



# Standard Test Method for Visual Assessment of Exfoliation Corrosion Susceptibility of 5XXX Series Aluminum Alloys (ASSET Test)<sup>1</sup>

This standard is issued under the fixed designation G 66; 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.

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 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 Water<sup>2</sup>

G 15 Terminology Relating to Corrosion and Corrosion Testing<sup>3</sup>

## 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 G 15).

## 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.<sup>4</sup> 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.

7.2 *Purity of Water*—Distilled or deionized water conforming to Specification D 1193. 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 <sub>4</sub> Cl	(1.0 M)
NH <sub>4</sub> NO <sub>3</sub>	(0.25 M)
(NH <sub>4</sub> ) <sub>2</sub> C <sub>4</sub> H <sub>4</sub> O <sub>6</sub>	(0.01 M)
H <sub>2</sub> O <sub>2</sub>	(0.09 M)

8.1.2 Dissolve 53.5 g ammonium chloride (NH<sub>4</sub>Cl), 20.0 g ammonium nitrate (NH<sub>4</sub>NO<sub>3</sub>), 1.8 g ammonium tartrate ((NH<sub>4</sub>)<sub>2</sub>C<sub>4</sub>H<sub>4</sub>O<sub>6</sub>), and 10 mL of 30 % stock solution hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) in a small amount of water. After dissolving,

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.02.

<sup>4</sup> Aluminum Association Technical Report T1, “Exfoliation Corrosion Testing of Aluminum Alloys 5086 and 5456”.

Spowls, 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.

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.

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/m<sup>2</sup> (65 mL/in.<sup>2</sup>).

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).<sup>5</sup>

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
<i>N</i>	No appreciable attack
<i>P</i>	Pitting
<i>E</i>	Exfoliation

13.2 Descriptions of the various classifications, which are illustrated in Figs. 1 and 2, are as follows:<sup>6</sup>

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,

14.1.2 Product of material tested, including reference to applicable product specification,

14.1.3 Sampling procedure if other than that specified in referenced product specification,

14.1.4 A rating of the test specimens using the code given in Section 13,

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

14.1.6 Size, type, and number of replicate specimens; method of edge preparation, and

14.1.7 Solution volume to specimen surface area ratio.

## 15. Precision and Bias

15.1 *Precision*:

15.1.1 The precision of the data from this test method was evaluated by way of an interlaboratory test program using one commercial and one non-commercial tempers of Alloy 5086 with different levels of exfoliation corrosion susceptibility. Six

<sup>5</sup> 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.

<sup>6</sup> Glossy prints of Fig. 1 and Fig. 2 are available from ASTM Headquarters. Order PCN ADJG0066

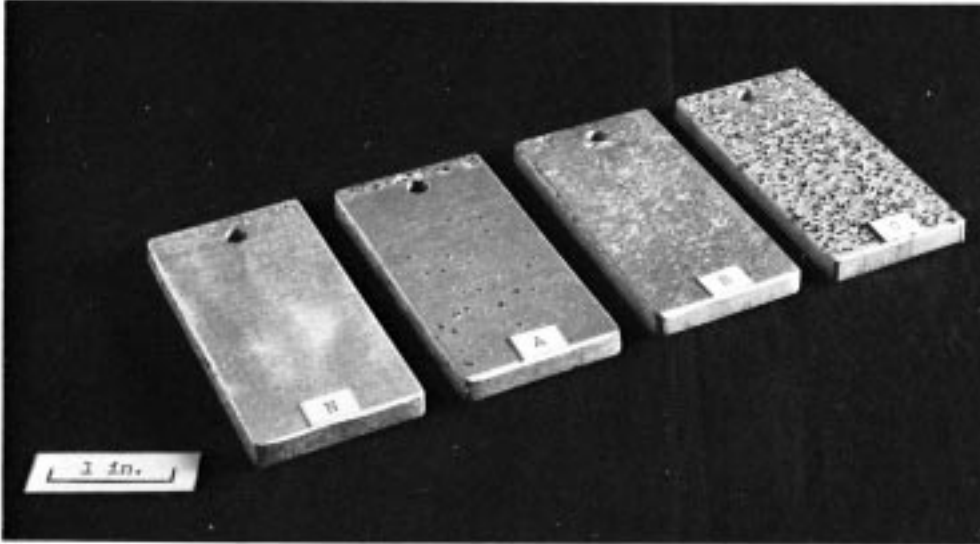


FIG. 1 ASSET Tested Exfoliation Resistant Specimens (N—No appreciable attack; A, B, C—Three Degrees of Pitting and Pit-Blistering)

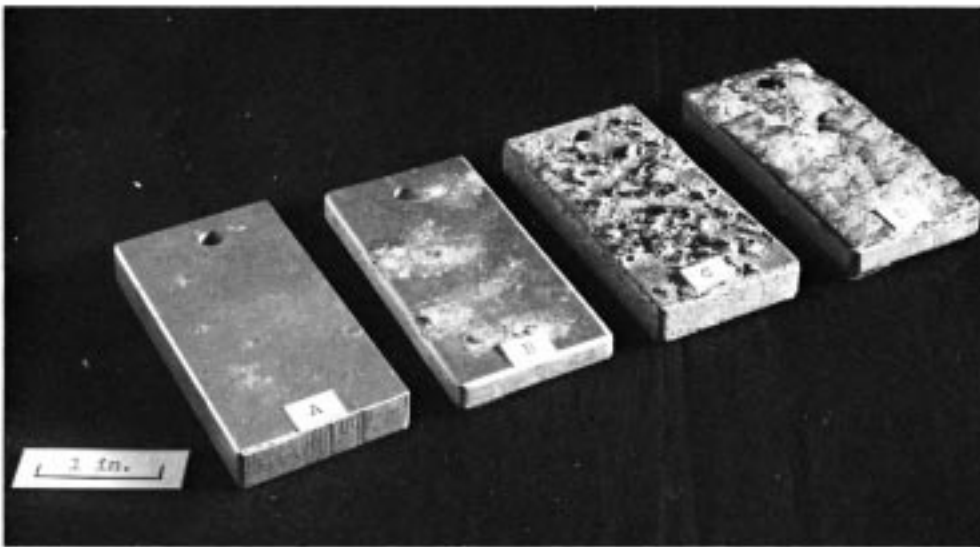


FIG. 2 ASSET Tested Exfoliation Susceptible Specimens (A, B, C, D—Four Degrees of Exfoliation)

laboratories, including experienced and inexperienced users, participated in the round robin. The laboratories received duplicate rough cut panels of each material which they prepared, exposed, and rated visually according to this test method.

15.1.2 Raw data from the laboratories are listed in Table 1 and plotted as a histogram in Fig. 3. All six laboratories easily differentiated between the two materials finding one resistant and one susceptible to exfoliation corrosion.

15.1.2.1 The data in Table 1 show the ratings were repeatable within laboratories. In each case the same rating was obtained for both panels tested by the individual laboratory.

15.1.2.2 The reproducibility of ratings among different

laboratories was consistent for the resistant material with all laboratories obtaining pitting ratings. In the case of the exfoliation susceptible material the ratings between laboratories were within one rating category.

15.2 Bias:

15.2.1 The procedure in Test Method G 66 has no bias because the exfoliation rating is defined only in terms of this test method.

16. Keywords

16.1 aluminum alloys; 5XXX aluminum alloys; exfoliation corrosion; pitting corrosion; visual inspection

TABLE 1 ASSET (Test Method G 66) Round Robin Results

Laboratory No.	Material Exfoliation Type	Volume/Area (L/m <sup>2</sup> )	Machined	Specimens/Container	Visual Ratings
1	Resistant	100	Yes	2	PA
1	Resistant	100	Yes	2	PA
1	Susceptible	100	Yes	2	EC
1	Susceptible	100	Yes	2	EC
2	Resistant	83	No	1	PA
2	Resistant	83	No	1	PA
2	Susceptible	83	No	1	EC
2	Susceptible	83	No	1	EC
3	Resistant	118	Yes	2	PA
3	Resistant	118	Yes	2	PA
3	Susceptible	118	Yes	2	EC
3	Susceptible	118	Yes	2	EC
4	Resistant	159	No	1	PA
4	Resistant	159	No	1	PA
4	Susceptible	159	No	1	ED
4	Susceptible	159	No	1	ED
5	Resistant	115	No	1	PA
5	Resistant	115	No	1	PA
5	Susceptible	115	No	1	EC
5	Susceptible	115	No	1	EC
6	Resistant	211	No	4	PB
6	Resistant	211	No	4	PB
6	Susceptible	211	No	4	ED
6	Susceptible	211	No	4	ED

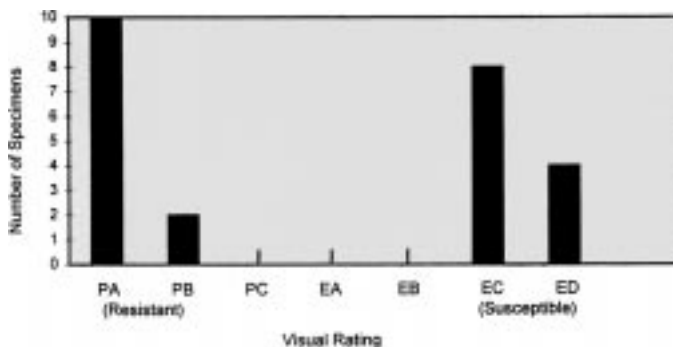


FIG. 3 Histogram of Visual Exfoliation Ratings by Six Laboratories for Two Tempers of 5086 Plate Tested by ASSET (Data in Table 1)

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