Designation: C703 – 72 (Reapproved 2016)

Standard Test Methods for Spalling Resistance of Porcelain Enameled Aluminum[1]

This standard is issued under the fixed designation C703; 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.

INTRODUCTION

This test, using an ammonium chloride or antimony trichloride test solution, covers an accelerated procedure for determining the resistance of porcelain enamel coatings on aluminum and aluminum alloys to spontaneous loss of adhesion (spalling) resulting from exposure to moisture, weathering, or other environmental stress.

1. Scope

1.1 These test methods cover accelerated determination of the resistance of porcelain enamel coatings on aluminum alloys to spalling from exposure to moisture or weathering. Test Method A,[2] using a 5 % solution of ammonium chloride, requires 96-h immersion while Test Method B,[3] using a 1 % solution of antimony trichloride, is completed after 20 h of immersion. The spalling tendency is evaluated by the same criteria in both methods. While either method is suitable for magnesium silicon alloys, such as 6061, Test Method B is preferred for simple alloys or commercially pure aluminum, such as 1100.

1.2 The test methods appear in the following order:

2. Terminology

2.1 *Definitions:*

2.1.1 *spalling*—a defect characterized by separation of the porcelain enamel from the aluminum base metal without apparent external cause. Spalling can result from the use of improper alloys or enamel formulations, incorrect pretreatment of the base metal, or faulty application and firing procedures.

3. Significance and Use

3.1 It is difficult to overemphasize the importance of the spall test. Porcelain enameled aluminum that fails this test will probably spall in service if subjected to moisture or weathering.

TEST METHOD A—AMMONIUM CHLORIDE

4. Apparatus

4.1 *Container,* glass or plastic, large enough to immerse the test area of the specimen completely and hold a minimum of 3 mL of solution per square centimetre of the immersed surface. No metal other than the base metal of the specimen may be exposed to the test solution.

5. Reagent

5.1 *Ammonium Chloride Solution (5 %)*—The test solution, freshly prepared, shall consist of 5 parts, by mass, of ammonium chloride (NH_4Cl) (technical grade is adequate) dissolved in 95 parts, by mass, of water. Deionized or distilled water is preferred, but in the case of very large production pieces, tap water may be used. Sufficient solution shall be prepared to permit complete immersion of the specimens.

6. Test Specimens

6.1 Full-size production pieces should be tested. When this is not practical, specimens approximately 4 by 6 in. (102 by 152 mm), cut from production parts should be tested. At least one representative specimen from each "job" or each 1000 ft^2 (93 m^2) of production, whichever is applicable, should be spall tested. Spall-tested pieces should not be shipped.

6.2 Test production parts in as-produced condition.

7. Procedure

7.1 Immerse the test specimens completely in the NH_4Cl solution at room temperature. Large production pieces may be

[1] These test methods are under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and are the direct responsibility of Subcommittee B08.12 on Materials for Porcelain Enamel and Ceramic-Metal Systems.

Current edition approved Nov. 1, 2016. Published November 2016. Originally approved in 1972. Last previous edition approved in 2011 as C703 – 72 (2011). DOI: 10.1520/C0703-72R16.

[2] Method A is based on Bulletin AL-1a, *Recommended Test Methods for Evaluation and Control of Quality of Porcelain Enamel on Aluminum* , of the Porcelain Enamel Institute.

[3] Method B is based on Bulletin T-51, *Antimony Trichloride Spall Test of Porcelain Enameled Aluminum*, of the Porcelain Enamel Institute.

immersed in a container made by lining a packing box with polyethylene plastic sheeting.

7.2 Make a visual inspection after 96 h of immersion.

NOTE 1—Variations of this test may be used for studies of processing variables. These include scored or deformed samples, 24 h inspection, and multiple cycles of 96 h each.

8. Evaluation

8.1 Any of the following types of spall that develop within 96 h shall constitute failure:

8.1.1 Any spall area (revealing bare metal) extending ⅛ in. (3 mm), or more, in from an edge and more than 1 in. (25 mm) in length,

8.1.2 Any spall area on the interior surface (not touching an edge) that is more than ⅛ in. (3 mm) to its maximum dimension, or

8.1.3 More than six visible spall spots per 1 ft^2 (929 cm^2) on the interior surface.

9. Report

9.1 The report shall include the following:

9.1.1 Evaluation of the specimen as passing or failing the immersion test in 96 h,

9.1.2 Complete identification of the specimen, including the basis metal, metal pretreatment, enamel slip formulation, firing time and temperature, date, and any other pertinent processing information, and

9.1.3 Size, description of part, number of specimens tested, and ratio of parts tested to parts produced.

TEST METHOD B—ANTIMONY TRICHLORIDE

10. Apparatus

10.1 *Container,* glass or plastic, large enough to immerse all or part of the test specimen and hold a minimum of 3 mL of solution per square centimetre of specimen surface.

10.2 *Steel Rod or Mandrel* of ½-in. (12.7-mm) diameter.

11. Reagent

11.1 *Antimony Trichloride Solution* (1 %)—The test solution, freshly prepared, shall consist of 1 part, by mass, of antimony trichloride ($SbCl_3$) (technical grade is adequate) dissolved in 99 parts, by mass, of water (10 g/L). Tap water may be used. Stir the solution thoroughly to disperse the antimony trichloride.

11.2 Discard the test solution when it becomes 3 days old or when the 1 gal/195 $in.^2$ (3 mL/cm^2) limit is reached, whichever occurs first.

12. Test Specimens

12.1 When practical, full-size production pieces may be tested; otherwise, specimens approximately 4 by 6 in. (102 by 152 mm), cut from production parts or a 4-in. (102-mm) wide cross-sectioned cut sample from a production piece should be tested. It is necessary that the metal-enamel interface is exposed to the test solution. Cut edges or the cracks in the enamel caused by bending over a mandrel usually accomplish this. If not, the interface should be exposed by scoring the enamel. At least one representative specimen from each "job" or each 1000 ft^2 (93 m^2) of production, whichever is applicable, should be spall tested.

12.2 Test production parts in as-produced condition.

12.3 Wherever practical (such as porcelain enameled sheet material) specimens cut from production pieces should be bent to a 45° angle over a ½-in. (12.7-mm) diameter rod or mandrel.

13. Procedure

13.1 Immerse as much of the test specimen as practical in the $SbCl_3$ solution at room temperature. If a sample has been bent over a mandrel, the bent portion must be immersed in the solution.

13.2 After 20 h of immersion, rinse the sample in water and scrub with a sponge to remove loose glass flakes (particularly on the bent areas).

14. Evaluation

14.1 On flat or non-deformed areas, any of the following types of spall that develop within 20 h shall constitute failure:

14.1.1 Any spall area (revealing bare metal) extending ⅛ in. (3 mm), or more, in from an edge and more than 1 in. (25 mm) in length.

14.1.2 Any spall area on the interior surface (not touching an edge) that is more than ⅛ in. (3 mm) in its maximum dimension, or

14.1.3 More than six visible spall spots per 1 ft^2 (929 cm^2) on the interior surface. Disregard pinholes up to 1/16-in. (1.5-mm) diameter.

14.2 The evaluation of spall on the formed (bent) enamel areas has not been standardized for all aluminum alloys used in sheet form. While spalling on bends indicates a potential field failure of production pieces, absence of spalling on bends may not be indicative of good porcelain adherence of all aluminum alloys in sheet form. For 1100 clad alloy, the following spall developing in 20 h shall constitute failure:

14.2.1 Any spall area on the bent portion (excluding the edges) that is more than ⅛ in. (3 mm) wide or more than ½ in. (12.7 mm) in length.

15. Report

15.1 The report shall include the following:

15.1.1 Evaluation of the specimens as passing or failing the immersion test in 20 h, for the bent or deformed and flat areas, respectively,

15.1.2 Complete identification of the specimen, including the basis metal, metal pretreatment, enamel slip formulation, firing time and temperature, date, and any other pertinent processing information, and

15.1.3 Size, description of parts, number of specimens tested, and ratio of parts tested to parts produced.

16. Precision and Bias

16.1 No justifiable statements can be made regarding the precision and bias of these test methods because it is designed for application to full-size production parts, with the result that

variables due to design, metal composition, fabrication, and metal processing, as well as porcelain enameling, are introduced into the results.