



Designation: A262 – 15

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.

Current edition approved Sept. 1, 2015. Published September 2015. Originally approved in 1943. Last previous edition approved in 2014 as A262 – 14. DOI: 10.1520/A0262-15.

*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.

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.

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.

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 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 $250\times$ to $500\times$ for wrought steels and at about $250\times$ for cast steels.

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

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

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

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

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

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

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

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



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

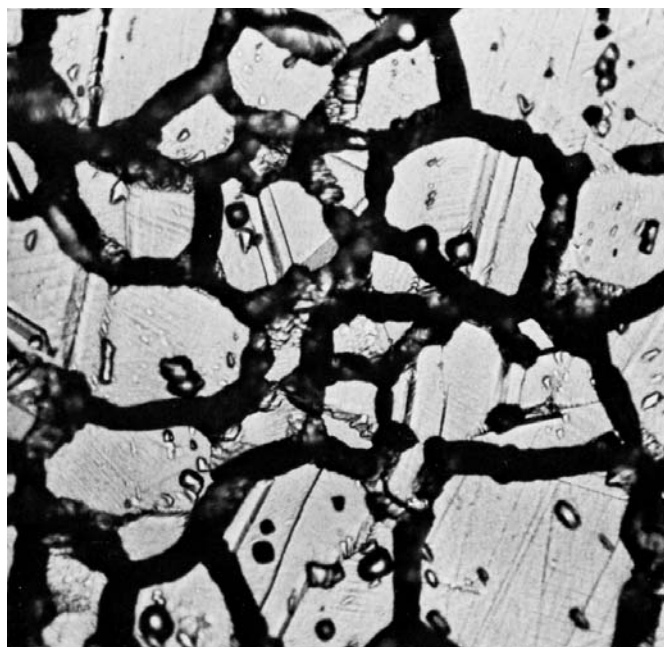


FIG. 3 Ditch Structure (500×) (One or More Grains Completely Surrounded by Ditches)

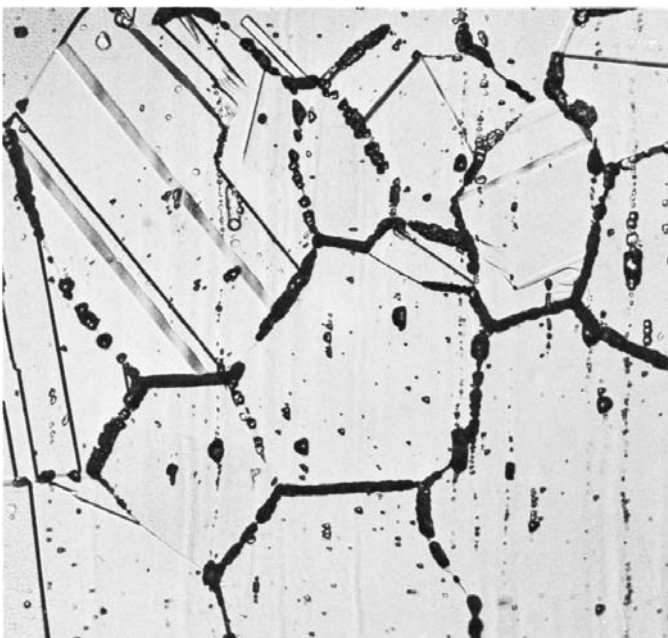


FIG. 2 Dual Structure (250×) (Some Ditches at Grain Boundaries in Addition to Steps, but No One Grain Completely Surrounded)

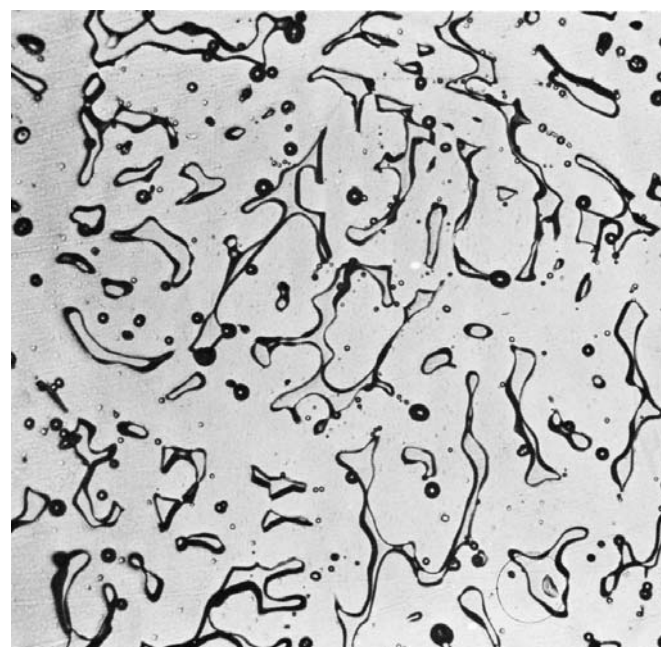


FIG. 4 Isolated Ferrite Pools (250×) (Observed in Castings and Welds. Steps Between Austenite Matrix and Ferrite Pools)

11.3.6 *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/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