



Designation: A 262 – 01

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

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This standard has been approved for use by agencies of the 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 3 to 7, inclusive),

1.1.2 *Practice B*—Ferric Sulfate–Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections 8 to 14, inclusive),

1.1.3 *Practice C*—Nitric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections 15 to 21, inclusive),

1.1.4 *Practice E*—Copper–Copper Sulfate–Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections 22 to 31, inclusive), and

1.1.5 *Practice F*—Copper–Copper Sulfate–50 % Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Molybdenum-Bearing Cast Austenitic Stainless Steels (Sections 32 to 38, inclusive).

1.2 The following factors govern the application of these practices:

1.2.1 Susceptibility to intergranular attack associated with the precipitation of chromium carbides is readily detected in all six tests.

1.2.2 Sigma phase in wrought chromium-nickel-molybdenum steels, which may or may not be visible in the microstructure, can result in high corrosion rates only in nitric acid.

1.2.3 Sigma phase in titanium or columbium stabilized alloys and cast molybdenum-bearing stainless alloys, which may or may not be visible in the microstructure, can result in high corrosion rates in both the nitric acid and ferric sulfate–sulfuric acid solutions.

1.3 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 precipi-

tates. These specimens will have low corrosion rates in certain corrosion tests and therefore can be eliminated (screened) from testing as “acceptable.”

1.4 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.

1.5 In most cases either the 24-h copper–copper sulfate–16 % sulfuric acid test or the 120-h ferric sulfate–sulfuric acid test, combined with the oxalic acid etch test, will provide the required information in the shortest time. All stainless grades listed in the accompanying table may be evaluated in these combinations of screening and corrosion tests, except those specimens of molybdenum-bearing grades (for example 316, 316L, 317, and 317L), which represent steel intended for use in nitric acid environments.

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

1.7 Only those stainless steel grades are listed in Table 1 for which data on the application of the oxalic acid etch test and on their performance in various quantitative evaluation tests are available.

1.8 Extensive test results on various types of stainless steels evaluated by these practices have been published in Ref (1).²

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

1.10 *This standard does not purport to address all of the safety problems, 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 statements are given in 5.6, 11.1.1, 11.1.9, and 35.1.)

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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² The boldface numbers in parentheses refer to the list of references found at the end of these practices.

2. Referenced Documents

2.1 ASTM Standards:

A 370 Test Methods and Definitions for Mechanical Testing of Steel Products³

2.2 ISO Standard:

ISO 5651-2 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 Acid⁴

PRACTICE A—OXALIC ACID ETCH TEST FOR CLASSIFICATION OF ETCH STRUCTURES OF AUSTENITIC STAINLESS STEELS 2

3. Scope

3.1 The oxalic acid etch test is used for acceptance of material but not for rejection of material. This may be used in connection with other evaluation tests to provide a rapid method for identifying those specimens that are certain to be free of susceptibility to rapid intergranular attack in these other tests. Such specimens have low corrosion rates in the various hot acid tests, requiring from 4 to 240 h of exposure. These specimens are identified by means of their etch structures, which are classified according to the following criteria:

3.2 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.

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 possibly nonacceptable performance in that particular test. Specimens having acceptable etch structures need not be subjected to the hot acid test. Specimens having nonacceptable etch structures must be tested in the specified hot acid solution.

3.3 The grades of stainless steels and the hot acid tests for which the oxalic acid etch test is applicable are listed in Table 2.

3.4 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).

4. 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 1).

4.3 *Variable Resistance* (Note 1).

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.

TABLE 1 Application of Evaluation Tests for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels

NOTE 1—For each corrosion test, the types of susceptibility to intergranular attack detected are given along with the grades of stainless steels in which they may be found. These lists may contain grades of steels in addition to those given in the rectangles. In such cases, the acid corrosion test is applicable, but not the oxalic acid etch test.

NOTE 2—The oxalic acid etch test may be applied to the grades of stainless steels listed in the rectangles when used in connection with the test indicated by the arrow.

OXALIC ACID ETCH TEST

↓	↓	↓	↓
AISI ^A : 304, 304L	AISI: 304, 304L, 316, 316L, 317, 317L	AISI: 201, 202, 301, 304, 304L, 304H, 316, 316L, 316H, 317, 317L, 321, 347	ACI: CF-3M, CF-8M,
ACI ^B : CF-3, CF-8	ACI: CF-3, CF-8, CF-3M, CF-8M		
Nitric Acid Test ^C (240 h in boiling solution)	Ferric Sulfate–Sulfuric Acid Test (120 h in boiling solution)	Copper–Copper Sulfate–Sulfuric Acid Test (24 h in boiling solution)	Copper–Copper Sulfate–50 % Sulfuric Acid Testing Boiling Solution
Chromium carbide in: 304, 304L, CF-3, CF-8 Chromium carbide and sigma phase in: ^D 316, 316L, 317, 317L, 321, 347, CF-3M, CF-8M End-grain in: all grades	Chromium carbide in: 304, 304L, 316, 316L, 317, 317L, CF-3, CF-8 Chromium carbide and sigma phase in: 321, CF-3M, CF-8M ^E	Chromium carbide in: 201, 202, 301, 304, 304L, 316, 316L, 317, 317L, 321, 347	Chromium carbide in: CF-3M, CF-8M

^A AISI: American Iron and Steel Institute designations for austenitic stainless steels.

^B ACI: Alloy Casting Institute designations.

^C The nitric acid test may be also applied to AISI 309, 310, 348, and AISI 410, 430, 446, and ACI CN-7M.

^D Must be tested in nitric acid test when destined for service in nitric acid.

^E To date, no data have been published on the effect of sigma phase on corrosion of AISI 347 in this test.

TABLE 2 Applicability of Etch Test

	AISI Grade No.	ACI Grade No.
Practice B—Ferric Sulfate—Sulfuric Acid Test	304, 304L, 316, 316L, 317, 317L	CF-3, CF-8, CF-3M, CF-8M
Practice C—Nitric Acid Test	304, 304L	CF-8, CF-3
Practice E—Copper—Copper Sulfate—16 % Sulfuric Acid Test	201, 202, 301, 304, 304L, 304H, 316, 316L, 316H, 317, 317L, 321, 347	...
Practice F—Copper—Copper Sulfate—50 % Sulfuric Acid Test	...	CF-8M, CF-3M

4.7 *Electrodes of the 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.

4.8 *Electrolyte*—Oxalic acid, (H₂C₂O₄·2H₂O), reagent grade, 10 weight % solution.

NOTE 1—The variable resistance and the ammeter are placed in the circuit to measure and control the current on the specimen to be etched.

5. Preparation of Test Specimens

5.1 *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.).

5.2 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 surface to be exposed in service. Only such surface finishing should be performed as is required to remove foreign material and obtain a standard, uniform finish as described in 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.

5.3 *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, ½, ⅓, ¼, ⅕, and ⅙, 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 area of 1 cm² or more is desirable. If any cross-sectional dimension is less than 1 cm, a minimum length of 1 cm should be polished. When the available length is less than 1 cm, a full cross section should be used.

5.4 *Etching Solution*—The solution used for etching is prepared by adding 100 g of reagent grade oxalic acid crystals

(H₂C₂O₄·2H₂O) 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 A/cm² 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.

5.6 Etching Precautions:

5.6.1 **Caution**—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.

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 % HNO₃.

5.6.3 The temperature of the etching solution gradually increases during etching. The temperature should be kept below 50°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, (NH₄)₂S₂O₈, may be used in place of oxalic acid. An etch of 5 or 10 min at 1 A/cm² in a solution at room temperature readily develops step structures on such specimens.

6. Classification of Etch Structures

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

6.2 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.

6.3 The etch structures are classified into the following types (Note 2):

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

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

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

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

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

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

6.3.7 *End-Grain Pitting II* (Fig. 7)—Structure contains numerous, deep end-grain pits at 500 \times . (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.

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

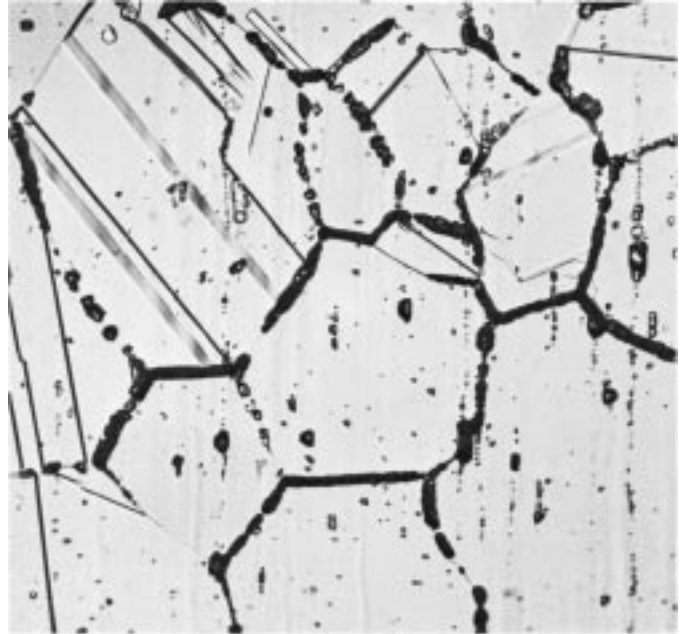


FIG. 2 Dual Structure (250 \times) (Some ditches at grain boundaries in addition to steps, but no one grain completely surrounded)

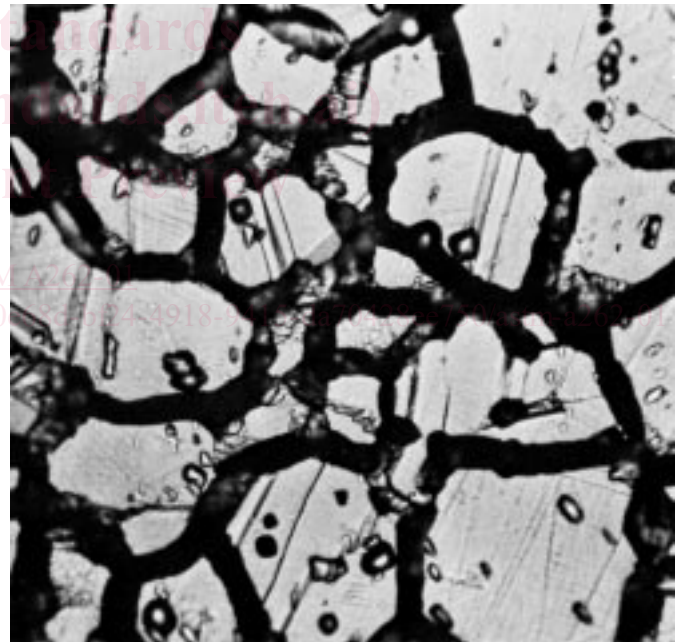


FIG. 3 Ditch Structure (500 \times) (One or more grains completely surrounded by ditches)



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

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.

6.5 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

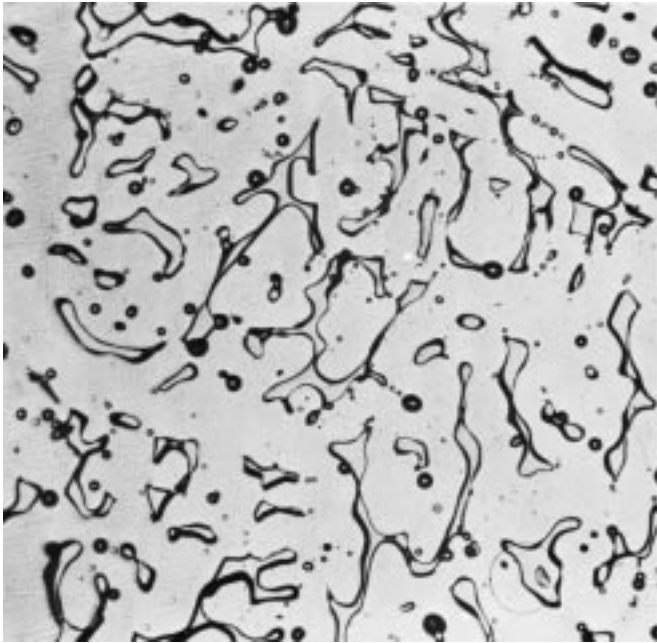
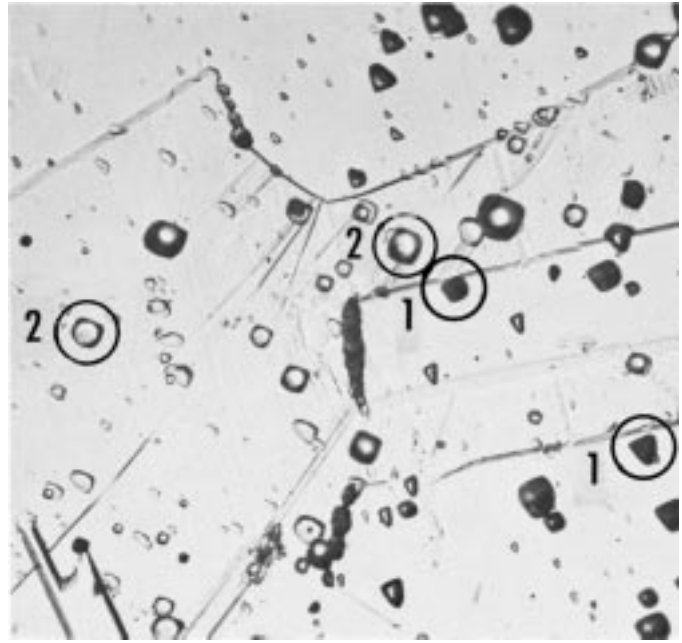


FIG. 4 Isolated Ferrite Pools (250×) (Observed in castings and welds. Steps between austenite matrix and ferrite pools)



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))

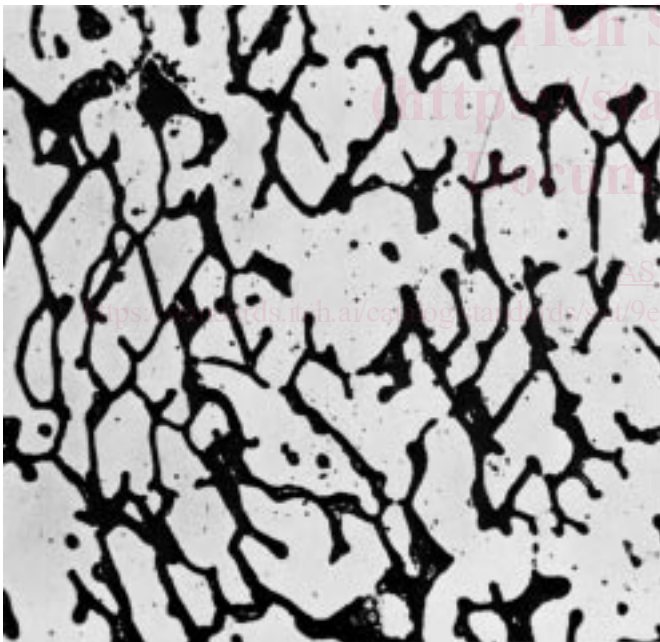
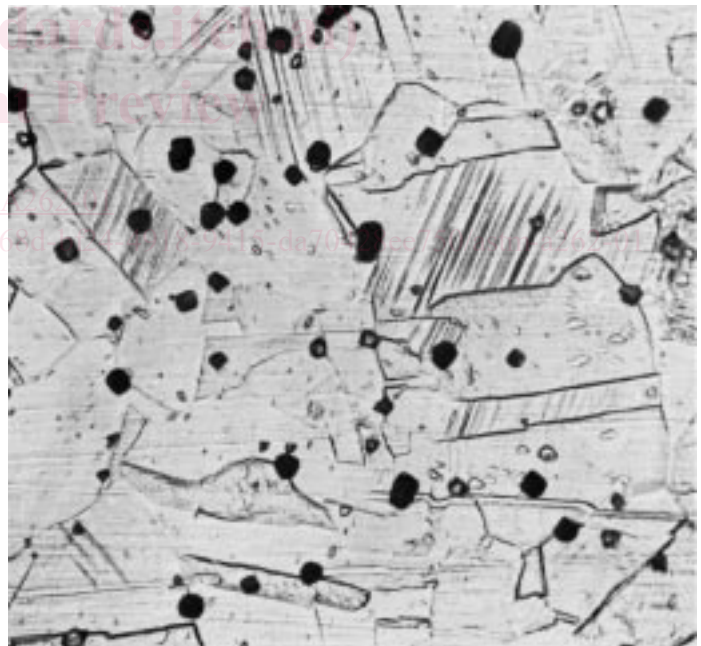


FIG. 5 Interdendritic Ditches (250×) (Observed in castings and welds. Deep interconnected ditches)



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×)

whenever it is specified. Such sharp, deep pits should not be confused with the shallow pits shown in Fig. 1 and Fig. 6.

7. Use of Etch Structure Classifications

7.1 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. Important characteristics of each of these tests are described below.

7.2 Practice B—Ferric Sulfate–Sulfuric Acid Test is a 120-h test in boiling 50 % solution that detects susceptibility to

intergranular attack associated primarily with chromium carbide precipitate. It does not detect susceptibility associated with sigma phase in wrought chromium-nickel-molybdenum stainless steels (316, 316L, 317, 317L), which is known to lead to rapid intergranular attack only in certain nitric acid environments. It does not detect susceptibility to end grain attack,

which is also found only in certain nitric acid environments. The ferric sulfate-sulfuric acid test does reveal susceptibility associated with a sigma-like phase constituent in stabilized stainless steels, AISI 321 and 347, and in cast chromium-nickel-molybdenum stainless steels (CF-8M, CF-3M, C6-8M, and CG-3M).

7.3 *Practice C—Nitric Acid Test* is a 240-h test in boiling, 65 % nitric acid that detects susceptibility to rapid intergranular attack associated with chromium carbide precipitate and with sigma-like phase precipitate. The latter may be formed in molybdenum-bearing and in stabilized grades of austenitic stainless steels and may or may not be visible in the microstructure. This test also reveals susceptibility to end grain attack in all grades of stainless steels.

7.4 *Practice E—Copper–Copper Sulfate–16 % Sulfuric Acid Test* is a 24-h test in a boiling solution containing 16 % sulfuric acid and 6 % copper sulfate with the test specimen embedded in metallic copper shot or grindings, which detects susceptibility to intergranular attack associated with the precipitation of chromium-rich carbides. It does not detect susceptibility to intergranular attack associated with sigma phase or end-grain corrosion, both of which have been observed to date only in certain nitric acid environments.

7.5 *Practice F—Copper–Copper Sulfate–50 % Sulfuric Acid Test* is a 120-h test in a boiling solution that contains 50 % sulfuric acid, copper sulfate, and metallic copper and that detects susceptibility to intergranular attack associated with the precipitation of chromium-rich carbides. It does not detect susceptibility to attack associated with sigma phase.

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

8. Scope <https://standards.iteh.ai/catalog/standards/sist/9e50>

8.1 This practice describes the procedure for conducting the boiling 120-h ferric sulfate–50 % sulfuric acid test (Note 3) 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.

NOTE 3—See Practice A for information on the most appropriate of the several test methods available for the evaluation of specific grades of stainless steel.

8.1.1 The ferric sulfate–sulfuric acid test detects susceptibility to intergranular attack associated with the precipitation of chromium carbides in unstabilized austenitic stainless steels. It does not detect susceptibility to intergranular attack associated with sigma phase in wrought austenitic stainless steels containing molybdenum, such as Types 316, 316L, 317, and 317L. The ferric sulfate–sulfuric acid test will detect intergranular corrosion associated with sigma phase in the cast stainless steels CF-3M and CF-8M.

NOTE 4—To detect susceptibility to intergranular attack associated with sigma phase in austenitic stainless steels containing molybdenum, the

nitric acid test, Practice C, should be used.

8.2 In stabilized stainless steel, Type 321 (and perhaps 347) and cast austenitic stainless steels containing molybdenum such as Types CF-8M, CF-3M, CG-8M, and CG-3M, the ferric sulfate–sulfuric acid test detects susceptibility associated with precipitated chromium carbides and with a sigma phase that may be invisible in the microstructure.

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

8.4 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).

9. Rapid Screening Test

9.1 Before testing in the ferric sulfate sulfuric acid test, specimens of certain grades of stainless steels (see Table 3) 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 3.

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 nonacceptable etch structures must be tested in the ferric sulfate-sulfuric acid test.

10. Apparatus

10.1 The apparatus (Note 6) is illustrated in Fig. 8.

TABLE 3 Use of Etch Structure Classifications from the Oxalic Acid Etch Test with Ferric Sulfate–Sulfuric Acid Test

NOTE 1—Grades AISI 321 and 347 cannot be screened because these grades may contain a type of sigma phase which is not visible in the etch structure but which may cause rapid corrosion in the ferric sulfate-sulfuric acid test.

Grade	Acceptable Etch Structures	Nonacceptable Etch Structures ^A
AISI 304	Step, dual, end grain, I & II	Ditch
AISI 304L	Step, dual, end grain, I & II	Ditch
AISI316	Step, dual, end grain, I & II	Ditch
AISI316L	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
AISI 321	None	...
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

^A Specimens having these structures must be tested in the ferric sulfate-sulfuric acid test.