



Designation: **A262—13 A262 – 14**

## Standard Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels<sup>1</sup>

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 ( $\epsilon$ ) 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 ~~34~~ to ~~713~~, inclusive),

1.1.2 *Practice B*—~~Ferric Sulfate–Sulfuric~~~~Sulfate–Sulfuric~~ Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections ~~814~~ to ~~1425~~, inclusive),

1.1.3 *Practice C*—Nitric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections ~~1526~~ to ~~2136~~, inclusive),

1.1.4 *Practice E*—Copper–Copper Sulfate–Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections ~~2237~~ to ~~3146~~, 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 ~~3247~~ to ~~3858~~, 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~~~~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.

NOTE 1—See [Appendix X1](#) for information regarding test selection.

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 ~~problems, 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. (Specific ~~precautionary~~ Some specific hazards statements are given in ~~5.610.1, 11.1.120.1.1, 11.1.920.1.9, 31.3, 34.4, 53.1.1, and 35.153.1.10~~).*

<sup>1</sup> 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. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

[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:<sup>3</sup>

[ASME Boiler & Pressure Vessel Code, Section IX](#)

### 2.3 ACS Specifications:<sup>4</sup>

[Reagent Chemicals, Specifications and Procedures](#)

### 2.4 ISO Standard:<sup>5</sup>

[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<sup>6</sup> 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)<sup>7</sup>

## 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 The oxalic acid etch test is used for acceptance of material but not for rejection of material. 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, requiring from 4 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 following criteria: 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 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. 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.

3.2.1 Each practice 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.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

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

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

<sup>6</sup> 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.

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

4.5 Extra-low-carbon grades, and stabilized grades, such as 304L, 316L, 317L, 321, and 347, are tested after sensitizing heat treatments at 650 to 675°C (1200 to 1250°F), which is the range of maximum carbide precipitation. These sensitizing treatments must be applied before the specimens are submitted to the oxalic acid etch test. The most commonly used sensitizing treatment is 1 h at 675°C (1250°F). 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

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

4.2 *Ammeter*—Range 0 to 30 A (**Note 2**).

4.3 *Variable Resistance* (**Note 2**).

4.4 *Cathode*—A cylindrical piece of stainless steel or, preferably, a 1-qt (0.946-L) stainless steel beaker.

4.5 *Large Electric Clamp*—To hold specimen to be etched.

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

7.1 *Electrodes of the Etching Cell—Etching Cell*: The specimen to be etched is made the anode, and a stainless steel beaker or a piece of stainless steel as large as the specimen to be etched is made the cathode.

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 *Electrolyte—Metallurgical Microscope*—Oxalic acid, (HFor examination of  $\text{Fe}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ ), reagent grade, 10 weight % solution. etched microstructures at 250 to 500 diameters.

**NOTE 2**—The variable resistance and the ammeter are placed in the circuit to measure and control the current on the specimen to be etched.

## 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. Preparation of Sampling and Test Specimens

9.1 *Cutting*—Sawing is preferred to shearing, especially on the extra-low-carbon grades. Shearing cold works adjacent metal and affects the response to subsequent sensitization. Microscopical examination of an etch made on a specimen containing sheared edges, should be made on metal unaffected by shearing. A convenient specimen size is 25 by 25 mm (1 by 1 in.). 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 intent is to test a specimen representing as nearly as possible the surface of the material as it will be used in service. Therefore, the preferred sample is a cross section including the product surface to be exposed in service. Only such surface finishing of the product surface should be performed as is required to remove foreign material and obtain a standard, uniform finish as described in material 5.3. For very heavy sections, specimens should be machined to represent the appropriate surface while maintaining reasonable specimen size for convenient testing. Ordinarily, removal of more material than necessary will have little influence on the test results. However, in the special case of surface carburization (sometimes encountered, for instance, in tubing or castings when lubricants or binders containing carbonaceous materials are employed) it may be possible by heavy grinding or machining to completely remove the carburized surface. Such treatment of test specimens is not permissible, except in tests undertaken to demonstrate such effects.

9.3 *Polishing*—On all types of materials, cross sectional surfaces should be polished for etching and microscopical examination. Specimens containing welds should include base plate, weld heat-affected zone, and weld metal. Scale should be removed from the area to be etched by grinding to an 80- or 120-grit finish on a grinding belt or wheel without excessive heating and then polishing on successively finer emery papers, No. 1, Whenever  $\frac{1}{2}$ ,  $\frac{1}{6}$ ,  $\frac{2}{6}$ , and  $\frac{3}{6}$ , or finer. This polishing operation can be carried out in a relatively short time since all large scratches need not be removed. Whenever practical, a polished practical, use a cross-sectional area of  $1 \text{ cm}^2$  or more is desirable. more. If any cross-sectional dimension is less than 1 cm, a minimum length of 1 cm should be polished. When the available length is 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 should be used. section.

5.4 *Etching Solution*—The solution used for etching is prepared by adding 100 g of reagent grade oxalic acid crystals ( $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ ) to 900 mL of distilled water and stirring until all crystals are dissolved.

5.5 *Etching Conditions*—The polished specimen should be etched at  $1 \text{ A/cm}^2$  for 1.5 min. To obtain the correct current density:

5.5.1 The total immersed area of the specimen to be etched should be measured in square centimetres, and

5.5.2 The variable resistance should be adjusted until the ammeter reading in amperes is equal to the total immersed area of the specimen in square centimetres.

9.4 *Etching Precautions: Polishing*—

5.6.1 **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. 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.

5.6.2 A yellow-green film is gradually formed on the cathode. This increases the resistance of the etching cell. When this occurs, the film should be removed by rinsing the inside of the stainless steel beaker (or the steel used as the cathode) with an acid such as 30%  $\text{HNO}_3$ .

5.6.3 The temperature of the etching solution gradually increases during etching. The temperature should be kept below  $50^\circ\text{C}$  by alternating two beakers. One may be cooled in tap water while the other is used for etching. The rate of heating depends on the total current (ammeter reading) passing through the cell. Therefore, the area etched should be kept as small as possible while at the same time meeting the requirements of desirable minimum area to be etched.

5.6.4 Immersion of the clamp holding the specimen in the etching solution should be avoided.

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

5.8 On some specimens containing molybdenum (AISI 316, 316L, 317, 317L), which are free of chromium carbide sensitization, it may be difficult to reveal the presence of step structures by electrolytic etching with oxalic acid. In such cases, an electrolyte of a 10% solution of ammonium persulfate,  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ , may be used in place of oxalic acid. An etch of 5 or 10 min at  $1 \text{ A/cm}^2$  in a solution at room temperature readily develops step structures on such specimens.



**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<sup>2</sup> 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<sub>3</sub>.

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<sup>2</sup> in a solution at room temperature readily develops step structures on such specimens.

**11. Classification of Etch Structures**

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

11.2 The Examine the etched cross-sectional areas should be thoroughly examined by complete traverse from inside to outside diameters of rods and tubes, from face to face on plates, and across all zones such as weld metal, weld-affected zones, and base plates on specimens containing welds. 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 The Classify the etch structures are classified into the following types (Note 32):

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



FIG. 1 Step Structure (500×) (Steps 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 etched under standard conditions: 10 % oxalic acid, room temperature, 1.5 min at 1 A/cm<sup>2</sup>.

6.4 The evaluation of etch structures containing steps only 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, the steel should be evaluated as a ditch structure. Areas near surfaces should be examined for evidence of surface carburization.

6.4.1 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 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 (The evaluation of etch structures containing only steps and of those showing grains completely surrounded by 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 ditches in every field can 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 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 Fig. 1 and structure. Fig. 6.

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 Fig. 1 and Fig. 6.

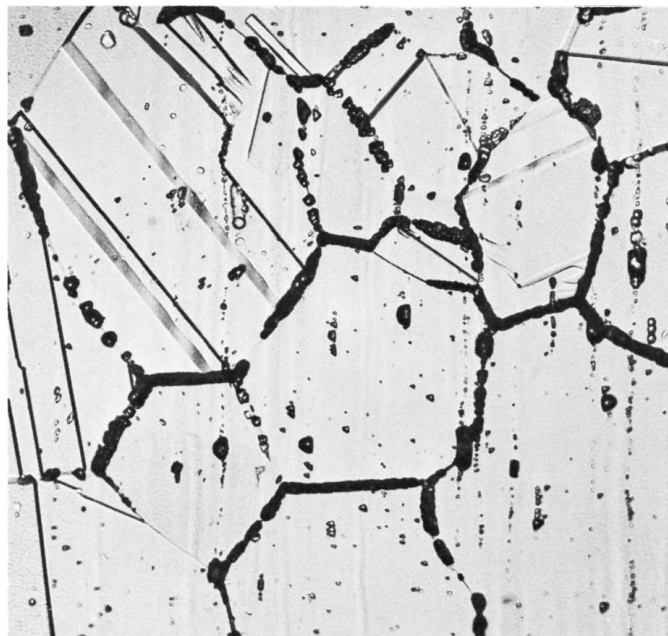


FIG. 2 Dual Structure (250×) (Some ditches at grain boundaries in addition to steps, but no one grain completely surrounded)

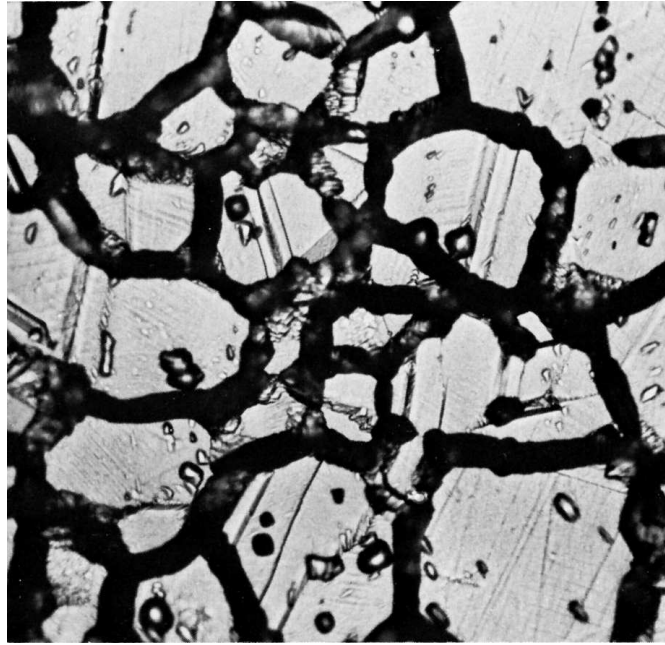


FIG. 3 Ditch Structure (500x) (One or more grains completely surrounded by ditches)

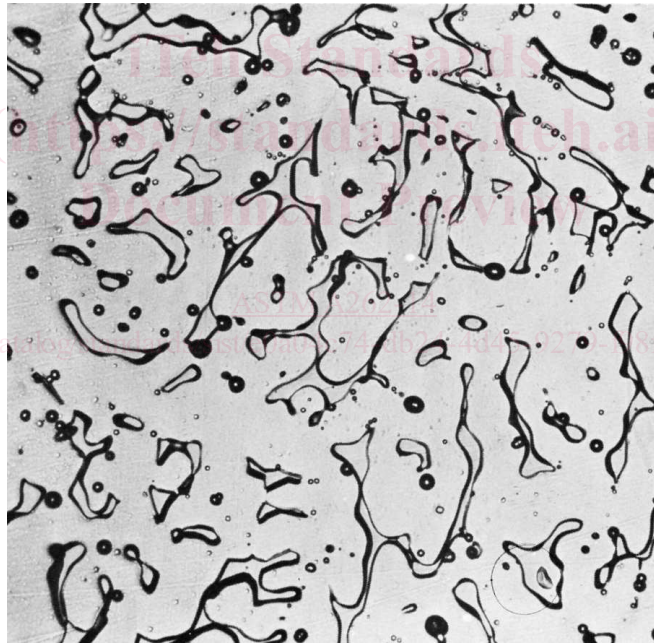


FIG. 4 Isolated Ferrite Pools (250x) (Observed in castings and welds. Steps between austenite matrix and ferrite pools)

## 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.

### ~~PRACTICE B—FERRIC SULFATE-SULFURIC ACID TEST FOR DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEELS (3)~~

## 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.



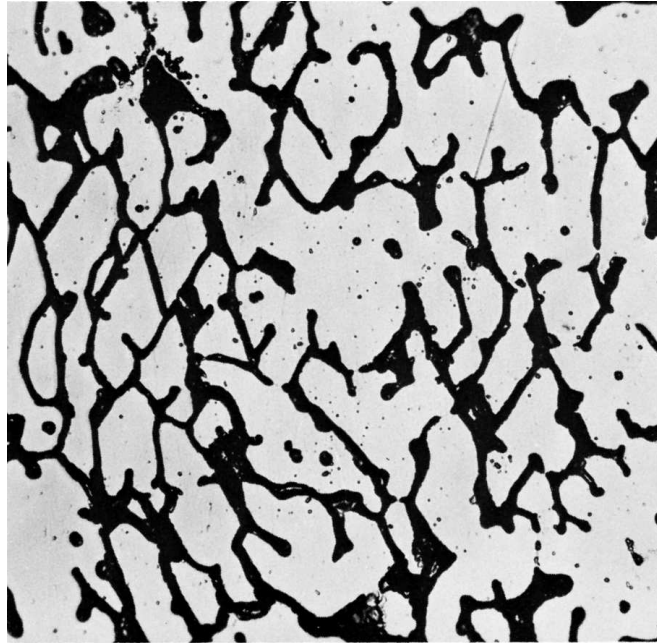
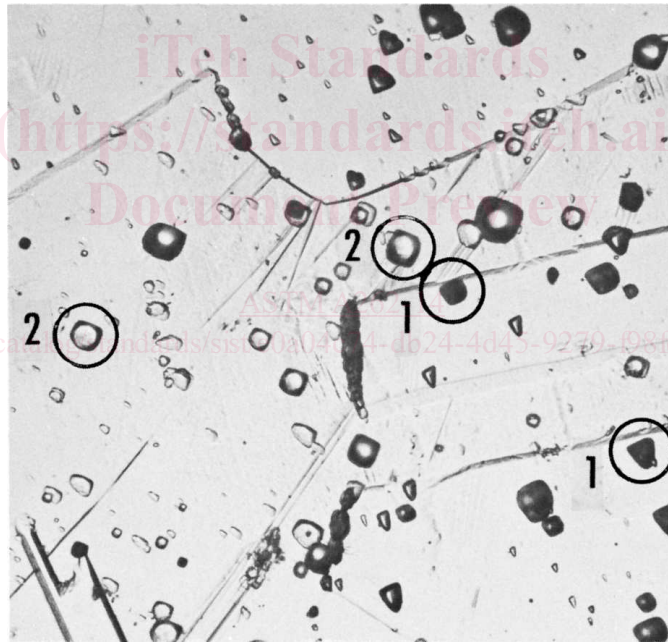


FIG. 5 Interdendritic Ditches (250x) (Observed in castings and welds. Deep interconnected ditches)



<https://standards.iteh.ai/catalog/standards/sist/a096174-4024-4d35-9230-08ac2875652/astm-a262-14>

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.

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.

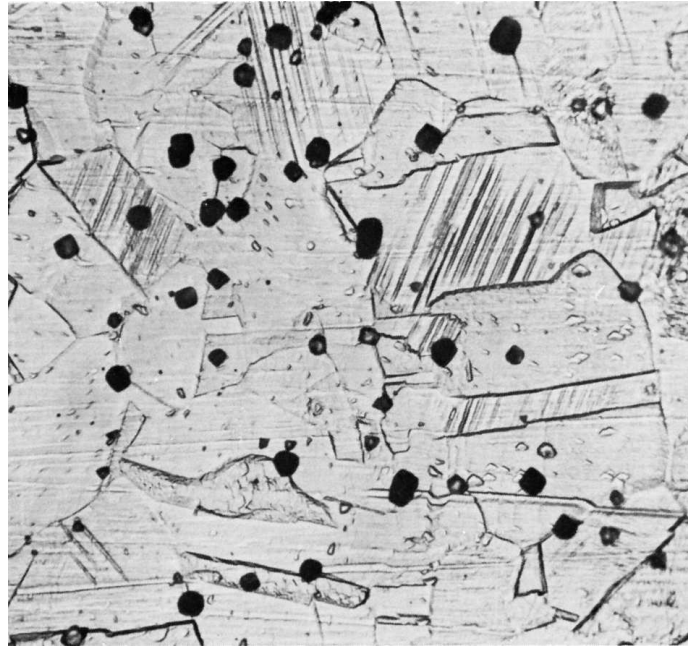
FIG. 6 End Grain Pitting I (500x) (A few deep end grain pits (see 1 in figure) and shallow etch pits (2))

**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.





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.

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×)

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

8.2 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 length of time of heating used for this sensitizing treatment determines the maximum permissible corrosion rate for such grades in the ferric sulfate-sulfuric acid test. The most commonly used sensitizing treatment is 1 h at 675°C (1250°F).

## 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-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.

9.1.1 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 must 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**

Grade	Acceptable Etch Structures	Suspect Etch Structures <sup>A</sup>
AISI 304	Step, dual, end grain, I & II	Ditch
AISI 304L	Step, dual, end grain, I & II	Ditch
AISI 316	Step, dual, end grain, I & II	Ditch
AISI 316L	Step, dual, end grain, I & II	Ditch
AISI 317	Step, dual, end grain, I & II	Ditch
AISI 317L	Step, dual, end grain, I & II	Ditch
ACI-CF-3	Step, dual, isolated ferrite pools	Ditch, interdendritic ditches
ACI-CF-8	Step, dual, isolated ferrite pools	Ditch, interdendritic ditches
ACI-CF-3M	Step, dual, isolated ferrite pools	Ditch, interdendritic ditches
ACI-CF-8M	Step, dual, isolated ferrite pools	Ditch, interdendritic ditches

**TABLE 1 Use of Etch Structure Classifications from the Oxalic Acid Etch Test with Ferric Sulfate-Sulfuric Acid Test<sup>A</sup>**

Grade	Acceptable Etch Structures	Suspect Etch Structures <sup>B</sup>
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

<sup>A</sup> 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.

<sup>B</sup> Specimens having these structures must shall be tested in the ferric sulfate-sulfuric acid test.

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 (Note 5) is illustrated in Fig. 8.

18.1.1 An Allihn or Soxhlet condenser with a minimum of four bulbs and with a 45/50 ground glass joint. Overall length about 330 mm (13 in.), condensing section, 9 ground glass joint to match that of the flask. 1/2 in. (241 mm).

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 45/50 ground glass joint. The ground glass opening is somewhat over 38 mm (1 ground glass joint to match that of the condenser. The flask opening limits the size of the specimen; 1/2 in.) wide. a larger opening is desirable.

18.1.3 Glass cradle (Note 4)—The glass cradle (Can Note 4) can be supplied by a glass-blowing shop. To pass through the ground glass joint on the Erlenmeyer flask, the width of the cradle should not exceed 38 mm (It 1/2 in.), and the front-to-back distance must be such that the cradle will fit the 34 mm (It must be sized so as to fit, with the specimen, through the flask 1/2 in.) diameter opening. It should have three or four holes to increase circulation 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—Boiling chips must be used Used to prevent bumping.

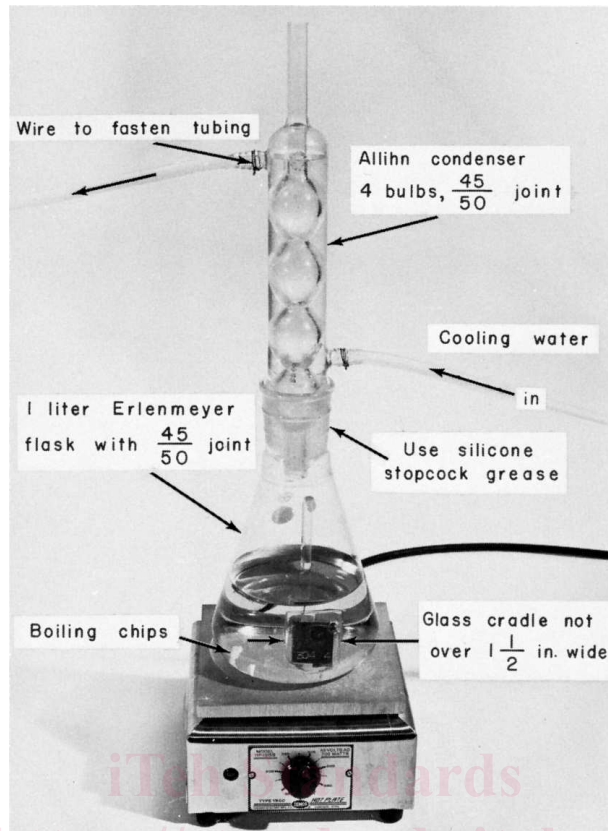


FIG. 8 Apparatus for Ferric Sulfate-Sulfuric Acid Test

18.1.5 High Vacuum Silicone Grease—A silicone grease is recommended for the ground glass joint.

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

18.1.7 During An testing, there analytical balance 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. 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—A device such as an electrically heated hot plate that provides heat for continuous boiling of the solution. For storage of prepared specimens prior to testing.

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

NOTE 5—No substitutions for this equipment may be used. The cold-finger type of condenser with standard Erlenmeyer flasks may not be used.

## 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 ( $\text{H}_2(\text{SO}_4)$ ), 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: (Warning—Protect the eyes and use rubber gloves for handling acid. Place the test flask under a hood. 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 distilled water in a 500-mL graduate Type IV reagent water and pour into the Erlenmeyer flask.

20.1.3 Then measure 236.0 mL of reagent-grade sulfuric acid of a concentration that must be in the range from 95.0 to 98.0 % by weight in a 250-mL graduate 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.