



Designation: A262 – 14

Standard Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels¹

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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

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

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from American Society of Mechanical Engineers (ASME), ASME International Headquarters, Two Park Ave., New York, NY 10016-5990, http://www.asme.org.

⁴ Available from American Chemical Society (ACS), 1155 Sixteenth Street, NW, Washington, DC 20036, http://www.acs.org

¹ These practices are under the jurisdiction of ASTM Committee A01 on Steel, Stainless Steel and Related Alloys and are the direct responsibility of Subcommittee A01.14 on Methods of Corrosion Testing.

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*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 Society⁶ 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.

⁵ Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, <http://www.iso.org>.

⁶ For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

⁷ The boldface numbers in parentheses refer to a list of references at the end of this standard.

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 ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) 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 ($(\text{NH}_4)_2\text{S}_2\text{O}_8$) 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 cm² 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/cm² 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/cm² 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.

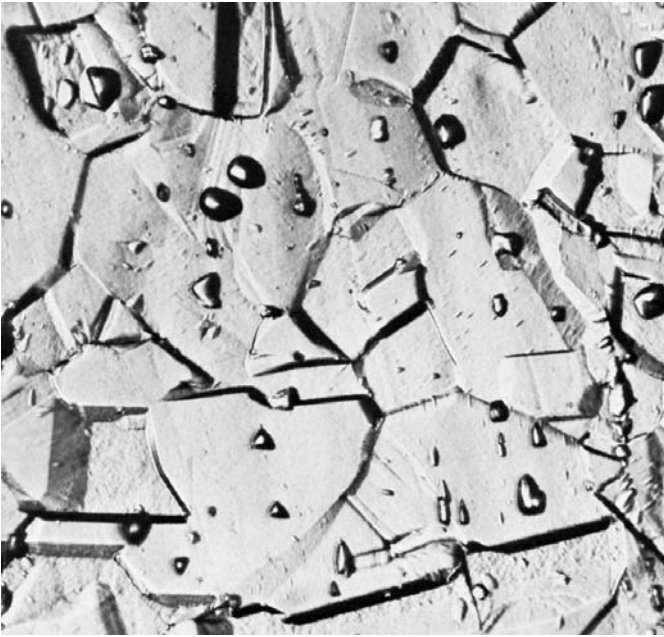


FIG. 1 Step Structure (500x) (Steps between grains, no ditches at grain boundaries)

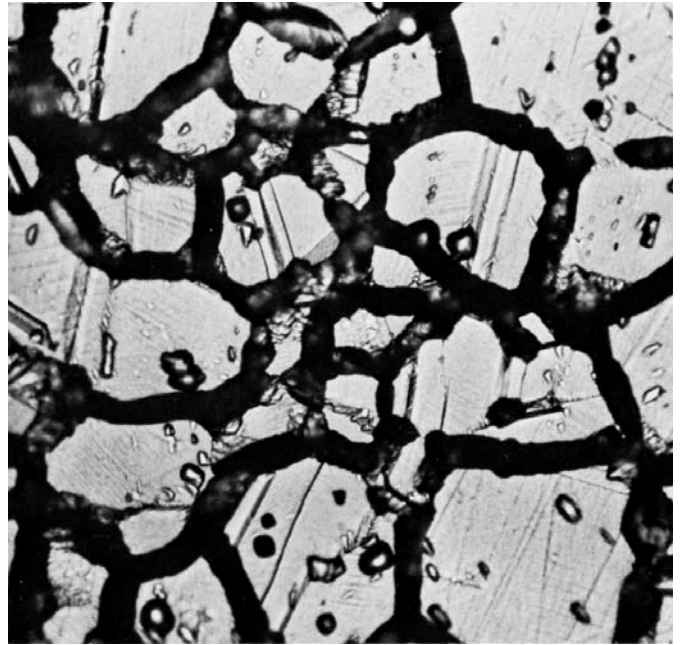


FIG. 3 Ditch Structure (500x) (One or more grains completely surrounded by ditches)

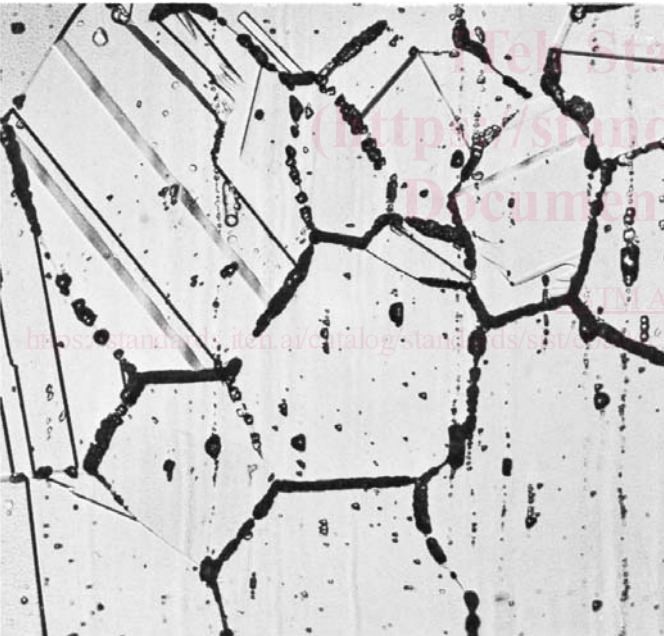


FIG. 2 Dual Structure (250x) (Some ditches at grain boundaries in addition to steps, but no one grain completely surrounded)

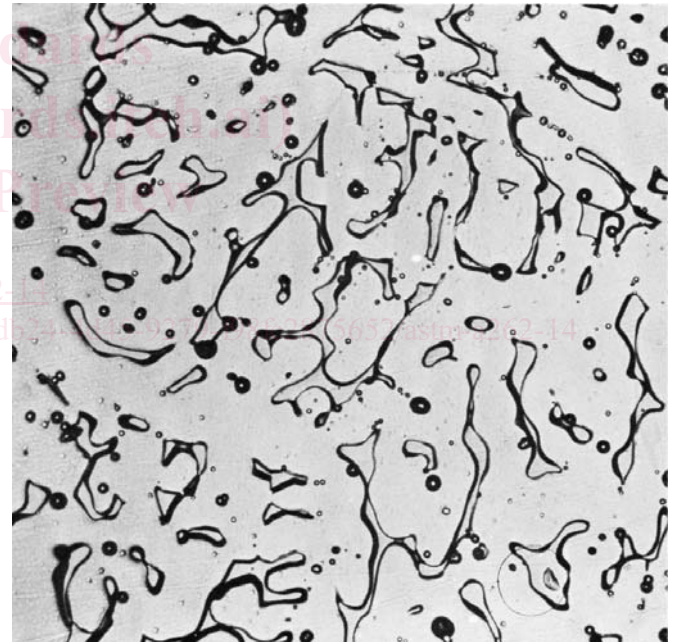


FIG. 4 Isolated Ferrite Pools (250x) (Observed in castings and welds. Steps between austenite matrix and ferrite pools)

11.3.6 *End-Grain Pitting I* (Fig. 6)—Structure contains a few deep end-grain pits along with some shallow etch pits at 500x. (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 500x. (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/cm².

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

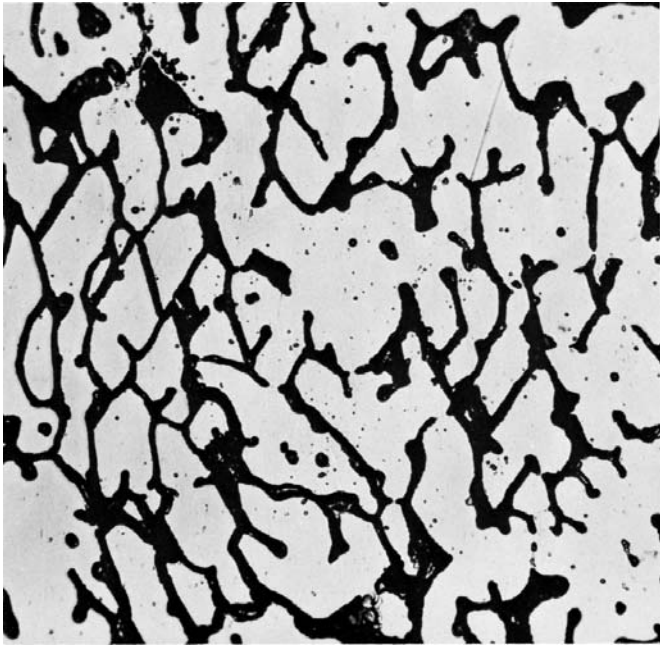
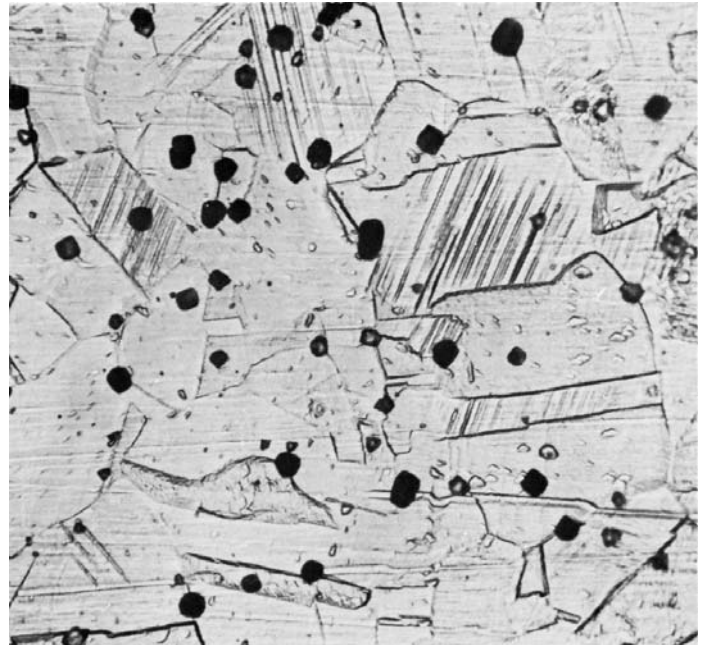
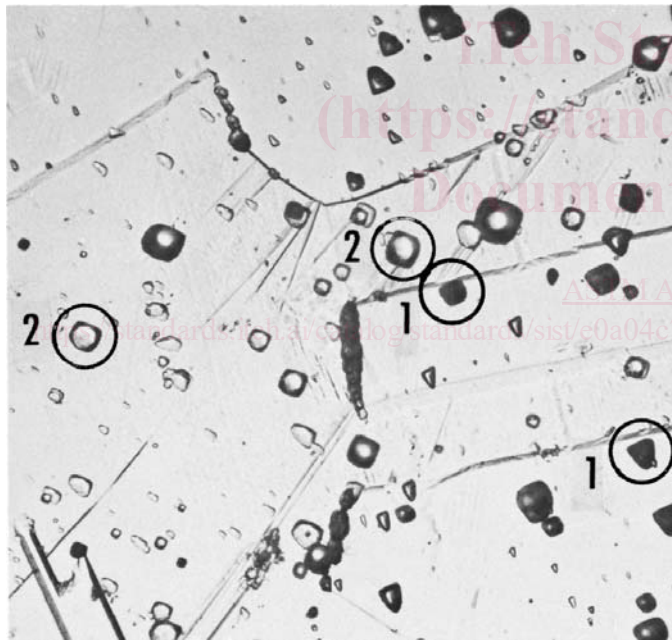


FIG. 5 Interdendritic Ditches (250x) (Observed in castings and welds. Deep interconnected ditches)



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

FIG. 7 End Grain Pitting II (500x)



NOTE 1—To differentiate between the types of pits, use a magnification of 500x 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 (500x) (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

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 Fig. 1 and Fig. 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.

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.

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.

18.1.3 Glass cradle (Note 4)—Can be supplied by a glass-blowing shop. It must be sized so as to fit, with the specimen,

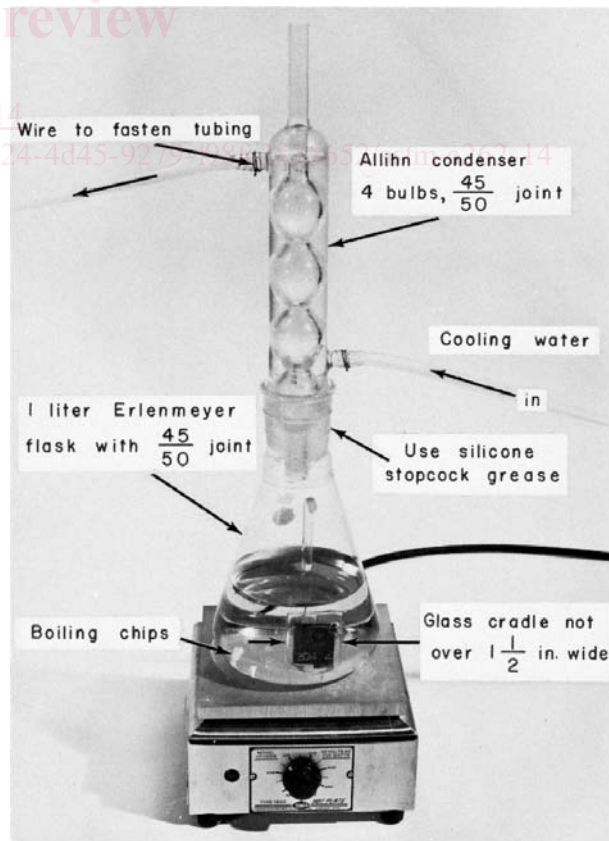


FIG. 8 Apparatus for Ferric Sulfate-Sulfuric Acid Test

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.

through the flask opening. It must be designed to allow free flow of the testing solution around the specimen.

NOTE 4—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 5—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* ($\text{Fe}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$), about 75 % ($\text{Fe}_2(\text{SO}_4)_3$) 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* ($\text{H}_2(\text{SO}_4)_4$), 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 6—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 6).

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 7—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 cm².

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 6**.)

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 8**), 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, $d = 7.9 \text{ g}/\text{cm}^3$

for chromium-nickel-molybdenum steels, $d = 8.00 \text{ g}/\text{cm}^3$

NOTE 8—Conversion factors to other commonly used units for corrosion rates are as follows:

Millimetres per month $\times 0.04 =$ inches per month

Millimetres per month $\times 0.47 =$ inches per year

Millimetres per month $\times 12 =$ millimetres per year

Millimetres per month $\times 472 =$ mils per year

Millimetres per month $\times 1000 \times \text{density}/3 =$ milligrams per square decimetre per day

Millimetres per month $\times 1.39 \times \text{density} =$ 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 9—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.

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 (HNO₃ **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

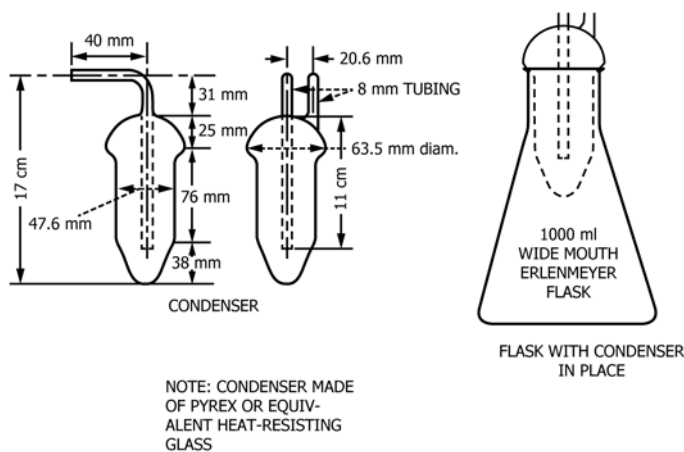


FIG. 9 Flask and Condenser for Nitric Acid Test

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

NOTE 11—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 cm².

33.5 Degrease the specimen using suitable nonchlorinated agents, such as soap and lukewarm water, or acetone (Note 12). 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 12—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/cm² (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 13—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 14). 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 14—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