Designation: G67 – 13

Standard Test Method for Determining the Susceptibility to Intergranular Corrosion of 5XXX Series Aluminum Alloys by Mass Loss After Exposure to Nitric Acid (NAMLT Test)[1]

This standard is issued under the fixed designation G67; 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 describes a procedure for constant immersion intergranular corrosion testing of 5XXX series aluminum alloys.

1.2 This test method is applicable only to wrought products.

1.3 This test method covers type of specimen, specimen preparation, test environment, and method of exposure.

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

1.5 *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]

D1193 Specification for Reagent Water

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

3. Summary of Test Method

3.1 This test method consists of immersing test specimens in concentrated nitric acid at 30°C (86°F) for 24 h and determining the mass loss per unit area as a measure of susceptibility to intergranular corrosion.

4. Significance and Use

4.1 This test method provides a quantitative measure of the susceptibility to intergranular corrosion of Al-Mg and Al-Mg-Mn alloys. The nitric acid dissolves a second phase, an aluminum-magnesium intermetallic compound (βAl-Mg), in preference to the solid solution of magnesium in the aluminum matrix. When this compound is precipitated in a relatively continuous network along grain boundaries, the effect of the preferential attack is to corrode around the grains, causing them to fall away from the specimens. Such dropping out of the grains causes relatively large mass losses of the order of 25 to 75 mg/cm^2 (160 to 480 mg/in^2), whereas, samples of intergranular-resistant materials lose only about 1 to 15 mg/cm^2 (10 to 100 mg/in^2). When the βAl-Mg compound is randomly distributed, the preferential attack can result in intermediate mass losses. Metallographic examination is required in such cases to establish whether or not the loss in mass is the result of intergranular attack.

4.2 The precipitation of the second phase in the grain boundaries also gives rise to intergranular corrosion when the material is exposed to chloride-containing natural environments, such as seacoast atmospheres or sea water. The extent to which the alloy will be susceptible to intergranular corrosion depends upon the degree of precipitate continuity in the grain boundaries. Visible manifestations of the attack may be in various forms such as pitting, exfoliation, or stress-corrosion cracking, depending upon the morphology of the grain structure and the presence of sustained tensile stress.[3]

5. Interferences

5.1 If all loose particles are not removed during cleaning after exposure, the mass loss will be low relative to the amount of corrosion that actually occurred.

6. Apparatus

6.1 *Nonmetallic Container*—A suitable inert, nonmetallic container should be used to contain the nitric acid and

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[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 Association, Inc.

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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] Craig, H. L. Jr., "Nitric Acid Weight Loss Test for the H116 and H117 Tempers of 5086 and 5456 Aluminum Alloys," *Localized Corrosion—Cause of Metal Failure, ASTM STP 516*, ASTM, 1972, pp. 17–37.

specimens during the period of the test. The use of individual beakers for each specimen is recommended; however, the immersion of multiple specimens in the same container is acceptable.

6.1.1 The specimens should be situated in the container so that none of the major surfaces is in total contact with the walls of the container. Also, specimens should be isolated electrically from one another. A recommended method of positioning the specimens is to incline them so that the edges rest on the bottom and side wall of the container.

6.1.2 The container should have a loose fitting cover to reduce evaporation and to confine any fumes evolved by the acid.

7. Reagents

7.1 *Purity of Reagents*—The nitric acid (HNO_3) test solution shall be reagent grade and have a specific gravity within the range of 1.4134 to 1.4218 (70 to 72 weight %). The sodium hydroxide (NaOH) solution used for etching and the HNO_3 (70 to 72 weight %) used for desmutting shall also be reagent grade.

7.2 *Purity of Water*—Use water conforming to Specification D1193 Type IV to prepare the NaOH solution and for rinsing purposes.

8. Test Solution

8.1 Use sufficient test solution to fully immerse the specimens and constitute a volume to specimen surface area ratio of at least 30 L/m^2 (19 mL/in^2).

8.2 Maintain the test solution temperature at 30 ± 0.1°C (86 ± 0.2°F).

9. Sampling

9.1 The specific location of samples in a mill product, the number of samples that should be tested, and so forth, are outside the scope of this standard.

10. Test Specimens

10.1 Prepare specimens with dimensions 50 mm by 6 mm (2 in. by 0.25 in.) by product thickness. The 50-mm dimension shall be parallel to the longitudinal direction of the product.

10.2 If the thickness of the product is greater than 25 mm (1 in.), reduce it by one half or to 25 mm, whichever is less, while retaining one original as-fabricated surface. All sawn surfaces shall be machined to the final dimensions.

10.3 Take a minimum of two specimens from each sample.

11. Procedure

11.1 Smooth all edges with a fine file or fine emery paper (320).

11.2 Identification of specimens can be achieved by either scribing or stamping. Confine identification of specimens to an as-fabricated surface.

11.3 Measure all three dimensions to the nearest 0.02 mm (0.001 in.), and calculate the total surface area.

11.4 Immerse the specimens in 5 % NaOH solution at 80°C (176°F) for 1 min followed by a water rinse, a 30-s immersion in HNO_3 (desmut), and a water rinse. Allow the specimens to air dry. Do not wipe dry with a cloth or paper towel.

11.5 Weigh the specimens to no more than ±1.0 mg, preferably 0.1 mg.

11.6 Immerse the specimens in the test solution for 24 h.

11.7 Remove the specimens, and rinse with water while brushing with a stiff plastic bristle brush, for example, a toothbrush, to remove all adhering particles. Allow to air dry.

11.8 Weigh the specimens and determine mass losses to no more than ±1.0 mg, preferably 0.1 mg. Reclean to obtain a constant weight in accordance with Practice G1.

11.9 Calculate the mass losses per unit area and express as mg/cm^2 to the nearest whole number.

NOTE 1—Optional—To ascertain the morphology of corrosion, the specimen can be cross-sectioned, polished, and examined metallographically after the mass loss determination.

12. Report

12.1 The report should contain the following information:

12.1.1 Alloy identification,

12.1.2 Product and temper of material,

12.1.3 Sampling procedure particularly with respect to location in the product and the original material thickness,

12.1.4 Mass loss per unit area,

12.1.5 Any deviation in test procedure from that set forth in preceding paragraphs,

12.1.6 Solution volume to specimen surface area ratio,

12.1.7 Specimen dimensions, and

12.1.8 Number of specimens tested in each container.

13. Precision and Bias

13.1 *Statement on Precision:*

13.1.1 Reproducibility of the mass loss results decreases with increased susceptibility to intergranular attack. Data on the precision of test results obtained by this test method derived from interlaboratory tests of 5086 and 5456 alloys are shown in ASTM STP 516.[3] The standard deviations for test results among six different laboratories ranged from 0.2 to 3.9 mg/cm^2 for resistant materials with mean mass losses ranging from 1 to 8 mg/cm^2 and from 5.5 to 6.6 mg/cm^2 for susceptible materials with mean losses ranging from 30 to 44 mg/cm^2 (see Footnote 4). The coefficient of variation is 0.3 ± 0.1.

13.1.2 The repeatability/variation within a laboratory is about 0.05 coefficient of variation.

13.1.3 To provide an indication when some inadvertent deviation from the correct test condition 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 mass loss each time it is included in the test.

13.1.4 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.

13.2 *Bias*—The procedure in this test method has no bias because the value of mass loss due to intergranular corrosion is defined only in terms of this test method.

14. Keywords

14.1 5xxx aluminum alloys; aluminum alloys; grain boundary sensitization; intercrystalline corrosion; intergranular corrosion; mass loss; nitric acid; pitting corrosion

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