1973

Designation: A 393 – 63ε

Standard Recommended Practice for CONDUCTING ACIDIFIED COPPER SULFATE TEST FOR INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEEL[1]

This Standard is issued under the fixed designation A 393; 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.

The committee responsible for this standard has voted its withdrawal. In the absence of substantial reasons that it should be continued, the Society will approve withdrawal from publication in April 1974.

ε NOTE—Editorial changes were made in Section 6 in July 1969.

1. Scope

1.1 This recommended practice describes the procedure for conducting the acidified copper sulfate test employed to measure the relative susceptibility of austenitic stainless steels to intergranular attack (Note 1). The presence or absence of attack in this test is not necessarily a measure of the performance of the material in other corrosive environments.

NOTE 1—This method is less sensitive than those described in ASTM Recommended Practice A 262, for Detecting Susceptibility to Intergranular Attack in Stainless Steels,[2] and will detect only severe sensitization.

1.2 The test is sometimes used to evaluate the heat treatment accorded "as-received" material. It is also used to check the effectiveness of stabilized or low-carbon (0.03 percent carbon, max) types of stainless steel to prevent intergranular attack.

1.3 This recommended practice may be applied to wrought products (including tubes), castings, and weld metal of austenitic stainless steel.

2. Apparatus

2.1 *Container*—A suitable glass or other inert container for the acidified copper sulfate solution, fitted with a condenser capable of avoiding loss of acid by evaporation. A 1-liter, wide-mouth Erlenmeyer flask (Note 2) equipped with either a 30-in. reflux condenser or a bulb-type condenser, such as used in Recommended Practice A 262, has been found satisfactory. If a reflux condenser is used, its connection to the flask should be made through a ground-glass joint. Rubber or cork stoppers are not suitable.

NOTE 2—Some samples, such as from castings or heavy bar stock, may require the use of a larger Erlenmeyer flask.

2.2 *Specimen Supports*—Glass hooks, stirrups, or cradles may be used for supporting the specimens in the flask. Their design should prevent specimens coming in contact with each other when tested in the same container.

2.3 *Heater*—A means for heating the test solution and of keeping it boiling throughout the test period. A gas or electrically heated hot plate is satisfactory for this purpose.[3]

3. Reagent

3.1 *Acidified Copper Sulfate Solution*—Dissolve 100 g of copper sulfate ($CuSO_4 \cdot 5H_2O$) in 700 ml of distilled water, add 100 ml of sulfuric acid (H_2SO_4, cp, sp gr 1.84), and make up to 1000 ml with distilled water.

NOTE 3—This solution will contain approximately 6 percent of anhydrous $CuSO_4$ and 16 percent of H_2SO_4 (by weight).

4. Test Specimens

4.1 *Size:*

4.1.1 The size of the test specimen and method of selecting it shall be that specified in the product specification or by agreement

[1] This recommended practice is under the jurisdiction of ASTM Committee A-1 on Steel, Stainless Steel and Related Alloys and is the direct responsibility of Subcommittee A01.14 on Methods of Corrosion Testing.

Current edition effective Sept. 30, 1963. Originally issued 1955. Replaces A 393-55 T.

[2] *Annual Book of ASTM Standards*, Part 3.

[3] A Glascol heater with a Variac has been found suitable for this purpose.

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between the purchaser and seller as stated in the purchase contract. Consideration should be given to the volume of solution to be used, whether the product is wrought or cast, how the specimen is to be selected, and the method of bending the specimen for inspection. The size of the specimen should permit easy entrance and removal through the relatively narrow neck of the Erlenmeyer flask.

4.1.2 Weld metal specimens shall be prepared in accordance with AWS Specification A 5.4, for Corrosion-Resisting Chromium and Chromium-Nickel Steel Covered Welding Electrodes.[4]

4.1.3 The sizes given in Table I may be used as guides for referee purposes. The exact length will be governed by the equipment used for bending the specimen.

4.2 *Sheared Edges*—When specimens are cut by shearing, the sheared edges shall be refinished by machining or wet grinding prior to testing.

4.3 *Surface Finishing:*

4.3.1 Any scale on the "as-received" surface should be removed by mechanical finishing. Unless it is desired to test specimens with some particular surface finish (such as cold-rolled strip or sheet in the "as-received" condition), all surfaces of the specimen, including edges, should be finished with 120-grit iron-free aluminum oxide abrasive, taking care to avoid overheating.

4.3.2 *Cleaning*—The specimen shall be degreased using a suitable solvent, such as acetone, a mixture of alcohol and ether, or a vapor degreaser, and then dried.

4.4 *Sensitizing Treatment:*

4.4.1 *"As-received" Material*—All material in the "as-received" condition should be capable of meeting this test.

4.4.2 *Stabilized and Low-Carbon Types*—For some purposes, it may be desirable to check the effectiveness of stabilized materials and 0.03 percent max carbon types to prevent intergranular attack. The following conditions are commonly used for all types of material: 1 h in air at a temperature of 1250 F (±25 F) followed by an air cool.

NOTE 4—Heating test specimens in air is preferred. They should not be treated in atmospheres which will carburize them nor in contact with high-carbon steels or any other source of carbon.

NOTE 5—Heat-treated material should be descaled and finished for test as prescribed in 4.3.1. Test specimens from cold-rolled sheet or strip may be tested in the "as-sensitized" condition.

5. Test Conditions

5.1 *Volume of Solution*—A sufficient quantity of the acidified copper sulfate test solution to cover the specimens and to provide a volume of at least 50 ml/in.^{2} (8 ml/cm^2) of specimen surface area shall be used.

5.2 *Number of Specimens per Flask*—The best practice is to use a separate container for each specimen. However, it is acceptable to treat as many as three specimens in the same container, provided they are all the same grade and that the specimen supports prevent the specimens from contacting each other and provided the solution volume to sample area ratio is maintained. If any specimen tested in the same flask fails the test, new specimens representative of the failed material shall be retested separately.

5.3 *Boiling Time:*

5.3.1 After the test specimen has been placed in the test solution, it should be brought to a boil and kept boiling throughout the test period.

NOTE 6—"Bumping" of the solution while boiling may be minimized by the use of glass beads, sections of porcelain crucibles, abrasive aluminum oxide rings, or boiling stones.

5.3.2 The test should consist of one 72-h boiling period unless otherwise specified.

NOTE 7—When more than one 72-h boiling period is specified, fresh test solution should be used for each period.

6. Bend Test

6.1 *Bending:*

6.1.1 The test specimen for material purchased in the annealed condition shall be bent 180 deg over a diameter equal to the thickness of the section or the diameter of the rod unless otherwise specified in the product specification. In no case, however, shall the specimen be bent over a smaller radius or through a greater angle than that specified for material purchased in other than the annealed condition.

6.1.2 Castings with heavy walls have large

[4] Available from American Welding Society, 2501 North West 7th St., Miami, Fla. 33125.

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grain size and low ductility which may not permit a 180-deg bend. In cases of low ductility, duplicate specimens should be made of the material. One of the specimens should not be exposed to the acidified $CuSO_4$ solution, and the bend test on this specimen will serve as a standard to determine the maximum angle of bend without causing cracks in the unexposed condition. The specimen exposed to the test solution is bent similarly and the results are compared.

6.1.3 Sheet samples shall be bent to an "S" shape with the surface on both sides of the rolled samples tested in tension.

NOTE 8—The "S" shape bend ensures detection of intergranular attack resulting from carburization of one surface of sheet material during the final stages of rolling.

6.1.4 Samples machined from round sections or cast material shall have the curved or original surface on the outside of the bend.

6.1.5 The specimens are generally bent by holding in a vise and starting the bend with a hammer. It is generally completed by bringing the two ends together in the vise. Heavy specimens may require bending in a fixture of suitable design.

6.1.6 Tubular products should be bent in accordance with the flattening test prescribed in the product specification.

NOTE 9—A typical flattening test is described in ASTM Specification A 213, for Seamless Alloy-Steel Boiler, Superheater, and Heat Exchanger Tubes.[5]

6.2 *Evaluation*—The U-bend shall be examined under low (5× to 15×) magnification. The appearance of fissures or cracks indicates the presence of intergranular attack.

NOTE 10—Cracking that originates at the edges of the specimen should be disregarded. The appearance of deformation lines, wrinkles, or "orange peel" on the surface, without accompanying cracks or fissures, should be disregarded also.

NOTE 11—For referee purposes, where the above evaluation is questioned, the presence or absence of grain boundary attack shall be determined by metallographic examination of a cross section of the specimen at a magnification of 100× to 250×.

[5] *Annual Book of ASTM Standards*, Part 1.

TABLE 1 Sizes of Test Specimens

Type of Material	Size of Test Specimen
Wrought wire or rod:	
Up to 1/4 in. in diameter, incl	full diameter by 3 to 5 in. long
Over 1/4 in. in diameter	cylindrical segment 3/4 in. thick by 1 in. (max) wide by 3 to 5 in. long[a]
Wrought sheet, strip, plates, or flat rolled products:	
Up to 3/16 in. thick, incl	full thickness by 3/8 to 1/2 in. wide by 3 to 5 in. long
Over 3/16 in. thick	3/16 in. thick by 3/8 to 1/2 in. wide by 3 to 5 in. long[a]
Tubing:	
Up to 1 1/2 in. in diameter, incl	full ring, 1 in. wide[c]
Over 1 1/2 in. in diameter	a circumferential segment 3 to 5 in. long cut from a 1-in. wide ring[d]
Castings[e]	3/16 in. thick by 3/8 to 1/2 in. wide by 3 to 5 in. long

[a] When bending such specimens, the curved surface shall be on the outside of the bend.
[b] One surface shall be an original surface of the material under test and it shall be on the outside of the bend. It shall be finished as prescribed in 4.3.1. Cold-rolled strip or sheets may be tested in the "as-received" condition.
[c] Ring sections are not flattened or subjected to any mechanical work before they are subjected to the test solution.
[d] Specimens from welded tubes over 1 1/2 in. in diameter shall be taken with the weld on the axis of the bend.
[e] Specimens to be machined from keel blocks or as near the outside surface of the casting as practical.

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