Standard Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels[1]

This standard is issued under the fixed designation A262; 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 These practices cover the following five tests:

1.1.1 *Practice A*—Oxalic Acid Etch Test for Classification of Etch Structures of Austenitic Stainless Steels (Sections 4 to 13, inclusive),

1.1.2 *Practice B*—Ferric Sulfate-Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections 14 to 25, inclusive),

1.1.3 *Practice C*—Nitric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections 26 to 36, inclusive),

1.1.4 *Practice E*—Copper–Copper Sulfate–Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections 37 to 46, inclusive), and

1.1.5 *Practice F*—Copper–Copper Sulfate–50 % Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Molybdenum-Bearing Austenitic Stainless Steels (Sections 47 to 58, inclusive).

1.2 The Oxalic Acid Etch Test is a rapid method of identifying, by simple etching, those specimens of certain stainless steel grades that are essentially free of susceptibility to intergranular attack associated with chromium carbide precipitates. These specimens will have low corrosion rates in certain corrosion tests and therefore can be eliminated (screened) from testing as "acceptable." The etch test is applicable only to those grades listed in the individual hot acid tests and classifies the specimens either as "acceptable" or as "suspect."

1.3 The ferric sulfate-sulfuric acid test, the copper–copper sulfate–50 % sulfuric acid test, and the nitric acid test are based on weight loss determinations and, thus, provide a quantitative measure of the relative performance of specimens evaluated. In contrast, the copper–copper sulfate–16 % sulfuric acid test is based on visual examination of bend specimens and, therefore, classifies the specimens only as acceptable or nonacceptable.

1.4 The presence or absence of intergranular attack in these tests is not necessarily a measure of the performance of the material in other corrosive environments. These tests do not provide a basis for predicting resistance to forms of corrosion other than intergranular, such as general corrosion, pitting, or stress-corrosion cracking.

NOTE 1—See Appendix X1 for information regarding test selection.

1.5 The values stated in SI units are to be regarded as standard. The inch-pound equivalents are in parentheses and may be approximate.

1.6 *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.* Some specific hazards statements are given in 10.1, 20.1.1, 20.1.9, 31.3, 34.4, 53.1.1, and 53.1.10.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A370 Test Methods and Definitions for Mechanical Testing of Steel Products

A380/A380M Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems

D1193 Specification for Reagent Water

E3 Guide for Preparation of Metallographic Specimens

2.2 *ASME Code:*[3]

ASME Boiler & Pressure Vessel Code, Section IX

2.3 *ACS Specifications:*[4]

Reagent Chemicals, Specifications and Procedures

*A Summary of Changes section appears at the end of this standard

2.4 *ISO Standard:*[5]

ISO 3651-2 Determination of Resistance to Intergranular Corrosion of Stainless Steels—Part 2: Ferritic, Austenitic, and Ferritic-Austenitic (Duplex) Stainless Steels—Corrosion Test in Media Containing Sulfuric Acid

3. Purity of Reagents

3.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society[6] where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the test result.

3.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D1193.

PRACTICE A—OXALIC ACID ETCH TEST FOR CLASSIFICATION OF ETCH STRUCTURES OF AUSTENITIC STAINLESS STEELS (1)[7]

4. Scope

4.1 The Oxalic Acid Etch Test is used for acceptance of wrought or cast austenitic stainless steel material but not for rejection of material. Use of A262 Practice A as a stand-alone test may reject material that the applicable hot acid test would find acceptable; such use is outside the scope of this practice.

4.2 This test is intended to be used in connection with other evaluation tests described in these practices to provide a rapid method for identifying qualitatively those specimens that are certain to be free of susceptibility to rapid intergranular attack in these other tests. Such specimens have low corrosion rates in the various hot acid tests which require from 15 to 240 h of exposure. These specimens are identified by means of their etch structures, which are classified according to the criteria given in Section 11.

4.3 The Oxalic Acid Etch Test may be used to screen specimens intended for testing in Practice B—Ferric Sulfate-Sulfuric Acid Test, Practice C—Nitric Acid Test, Practice E—Copper-Copper Sulfate–16 % Sulfuric Acid Test, and Practice F—Copper-Copper Sulfate–50 % Sulfuric Acid Test.

4.4 Each of these other practices contains a table showing which classifications of etch structures on a given stainless steel grade are equivalent to acceptable or suspect performance in that particular test. Specimens having acceptable etch structures need not be subjected to the hot acid test. Specimens having suspect etch structures must be tested in the specified hot acid solution.

4.5 There are two classes of specimens to be considered: base metal, and process-affected metal.

4.5.1 Process-affected metal contains any condition that affects the corrosion properties of the material in a non-uniform way, such as (but not limited to) welds; carburized. nitrided, or oxidized surfaces; mechanical deformation; and areas affected by heat. Base metal has none of these conditions.

4.5.2 Because Practices B, C, and F involve immersing the entire specimen and averaging the mass loss over the total specimen area, and because welding, carburization, mechanical deformation, and the like affect only part of a specimen, the presence of process-affected metal in a specimen can affect the test result in an unpredictable way depending on the proportions of the area affected.

4.5.3 If the presence of these or other localized conditions is a concern to the purchaser, then tests that do not average the mass loss over the total specimen surface area, such as Practice A, the Oxalic Acid Etch Test, or Practice E, the Copper–Copper Sulfate–Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels, should be considered.

5. Summary of Practice

5.1 A specimen representative of the material to be evaluated is polished to a specified finish and over-etched using oxalic acid electrolytically. The etched specimen is then examined using a metallurgical microscope. The etched structure is compared with reference photographs to determine whether the material is acceptable or suspect. Suspect material is then subjected to the specified hot acid immersion test.

6. Significance and Use

6.1 Use of the etch test allows rapid acceptance of specific lots of material without the need to perform time-consuming and costly hot acid immersion tests on those lots.

7. Apparatus

7.1 *Etching Cell:*

7.1.1 An etching cell may be assembled using components as described in this section. Alternatively, a commercial electropolisher/etcher (as used for metallographic sample preparation) may be used for small specimens provided the current density requirement of 10.2 is met.

7.1.2 *Source of Direct Current*—Battery, generator, or rectifier capable of supplying about 15 V and 20 A.

7.1.3 *Ammeter*—For direct current; used to measure the current on the specimen to be etched.

7.1.4 *Variable Resistance*—Used to control the current on the specimen to be etched.

7.1.5 *Cathode*—A stainless steel container, for example, a 1-L (1-qt) stainless steel beaker.

7.1.5.1 *Alternate Cathode*—A piece of flat stainless steel at least as large as the specimen surface.

7.1.6 *Electrical Clamp*—To hold the specimen to be etched and to complete the electrical circuit between the specimen and the power source such that the specimen is the anode of the cell.

7.1.7 The power source, resistor, and ammeter must be sized appropriately for providing and controlling the current as specified in 10.2 of this practice.

7.1.8 As described, the electrolyte container is the cathode; it may be a stainless steel beaker or fabricated from stainless steel such as by welding a section of tube or pipe to a flat plate or sheet. Alternatively, the electrolyte container may be glass (or other non-conducting, corrosion resisting material) in lieu of a stainless steel container, and the cathode may be a flat plate or sheet of a corrosion resisting alloy. In this latter case, the flat surface of the cathode must be at least as large as, facing, and approximately centered on, the prepared surface of the specimen. Other configurations of the electrodes might not provide uniform etching over the specimen surface. In any case, the size and shape of the specimen dictate the size and construction of the etching cell and of the power source and controls. The overriding principle is that the etch needs to be uniform over the surface to be examined.

7.2 *Metallurgical Microscope*—For examination of etched microstructures at 250 to 500 diameters.

8. Reagents and Materials

8.1 *Etching Solution (10 %)*—Dissolve 100 g of reagent grade oxalic acid crystals ($H_2C_2O_4 \cdot 2H_2O$) in 900 mL of reagent water. Stir until all crystals are dissolved.

8.1.1 *Alternate Etching Solution* (See 10.7)—Dissolve 100 g of reagent grade ammonium persulfate ($(NH_4)_2S_2O_8$) in 900 mL of reagent water. Stir until dissolved.

9. Sampling and Test Specimens

9.1 The specified hot acid test provides instructions for sampling and for specimen preparation such as a sensitization heat treatment. Additional instructions specific to Practice A follow:

9.2 The preferred specimen is a cross-section including the product surface to be exposed in service. Only such finishing of the product surface should be performed as is required to remove foreign material.

9.3 Whenever practical, use a cross-sectional area of 1 cm^2 or more. If any cross-sectional dimension is less than 1 cm, then the other dimension of the cross-section should be a minimum of 1 cm. When both dimensions of the product are less than 1 cm, use a full cross section.

9.4 *Polishing*—On all types of materials, polish cross sectional surfaces through CAMI/ANSI 600 [FEPA/ISO P1200] in accordance with Guide E3 prior to etching and examination. Not all scratches need to be removed.

10. Procedure

10.1 (**Warning—**Etching should be carried out under a ventilated hood. Gas, which is rapidly evolved at the electrodes with some entrainment of oxalic acid, is poisonous and irritating to mucous membranes.)

10.2 Etch the polished specimen at 1 A/cm^2 for 1.5 min.

10.2.1 To obtain the correct specified current density:

10.2.1.1 Measure the total immersed area of the specimen to be etched in square centimetres.

10.2.1.2 Adjust the variable resistance until the ammeter reading in amperes is equal to the total immersed area of the specimen in square centimetres.

10.3 A yellow-green film is gradually formed on the cathode. This increases the resistance of the etching cell. When this occurs, remove the film by rinsing the inside of the stainless steel beaker (or the steel used as the cathode) with an acid such as 30 % HNO_3.

10.4 The temperature of the etching solution gradually increases during etching. Keep the temperature below 50°C. This may be done by alternating two containers. One may be cooled in tap water while the other is used for etching.

10.4.1 The rate of heating depends on the total current (ammeter reading) passing through the cell. Therefore, keep the area to be etched as small as possible while at the same time meeting the requirements of desirable minimum area to be etched.

10.5 Avoid immersing the clamp holding the specimen in the etching solution.

10.6 *Rinsing*—Following etching, rinse the specimen thoroughly in hot water and then in acetone or alcohol to avoid crystallization of oxalic acid on the etched surface during drying.

10.7 It may be difficult to reveal the presence of step structures on some specimens containing molybdenum (AISI 316, 316L, 317, 317L), which are free of chromium carbide sensitization, by electrolytic etching with oxalic acid. In such cases, an alternate electrolyte of ammonium persulfate may be used in place of oxalic acid. (See 8.1.1.) An etch for 5 or 10 min at 1 A/cm^2 in a solution at room temperature readily develops step structures on such specimens.

11. Classification of Etch Structures

11.1 Examine the etched surface on a metallurgical microscope at 250× to 500× for wrought steels and at about 250× for cast steels.

11.2 Examine the etched cross-sectional areas thoroughly by complete traverse from inside to outside diameters of rods and tubes, from face to face on plates.

11.2.1 Microscopical examination of a specimen shall be made on metal unaffected by cold-working, carburization, welding, and the like. If any of these conditions are found, note their presence in the report.

11.3 Classify the etch structures into the following types (Note 2):

11.3.1 *Step Structure* (Fig. 1)—Steps only between grains, no ditches at grain boundaries.

11.3.2 *Dual Structure* (Fig. 2)—Some ditches at grain boundaries in addition to steps, but no single grain completely surrounded by ditches.

11.3.3 *Ditch Structure* (Fig. 3)—One or more grains completely surrounded by ditches.

11.3.4 *Isolated Ferrite* (Fig. 4)—Observed in castings and welds. Steps between austenite matrix and ferrite pools.

11.3.5 *Interdendritic Ditches* (Fig. 5)—Observed in castings and welds. Deep interconnected ditches.

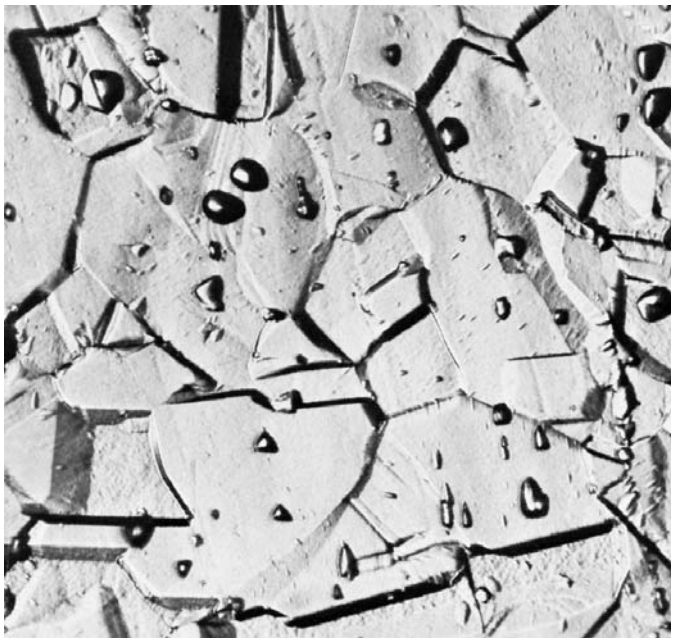


FIG. 1 Step Structure (500×) (Steps Between Grains, No Ditches at Grain Boundaries)

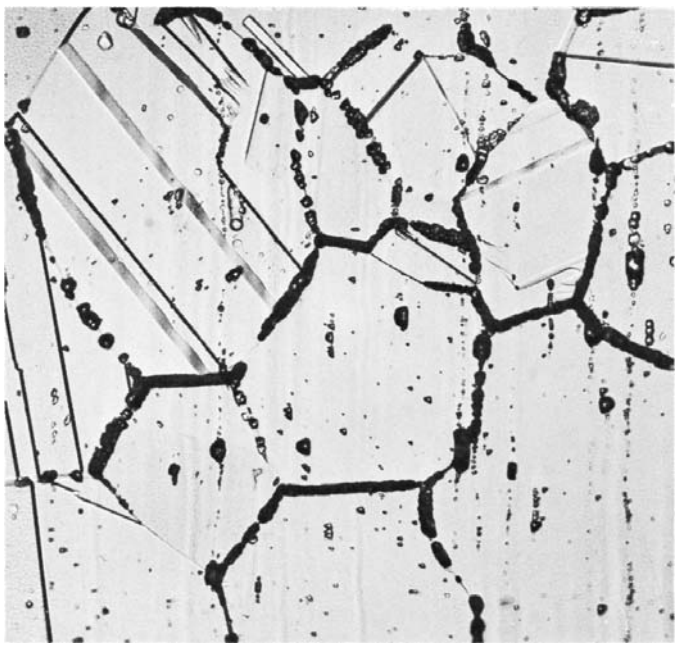


FIG. 2 Dual Structure (250×) (Some Ditches at Grain Boundaries in Addition to Steps, but No One Grain Completely Surrounded)

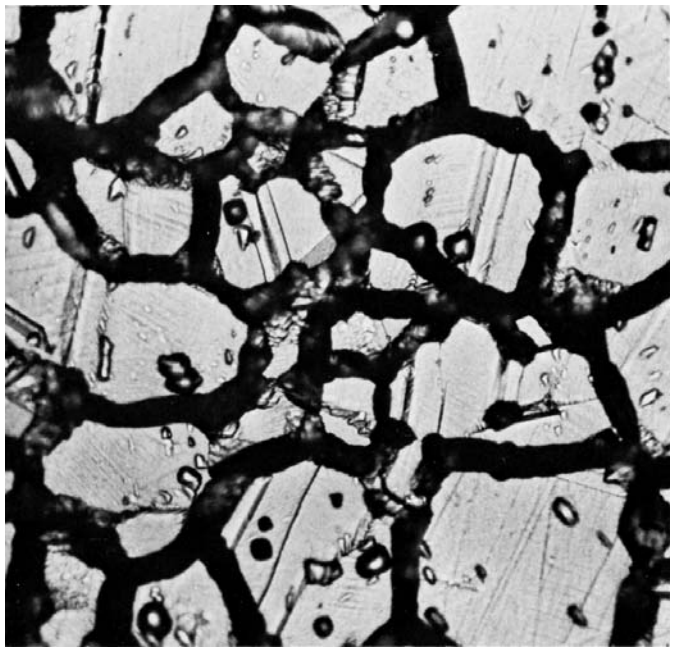


FIG. 3 Ditch Structure (500×) (One or More Grains Completely Surrounded by Ditches)

FIG. 4 Isolated Ferrite Pools (250×) (Observed in Castings and Welds. Steps Between Austenite Matrix and Ferrite Pools)

11.3.6 *End-Grain Pitting I* (Fig. 6)—Structure contains a few deep end-grain pits along with some shallow etch pits at 500×. (Of importance only when the nitric acid test is used.)

11.3.7 *End-Grain Pitting II* (Fig. 7)—Structure contains numerous, deep end-grain pits at 500×. (Of importance only when nitric acid test is used.)

NOTE 2—All photomicrographs were made with specimens that were etched under standard conditions: 10 % oxalic acid, room temperature, 1.5 min at 1 A/cm^2.

11.4 The evaluation of etch structures containing only steps and of those showing grains completely surrounded by ditches in every field can be carried out relatively rapidly. In cases that appear to be dual structures, more extensive examination is required to determine if there are any grains completely encircled. If an encircled grain is found, classify the steel as a ditch structure.

11.4.1 On stainless steel castings (also on weld metal), the steps between grains formed by electrolytic oxalic acid etching tend to be less prominent than those on wrought materials or

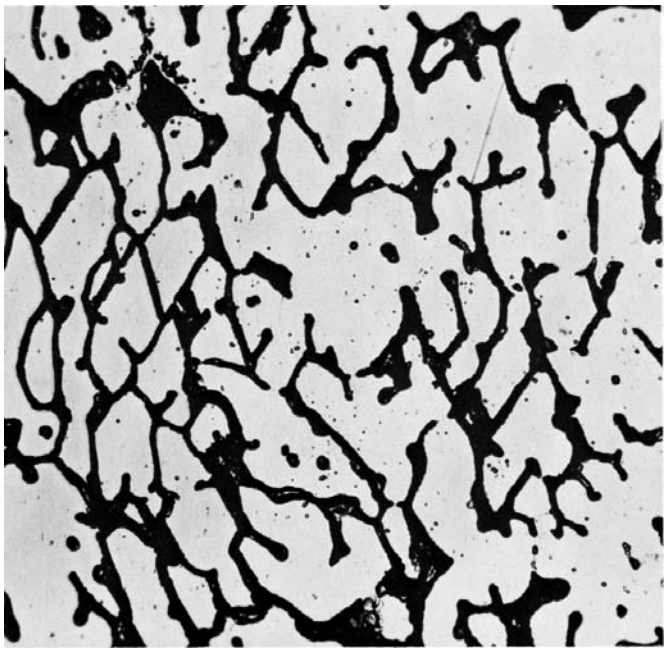
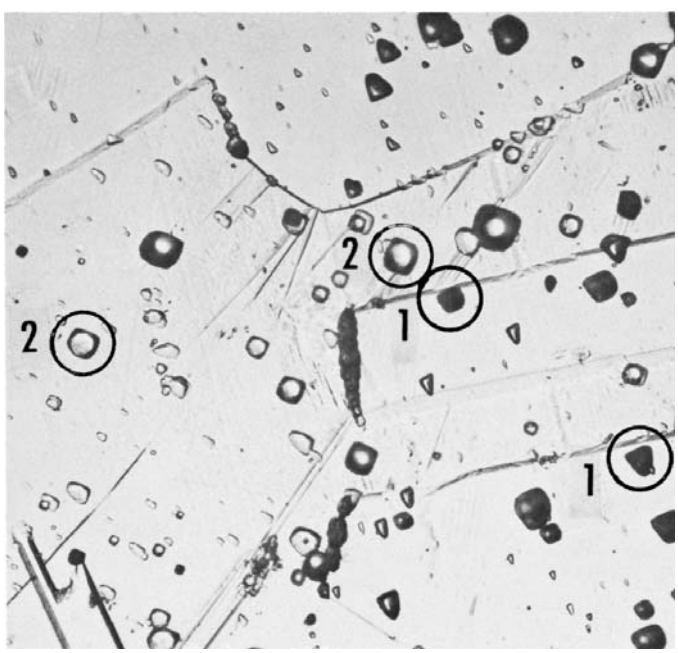


FIG. 5 Interdendritic Ditches (250×) (Observed in Castings and Welds. Deep Interconnected Ditches)

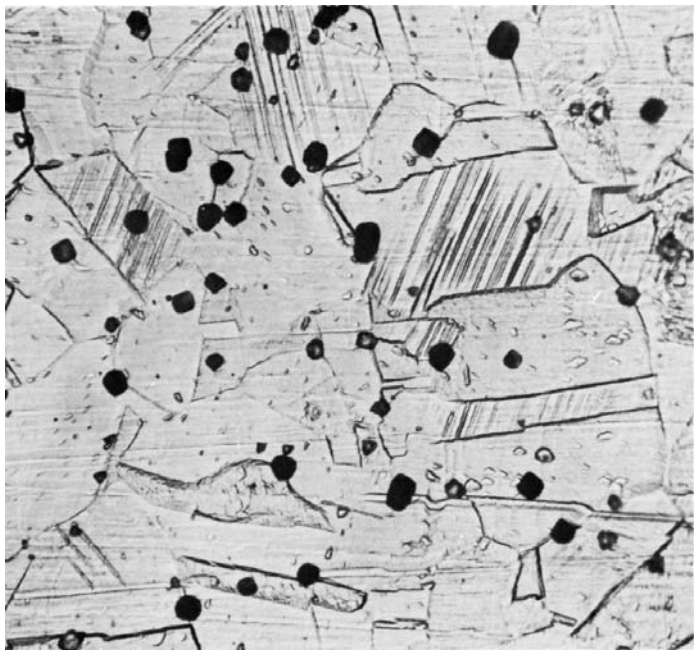


NOTE 1—To differentiate between the types of pits, use a magnification of 500× and focus in the plane of etched surface. The pits which now appear completely black are end grain pits.

FIG. 6 End Grain Pitting I (500×) (A Few Deep End Grain Pits (See 1 in Figure) and Shallow Etch Pits (2))

are entirely absent. However, any susceptibility to intergranular attack is readily detected by pronounced ditches.

11.4.2 Some wrought specimens, especially from bar stock, may contain a random pattern of pits. If these pits are sharp and so deep that they appear black (Fig. 7) it is possible that the specimen may be susceptible to end grain attack in nitric acid only. Therefore, even though the grain boundaries all have step



NOTE 1—This or a greater concentration of end grain pits at 500× (using standard etching conditions) indicates that the specimen must be tested when screening is for nitric acid test.

FIG. 7 End Grain Pitting II (500×)

structures, specimens having as much or more end grain pitting than that shown in Fig. 7 cannot be safely assumed to have low nitric acid rates and should be subjected to the nitric acid test whenever it is specified. Such sharp, deep pits should not be confused with the shallow pits shown in Figs. 1 and 6.

12. Use of Etch Structure Classifications

12.1 The use of these classifications depends on the hot acid corrosion test for which stainless steel specimens are being screened by etching in oxalic acid and is described in each of the practices.

13. Precision and Bias

13.1 *Precision and Bias*—No information is presented about either the precision or bias of Practice A—Oxalic Acid Etch Test for classification of Etch Structures of Austenitic Stainless Steels since the test result is nonquantitative.

PRACTICE B—FERRIC SULFATE–SULFURIC ACID TEST FOR DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEELS (2)

14. Scope

14.1 This practice describes the procedure for conducting the boiling 120-h ferric sulfate–50 % sulfuric acid test which measures the susceptibility of austenitic stainless steels to intergranular attack.

14.2 The presence or absence of intergranular attack in this test is not necessarily a measure of the performance of the material in other corrosive environments. The test does not provide a basis for predicting resistance to forms of corrosion

other than intergranular, such as general corrosion, pitting, or stress-corrosion cracking.

15. Summary of the Ferric Sulfate-Sulfuric Acid Practice B

15.1 A specimen representative of the material to be evaluated is immersed in a boiling solution of ferric sulfate and sulfuric acid for a specified time. The resulting mass loss is converted to a corrosion rate, which is compared to a specified maximum value to determine whether the material has the resistance to attack expected of the grade of material being tested.

16. Significance and Use

16.1 The ferric sulfate-sulfuric acid test detects susceptibility to intergranular attack associated primarily with chromium carbide precipitate in unstabilized austenitic stainless steels, and to intergranular attack associated with sigma phase.

16.2 The corrosion potential of the ferric sulfate-sulfuric acid test has been reported as 0.6 V versus a standard calomel electrode (SCE), as compared with 0.75 to 1.0 V for Practice C, and 0.1 V for Practices E and F. (**3**)

NOTE 3—A higher corrosion potential indicates more severely oxidizing conditions.

17. Rapid Screening Test

17.1 Before testing in the ferric sulfate-sulfuric acid test, specimens of certain grades of stainless steels (see Table 1) may be given a rapid screening test in accordance with procedures given in Practice A, Oxalic Acid Etch Test for Classification of Etch Structures of Austenitic Stainless Steels. Preparation, etching, and the classification of etch structures are described therein. The use of etch structure evaluations in connection with the ferric sulfate-sulfuric acid test is specified in Table 1.

17.2 Heat treat the material in accordance with 22.1 prior to performing the etch test.

TABLE 1 Use of Etch Structure Classifications from the Oxalic Acid Etch Test with Ferric Sulfate-Sulfuric Acid Test[A]

Grade	Acceptable Etch Structures	Suspect Etch Structures[B]
304	Step, dual, end grain, I & II	Ditch
304L	Step, dual, end grain, I & II	Ditch
316	Step, dual, end grain, I & II	Ditch
316L	Step, dual, end grain, I & II	Ditch
317	Step, dual, end grain, I & II	Ditch
317L	Step, dual, end grain, I & II	Ditch
CF-3	Step, dual, isolated ferrite pools	Ditch, interdendritic ditches
CF-8	Step, dual, isolated ferrite pools	Ditch, interdendritic ditches
CF-3M	Step, dual, isolated ferrite pools	Ditch, interdendritic ditches
CF-8M	Step, dual, isolated ferrite pools	Ditch, interdendritic ditches

[A] Grades not listed in this table either have not been evaluated for use of Practice A with Practice B or have been found to give acceptable results in the etch test while giving unacceptable results in Practice B. In the latter case Practices A would pass material that should have been subjected to the ferric sulfate-sulfuric acid test.
[B] Specimens having these structures shall be tested in the ferric sulfate-sulfuric acid test.

17.3 Ignore "process-affected" areas (see Section 21); application of the ferric sulfate-sulfuric acid test to process-affected areas is currently outside the scope of Practice B.

17.4 Corrosion test specimens having acceptable etch structures in the Oxalic Acid Etch Test will be essentially free of intergranular attack in the ferric sulfate-sulfuric acid test. Such specimens are acceptable without testing in the ferric sulfate-sulfuric acid test. All specimens having suspect etch structures shall be tested in the ferric sulfate-sulfuric acid test.

18. Apparatus

18.1 The apparatus is illustrated in Fig. 8.

NOTE 4—Other ground glass joints, such as the 45/40 joint may also be used.

18.1.1 An *Allihn condenser* with a minimum of four bulbs and with a ground glass joint to match that of the flask.

18.1.1.1 Substitutions for this condenser or flask are not allowed. Specifically, the cold-finger type of condenser with standard Erlenmeyer flasks shall not be used. Corrosion rates obtained using the cold-finger type of condenser are lower than those obtained using the Allihn type of condenser whether due to loss of vapor or to higher oxygen content in the solution or both. Such lower corrosion rates lead to acceptance of material that should be rejected.

18.1.2 A 1-L Erlenmeyer flask with a ground glass joint to match that of the condenser. The flask opening limits the size of the specimen; a larger opening is desirable.

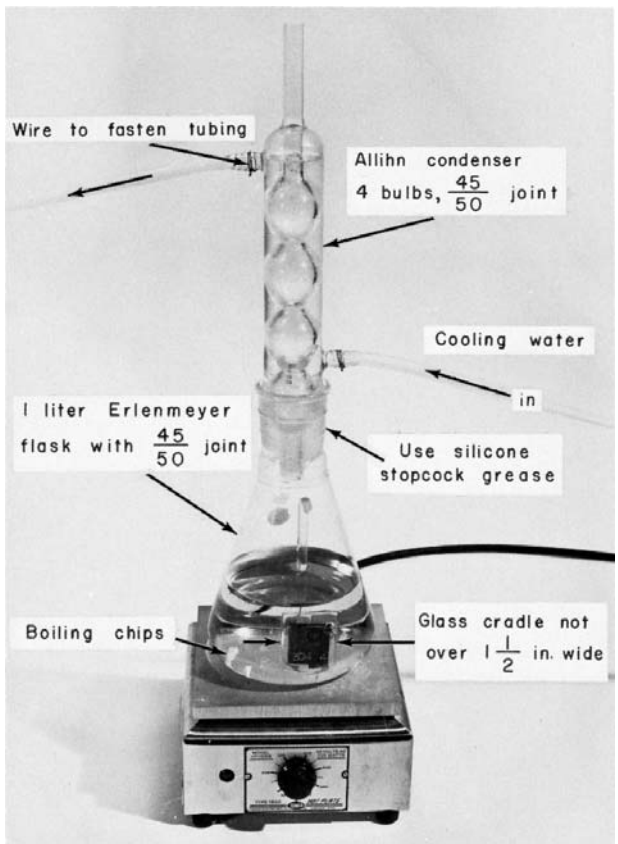


FIG. 8 Apparatus for Ferric Sulfate-Sulfuric Acid Test

18.1.3 *Glass cradle* (Note 5)—Can be supplied by a glass-blowing shop. It must be sized so as to fit, with the specimen, through the flask opening. It must be designed to allow free flow of the testing solution around the specimen.

NOTE 5—Other equivalent means of specimen support, such as glass hooks or stirrups, may also be used.

18.1.4 *Boiling Chips*—Used to prevent bumping.

18.1.5 *High Vacuum Silicone Grease*—For the ground glass joint.

18.1.6 *Hot plate,* capable of providing heat for continuous boiling of the solution.

18.1.7 An *analytical balance* capable of weighing to the nearest 0.001 g.

NOTE 6—During testing, there is some deposition of iron oxides on the upper part of the Erlenmeyer flask. This can be readily removed, after test completion, by boiling a solution of 10 % hydrochloric acid in the flask.

18.1.8 *Desiccator*—For storage of prepared specimens prior to testing.

19. Reagents and Materials

19.1 *Ferric Sulfate Hydrate* ($Fe_2(SO_4)_3 \cdot xH_2O$), about 75 % ($Fe_2(SO_4)_3$) by mass.

19.1.1 Ferric sulfate is a specific additive that establishes and controls the corrosion potential. Substitutions are not permitted.

19.2 *Sulfuric Acid* ($H_2(SO)_4$), 95.0 to 98.0 % by mass.

20. Ferric Sulfate-Sulfuric Acid Test Solution

20.1 Prepare 600 mL of 50 % (49.4 to 50.9 %) solution as follows:

20.1.1 (**Warning—**Protect the eyes and use rubber gloves for handling acid. Place the test flask under a hood.)

20.1.2 First, measure 400.0 mL of Type IV reagent water and pour into the Erlenmeyer flask.

20.1.3 Then measure 236.0 mL of reagent-grade sulfuric acid. Add the acid slowly and with constant stirring to the water in the Erlenmeyer flask to avoid boiling by the heat evolved.

NOTE 7—Loss of vapor results in concentration of the acid.

20.1.4 Weigh 25 g of reagent-grade ferric sulfate to the nearest 0.1 g and add to the sulfuric acid solution.

20.1.5 Drop boiling chips into the flask.

20.1.6 Lubricate ground glass joint with silicone grease.

20.1.7 Cover flask with condenser and circulate cooling water.

20.1.8 Boil the solution until all ferric sulfate is dissolved (see Note 7).

20.1.9 (**Warning—**It has been reported that violent boiling resulting in acid spills can occur. It is important to ensure that the concentration of acid does not increase and that an adequate number of boiling chips (which are resistant to attack by the test solution) are present.)

21. Sampling

21.1 Obtain and prepare only base metal samples.

21.1.1 There are two classes of specimens to be considered: base metal, and process-affected metal. Process-affected metal contains any condition that affects the corrosion properties of the material in a non-uniform way, such as (but not limited to) welds; carburized. nitrided, or oxidized surfaces; mechanical deformation; and areas affected by heat. Base metal has none of these conditions.

21.1.2 The Practice B test involves immersing the entire specimen and averaging the mass loss over the entire surface of the specimen. Welding, carburization, mechanical deformation, and the like, affect only part of a specimen.

21.1.3 The mass loss rate from process-affected metal is expected to differ from that from base metal; the presence of process-affected metal in a specimen will affect the calculated test result in an unpredictable way.

21.1.4 If the presence of these or other localized conditions is a concern to the purchaser, then tests that do not average the mass loss over the total specimen surface area, such as Practice A, the Oxalic Acid Etch Test, or Practice E, the Copper–Copper Sulfate–16% Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels, should be considered. Details of the test and acceptance criteria shall be as agreed by the purchaser and producer.

21.2 Unless otherwise specified by the purchaser, the procedures for obtaining representative base metal samples, for removing the specimens from the samples, and the number of specimens shall be at the discretion of the producer.

22. Preparation of Test Specimens

22.1 Heat treat extra-low carbon and stabilized grades at 650 to 675°C (1200 to 1250°F), which is the range of maximum carbide precipitation, prior to testing. The length of time of heating, and the method of subsequent cooling used for this sensitizing treatment together with the corresponding maximum permissible corrosion rate shall be as agreed between the material producer and purchaser.

NOTE 8—The most commonly used sensitizing treatment is 1 h at 675°C (1250°F).

22.2 Prepare the specimens, each having a total surface area of 5 to 20 cm^2.

22.3 Where feasible for the product form, grind all the specimen surfaces using CAMI/ANSI 120 [FEPA/ISO P120] paper-backed, wet or dry, closed coated abrasive paper, with water as a coolant. If abrasive paper is used dry, polish slowly to avoid overheating. Do not use abrasives with grinding aids; some grinding aids contain fluorides that can affect the measured corrosion rate.

22.4 Remove all traces of oxide scale and heat tint formed during heat treatments. Any scale that cannot be removed by grinding (for example, in stamped numbers) may be removed by using one of the pickling solutions described in Practice A380/A380M, Table A1.1. (Residual oxide scale causes galvanic action and consequent activation in the test solution.)

22.5 Measure the specimens, including the inner surfaces of any holes, to the nearest 0.05 mm (0.001 in.) and calculate the total exposed area.

22.6 Degrease the specimens using suitable nonchlorinated agents, such as soap and lukewarm water, or acetone. Dry the

specimens and weigh each one to the nearest 0.001 g. Store the specimens in a desiccator until the test is to be performed.

23. Procedure

23.1 If the test solution is not already boiling, bring it to boiling.

23.1.1 Keep the flask covered with the condenser (with cooling water flowing) except when inserting or removing specimens. (See Note 7.)

23.2 Turn off the heat source and allow the boiling to subside.

23.3 Place specimens in glass cradles.

23.4 Uncover the flask.

23.5 Insert the specimens.

23.6 Replace the condenser immediately, restore cooling water flow, and turn on the heat source.

23.7 Mark the liquid level on the flask to provide a check on vapor loss, which would result in concentration of the acid. If there is an appreciable change in the level, repeat the test with fresh solution and reground and reweighed specimens.

23.8 Continue the immersion of the specimens for a total of 120 h (five days), then remove the specimens, rinse in water or acetone, and dry.

23.9 Weigh the specimens and subtract the new weights from original weights.

23.10 Intermediate weighings are usually not necessary. The test can be run without interruption for 120 h. However, if preliminary results are desired, the specimens can be removed at any time for weighing.

23.11 Changes to the solution during the 120-h test periods are not necessary.

23.12 If the corrosion rate is extraordinarily high, as evidenced by a change in the color (from yellow to green) of the solution, additional ferric sulfate inhibitor may need to be added during the test. If the total weight loss of all the specimens in a flask exceeds 2 g, more ferric sulfate must be added. (During the test, ferric sulfate is consumed at a rate of 10 g for each 1 g of dissolved stainless steel.)

23.13 Several specimens may be tested simultaneously. The number (3 or 4) is limited only by the number of glass cradles that can be fitted into the flask.

24. Calculation and Report

24.1 The effect of the acid solution on the material is measured by determining the loss of weight of the specimen. The corrosion rates should be reported as millimetres of penetration per month (Note 9), calculated as follows:

$$\text{Millimetre per month} = (7305 \times W)/(A \times t \times d) \quad (1)$$

where:

t = time of exposure, h,
A = area, cm^2,
W = weight loss, g, and
d = density, g/cm^3

for chromium-nickel steels, d = 7.9 g/cm^3
for chromium-nickel-molybdenum steels, d = 8.00 g/cm^3

NOTE 9—Conversion factors to other commonly used units for corrosion rates are as follows:
Millimetres per month × 0.04 = inches per month
Millimetres per month × 0.47 = inches per year
Millimetres per month × 12 = millimetres per year
Millimetres per month × 472 = mils per year
Millimetres per month × 1000 × density/3 = milligrams per square decimetre per day
Millimetres per month × 1.39 × density = grams per square metre per hour

25. Precision and Bias

25.1 *Precision*—The precision of Practice B is being determined.

25.2 *Bias*—This practice has no bias because the resistance to intergranular corrosion is defined only in terms of this practice.

PRACTICE C—NITRIC ACID TEST FOR DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEELS

26. Scope

26.1 This practice describes the procedure for conducting the boiling nitric acid test (**2**) as employed to measure the relative susceptibility of austenitic stainless steels to intergranular attack.

26.2 The presence or absence of intergranular attack in this test is not necessarily a measure of the performance of the material in other corrosive environments; in particular, it does not provide a basis for predicting resistance to forms of corrosion other than intergranular, such as general corrosion, pitting, or stress-corrosion cracking.

27. Summary of Test Method C, the Nitric Acid Test

27.1 A specimen representative of the material to be evaluated is immersed in a boiling solution of nitric acid for a specified time. The resulting mass loss is converted to a corrosion rate, which is compared to a specified maximum value to determine whether the material has the resistance to attack expected of the grade of material being tested.

28. Significance and Use

28.1 The nitric acid test detects susceptibility to rapid intergranular attack associated with chromium carbide precipitate

28.2 The corrosion potential of the nitric acid test (Practice C) has been reported as 0.75 to 1.0 V versus a standard calomel electrode as compared with 0.6 V for Practice B, and 0.1 V for Practices E and F. (**3**)

NOTE 10—Higher corrosion potential indicates more severely oxidizing conditions. The high corrosion potential of the nitric acid test suggests that it should be invoked only when the material is destined for nitric acid service.

29. Rapid Screening Test

29.1 Before testing in the nitric acid test, specimens of certain grades of stainless steel, as given in Table 2, may be

TABLE 2 Use of Etch Structure Classification from Oxalic Acid Etch Test with Nitric Acid Test[A]

Grade	Acceptable Etch Structures	Suspect Etch Structures[B]
AISI 304	Step, dual, end grain I	Ditch, end grain II
AISI 304L	Step, dual, end grain I	Ditch, end grain II
ACI CF-8	Step, dual, isolated ferrite pools	Ditch, interdendritic ditches
ACI CF-3	Step, dual, isolated ferrite pools	Ditch, interdendritic ditches

[A] Grades not listed in this table either have not been evaluated for use of Practice A with Practice B or have been found to give acceptable results in the etch test while giving unacceptable results in Practice B. In the latter case Practice A would pass material that should have been subjected to the ferric sulfate-sulfuric acid test.
[B] Specimens having these structures shall be tested in the nitric acid test.

given a rapid screening test in accordance with procedures given in Practice A, Oxalic Acid Etch Test for Classification of Etch Structures of Austenitic Stainless Steels. The use of the etch structure evaluations in connection with the nitric acid test is specified in Table 2.

29.2 Heat treat the material in accordance with 33.1 prior to performing the etch test.

29.3 Ignore "process-affected" areas, if any (see Section 32); application of the nitric acid test to process-affected areas is currently outside the scope of Practice C.

29.4 Corrosion test specimens having acceptable etch structures in the Oxalic Acid Etch Test will be essentially free of intergranular attack in the nitric acid test; such specimens are acceptable without testing in the nitric acid test. All specimens having suspect etch structures shall be tested in the nitric acid test.

30. Apparatus

30.1 *Container*—A 1-L Erlenmeyer flask equipped with a cold finger-type condenser, as illustrated in Fig. 9.

30.2 *Specimen Supports*—Glass hooks, stirrups, or cradles for supporting the specimens in the flask fully immersed at all times during the test and so designed that specimens tested in the same container do not come in contact with each other.

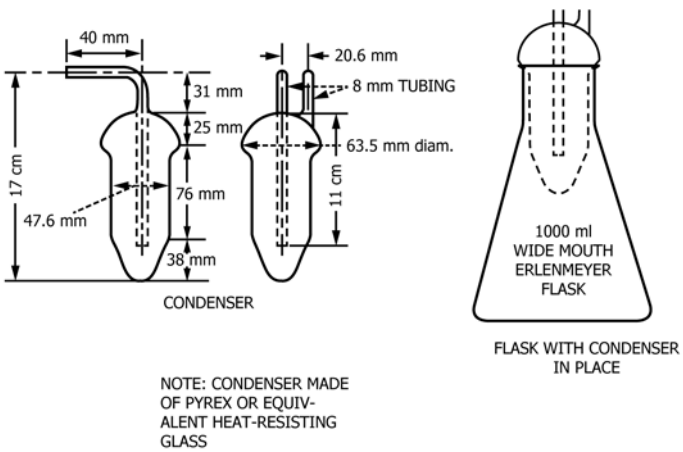


FIG. 9 Flask and Condenser for Nitric Acid Test

30.3 *Heater*—A means for heating the test solutions and of keeping them boiling throughout the test period. An electrically heated hot plate is satisfactory for this purpose.

30.4 *Balance*—An analytical balance capable of weighing to at least the nearest 0.001 g.

30.5 *Desiccator*—For storage of prepared specimens prior to testing.

31. Nitric Acid Test Solution

31.1 The test solution shall be 65.0 ± 0.2 weight % as nitric acid determined by analysis.

31.2 Prepare this solution by adding reagent grade nitric acid (HNO_3 Table 3) to reagent water at the rate of 108 mL of reagent water per litre of reagent nitric acid.

31.3 (**Warning—**Protect the eyes and use rubber gloves for handling acid. Place the test flask under a hood.)

31.4 The nitric acid used shall conform to the American Chemical Society Specifications for Reagent Chemicals and the additional requirements of this test method as shown in Table 3.

32. Sampling

32.1 Obtain and prepare only base metal samples.

32.1.1 There are two classes of specimens to be considered: base metal, and process-affected metal. Process-affected metal contains any condition that affects the corrosion properties of the material in a non-uniform way, such as (but not limited to) welds; carburized. nitrided, or oxidized surfaces; mechanical deformation; and areas affected by heat. Base metal has none of these conditions.

32.1.2 The Practice C test involves immersing the entire specimen and averaging the mass loss over the entire surface of the specimen. Welding, carburization, mechanical deformation, and the like, affect only part of a specimen.

32.1.3 The mass loss rate from process-affected metal is expected to differ from that from base metal; the presence of process-affected metal in a specimen will affect the calculated test result in an unpredictable way.

32.1.4 If the presence of these or other localized conditions is a concern to the purchaser, then tests that do not average the mass loss over the total specimen surface area, such as Practice A, the Oxalic Acid Etch Test, or Practice E, the Copper–Copper Sulfate–Sulfuric Acid Test for Detecting Susceptibility to

TABLE 3 Nitric Acid Composition Limits

	Minimum	Maximum
Nitric Acid (HNO_3), mass percent	69.0	71.0
Ash, ppm	...	5
Chloride as Cl, ppm	...	0.5
Sulfate, as (SO_4), ppm	...	1
Arsenic (As), ppm	...	0.01
Heavy metals, as Pb, ppm	...	0.2
Iron, (Fe), ppm	...	0.2
Additional limits per Practices A262		
Fluorine (F), ppm	...	1
Phosphate (PO_4), ppm	...	0.2

Intergranular Attack in Austenitic Stainless Steels, should be considered. Details of the test and acceptance criteria shall be as agreed by the purchaser and producer.

32.2 Unless otherwise specified by the purchaser, the procedures for obtaining representative base metal samples, for removing the specimens from the samples, and the number of specimens shall be at the discretion of the producer.

32.3 When specimens are cut by shearing, the sheared edges shall be refinished by machining or grinding prior to testing.

33. Preparation of Test Specimens

33.1 Heat treat extra-low carbon and stabilized grades at 650 to 675°C (1200 to 1250°F), which is the range of maximum carbide precipitation, prior to testing. The length of time of heating, and the method of subsequent cooling used for this sensitizing treatment together with the corresponding maximum permissible corrosion rate shall be as agreed between the material producer and purchaser.

NOTE 11—The most commonly used sensitizing treatment is 1 h at 675°C (1250°F).

NOTE 12—The size and shape of the specimen must be considered with respect to available facilities for accurate weighing and the volume of test solution to be used. Normally, the maximum convenient weight of a specimen is about 100 g. In the case of bar, wire, and tubular products, the proportion of the total area represented by the exposed cross section may influence the results. Cross-sectional areas in these products may be subject to end grain attack in nitric acid. The proportion of end grain in the specimen should therefore be kept low unless such surface is actually to be exposed in service involving nitric acid. In this latter case, the proportion of end grain in the specimen should be kept high.

33.2 Where feasible for the product form, grind all the specimen surfaces using CAMI/ANSI 120 [FEPA/ISO P120] paper-backed, wet or dry, closed coated abrasive paper, with water as a coolant. If abrasive paper is used dry, polish slowly to avoid overheating. Do not use abrasives with grinding aids; some grinding aids contain fluorides that can affect the measured corrosion rate.

33.3 Remove all traces of oxide scale and heat tint formed during heat treatments. Any scale that cannot be removed by grinding (for example, in stamped numbers) may be removed by using one of the pickling solutions described in Practice A380/A380M, Table A1.1.

33.4 Measure the specimen, including the inner surfaces of any holes to the nearest 0.05 mm (0.001 in.), and calculate the total exposed area in cm^2.

33.5 Degrease the specimen using suitable nonchlorinated agents, such as soap and lukewarm water, or acetone (Note 13). Dry the specimens and weigh each one to the nearest 0.001 g. Store the specimens in a desiccator until the test is to be performed.

NOTE 13—The cleaning treatment described may be supplemented by immersing the specimen in nitric acid (for example, 20 weight % at 49 to 60°C (120 to 140°F)) for 20 min, followed by rinsing, drying, and weighing. In the case of small-diameter tubular specimens which cannot be conveniently resurfaced on the inside, it is desirable to include in the preparation an immersion in boiling nitric acid (65 %) for 2 to 4 h using the same apparatus as for the actual test. The purpose of these treatments is to remove any surface contamination that may not be accomplished by the regular cleaning method and which may increase the apparent weight loss of the specimen during the early part of the test.

33.6 The standard test is to test only one specimen of each material or lot of material. However, in case of dispute, the use of at least two specimens for check purposes is recommended.

34. Procedure

34.1 Use a sufficient quantity of the nitric acid test solution to cover the specimens and to provide a volume of at least 20 mL/cm^2 (125 $mL/in.^2$) of specimen surface. Normally, a volume of about 600 mL is used.

34.2 Use a separate container for each test specimen.

34.2.1 As many as three specimens may be tested in the same container provided that they all are of the same grade and all show satisfactory resistance to corrosion.

34.2.2 If more than one of the specimens tested in the same container fail to pass the test, retest all the specimens in separate containers.

NOTE 14—Excessive corrosion of one specimen may result in accelerated corrosion of the other specimens tested with it. Excessive corrosion may often be detected by changes in the color of the test solution, and it may be appropriate to provide separate containers for such specimens without waiting until the end of the test period. A record should be made showing which specimens were tested together.

34.3 After the specimens have been placed in the acid in the container, pass cooling water through the condenser, bring the acid to a boil on the hot plate, and keep boiling throughout the test period (Note 15). After each test period, rinse the specimens with water and treat by scrubbing with rubber or a nylon brush under running water to remove any adhering corrosion products, then dry and weigh them. Drying may be facilitated, if desired, by dipping the specimens in acetone after they are scrubbed.

34.4 (**Warning—**It has been reported that violent boiling resulting in acid spills can occur. It is important to ensure that the concentration of acid does not increase and that an adequate number of boiling chips (which are resistant to attack by the test solution) are present.)

NOTE 15—Take care to prevent contamination of the testing solution, especially by fluorides, either before or during the test. Experience has shown that the presence of even small amounts of hydrofluoric acid will increase the corrosion rate in the nitric acid test. It is not permissible, for example, to conduct nitric-hydrofluoric acid tests in the same hood with nitric acid tests.

34.5 The standard test consists of five boiling periods of 48 h each with a fresh test solution being used in each period.

34.5.1 A combination of one 48-h period and two 96-h periods (not necessarily in that order) instead of five 48-h test periods may be used if so agreed by the purchaser.

35. Calculation and Report

35.1 *Calculation*—The effect of the acid on the material shall be measured by determining the loss of weight of the specimen after each test period and for the total of the test periods. Using Eq 1, calculate the corrosion rate for each specimen for each test period, and for the total of the test periods.

35.2 *Report*—Report the calculated corrosion rates for the individual periods in chronological order, as well as the

average for the five test periods. If the modified test periods (34.5.1) are used, then identify each result as to the sequence and length of the test period.

36. Precision and Bias

36.1 *Precision*—The precision of Practice C is being determined.

36.2 *Bias*—This practice has no bias because the resistance to intergranular corrosion is defined only in terms of this practice.

PRACTICE E—COPPER-COPPER SULFATE–16 % SULFURIC ACID TEST FOR DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEELS (4, 5)

37. Scope

37.1 This practice describes the procedure by which the copper–copper sulfate–16 % sulfuric acid test is conducted to determine the susceptibility of austenitic stainless steels to intergranular attack. The presence or absence of intergranular corrosion in this test is not necessarily a measure of the performance of the material in other corrosive media. The test does not provide a basis for predicting resistance to other forms of corrosion, such as general corrosion, pitting, or stress-corrosion cracking.

38. Rapid Screening Test

38.1 Before testing in the copper–copper sulfate–16 % sulfuric acid test, specimens of certain grades of stainless steel (see Table 4) may be given a rapid screening test in accordance with the procedures given in Practice A (Sections 4 through 13). Preparation, etching, and the classification of etch structures are described therein. The use of etch-structure evaluations in connection with the copper–copper sulfate–16 % sulfuric acid test is specified in Table 4.

38.1.1 Corrosion test specimens having acceptable etch structures in the Oxalic Acid Etch Test will be essentially free of intergranular attack in the copper–copper sulfate–16 % sulfuric acid test. Such specimens are acceptable without testing in the copper–copper sulfate–16 % sulfuric acid test. All specimens having suspect etch structures must be tested in the copper–copper sulfate–16 % sulfuric acid test.

38.1.2 Heat treat the material when required by and in accordance with 43.3.1 prior to performing the etch test.

TABLE 4 Use of Etch Structure Classifications from the Oxalic Acid Etch Test with the Copper–Copper Sulfate–16 % Sulfuric Acid Test

Grade	Acceptable Etch Structures	Suspect Etch Structures [A]
AISI 201	Step, dual, end grain I and II	Ditch
AISI 202	Step, dual, end grain I and II	Ditch
AISI 301	Step, dual, end grain I and II	Ditch
AISI 304	Step, dual, end grain I and II	Ditch
AISI 304L	Step, dual, end grain I and II	Ditch
AISI 304H	Step, dual, end grain I and II	Ditch
AISI 316	Step, dual, end grain I and II	Ditch
AISI 316L	Step, dual, end grain I and II	Ditch
AISI 316H	Step, dual, end grain I and II	Ditch
AISI 317	Step, dual, end grain I and II	Ditch
AISI 317L	Step, dual, end grain I and II	Ditch
AISI 321	Step, dual, end grain I and II	Ditch
AISI 347	Step, dual, end grain I and II	Ditch

[A] Specimens having these structures must be tested in the copper–copper sulfate–16 % sulfuric acid test.

39. Summary of Practice

39.1 A suitable sample of an austenitic stainless steel, embedded in copper shot or grindings, is exposed to boiling acidified copper sulfate solution for 15 h. After exposure in the boiling solution, the specimen is bent. Intergranular cracking or crazing is evidence of susceptibility.

39.2 *Alternative Testing Procedures:*

39.2.1 Unless prohibited by the purchaser in the purchase order, the supplier is permitted to meet the requirements of Practice E by performing a test in accordance with ISO 3651–2, Method A, provided that the testing period shall be a minimum of 15 h. When a sensitization treatment is required, sensitization heat treatment T1 [700°C ± 10°C (1292°F ± 18°F), 30 min, water quench] shall be used unless the supplier and purchaser shall agree upon preparation of welded test pieces to be tested in the as-welded condition.

39.2.2 When this alternative test procedure is used, it shall be noted on the test report.

40. Apparatus

40.1 The basic apparatus is described in Section 18.

40.2 *Specimen Supports*—An open glass cradle capable of supporting the specimens and copper shot or grindings in the flask is recommended.

NOTE 16—It may be necessary to embed large specimens, such as from heavy bar stock, in copper shot on the bottom of the test flask. A copper cradle may also be used.

40.3 *Heat Source*—Any gas or electrically heated hot plate may be utilized for heating the test solution and keeping it boiling throughout the test period.

41. Acidified Copper Sulfate Test Solution

41.1 Dissolve 100 g of reagent grade 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 dilute to 1000 mL with distilled water.

NOTE 17—The solution will contain approximately 6 weight % of anhydrous $CuSO_4$ and 16 weight % of H_2SO_4.

42. Copper Addition

42.1 Electrolytic grade copper shot or grindings may be used. Shot is preferred for its ease of handling before and after the test.

42.2 A sufficient quantity of copper shot or grindings is to be used to cover all surfaces of the specimen whether it is in a vented glass cradle or embedded in a layer of copper shot on the bottom of the test flask.

42.3 The amount of copper used, assuming an excess of metallic copper is present, is not critical. The effective galvanic coupling between copper and the test specimen may have importance (6).

42.4 The copper shot or grindings may be reused if they are cleaned in warm tap water after each test.

43. Specimen Preparation

43.1 The size of the sample submitted for test and the area from which it is to be taken (end or middle of coil, midway surface and center, and so forth) is generally specified in the agreement between the purchaser and the seller. The testing apparatus dictates the final size and shape of the test specimen. The specimen configuration should permit easy entrance and removal through the neck of the test container.

43.1.1 Table 5 may be used as a guide to determine acceptable specimen sizes. There may be restrictions placed on specimen size by the testing apparatus.

43.1.2 Specimens obtained by shearing should have the sheared edges machined or ground off prior to testing. Care should be taken when grinding to avoid overheating or "burning." A "squared" edge is desirable.

43.2 Any scale on the specimens should be removed mechanically unless a particular surface finish is to be evaluated. Chemical removal of scale is permissible when this is the case. Mechanical removal of scale should be accomplished with 120-grit iron-free aluminum oxide abrasive.

43.2.1 Each specimen should be degreased using a cleaning solvent such as acetone, alcohol, ether, or a vapor degreaser prior to being tested.

43.3 All austenitic material in the "as-received" (mill-annealed) condition should be capable of meeting this test.

43.3.1 Specimens of extra-low-carbon and stabilized grades are tested after sensitizing heat treatments at 650 to 675°C (1200 to 1250°F), which is the range of maximum carbide precipitation. The most commonly used sensitizing treatment is 1 h at 675°C. Care should be taken to avoid carburizing or nitriding the specimens. The heat treating is best carried out in air or neutral salt.

NOTE 18—The sensitizing treatment 675°C is performed to check the effectiveness of stabilized and 0.03 % maximum carbon materials in resisting carbide precipitation, hence, intergranular attack.

TABLE 5 Sizes of Test Specimens

Type of Material	Size of Test Specimen
Wrought wire or rod:	
Up to 6 mm (¼ in.) in diameter, incl	Full diameter by 75 mm (3 in.) (min) long
Over 6 mm (¼ in.) in diameter	Cylindrical segment 6 mm (¼ in.) thick by 25 mm (1 in.) (max) wide by 75 to 125 mm (3 to 5 in.) long [A]
Wrought sheet, strip, plates, or flat rolled products:	
Up to 5 mm (3/16 in.) thick, incl	Full thickness by 9 to 25 mm (⅛ to 1 in.) wide by 75 mm (3 in.) (min) long
Over 5 mm (3/16 in.) thick	5 to 13 mm (3/16 to ½ in.) thick by 9 to 25 mm (⅛ to 1 in.) wide by 75 mm (3 in.) (min) long [B]
Tubing:	
Up to 38 mm (1½ in.) in diameter, incl	Full ring, 25 mm (1 in.) wide [C]
Over 38 mm (1½ in.) in diameter	A circumferential segment 75 mm (3 in.) (min) long cut from a 25 mm (1-in.) wide ring [D]

[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. Cold-rolled strip or sheets may be tested in the thickness supplied.
[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 38 mm (1½ in.) in diameter shall be taken with the weld on the axis of the bend.

44. Test Conditions

44.1 The volume of acidified copper sulfate test solution used should be sufficient to completely immerse the specimens and provide a minimum of 8 mL/cm^2 (50 mL/in.^{2}) of specimen surface area.

44.1.1 As many as three specimens can be tested in the same container. It is ideal to have all the specimens in one flask to be of the same grade, but it is not absolutely necessary. The solution volume-to-sample area ratio is to be maintained.

44.1.2 The test specimen(s) should be immersed in ambient test solution, which is then brought to a boil and maintained boiling throughout the test period. Begin timing the test period when the solution reaches the boiling point.

NOTE 19—Measures should be taken to minimize bumping of the solution when glass cradles are used to support specimens. A small amount of copper shot (eight to ten pieces) on the bottom of the flask will conveniently serve this purpose.

44.1.3 The time of the test shall be a minimum of 15 h, unless a longer time is agreed upon between the purchaser and the producer. If not 15 h, the test time shall be specified on the test report. Fresh test solution would not be needed if the test were to run 48 or even 72 h. (If any adherent copper remains on the specimen, it may be removed by a brief immersion in concentrated nitric acid at room temperature.)

NOTE 20—Results in the literature indicate that this test is more sensitive if it is run for longer times (**3**, **7**).

45. Bend Test

45.1 The test specimen shall be bent through 180° and over a diameter equal to the thickness of the specimen being bent (see Fig. 10). In no case shall the specimen be bent over a

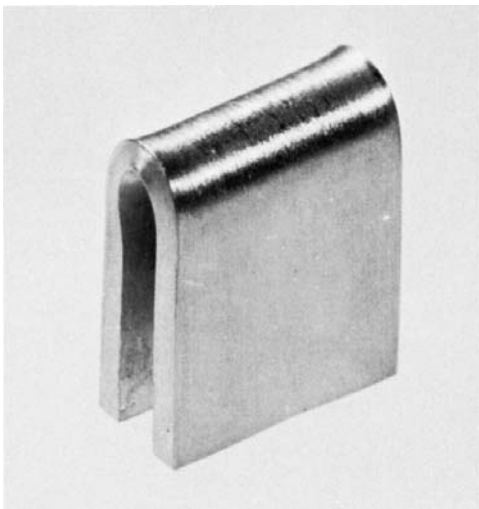


FIG. 10 A Bent Copper–Copper Sulfate–Sulfuric Acid Test Specimen

smaller radius or through a greater angle than that specified in the product specification. In cases of material having low ductility, such as severely cold worked material, a 180° bend may prove impractical. Users shall inform those conducting the Practice E test when the material is in the low ductility highly stressed condition, such as highly cold worked material. Determine the maximum angle of bend without causing cracks in such material by bending an untested specimen of the same configuration as the specimen to be tested. After exposure to the acidified copper–copper sulfate sulfuric acid test solution, the maximum angle of bend without causing cracks as determined from untested low ductility specimens shall be utilized in evaluation of the specimens exposed to the acidified copper–copper sulfate sulfuric acid test solution. The angle of bend utilized in evaluating tested specimens shall be reported.

45.1.1 Duplicate specimens shall be obtained from sheet material so that both sides of the rolled samples may be bent. This will assure detection of intergranular attack resulting from carburization of one surface of sheet material during the final stages of rolling.

NOTE 21—Identify the duplicate specimen in such a manner as to ensure both surfaces of the sheet material being tested are subjected to the tension side of the bends.

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

45.1.3 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. An air or hydraulic press may also be used for bending the specimens.

45.1.4 Tubular products should be flattened in accordance with the flattening test, prescribed in Test Methods and Definitions A370.

45.1.5 When agreed upon between the purchaser and the producer, the following shall apply to austenitic stainless steel plates 4.76 mm (0.1875 in.) and thicker:

45.1.5.1 Samples shall be prepared according to Table 5.

45.1.5.2 The radius of bend shall be two times the sample thickness, and the bend axis shall be perpendicular to the direction of rolling.

45.1.5.3 Welds on material 4.76 mm (0.1875 in.) and thicker shall have the above bend radius, and the weld-base metal interface shall be located approximately in the centerline of the bend.

45.1.5.4 Face, root, or side bend tests may be performed, and the type of bend test shall be agreed upon between the purchaser and the producer. The bend radius shall not be less than that required for mechanical testing in the appropriate material specification (for base metal) or in ASME Code Section IX (for welds).

46. Evaluation

46.1 The bent specimen shall be examined under low (5 to 20×) magnification (see Fig. 11). The appearance of fissures or

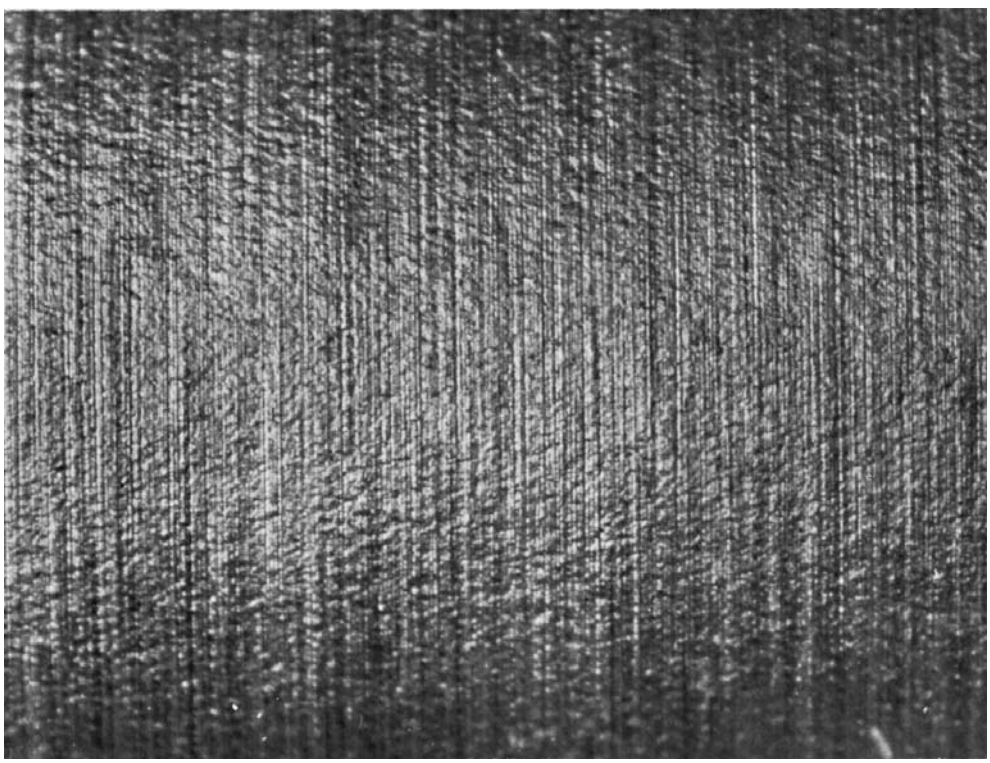


FIG. 11 Passing Test Specimen—View of the Bent Area (20× Magnification Before Reproduction)

cracks indicates the presence of intergranular attack (see Fig. 12).

46.1.1 When an evaluation is questionable (see Fig. 13), the presence or absence of intergranular attack shall be determined by the metallographic examination of the outer radius of a longitudinal section of the bend specimen at a magnification of 100 to 250×.

46.1.2 Cracking that originates at the edge of the specimen shall be disregarded. The appearance of deformation lines, wrinkles, or "orange peel" on the surface, without accompanying cracks or fissures, shall be disregarded also.

46.1.3 Cracks suspected as arising through poor ductility shall be investigated by bending a similar specimen that was not exposed to the boiling test solution. A visual comparison between these specimens should assist in interpretation.

PRACTICE F—COPPER-COPPER SULFATE–50 % SULFURIC ACID TEST FOR DETERMINING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEELS

47. Scope

47.1 This practice describes the procedure for conducting the boiling copper–copper sulfate–50 % sulfuric acid test, which measures the susceptibility of stainless steels to intergranular attack.

47.2 The presence or absence of intergranular attack in this test is not necessarily a measure of the performance of the material in other corrosive environments. The test does not provide a basis for predicting resistance to forms of corrosion other than intergranular, such as general corrosion, pitting, or stress-corrosion cracking.

48. Summary of Test Method F, the Copper–Copper Sulfate–50% Sulfuric Acid Test

48.1 A specimen representative of the material to be evaluated is immersed in a boiling solution of copper sulfate and sulfuric acid for a specified time. A piece of copper is also immersed in the solution to maintain a constant corrosion potential. The resulting mass loss is converted to a corrosion rate, which is compared to a specified maximum value to determine whether the material has the resistance to attack expected of the grade of material being tested.

49. Significance and Use

49.1 The copper–copper sulfate–sulfuric acid test detects susceptibility to intergranular attack associated primarily with chromium carbide precipitate in unstabilized cast austenitic stainless steels and in certain wrought grades.

49.2 The copper–copper sulfate–sulfuric acid test does not detect susceptibility to intergranular attack associated primarily with sigma phase.

49.3 The corrosion potential of the copper–copper sulfate–sulfuric acid test has been reported as 0.1 V as compared with 0.6 V for Practice B, 0.75 to 1.0 V for Practice C, and 0.1 V for Practice E. (3)

NOTE 22—Higher corrosion potential indicates more severely oxidizing conditions.

50. Rapid Screening Test

50.1 Before testing in the copper–copper sulfate–50 % sulfuric acid test, specimens of certain grades of stainless steels (see Table 6) may be given a rapid screening test in accordance with procedures given in Practice A, Oxalic Acid Etch Test for

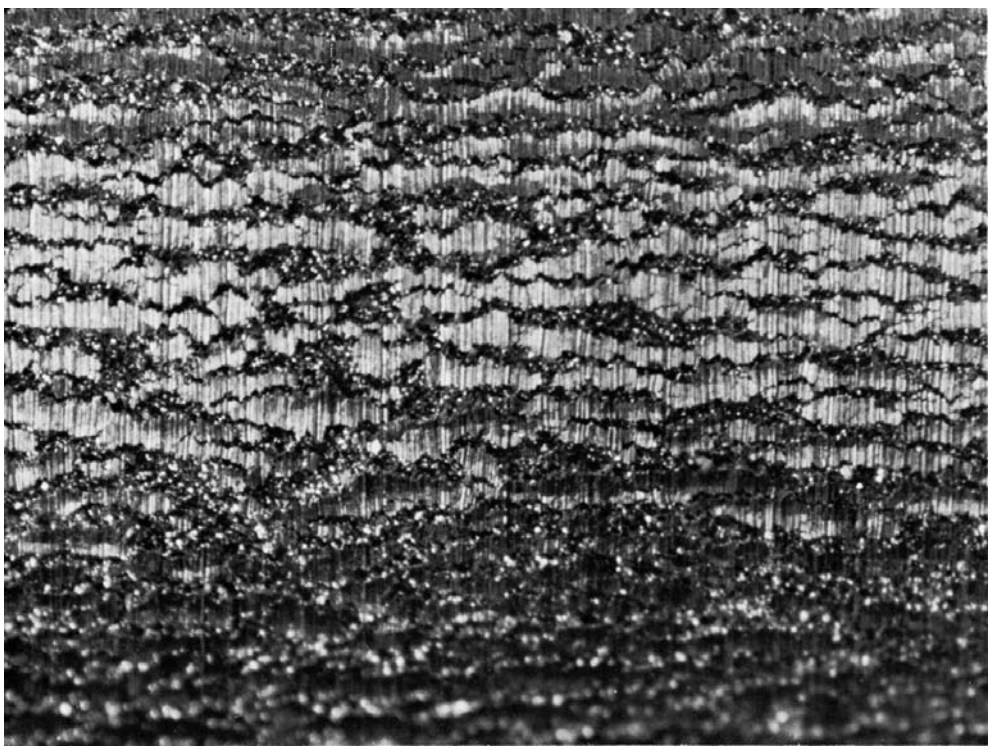


FIG. 12 Failing Test Specimen (Note the many intergranular fissures. Bent Area at 20× Magnification Before Reproduction.)

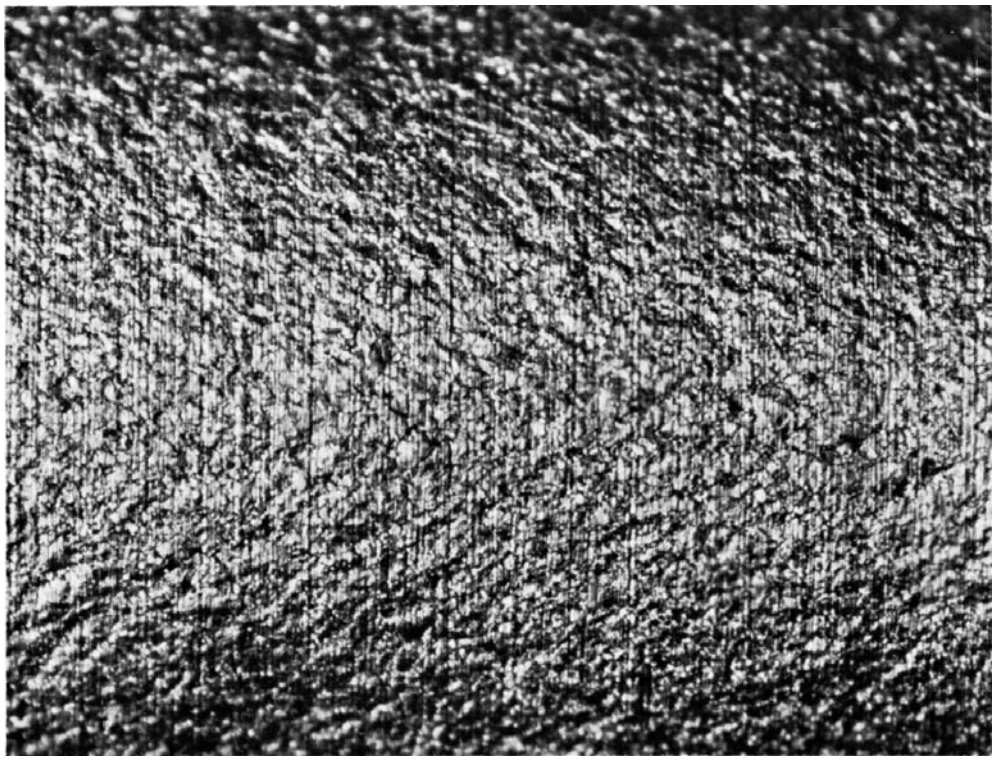


FIG. 13 Note the Traces of Intergranular Fissures and "Orange-Peel" Surface. Bent Area at 20× Magnification Before Reproduction.)

TABLE 6 Use of Etch Structure Classifications from the Oxalic Acid Etch Test With the Copper–Copper Sulfate–50 % Sulfuric Acid Test[A]

Grade	Acceptable Etch Structures	Suspect Etch Structures[B]
CF-3M	Step, dual, isolated ferrite	Ditch, interdendritic ditches
CF-8M	Step, dual, isolated ferrite	Ditch, interdendritic ditches

[A] Grades not listed in this table either have not been evaluated for use of Practice A with Practice F or have been found to give acceptable results in the etch test while giving unacceptable results in Practice F. In the latter case Practice A would pass material that should have been subjected to the copper–copper sulfate-sulfuric acid test.
[B] Specimens having these structures shall be tested in the copper–copper sulfate-sulfuric acid test

Classification of Etch Structures of Austenitic Stainless Steels. Preparation, etching, and the classification of etch structures are described therein. The use of etch structure evaluations in connection with the copper–copper sulfate–50 % sulfuric acid test is specified in Table 6.

50.2 Heat treat the material in accordance with 55.1 prior to performing the etch test.

50.3 Ignore "process-affected" areas (see 54.1.1); application of the etch test to these areas is currently outside the scope of Practice F.

50.4 Corrosion test specimens having acceptable etch structures in the Oxalic Acid Etch Test will be essentially free of intergranular attack in the copper–copper sulfate–50 % sulfuric acid test. Such specimens are acceptable without testing in the copper–copper sulfate–50 % sulfuric acid test. All specimens having suspect etch structures shall be tested in the copper–copper sulfate–50 % sulfuric acid test.

51. Apparatus

51.1 The basic apparatus is described in Section 18.

51.1.1 Substitutions for this condenser or flask are not allowed. Specifically, the cold-finger type of condenser with standard Erlenmeyer flasks shall not be used. Corrosion rates obtained using the cold-finger type of condenser are lower than those obtained using the Allihn type of condenser whether due to loss of vapor or to higher oxygen content in the solution or both.

52. Reagents and Materials

52.1 *Cupric Sulfate Pentahydrate* ($CuSO_4 \cdot 5H_2O$); about 64 % ($CuSO_4$) by mass.

52.1.1 Cupric sulfate is a specific additive that establishes and controls the corrosion potential. Substitutions are not permitted.

52.2 *Sulfuric Acid* (H_2SO_4), 95.0 to 98.0 % by mass.

52.3 A piece of copper metal about 3 by 20 by 40 mm (⅛ by ¾ by 1½ in.) with a bright, clean finish. An equivalent area of copper shot or chips may be used.

52.3.1 Wash, degrease, and dry the copper before use.

NOTE 23—A rinse in 5 % H_2SO_4 will clean corrosion products from the copper.

53. Copper–Copper Sulfate–50 % Sulfuric Acid Test Solution

53.1 Prepare 600 mL of test solution as follows:

53.1.1 (**Warning—**Protect the eyes and face by face shield and use rubber gloves and apron when handling acid. Place flask under hood.)

53.1.2 First, measure 400.0 mL of Type IV reagent water and pour into the Erlenmeyer flask.

53.1.3 Then measure 236.0 mL of reagent grade sulfuric acid. Add the acid slowly to the water in the Erlenmeyer flask to avoid boiling by the heat evolved. (Note 7.)

53.1.4 Weigh 72 g of reagent grade copper sulfate ($CuSO_4 \cdot 5 H_2O$) and add to the sulfuric acid solution.

53.1.5 Place the copper piece into one glass cradle and put it into the flask.

53.1.6 Drop boiling chips into the flask.

53.1.7 Lubricate the ground-glass joint with silicone grease.

53.1.8 Cover the flask with the condenser and circulate cooling water.

53.1.9 Heat the solution slowly until all of the copper sulfate is dissolved.

53.1.10 (**Warning—**It has been reported that violent boiling resulting in acid spills can occur. It is important to ensure that the concentration of acid does not increase and that an adequate number of boiling chips (which are resistant to attack by the test solution) are present.)

54. Sampling

54.1 Obtain and prepare only base metal samples.

54.1.1 There are two classes of specimens to be considered: base metal, and process-affected metal. Process-affected metal contains any condition that affects the corrosion properties of the material in a non-uniform way, such as (but not limited to) welds; carburized, nitrided, or oxidized surfaces; mechanical deformation; and areas affected by heat. Base metal has none of these conditions.

54.1.2 The Practice F test involves immersing the entire specimen and averaging the mass loss over the entire surface of the specimen. Welding, carburization, mechanical deformation, and the like, affect only part of a specimen.

54.1.3 The mass loss rate from process-affected metal is expected to differ from that from base metal; the presence of process-affected metal in a specimen will affect the calculated test result in an unpredictable way.

54.1.4 If the presence of these or other localized conditions is a concern to the purchaser, then tests that do not average the mass loss over the total specimen surface area, such as Practice A, the Oxalic Acid Etch Test, or Practice E, the Copper–Copper Sulfate–16% Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels, should be considered. Details of the test and acceptance criteria shall be as agreed by the purchaser and producer.

54.2 Unless otherwise specified by the purchaser, the procedures for obtaining representative base metal samples, for removing the specimens from the samples, and the number of specimens shall be at the discretion of the producer.

55. Preparation of Test Specimens

55.1 Heat treat extra-low carbon and stabilized grades at 650 to 675°C (1200 to 1250°F), which is the range of maximum carbide precipitation, prior to testing. The length of time of heating, and the method of subsequent cooling used for this sensitizing treatment together with the corresponding maximum permissible corrosion rate shall be as agreed between the material producer and purchaser.

NOTE 24—The most commonly used sensitizing treatment is 1 h at 675°C (1250°F).

55.2 Prepare the specimens, each having a total surface area of 5 to 20 cm^2.

55.3 Where feasible for the product form, grind all the specimen surfaces using CAMI/ANSI 120 [FEPA/ISO P120] paper-backed, wet or dry, closed coated abrasive paper, with water as a coolant. If abrasive paper is used dry, polish slowly to avoid overheating. Do not use abrasives with grinding aids; some grinding aids contain fluorides that can affect the measured corrosion rate.

55.4 Remove all traces of oxide scale and heat tint formed during heat treatments. Any scale that cannot be removed by grinding (for example, in stamped numbers) may be removed by using one of the pickling solutions described in Practice A380/A380M, Table A1.1. (Residual oxide scale causes galvanic action and consequent activation in the test solution.)

55.5 Measure the specimens, including the inner surfaces of any holes, to the nearest 0.05 mm (0.001 in.) and calculate the total exposed area.

55.6 Degrease the specimens using suitable nonchlorinated agents, such as soap and lukewarm water, or acetone. Dry the specimens and weigh each one to the nearest 0.001 g. Store the specimens in a desiccator until the test is to be performed.

56. Procedure

56.1 If the test solution is not already boiling, bring it to boiling.

56.1.1 Keep the flask covered with the condenser (with cooling water flowing) except when inserting or removing specimens. (See Note 7.)

56.2 Turn off the heat source and allow the boiling to subside.

56.3 Place the specimen in a second glass cradle.

56.4 Uncover the flask.

56.5 Insert the specimens.

56.6 Replace the condenser immediately, restore cooling water flow, and turn on the heat source.

56.7 Mark the liquid level on the flask to provide a check on vapor loss, which would result in concentration of the acid. If there is an appreciable change in the level, repeat the test with fresh solution and a reground specimen.

56.8 Continue immersion of the specimen for 120 h, then remove the specimen, rinse in water and acetone, and dry. If any adherent copper remains on the specimen, it may be removed by a brief immersion in concentrated nitric acid at room temperature.

56.9 Weigh the specimen and subtract the weight from the original weight.

56.10 Intermediate weighings are usually not necessary; the test can be run without interruption. However, if preliminary results are desired, the specimen can be removed at any time for weighing.

56.11 Changes to the solution during the 120-h test period are not necessary.

57. Calculation and Report

57.1 The effect of the acid solution on the material is measured by determining the loss of weight of the specimen. The corrosion rate should be reported as millimetres of penetration per month (Note 9) calculated using Eq 1.

58. Precision and Bias

58.1 *Precision*—The precision of Practice F is being determined.

58.2 *Bias*—This practice has no bias because the resistance to intergranular corrosion is defined only in terms of this practice.

59. Keywords

59.1 austenitic stainless steel; copper sulfate; corrosion testing; etch structures; ferric sulfate; intergranular corrosion; nitric acid; oxalic acid

APPENDIX

Nonmandatory Information

X1. APPLICATION OF THESE TEST METHODS

X1.1 General

X1.1.1 These test methods detect one or more of three types of susceptibility to intergranular attack: chromium carbide, sigma phase, and end-grain. The choice of test method is affected by the intended service, the type or types of attack expected from that service, and the grade of material to be evaluated.

X1.1.2 These practices describe the procedures by which the tests are conducted to determine the susceptibility of austenitic stainless steels to intergranular attack. The presence or absence of intergranular corrosion in these tests is not necessarily a measure of the performance of the material in other corrosive media. The tests do not provide a basis for predicting resistance to other forms of corrosion, such as general corrosion, pitting, or stress-corrosion cracking.

X1.1.3 Susceptibility to intergranular attack associated with the precipitation of chromium carbides is readily detected in all five tests.

X1.1.4 Sigma phase may be present in wrought chromium-nickel-molybdenum steels, in titanium-or columbium-stabilized alloys, and in cast molybdenum-bearing stainless alloys. Such sigma phase may or may not be visible in the microstructure depending on the etching technique and magnification used. Not all of the test methods can detect sigma phase; see the discussions below.

X1.1.5 In most cases either the 15-h copper–copper sulfate–16 % sulfuric acid test or the 120-h ferric sulfate-sulfuric acid test, combined with the Oxalic Acid Etch Test, will provide the required information in the shortest time. All stainless grades listed in this appendix may be evaluated in these combinations of screening and corrosion tests, except those specimens of molybdenum-bearing grades (for example 316, 316L, 317, and 317L), which represent steel intended for use in nitric acid environments.

X1.1.6 The 240-h nitric acid test should be applied to stabilized and molybdenum-bearing grades intended for service in nitric acid and to all stainless steel grades that might be subject to end grain corrosion in nitric acid service.

X1.1.7 Extensive test results on various types of stainless steels evaluated by these practices have been published in (**8**).

PRACTICE A—OXALIC ACID ETCH TEST

X1.2 The Oxalic Acid Etch Test is used for acceptance of material but not for rejection of material. This may be used in connection with other evaluation tests to provide a rapid method for identifying those specimens that are certain to be free of susceptibility to rapid intergranular attack in these other tests.

X1.2.1 The etch test is suitable for use only when it is listed in the applicable table under the specified hot acid test.

X1.2.2 Grades not listed in the applicable table either have not been evaluated for use of Practice A with that hot acid test, or have been found to give acceptable results in the etch test while giving unacceptable results in the hot acid test. In the latter case the etch test would pass material that should have been rejected.

X1.2.3 When listed, the etch test can reduce the time required to determine whether the material represented by the specimen will have a low corrosion rate in that hot acid test. However, when the etch test shows a suspect structure, the specified hot acid must be performed to avoid rejecting good material.

PRACTICE B—FERRIC SULFATE-SULFURIC ACID TEST

X1.3 Practice B—Ferric sulfate-sulfuric acid test is a 120-h test in boiling solution.

X1.3.1 The ferric sulfate-sulfuric acid test may be used to evaluate the heat treatment accorded as-received material. It may also be used to check the effectiveness of stabilizing columbium or titanium additions and of reductions in carbon content in preventing susceptibility to rapid intergranular attack. It may be applied to wrought products (including tubes), castings, and weld metal.

X1.3.2 The ferric sulfate-sulfuric acid test detects susceptibility to intergranular attack associated primarily with chromium carbide precipitate in the unstabilized austenitic stainless steels 304, 304L, 316, 316L, 317, 317L, CF-3, CF-8, CF3M, CF8M, CG3M, and CG8M; to intergranular attack associated with sigma phase in 321, 347, CF-3M, CF-8M, CG3M, and CG8M. It also reveals susceptibility associated with a sigma-like phase constituent in stabilized stainless steels 321 and 347, and in cast chromium-nickel-molybdenum stainless steels CF-3M, CF-8M, CG-3M, and CG-8M.

X1.3.3 The ferric sulfate-sulfuric acid test does not detect susceptibility to intergranular attack associated primarily with sigma phase in wrought chromium-nickel-molybdenum stainless steels (316, 316L, 317, 317L), which is known to lead to rapid intergranular attack in certain nitric acid environments. It does not detect susceptibility to end grain attack, which is also found in certain nitric acid environments.

NOTE X1.1—To detect susceptibility to intergranular attack associated with sigma phase in austenitic stainless steels containing molybdenum, the nitric acid test, Practice C, should be used.

X1.3.4 The Oxalic Acid Etch Test (Practice A) may be used to screen certain grades from testing in the ferric sulfate-sulfuric acid test; see Table 1. Grades not listed in Table 1 either have not been evaluated for use of Practice A with the ferric sulfate-sulfuric acid test or have been found to give acceptable results in the etch test while giving unacceptable results in the ferric sulfate–sulfuric acid test, thus passing material that should be rejected.

PRACTICE C—NITRIC ACID TEST

X1.4 Practice C—Nitric test is a 240-h test in boiling solution.

X1.4.1 The boiling nitric acid test may be used to evaluate the heat treatment accorded "as-received" material. It is also sometimes used to check the effectiveness of stabilizing elements and of reductions in carbon content in preventing susceptibility to rapid intergranular attack. This practice may be applied to wrought products (including tubes), castings, and weld metal of the various grades of stainless steel

X1.4.2 Intergranular attack in nitric acid is associated with one or more of the following: intergranular precipitation of chromium carbides, sigma or transition phases in molybdenum-bearing grades, and sigma phase constituents in stabilized grades. The boiling nitric acid test should not be used for extra-low-carbon molybdenum-bearing grades unless the material tested is to be used in nitric acid service.

X1.4.3 The Practice C test detects susceptibility to rapid intergranular attack associated with chromium carbide precipitate and with sigma-like phase precipitate. The latter may be formed in molybdenum-bearing and in stabilized grades of austenitic stainless steels and may or may not be visible in the microstructure. This test also reveals susceptibility to end grain attack in all grades of stainless steels.

X1.4.4 The nitric acid test detects susceptibility to intergranular attack associated primarily with chromium carbide precipitate in 304, 304L, 316, 316L, 317, 317L, 321, 347, CF-3, CF-8, CF-3M, and CF-8M; to intergranular attack associated with sigma phase in 316, 316L, 317, 317L, 321,

347, CF-3M, and CF-8M; and to end-grain attack in 304, 304L, 316, 316L, 317, 317L, 321, and 347. The nitric acid test may be also applied to 309, 310, 348, 410, 430, 446, and CN-7M. Those grades in which sigma phase may form must be tested in nitric acid test when destined for service in nitric acid.

X1.4.5 The Oxalic Acid Etch Test (Practice A) may be used to screen certain grades from testing in the nitric acid test; see Table 2. Grades not listed in Table 2 either have not been evaluated for use of Practice A with the nitric acid test or have been found to give acceptable results in the etch test while giving unacceptable results in the nitric acid test, thus passing material that should be rejected. Specifically, grades 316, 316L, 317, 317L, 347, and 321 cannot be screened because these steels may contain sigma phase not visible in the etch structure. This may cause rapid intergranular attack in the nitric acid test.

PRACTICE E—COPPER–COPPER SULFATE–16 % SULFURIC ACID TEST

X1.5 Practice E—Copper–Copper Sulfate–16 % Sulfuric Acid Test is a 15-h test in a boiling solution with the test specimen embedded in metallic copper shot or grindings. After exposure in the boiling solution, the specimen is bent.

X1.5.1 This test may be used to evaluate the heat treatment accorded as-received material. It may also be used to evaluate the effectiveness of stabilizing element additions (Cb, Ti, and so forth) and reductions in carbon content to aid in resisting intergranular attack. All wrought products and weld material of austenitic stainless steels can be evaluated by this test.

X1.5.2 Practice E indicates susceptibility to intergranular attack associated with the precipitation of chromium-rich carbides in 201, 202, 301, 304, 304L, 316, 316L, 317, 317L, 321, and 347.

X1.5.3 It does not detect susceptibility to intergranular attack associated with sigma phase or end-grain corrosion, both of which have been observed only in certain nitric acid environments.

X1.5.4 The Oxalic Acid Etch Test (Practice A) may be used to screen certain grades from testing in the copper–copper sulfate–16 % sulfuric acid test; see Table 4. Grades not listed in Table 4 either have not been evaluated for use of Practice A with the copper–copper sulfate-16 % sulfuric acid test or have been found to give acceptable tests in the etch test while giving unacceptable results in the copper–copper sulfate–16 % sulfuric acid test, thus passing material that should be rejected.

PRACTICE F—COPPER–COPPER SULFATE–50 % SULFURIC ACID TEST

X1.6 Practice F—Copper–Copper Sulfate–50 % Sulfuric Acid Test is a 120-h test in a boiling solution that contains metallic copper.

X1.6.1 This test detects susceptibility to intergranular attack associated with the precipitation of chromium-rich carbides in CF-3M, CF-8M, and 316Ti.

X1.6.2 This test does not detect susceptibility to attack associated with sigma phase.

X1.6.3 The Oxalic Acid Etch Test (Practice A) may be used to screen certain grades from testing in the copper–copper sulfate–50 % sulfuric acid test; see Table 6. Grades not listed in Table 6 either have not been evaluated for use of Practice A with the copper–copper sulfate–50 % sulfuric acid test or have been found to give acceptable results in the etch test while giving unacceptable results in the copper–copper sulfate–50 % sulfuric acid test, thus passing material that should be rejected.

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(2) For original descriptions of the boiling nitric acid test, see Huey, W. R., "Corrosion Test for Research and Inspection of Alloys," *Transactions, American Society of Steel Treating,* Vol 18 , 1930, p. 1126; also, "Report of Subcommittee IV on Methods of Corrosion Testing," *Proceedings,* ASTM, Vol 33, Part I, 1933, p. 187.

(3) Streicher, M. A., "Theory and Application of Evaluation Tests for Detecting Susceptibility to Intergranular Attack in Stainless Steels and Related Alloys-Problems and Opportunities," *Intergranular Corrosion of Stainless Alloys, ASTM STP 656*, R. F. Steigerwald ed., ASTM, 1978, pp. 3–84.

(4) The use of copper to accelerate the intergranular corrosion of sensitized austenitic stainless steels in copper sulfate–sulfuric acid was first described by H. J. Rocha in the discussion of a paper by Brauns, E., and Pier, G., *Stahl und Eisen,* Vol 75, 1955, p. 579.

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(1) For original description of ferric sulfate-sulfuric acid test, see Streicher, M. A., "Intergranular Corrosion Resistance of Austenitic Stainless Steels: A Ferric Sulfate-Sulfuric Acid Test," *ASTM Bulletin*, No. 229, April 1958, pp. 77–86.

(2) For details, see DeLong, W. B., "Testing Multiple Specimens of Stainless Steels in a Modified Boiling Nitric Acid Test Apparatus," *Symposium on Evaluation Tests for Stainless Steels, ASTM STP 93,* ASTM, 1950, p. 211.

(3) See *Industrial and Engineering Chemistry,* Vol 17, 1925, p. 756; also, "A.C.S. Analytical Reagents. Specifications Recommended by Committee on Analytical Reagents," American Chemical Society, March 1941.

SUMMARY OF CHANGES

Committee A01 has identified the location of selected changes to this standard since the last issue (A262 – 14) that may impact the use of this standard. (Approved Sept. 1, 2015.)

(1) Added Note 4 to 18.1, allowing other glass joints.
(2) Revised Apparatus requirements in 40.1 to refer to Section 18.
(3) Added new subsection 38.1.2 to Practice E to clarify required heat treatment the prior to performing the Rapid Screening (Etch) Test.

Committee A01 has identified the location of selected changes to this standard since the last issue (A262 – 13) that may impact the use of this standard. (Approved July 1, 2014.)

(1) Added new Section 3 on Purity of Reagents.
(2) Added four referenced documents.
(3) Re-wrote Practice A in test method format.
(4) Numbered the un-numbered notes in Figs. 6 and 7.
(5) Re-wrote Practice B; Renumbered subsequent paragraphs, notes, tables, and references to match.
(6) Corrected the constant in Eq 1.
(7) Re-wrote Practice C; Renumbered subsequent paragraphs, notes, tables, and references to match.
(8) Removed references to the multiple sample apparatus.
(9) Re-wrote Practice F in test-method format.
(10) Restricted specimen type to base metal.
(11) Added procedural text.
(12) Revised 45.1.