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Standard Guide for
Conducting Exfoliation Corrosion Tests in Aluminum Alloys[1]

This standard is issued under the fixed designation G 112; 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 guide differs from the usual ASTM standard in that it does not address a specific test. Rather, it is an introductory guide for new users of other standard exfoliation test methods, (see Terminology G 15 for definition of exfoliation).

1.2 This guide covers aspects of specimen preparation, exposure, inspection, and evaluation for conducting exfoliation tests on aluminum alloys in both laboratory accelerated environments and in natural, outdoor atmospheres. The intent is to clarify any gaps in existent test methods.

1.3 The values stated in SI units are to be regarded as the standard. The inch-pound units 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens[2]

G 15 Terminology Relating to Corrosion and Corrosion Testing[2]

G 34 Test Method for Exfoliation Corrosion Susceptibility in 2XXX and 7XXX Series Aluminum Alloys (EXCO Test)[2]

G 50 Practice for Conducting Atmospheric Corrosion Tests on Metals[2]

G 66 Method for Visual Assessment of Exfoliation Corrosion Susceptibility of 5XXX Series Aluminum Alloys (ASSET Test)[2]

G 85 Practice For Modified Salt Spray (Fog) Testing[2]

G 92 Practice for Characterization of Atmospheric Test Sites[2]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *panel*—a flat, rectangular specimen normally taken with the test surface parallel to the longitudinal and long-transverse dimensions of fabricated product. For thin sheet and extrusions, the thickness may be the full thickness of the part.

3.1.2 *sample*—a portion of a large piece, or an entire piece out of a group of many pieces, that is submitted for evaluation and considered representative of the larger piece or population. For castings and forgings, this may be an extra portion or prolongation, or in the case of small parts, an entire extra piece taken from a specific lot.

3.1.3 *specimen*—the actual test piece to be corrosion tested. Frequently this has a specific shape with prescribed dimensional tolerances and finishes.

3.1.4 *test plane*—the plane in the thickness of the sample that is being tested. Generally this is the fabricated surface or some specified interior plane. Interior planes typically used are: (*a*) T/10 = 10 % of the thickness removed, (this is representative of a minimal machining cut to obtain a flat surface), (*b*) T/4 = quarter plane, 25 % of the thickness removed, and (*c*) T/2 = midplane, 50 % of the thickness removed.

4. Significance and Use

4.1 Although there are ASTM test methods for exfoliation testing, they concentrate on specific procedures for test methodology itself. Existent test methods do not discuss material variables that can affect performance. Likewise they do not address the need to establish the suitability of an accelerated test for alloys never previously tested nor the need to correlate results of accelerated tests with tests in outdoor atmospheres and with end use performance.

4.2 This guide is a compilation of the experience of investigators skilled in the art of conducting exfoliation tests and assessing the degree and significance of the damage encountered. The focus is on two general aspects: guides to techniques

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[1] This guide 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 03.02.

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that will enhance the likelihood of obtaining reliable information, and tips and procedures to avoid pitfalls that could lead to erroneous results and conclusions.

4.3 The following three areas of testing are considered: the test materials starting with the "as-received" sample up through final specimen preparation, the corrosion test procedures including choice of test, inspection periods, termination point, and rating procedures, and analyses of results and methods for reporting them.

4.4 This guide is not intended as a specific corrosion test procedure by which to evaluate the resistance to exfoliation of an aluminum alloy product.

4.5 This guide is not intended as a basis for specifications, nor as a guide for material lot acceptance.

5. Material

5.1 *Sample Size*—Most exfoliation tests do not require any particular specimen size, but when beginning a new investigation it is best to obtain considerably more material than the minimum amount needed. About 50 to 100 % overage is recommended. This avoids the need of procuring a second sample, that may have a different response, to complete any confirmatory retests or extensions to a specific program.

5.2 *Sample Reproducibility*—The specific location of samples in a mill product, and the number of samples to take are beyond the scope of this guide. When testing large production items, a typical procedure is to test at both ends (front and rear), and to test at the side and at the mid-width if the product is 0.6 m (2 ft) or more in width. Thick products should be tested at various planes through the thickness.

5.2.1 In addition, some assessment should be made of the uniformity of a large sample, or of numerous small samples. Typical quick check methods would be to measure electrical conductivity or hardness. If the material variability has a pattern, for example, a difference between front and rear of a long extrusion, then this should be noted and the specimens segregated accordingly. If the variability is random, then multiple test specimens should be randomized.

5.3 *Sample Microstructure*—The directionality of the grain structure of aluminum alloys will markedly affect the susceptibility to exfoliation. When a product shape and alloy are being tested for the first time, it is advisable to macroetch full thickness by longitudinal and by transverse slices to establish the directionality and uniformity of the grain structure. Test panels are normally positioned such that the test surface is parallel to the plane in the product with the most elongated grain structure. Complex shaped parts, such as certain extrusions or die forgings, may have several categories of grain structures and grain flow that do not necessarily follow the part geometry. Grain structure of such parts must be determined by macroetching or from prior experience.

5.3.1 For a given temper condition, unrecrystallized, pancake shaped grains, that are long and wide but relatively thin, are the most susceptible. Pancake shaped recrystallized grains, as in sheet, are the next most susceptible. This is followed by the long, rod shaped grains found in extruded or rolled rod and bar with a symmetrical cross section, for example, circle, square, hex, or a rectangle with the width not more than twice the thickness. An equiaxed grain structure is the least susceptible to exfoliation, especially if the grain size is large. Often the recrystallized surface layer on products such as extrusions, forgings, or sheet will not exfoliate, even though it corrodes intergranularly.

5.4 *Sample Temper*—When a large sample is obtained as a stock item for use over a long time period, the extra material should be stored in a stable temper and at a low enough temperature so that no further precipitation will occur to alter the starting condition of the metal. The unaged W temper of 7XXX alloys is not stable and will continue to age harden at room temperature. Room temperature storage of such material should be limited to a couple of months at most. Natural aging of these alloys can be retarded almost completely by storing the material in a freezer at −40°C (−40°F) or colder. This factor is of even more importance in determination of mechanical properties than the investigation of corrosion resistance.

6. Selection of an ASTM Test Method

6.1 Selection of the appropriate ASTM test method(s) to use will depend primarily on the type of alloy and on the end use environment. When testing a new alloy or temper, a test method known to be applicable to the most similar commercial alloy is normally selected. The user is cautioned, however, that even small changes in alloy chemistry, or changes in processing method (for example, rapid solidification processes) can markedly effect resistance of an alloy and the appropriateness of a test method. Normally exfoliation tests are conducted on ingot metallurgy alloys, that tend to have the elongated grain structure prone to exfoliate. The known alloy applicability of the ASTM test methods are listed below. Included are some observed instances where a test method was found to be inappropriate, or at least produced results different than those observed on the initial qualification alloys.

6.1.1 It is advisable to initially employ more than one laboratory test method and determine whether they agree; or if not, which method is the most discriminating. One procedure for doing this is to apply different fabrication procedures to the metal that are known to generally affect resistance to exfoliation and determine which of the test methods best detects differences in the corresponding resistance to exfoliation. Fabrication variables that often affect resistance to exfoliation are variable quench cooling rates, slow quenches being adverse; and variable amounts of aging, underaged, or peak aged conditions generally being more susceptible than overaged conditions. (**1**)[3]

6.2 Test Method G 66 Acidified Salt Solution Exfoliation Test (ASSET) is used for 5XXX alloys containing 2.0 % or more magnesium. The round robin qualification tests for this test method were conducted on alloys 5086 (3.5 to 4.5 % Mg) and 5456 (4.7 to 5.5 % Mg). (**2**) However, Test Method G 66 (ASSET) gives problem free exfoliation indications with all 5XXX alloys.

6.3 Test Method G 34 Exfoliation Corrosion (EXCO) Test is intended for use with high strength 2XXX and 7XXX ingot

[3] The boldface numbers given in parentheses refer to a list of references at the end of the text.

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metallurgy alloys, a 96 h period being prescribed for the 2XXX alloys and a 48 h period for the 7XXX alloys.

6.3.1 For the 2XXX alloys, the round robin qualification tests were conducted on alloys 2024 and 2124 in the T351 and T851 tempers. The appropriateness of the method has not been fully established for all other 2XXX alloys. It has been reported as being too aggressive and nonrepresentative of performance in outdoor atmospheres for alloys 2219, 2419 and 2519 in the T851 tempers (**3**) and for various Al-Li alloys in both as-quenched and artificially aged tempers (**1**).

6.3.2 For the 7XXX alloys the round robin qualification tests were conducted on alloy 7075 in the T651, T7651, and T7351 tempers and alloy 7178 in the T651 and T7651 tempers. Experience has shown that the EXCO method can be used for 7050 and 7150 alloys in the T651, T6151, T7451, T7651, and T7751 tempers, but the test is somewhat more aggressive on these alloys (**4**). This method also was evaluated with copper free alloys such as 7021-T6 and 7146-T6, but generally an abbreviated exposure period of 16 to 24 h was used.

6.3.3 Exposure of the powder metallurgy alloys 7090 and 7091-T6 specimens to EXCO results in rapid dissolution and powdering of the specimen, due to continuous drop of the extremely fine grains. Four years of exposure of the same parts to seacoast atmosphere resulted only in mild general corrosion and no exfoliation (**5**).

6.4 Annex A2 of Practice G 85 Modified ASTM Acetic Acid Salt Intermittent Spray Test, (MASTMAASIS) was developed using alloys 2024, 2124, 7075, and 7178. This method usually is run in the wet bottom condition (some solution and high humidity always present). A dry bottom condition (no solution present and gradually falling humidity during the purge and non-spraying periods) has been recommended for 2XXX alloys.

6.4.1 The test cabinets used to conduct the MASTMAASIS test, and the salt fog tests subsequently described in 6.5 and 6.6, are produced by several suppliers. The fog delivery systems and cabinet geometry can differ and have gradually evolved. Consequently some cabinet to cabinet variability in test results is inherent, due primarily to variation in spray techniques and the relative humidity conditions during the non-spray portions of the cycle.

6.4.2 There is no record of the MASTMAASIS environment being unrealistically aggressive, causing exfoliation of a material that did not subsequently exfoliate in the seacoast. As such any occurrence of exfoliation in this test most likely indicates susceptibility under some service conditions. The converse of this statement has not been observed.

6.4.3 MASTMAASIS is not appropriate for 5XXX alloys, because it does not always detect exfoliation susceptibility in materials proven to be susceptible by other test methods.

6.4.4 MASTMAASIS has been used with some success on 6XXX series alloys. However, in some cases it caused severe intergranular corrosion that could be confused with exfoliation corrosion unless specimens are examined metallographically.

6.5 Annex A3 of Practice G 85 Seawater Acetic Acid Test (SWAAT) was developed using the same 5XXX, 2XXX, and 7XXX alloys as mentioned above for the ASSET and EXCO methods (**6**).

6.6 Practice G 85 Annex A4 (SALT/SO_2 Spray Testing) was developed using the same, 2XXX and 7XXX alloys as mentioned above for the EXCO method (**7**).

6.7 Both the methods in Annex A3 and Annex A4 of Practice G 85 result in more gelatinous corrosion products than does Annex A2. This tends to increase pitting corrosion on the specimens. Annex methods A2, A3, and A4 in Practice G 85 are not equivalent, and the user should determine which method best suits the alloys and applications under investigation.

7. Baseline Experience

7.1 The best check on the appropriateness of an accelerated test is to determine whether the results it produces agree with known service experience.

7.2 When there is no actual service experience, then exposure in a severe outdoor atmosphere known to produce exfoliation corrosion is a useful approximation of the conditions a part will encounter in service. The most frequently used environments are seacoast sites and highly industrialized urban locations. Selection of the particular environment to use can best be based on the intended end use. If there is no prior experience with the particular alloy being tested, then outdoor tests should be started as soon as possible to establish a baseline for eventual comparison.

7.3 Seacoast atmospheres are representative of the more extreme conditions most parts can encounter in service. However, it is noteworthy that "Seacoast Atmospheric Conditions" prevail only in the immediate vicinity of the seashore. Generally "seacoast" conditions no longer exist after 0.4 Km (0.25 mile) distance from the shoreline.

7.3.1 Significant differences have been noted in tests conducted at the two beach sites at Kure Beach, NC which are located 25 and 250 m (80 and 800 ft) from the shoreline (**8**).

7.3.2 A notable example of this effect is observed at the U.S. Army's exposure sites at Fort Sherman, at the Caribbean entrance to the Panama Canal. The Breakwater and Coastal sites are within sight of each other and have been photographed in one picture. However, the Breakwater site incurs direct saltwater spray from wave action of the Caribbean Sea, whereas the Coastal site is about 50 m (165 ft) from the shore and is protected from wave action by a coral reef. Depending on the season of the year and the length of exposure, corrosion rates of iron and steel were two to nine times higher for the Breakwater site compared with the Coastal site (**9**).

7.3.3 At least two years exposure is needed at a seacoast site in order to be considered a significant length of exposure. Materials with marked susceptibility to exfoliation normally begin to show some evidence of it within 6 to 24 months. Materials showing very mild susceptibility to exfoliation in accelerated tests may require as long as seven to nine years of exposure at a seacoast site to develop a similar degree of exfoliation (**10**).

8. Specimens

8.1 *Specimen Size*—There is no required specimen size or shape, but it is advisable not to use too small a specimen since visual inspection is a key interpretation method. Specimens should be at least 50 mm (2 in.) long and 25 mm (1 in.) or more

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