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

This standard is issued under the fixed designation G112; 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 various standard exfoliation test methods with consideration for specific aluminum alloy families (see Terminology G193 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 standard. The values given in parentheses after SI units are provided for information only and are not considered 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, 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]

G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens

G34 Test Method for Exfoliation Corrosion Susceptibility in 2XXX and 7XXX Series Aluminum Alloys (EXCO Test)

G50 Practice for Conducting Atmospheric Corrosion Tests on Metals

G66 Test Method for Visual Assessment of Exfoliation Corrosion Susceptibility of 5XXX Series Aluminum Alloys (ASSET Test)

G85 Practice for Modified Salt Spray (Fog) Testing

G92 Practice for Characterization of Atmospheric Test Sites

G193 Terminology and Acronyms Relating to Corrosion

2.2 *ASTM Adjunct:*[3]

Illustrations of Appearance Classifications (6 glossy photos)

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *panel, n*—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, n*—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, n*—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, n*—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

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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-E-PDF. Original adjunct produced in 1980.

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 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. Sample

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. An alternative practice is to use a standard first-step age that will stabilize the material and allow for variations in the subsequent second and third aging steps.

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 chemical composition, 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**).[4]

6.2 Test Method G66 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 % Mg to 4.5 % Mg) and 5456 (4.7 % Mg to 5.5 % Mg). (**2**) However, Test Method G66 (ASSET) gives problem-free exfoliation indications with all 5XXX alloys.

6.3 Test Method G34 Exfoliation Corrosion (EXCO) Test is intended for use with high strength 2XXX and 7XXX ingot 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 h 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 G85 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. This reflects experience and controlled tests that show 7xxx alloys to be more susceptible to wet bottom, but 2xxx alloys to be more susceptible to dry bottom; in fact, wet bottom underestimates seacoast exfoliation susceptibility for a number of 2xxx alloys.

6.4.1 The dry bottom condition has become the standard for 2XXX Al-Li alloys as it has been shown to accurately reflect seacoast performance for numerous Al-Li alloys and tempers (**6**).

[4] The boldface numbers in parentheses refer to a list of references at the end of this standard.

6.4.2 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.

6.4.3 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.

6.4.4 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.

6.4.5 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 G85 Seawater Acetic Acid Test (SWAAT) was developed using the same 5XXX, 2XXX, and 7XXX alloys as mentioned above for the ASSET and EXCO methods (**7**).

6.6 Practice G85 Annex A4 (SALT/SO_2 Spray Testing) was developed using the same, 2XXX and 7XXX alloys as mentioned above for the EXCO method (**8**).

6.7 Both the methods in Annex A3 and Annex A4 of Practice G85 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 G85 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 m and 250 m (80 ft and 800 ft) from the shoreline (**9**).

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 (**10**).

7.3.3 Similarly, there can be considerable variation among seacoasts sites due to ambient conditions such as temperature, humidity, airborne pollutants, and physical layout such as rocky coasts that increase seawater spray, the shape of the coastline, and prevailing wind direction. Controlled studies have been conducted that show time to develop exfoliation varying, but the various sites agreeing on the sample ranking with respect to exfoliation resistance. (**9**, **10**, **11**)

7.3.4 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 months 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 (**11**).

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 in width. This surface area permits visual interpretation as to whether any exfoliation is just protruding whiskers of metal, small flakes, or delamination of strips of metal. Typical sizes are: 38 mm to 50 mm by 100 mm (1.5 in. or 2 in. by 4 in.) for the Test Method G34 EXCO test, and the Test Method G66 ASSET test, 75 mm by 150 mm (3 in. by 6 in.) for the Practice G85 Modified Salt Fog tests, Annex A2 (MASTMAASIS), A3 (SWAAT) and A4 (SALT/SO_2), and 100 mm by 150 mm to 300 mm (4 in. by 6 in. to 12 in.) or larger for outdoor atmospheric tests.

8.1.1 Specimens from product forms with non-uniform grain structures, such as extrusions, rod, bar, and forgings, must be large enough to encompass all relevant grain flow regions. In some cases, the initial test plan may need to include multiple locations to determine whether severity varies among the locations. Subsequent testing would only need to sample the most susceptible site or sites.

8.2 *Specimen Identification and Records*—Considerable material may be lost in the testing of susceptible materials, so scribed or stenciled specimen numbers often are inadequate. Some sort of permanent identification should be used. One method for accelerated tests is to number the back side of the specimen and then mask off that area. A separate tag of a non-corrodible, non-conducting material is another method.

8.2.1 On-site tests frequently run for many years and may be evaluated by several persons. It is important, therefore, to have good initial records describing the original material, the specimens, the test purposes, and the intended periods of exposure. Clear records should also be maintained with descriptive remarks or documenting photographs for each inspection period.

8.3 *Specimen Machining*—Specimen edges may be sawed or machined. If panels are obtained by shearing, the edges should be dressed back by machining, sanding, or filing to a depth equal to or greater than the specimen thickness. The cladding should be removed from the test surface of specimens from alclad sheet and either removed or masked off on the back (non-test) surface. When machining panels for exposure of interior planes (T/10, T/2, and so forth.) the final machining cut should be a light one of 0.635 mm (0.025 in.) or less to avoid having a highly worked surface. The grain structure of such a worked surface may not exfoliate and instead create a misleading artifact by peeling off in one layer when the underlying structure corrodes. For very thick plate and other thick products, a good procedure is to saw off most of the material and machine only the last 2.5 mm (0.100 in.) or so. If any cosmetic differences (for example, color changes, scratches, surface roughness, and so forth.) are noted on the as-machined surfaces, they should be recorded. Subsequently the investigator should establish whether these visible differences had an effect on initiation or development of exfoliation.

8.4 *Surface Preparation*—Specimens should be degreased with a suitable solvent, and it is advisable to remove any mill scale by mechanical methods such as machining or sanding, and so forth, or by appropriate etching. A frequently used etch technique is to etch for 1 min in 5 % by weight sodium hydroxide solution at 80 °C (175 °F), rinse in water, desmut 30 s in concentrated nitric acid at room temperature, rinse with distilled or deionized water, and air dry.

8.5 *Specimen Framing*—Guidelines for outdoor exposure of metals are given in Practice G50. Specimens exposed outdoors should preferably be held in place by inert, non-conducting fasteners and holders. Any metallic fasteners must be galvanically compatible with the test specimens, or be insulated from them. It is advisable to have the panels offset from the mounting rack, regardless of the material of construction of the rack. Normal corrosion test procedures should be used to ensure that each specimen is electrically isolated from other specimens and from specimen holders. Ceramic, fiber, or plastic washers are often used to mount outdoor specimens and the crevice created between the washer and the test specimen may hasten the onset of exfoliation.

8.6 Many outdoor exposure tests expose the principal test surface skyward to incur maximum exposure to sunlight and airborne pollutants. However, experience has shown that the earthward surface usually is more prone to exfoliate than the skyward surface. Joint Aluminum Association-ASTM groups on atmospheric exfoliation testing have recommended earthward exposure to avoid washing of exfoliated surfaces by rainfall, which can remove species such as NaCl and pollutants. When conditions are not known for a particular test site or a new material, it may be advisable to initially use duplicate panels exposing the test surface both skyward and earthward. Single specimens can be used when the more critical exposure position has been established.

9. Initiation of Specimen Exposure

9.1 It is advisable to start short term tests, such as the 24 h ASSET test and the 48 h EXCO test, early in the day so that the specimens can be given an initial inspection before the end of the work day.

9.2 Corrosion will initiate and progress sooner during the warmer months at outdoor tests that experience appreciable seasonal changes in temperature and other climatic conditions. When possible, it is best to start outdoor tests at the beginning of the warmer seasons.

10. Test Controls

10.1 It is always advisable to include control specimens from known materials representing both high and low resistance to exfoliation. This is recommended for both accelerated and outdoor tests. Such controls verify the validity of a particular test and permit the investigator to make some assessment of the normalcy of a particular test run. For example, it cannot be concluded that a new material is resistant to exfoliation if the susceptible control specimen did not exfoliate to the usual degree. In outdoor tests, the condition of the susceptible control serves as an indicator of when a significant exposure period has been accrued. Controls are especially advisable in outdoor tests that encounter variable conditions in temperature, rainfall, airborne pollutants, and so forth, beyond the control of the investigator.

11. In-Test Inspection

11.1 *Periodic Inspection*—Even though there usually is a prescribed test period, it is a good practice to inspect the panels *in-situ* during the course of the exposure to note when exfoliation begins and how it progresses. Care should be taken so as not to dislodge any exfoliated metal from specimens showing appreciable corrosion. A specimen is usually removed from test when it becomes so severely exfoliated that there is a risk of the exfoliated metal falling off with continued exposure.

11.2 EXCO specimens, that are usually exposed for 48 h, can be inspected after 4 h to 6 h (or at the end of the first working day) and after 24 h exposure. Salt fog (MASTMAASIS and SWAAT) specimens can be inspected after periods of 3 days, 7 days, 10 days, 14 days, and 28 days. If the investigator has no idea what to expect of a new alloy or temper, it is advisable to expose replicate specimens that can be removed individually as significant progress in exfoliation is noted.

11.3 *Outdoor Tests*—Specimens exposed outdoors to natural atmospheres should be examined twice per year, or more often, during the first two years of exposure and at least yearly thereafter. In regions where the climate varies seasonally, some investigators prefer to make the biannual inspections in early spring and late fall rather than on a strict semiannual basis.

11.3.1 Frequently a specimen is photographed when exfoliation is first noted, and again when appreciable changes occur. Visual inspection may not be able to establish whether exfoliation is present on an atmospheric specimen showing only mild surface corrosion. In such cases it may be advisable to remove a small coupon from a corner for metallographic examination of the cross-section to establish the type of corrosion present. Specimens should be returned to test as quickly as possible, and care should be taken to avoid contamination of the test surface with materials not present at the outdoor site. Time spent out of the intended atmosphere should be recorded, along with any unintended circumstance or incident.

12. Duration or Termination of Exposure

12.1 In any environment, testing of individual specimens generally is terminated when they become so corroded that further exposure is likely to result in complete loss of the exfoliated metal, or when the material's performance is judged to be too poor to be of commercial interest.

12.2 *Accelerated Tests*—Standard tests generally are conducted for the recommended exposure period. If no appreciable exfoliation is observed on a new alloy or temper, the period can be doubled. If this still does not produce significant exfoliation it generally can be concluded that the material is not susceptible to exfoliation in that test method.

12.3 *Outdoor Tests*—Past experience has shown that materials that are very prone to exfoliation in service conditions will show marked exfoliation within four years exposure at severe outdoor sites, such as seacoast and certain highly industrialized urban areas. If test space is limited, specimens surviving this length of exposure at outdoor sites known to cause exfoliation, can be terminated and considered "not highly susceptible." However, some investigators now have programs of 20 or more years duration and the indication is that continued exposure will discriminate between materials with the "better and best" resistance. At this time there is no established time period after which it can be concluded that exfoliation will never occur. For long life applications, the limiting maximum exposure most likely has to be agreed upon by users and producers, based on the life expectancy of the product.

12.4 When long time outdoor tests are conducted, the investigator must realize that all outdoor environments are changeable. Most sites experience cyclic atmospheric conditions. Also these conditions may increase and decrease in corrosiveness, often as a function of surrounding environmental factors beyond the control of the investigator. This is highlighted by the current critical issue of acid precipitation, together with probable clean-up efforts. Ideally atmospheric conditions should be continuously monitored, by means such as those covered in Practice G92. This includes both collection of atmospheric data and periodic exposures of standard specimens of known response.