This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: A262 – 15 (Reapproved 2021)

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.

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, health, and environmental 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.

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

[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.

Current edition approved Sept. 1, 2021. Published September 2021. Originally approved in 1943. Last previous edition approved in 2015 as A262 – 15. DOI: 10.1520/A0262-15R21.

[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.

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

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. United States

Documents
Document Sharing Hub
Full document details: ASTM A262-15(2021)

2.2 *ASME Code:*[3]

BPVC ASME Boiler and Pressure Vessel Code, Section IX

2.3 *ACS Specifications:*[4]

ACS Reagent Chemicals Specifications and Procedures

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.

[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 St., NW, Washington, DC 20036, http://www.acs.org.

[5] Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://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.

AVA Documents
Document Sharing Hub

Full document details: ASTM A262-15(2021)

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):

AVA Documents
Document Sharing Hub

Full document details: ASTM A262-15(2021)

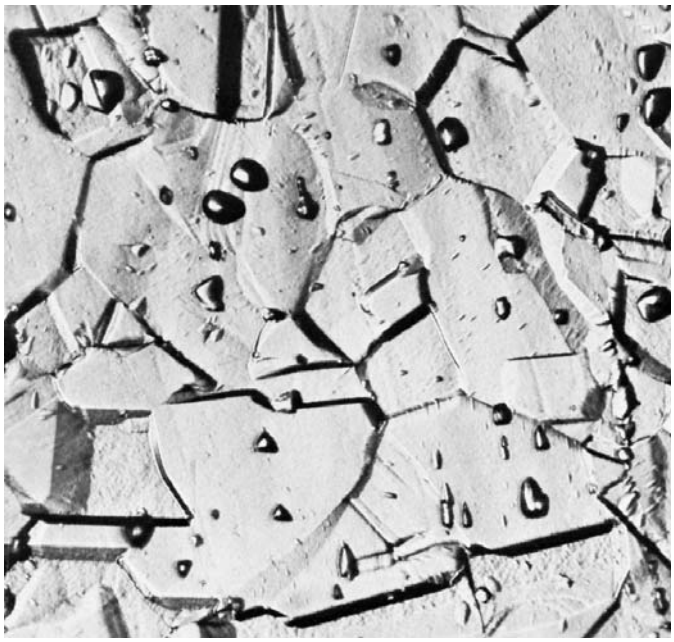


FIG. 1 Step Structure (500×) (Steps Between Grains, No Ditches at Grain Boundaries)

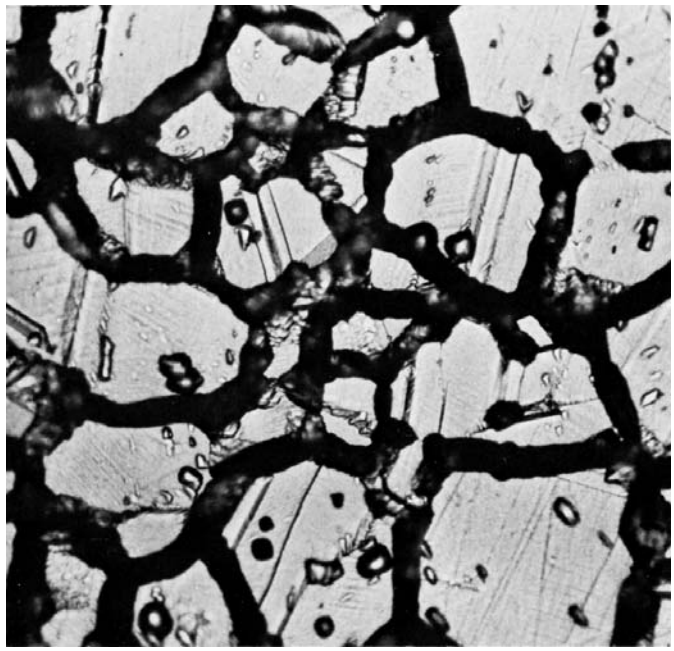


FIG. 3 Ditch Structure (500×) (One or More Grains Completely Surrounded by Ditches)

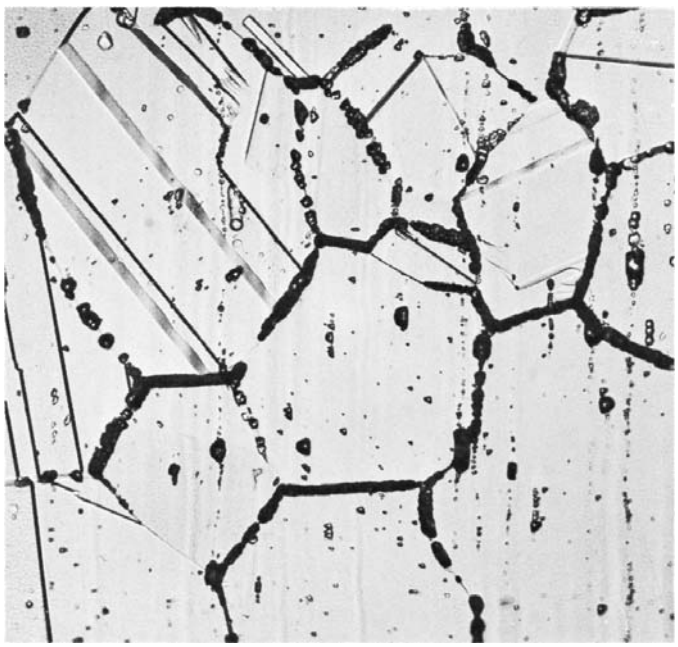


FIG. 2 Dual Structure (250×) (Some Ditches at Grain Boundaries in Addition to Steps, but No One Grain Completely Surrounded)

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

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.

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

Documents
Document Sharing Hub

Full document details: ASTM A262-15(2021)

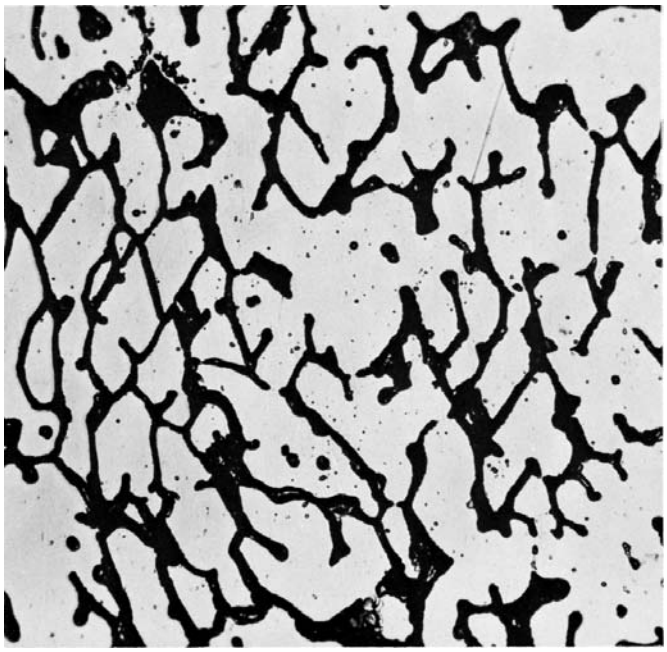
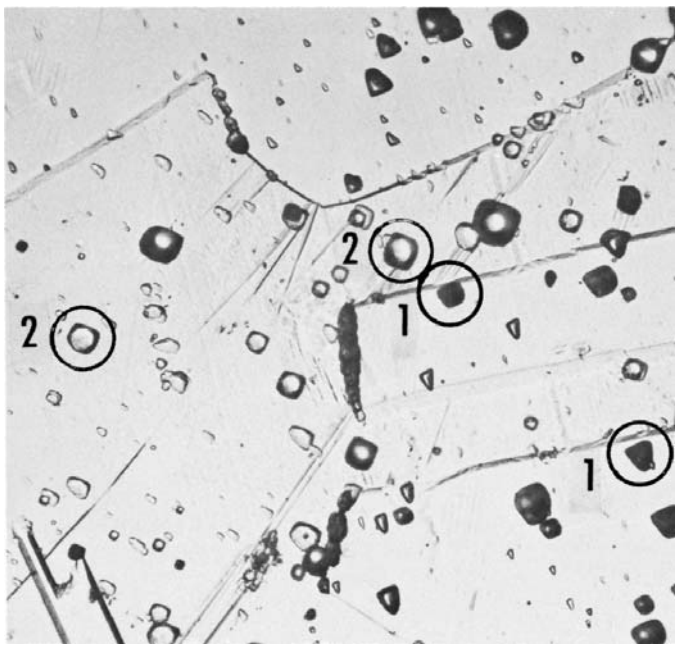


FIG. 5 Interdendritic Ditches (250×) (Observed in Castings and Welds. Deep Interconnected Ditches)

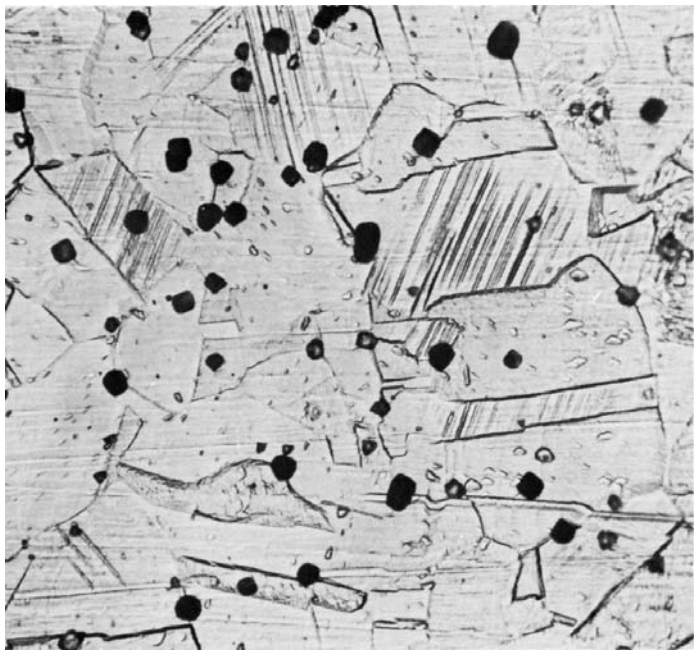


NOTE 1—To differentiate between the types of pits, use a magnification of 500× and focus in the plane of etched surface. The pits which now appear completely black are end grain pits.

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

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.



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×)

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 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 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.

AVA Documents
Document Sharing Hub

Full document details: ASTM A262-15(2021)

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.

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.

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.

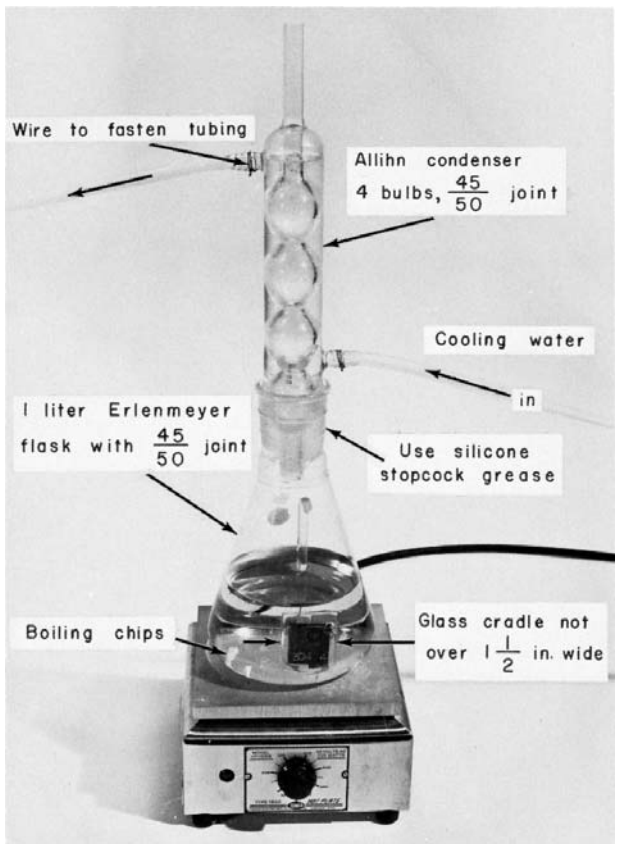


FIG. 8 Apparatus for Ferric Sulfate-Sulfuric Acid Test

AVA Documents
Document Sharing Hub

Full document details: ASTM A262-15(2021)