weight sodium hydroxide solution at 80°C (176°F), rinse in water, desmut 30 s in concentrated nitric acid at room temperature, rinse with distilled or deionized water, air dry.

NOTE 2—If specimens are not to be immersed in the test solution immediately, they should be stored in a desiccator maintained at less than 1 % relative humidity (use fresh desiccant such as activated alumina or anhydrous calcium sulfate).⁸

12.2 Use fresh solution at the start of each test.

12.3 Immerse the specimens vertically with the top edge of the specimens at least 25 mm (1 in.) below the surface of the solution and the bottom edge at least 25 mm above the bottom of the container.

12.4 Immerse the specimens in the test solution continuously for 24 h.

12.5 Rinse the specimens gently in running tap water immediately after removal from the solution, then soak in concentrated nitric acid at room temperature until they appear clean, again rinse in water, and air dry.

12.5.1 Air-blast drying is to be avoided in order to prevent mechanical removal of exfoliated metal.

13. Rating of Specimens

13.1 The following codes and classifications shall be used for reporting the visual appearance of corroded specimens.

Code	Classification
N	No appreciable attack
P	Pitting
E	Exfoliation

13.2 Descriptions of the various classifications, which are illustrated in Figs. 1 and 2,⁴ are as follows:

13.2.1 *N* (no appreciable attack)—Surface may be etched or discolored.

13.2.2 *P* (Pitting)—Includes discrete pitting or pit-blistering. In the latter case, attack results in a slight undercutting of the surface. Pitting or pit-blistering is to be expected and may occur in varying degrees of severity, as shown in Fig. 1; it should not be construed as exfoliation.

13.2.3 *E* (Exfoliation)—Visible lifting of the surface. A range of exfoliation can occur in varying degrees of severity, as shown in Fig. 2.

13.3 Ratings of different levels of pitting or exfoliation may be indicated *PA*, *PB*, *PC* or *EA*, *EB*, *EC*, *ED* as shown in Figs. 1 and 2.

14. Report

14.1 Report the following information:

14.1.1 Alloy and temper,

⁸ Drierite, (a special form of anhydrous calcium sulfate), available from W. A. Hammond Drierite Co., 120 Dayton Ave., Xenia, OH 45385, or an equivalent drying agent may be used. 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 committee¹, which you may attend.