



Standard Test Method for Exfoliation Corrosion Susceptibility in 2XXX and 7XXX Series Aluminum Alloys (EXCO Test)¹

This standard is issued under the fixed designation G 34; 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.

1. Scope

1.1 This test method covers a procedure for constant immersion exfoliation corrosion (EXCO) testing of high-strength 2XXX and 7XXX series aluminum alloys.

NOTE 1—This test method was originally developed for research and development purposes; however, it is referenced, in specific material specifications, as applicable for evaluating production material (refer to Section 14 on Precision and Bias).

1.2 This test method applies to all wrought products such as sheet, plate, extrusions, and forgings produced from conventional ingot metallurgy process.

1.3 This test method can be used with any form of specimen or part that can be immersed in the test solution.

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:²

D 1193 Specification for Reagent Water

E 3 Guide for Preparation of Metallographic Specimens

G 15 Terminology Relating to Corrosion and Corrosion Testing

G 112 Guide for Conducting Exfoliation Corrosion Tests in Aluminum Alloys

2.2 ASTM Adjuncts:

Illustrations (Enlarged Glossy Prints)³

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.

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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 Available from ASTM International Headquarters. Order Adjunct No. ADJG003402. Original adjunct produced in 1980.

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 (Terminology G 15).

4. Summary of Test Method

4.1 This test method provides an accelerated exfoliation corrosion test for 2XXX and 7XXX series aluminum alloys that involves the continuous immersion of test materials in a solution containing 4 M sodium chloride, 0.5 M potassium nitrate, and 0.1 M nitric acid at $25 \pm 3^\circ\text{C}$ ($77 \pm 5^\circ\text{F}$). The susceptibility to exfoliation is determined by visual examination, with performance ratings established by reference to standard photographs.

5. Significance and Use

5.1 This test method was originally developed for research and development purposes; however, it is referenced, in specific material specifications, as applicable for evaluating production material (refer to Section 14 on Precision and Bias).

5.2 Use of this test method provides a useful prediction of the exfoliation corrosion behavior of these alloys in various types of outdoor service, especially in marine and industrial environments.⁴ The test solution is very corrosive and represents the more severe types of environmental service, excluding, of course, unusual chemicals not likely to be encountered in natural environments.

5.3 The exfoliation ratings were arbitrarily chosen to illustrate a wide range in resistance to exfoliation in this test. However, it remains to be determined whether correlations can be established between EXCO test ratings and realistic service conditions for a given alloy. It is an ongoing activity of the

⁴ Ketcham, S. J., and Jeffrey, P. W., "Exfoliation Corrosion Testing of 7178 and 7075 Aluminum Alloys" (Report of ASTM G01.05 Interlaboratory Testing Program in Cooperation with the Aluminum Association); and 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.

Task Group on Exfoliation Corrosion of Aluminum Alloys (G01.05.02.08) to maintain outdoor exposure tests for this purpose. For example, it has been reported⁵ that samples of Al-Zn-Mg-Cu alloys rated EA or P in a 48-h EXCO test did not develop more than a slight amount of incipient exfoliation (EA) during six- to nine-year exposures to seacoast atmospheres, whereas, ED rated materials in most cases developed severe exfoliation within a year in the seacoast atmosphere. It is anticipated that additional comparisons will become available as the outdoor tests are extended.

6. Apparatus

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

7. Reagents

7.1 *Purity of Reagents*—The test solution shall be prepared with reagent grade sodium chloride (NaCl), potassium nitrate (KNO₃), and nitric acid (HNO₃).

7.2 *Purity of Water*—Distilled or deionized water shall be used to prepare test solution. The water purity must conform to Specification D 1193, Type IV reagent water, except that for this method, the values of chloride and sodium can be disregarded.

7.3 A test solution of the following composition shall be used:

NaCl	(4.0 M)
KNO ₃	(0.5 M)
HNO ₃	(0.1 M)

Dissolve 234 g of NaCl, 50 g of KNO₃ in water, and add 6.3 mL of concentrated HNO₃ (70 weight %). Dilute to 1 L. This solution has an apparent pH of 0.4.

7.4 The solution shall be maintained at a temperature of 25 ± 3°C (77 ± 5°F).

8. Sampling

8.1 Sampling procedures are not considered applicable to this test method, as they are often covered by product specifications. It is assumed that the test specimens are removed from representative samples of materials.

9. Test Specimens

9.1 Specimens may be of any practical size or shape. Nevertheless, for the results to be of most significance a specimen size of at least 50 by 100 mm (2 by 4 in.), or the equivalent, is recommended.

9.2 The edges of sawed specimens need not be machined, but specimens obtained by blanking or shearing shall have edges dressed by machining or filing to a depth equal to the thickness of the specimen to remove cold-worked metal.

9.3 Remove the cladding of alclad sheet by machining the test surface; remove or mask the cladding on the back side (non-test surface) also.

9.4 When removing test specimens from extrusions and forgings, take care to avoid specimen locations underneath flanges, ribs, etc., where the grain structure is usually variable.

10. Standardization

10.1 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 with known resistance. This control should exhibit the same degree of exfoliation each time it is included in the test.

10.2 The control may be any material of the alloy type included in the scope of this test method, preferably one with an intermediate degree of susceptibility (Fig. 1 and Fig. 2).

11. Procedure

11.1 Degrease the specimens with a suitable solvent.

11.2 Mask the back surfaces of the specimens to minimize corrosion of non-test areas. Protective coatings must have good adherence to avoid crevice corrosion beneath the coating; also, they should not contain leachable ions or protective oils that will influence the corrosion of the test surface. (See Figs. 1-6.)³

11.3 Use the solution in sufficient quantity to provide a volume-to-metal surface area ratio of 10 to 30 mL/cm² (65 to 200 mL/in.²). Include all exposed metal area in the determination of total surface area.

11.4 Use fresh solution at the start of each test. Do not change the solution even though the pH increases during the test. It is normal for the pH to increase from the initial apparent value of 0.4 to about 3 during the first several hours depending upon the amount of corrosion that occurs.

11.5 Immerse the specimens in the solution using rods or racks of inert material to support the specimens above the bottom of the container. Place the test surface upward in a horizontal position to prevent loss of exfoliated metal from the surface of the specimen. Do not concurrently immerse in the same container alloys containing less than 0.25 % copper with those containing greater amounts of copper.

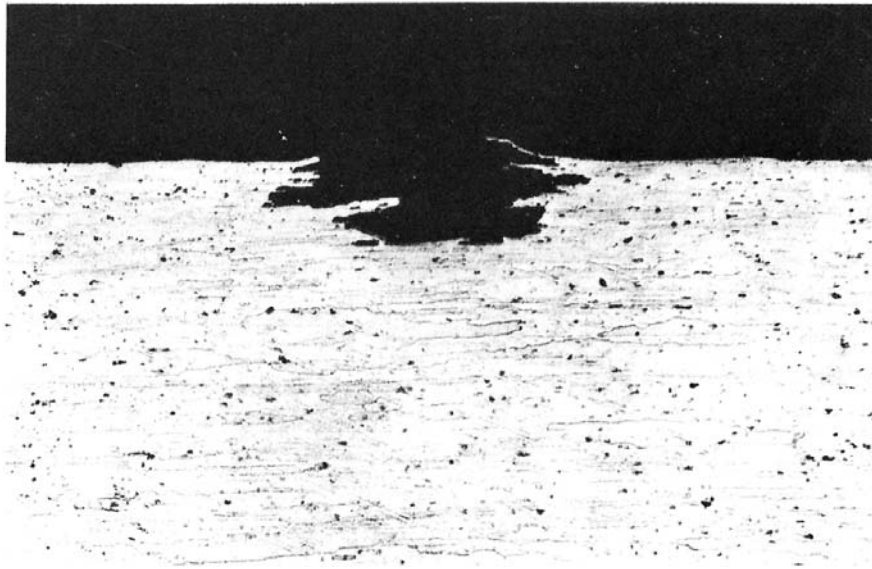
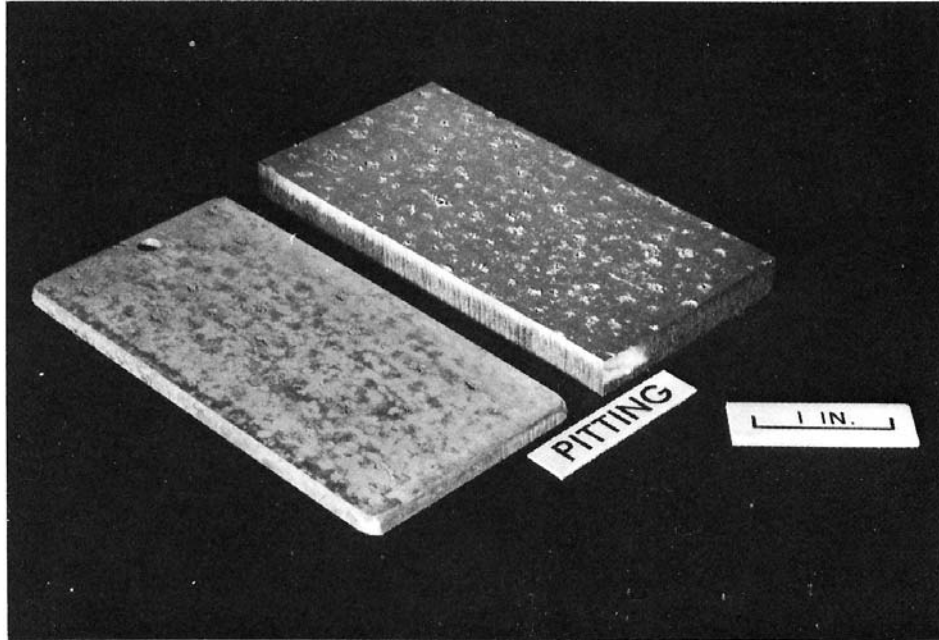
11.6 The following maximum periods of exposure are recommended for testing the alloy types indicated:

2XXX Series	96 h
7XXX Series	48 h

The length of time to develop exfoliation in material of a given alloy and temper may vary with the mill product form, with some materials developing severe exfoliation in much shorter periods than those listed. Therefore, inspect test specimens in place and rate in accordance with Section 12 at periods such as 5, 24, 48, and 72 h, and discontinue the exposure of a specimen when it has developed the most severe exfoliation rating (Fig. 6).

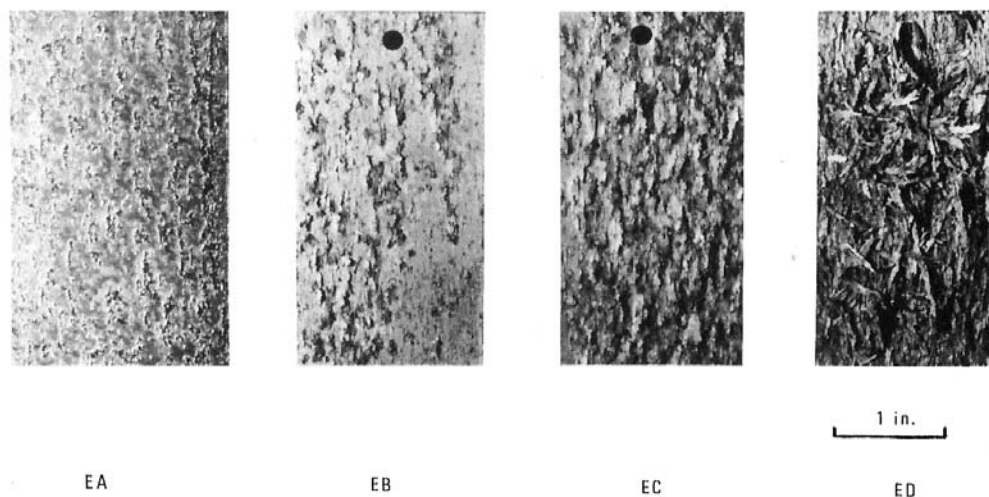
11.7 Rate the performance of test specimens in accordance with Section 12 immediately after discontinuation of the

⁵ Sprowls, D. O., Summerson, T. J., and Loftin, F. E., "Exfoliation Corrosion Testing of 7075 and 7178 Aluminum Alloys—Interim Report on Atmospheric Exposure Tests" (Report of ASTM G01.05.02 Interlaboratory Testing Program in Cooperation with the Aluminum Association); and Lifka, B. W. and Sprowls, D. O., "Relationship of Accelerated Test Methods for Exfoliation Resistance in 7XXX Series Aluminum Alloys With Exposure to a Seacoast Atmosphere," *Corrosion in Natural Environments, ASTM STP 558*, ASTM, 1974.



Undermining pitting that may form the surface give the appearance of incipient exfoliation (Keller's Etch; 100×).

FIG. 1 Examples of Pitting Corrosion



Exfoliation resulting from rapid lateral attack of selective boundaries or strata forming wedges of corrosion product that force layers of metal upward giving rise to a layered appearance (Keller's Etch; 100×).

FIG. 2 Four Degrees of Severity of Exfoliation Corrosion

exposure while the specimens are still wet or moist, taking into account all loose products of exfoliation lying on the test specimen or on the bottom of the container.

11.8 Clean exposed test specimens, if desired, by rinsing in water and soaking in concentrated nitric acid after the specimens have been inspected and rated.

12. Interpretation of Results

12.1 The following codes and classifications shall be used when reporting the visual rating of corroded specimens:

Classification	Code
No appreciable attack	N
Pitting	P
Exfoliation	EA through ED

12.2 Descriptions of the various classifications, which are illustrated in Figs. 1-6,³ are as follows:

12.2.1 *N*—No appreciable attack: Surface may be discolored or etched, but no evidence of pitting or exfoliation.

12.2.2 *P*—Pitting: Discrete pits, sometimes with a tendency for undermining and slight lifting of metal at the pit edges (Fig. 1).

12.2.3 *EA through ED*—Exfoliation (Figs. 1-6):

12.2.3.1 Visible separation of the metal into layers manifested in various forms, such as blisters, slivers, flakes, fairly continuous sheets, and sometimes granular particles resulting from disintegration of thin layers, depending upon the grain morphology of the sample. Various degrees of exfoliation with increasing penetration and loss of metal are illustrated in Fig. 2. Additional examples of the various ratings are shown in Figs. 3-6.

12.2.3.2 The formation of tiny pit-blisters or the dislodgement of an extremely thin surface layer of metal after only a few hours of exposure may resemble superficial exfoliation (EA), but can in fact result from undermining pitting. If continued exposure to the recommended periods in 11.6

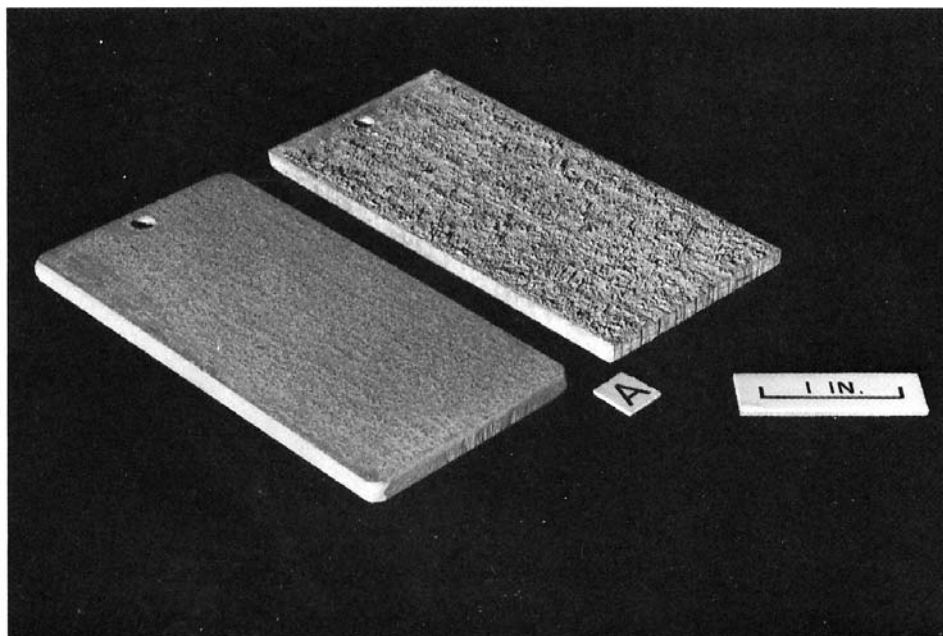


FIG. 3 Examples of Exfoliation Rating EA (Superficial): Tiny Blisters, Thin Slivers, Flakes or Powder, with only Slight Separation of Metal

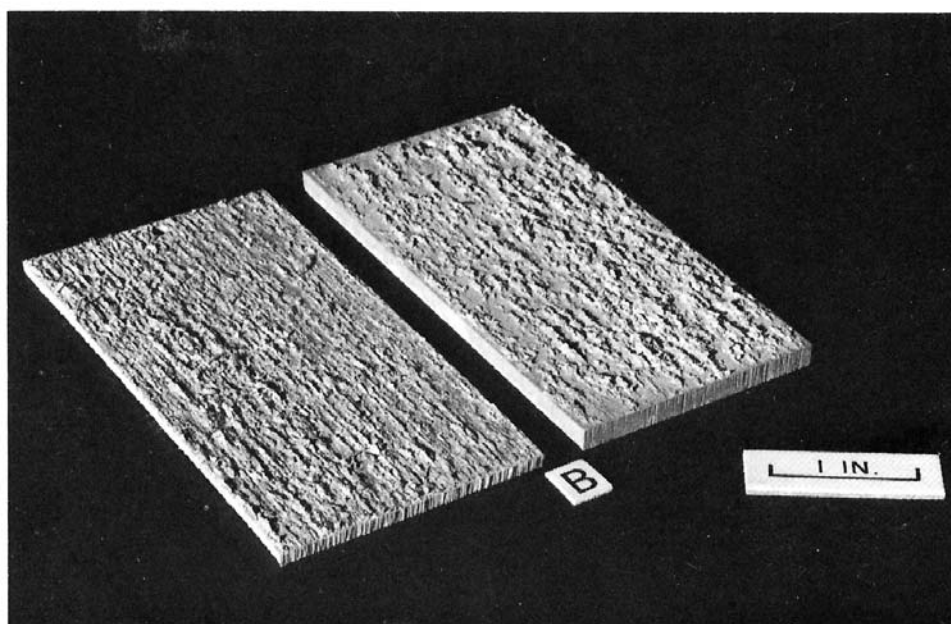


FIG. 4 Example of Exfoliation Rating EB (Moderate); Notable Layering and Penetration into the Metal

produces more corrosion but no evidence of advancing delamination, metallographic examination (see Guide E 3) will be required to determine whether the initial effect was truly exfoliation (Fig. 2) or undermining pitting (Fig. 1); in the latter case the rating should be P.

12.2.3.3 When exfoliation occurs in isolated sites, rate the worst localized condition observed.

12.3 The visual ratings are intended to be finite indications of the resistance to exfoliation, and care should be taken when

rating a series of test specimens to compare them with the photographs and captions in Figs. 1-6 rather than with each other. The final rating of a specimen shall be determined by the poorest classification observed during the exposure.

12.3.1 When it is difficult to classify a specimen, it is advisable to place it in the category of greater susceptibility.

NOTE 2—Enlarged glossy prints of Figs. 1-6 are available from ASTM International.³ These prints are more convenient to use and are of better

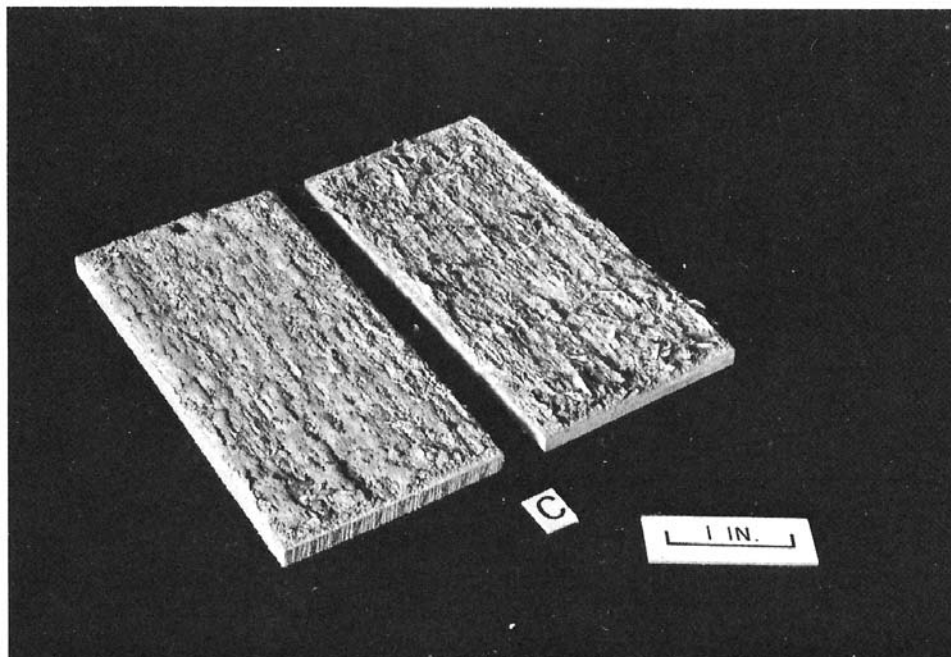


FIG. 5 Examples of Exfoliation Rating EC (Severe): Penetration to a Considerable Depth into the Metal

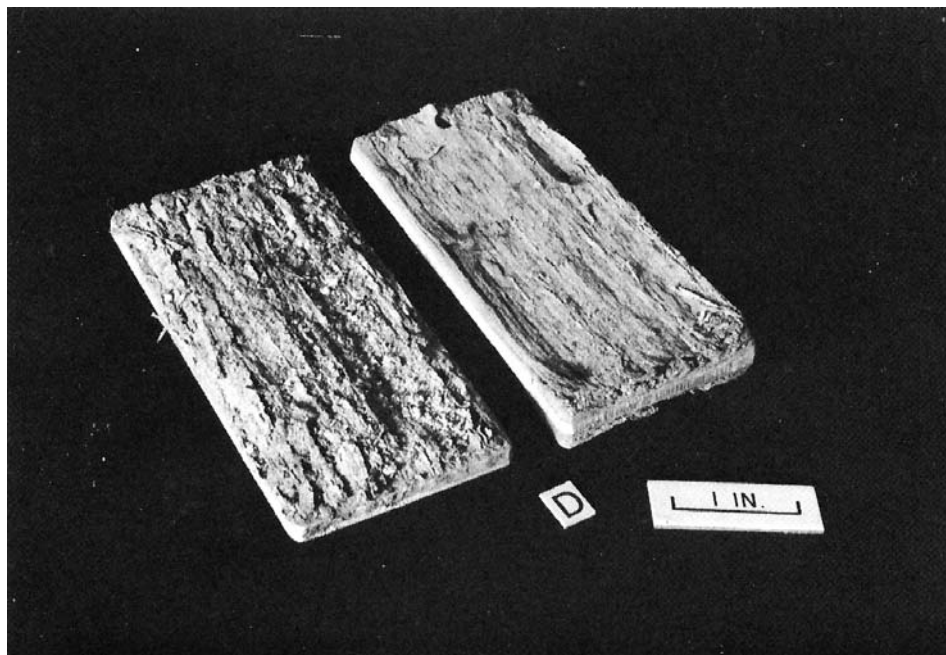


FIG. 6 Examples of Exfoliation Rating ED (Very Severe) (Similar to EC Except for Much Greater Penetration and Loss of Metal)

quality than the reproductions in the printed standard. The user is urged to obtain and use these prints.

13. Report

13.1 The report should contain the following essential information:

- 13.1.1 Alloy and temper of the material tested,
- 13.1.2 Mill product, section thickness, and the surface tested, including reference to applicable product specification,
- 13.1.3 Sampling procedure if other than that specified in referenced product specification,

13.1.4 A rating of the test specimens using the codes and classifications in Section 12, and

13.1.5 Notation of any deviation in test procedure from that set forth in preceding paragraphs.

13.2 Other information that may be desirable for certain types of reports includes:

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

13.2.2 Volume to surface ratio.

14. Precision and Bias

14.1 Precision:

14.1.1 The precision of the data from this test method was evaluated by way of an interlaboratory test program using two non-commercial tempers of Alloy 7075 with different levels of exfoliation corrosion susceptibility. Seven laboratories, including experienced and inexperienced users, participated in the round robin. The laboratories received rough machined panels, which they finish machined, exposed according to the procedure in this test method, and rated visually according to the photographs included in this test method.

14.1.2 The raw data from laboratories is listed in **Table 1** and plotted in **Fig. 7**. All seven laboratories rated the more susceptible T6X material as having severe or very severe exfoliation corrosion as designated by the ratings EC and ED. However, for the more resistant T7X temper there was no agreement as the visual ratings ranged from pitting only to very severe exfoliation as designated by the ratings P to ED. **Fig. 7** shows that there was no clustering of the ratings either, they seem to follow a uniform distribution.

14.1.2.1 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.

14.1.2.2 The reproducibility of ratings among different laboratories is shown most clearly in **Fig. 7**. In addition to the visual ratings, one of the laboratories measured the depth of corrosion using an ultrasonic technique (the laboratories reported original panel thickness) and the data showed not only that the two tempers had different amounts of exfoliation, but that there was good consistency within each temper as plotted in **Fig. 8**. Thus the laboratory to laboratory variation in ratings for the T7X material resulted from individual rater's interpretation of the photographs and wording used to define the visual rating system. Samples with resistance, that is, intermediate between highly susceptible and highly resistant samples can be the most difficult to rate visually, which is confirmed by these results. One of the reasons for this is corrosion debris that does not result from exfoliation corrosion as discussed in Guide **G 112**.

14.1.2.3 Based on the results from this interlaboratory test program, the visual rating system gives consistent ratings for

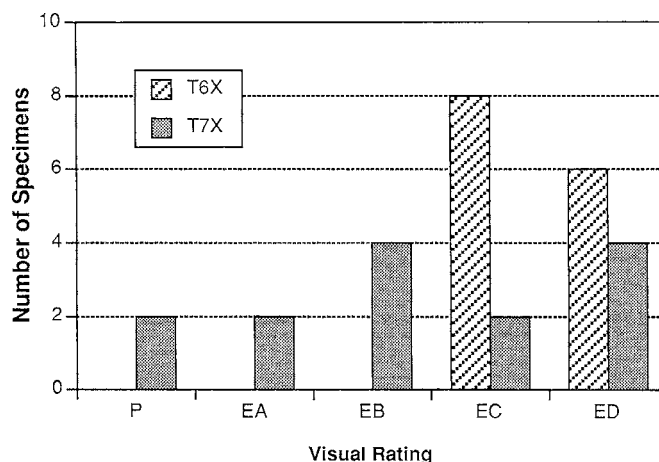


FIG. 7 Histogram of Visual Exfoliation Ratings by Seven Laboratories for Two Tempers of 7075 Plate Tested at the T/4 Plane

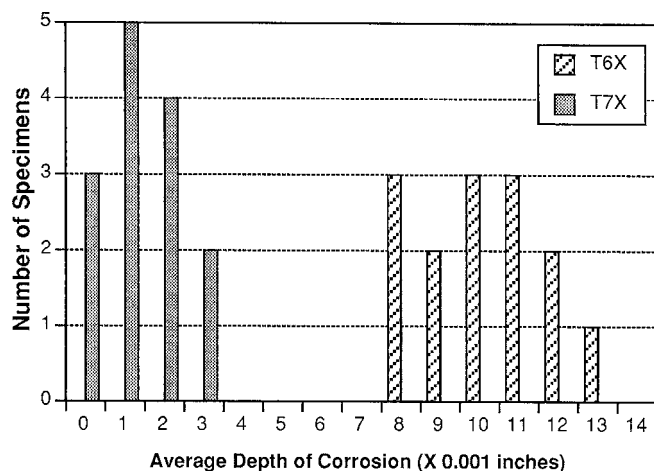


FIG. 8 Histogram of Average Corrosion Depths as Measured by Ultrasonic Inspection for Two Tempers of 7075 Plate at the T/4 Plane

highly susceptible samples, but produces a large amount of laboratory-to-laboratory variation for tempers with intermediate levels of resistance. Experience indicates that the visual ratings will produce more consistent results for highly resistant samples, such as 7075-T73X products, than it does for samples with intermediate resistance, such as T7X tested in this interlaboratory test program.

14.2 *Bias*—The procedure in Test Method G 34 has no bias because the exfoliation rating is defined only in terms of this test method.

15. Keywords

15.1 exfoliation corrosion; heat treatable aluminum alloys; immersion corrosion test; 2XXX aluminum alloys; 7XXX aluminum alloys

TABLE 1 Visual Ratings from Interlaboratory Test of Alloy 7075 Plate in Two Noncommercial Tempers

Laboratory	T6X		T7X	
	Specimen 1	Specimen 2	Specimen 1	Specimen 2
A	ED	ED	EB	EB
B	EC	EC	ED	ED
C	EC	EC	EA	EA
D	EC-ED	EC-ED	EC	EC
E	ED	ED	ED	ED
F	EC	EC	EB	EB
G	EC	EC	P	P

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