Designation: A 262 – 02a (Reapproved 2008)

Standard Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels[1]

This standard is issued under the fixed designation A 262; 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 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 precipitates. 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 15-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.

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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 ACI[B]: CF-3, CF-8	AISI: 304, 304L, 316, 316L, 317, 317L ACI: CF-3, CF-8, CF-3M, CF-8M	AISI: 201, 202, 301, 304, 304L, 304H, 316, 316L, 316H, 317, 317L, 321, 347	ACI: 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 (15 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.

2. Referenced Documents

2.1 *ASTM Standards:* [3]

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

2.2 *ISO Standard:*[4]

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

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.

[3] 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.

[4] Available from International Organization for Standardization (ISO), 1 rue de Varembé, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

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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

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

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

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, ($H_2C_2O_4 \cdot 2H_2O$), 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 , ⅛ , ¼ , and ⅜ , 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 cm^2 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 ($H_2C_2O_4 \cdot 2H_2O$) 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/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.

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 % HNO_3.

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, $(NH_4)_2S_2O_8$, may be used in place of oxalic acid. An etch of 5 or 10 min at 1 A/cm^2 in a solution at room temperature readily develops step structures on such specimens.

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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×. (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×. (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^2.

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.

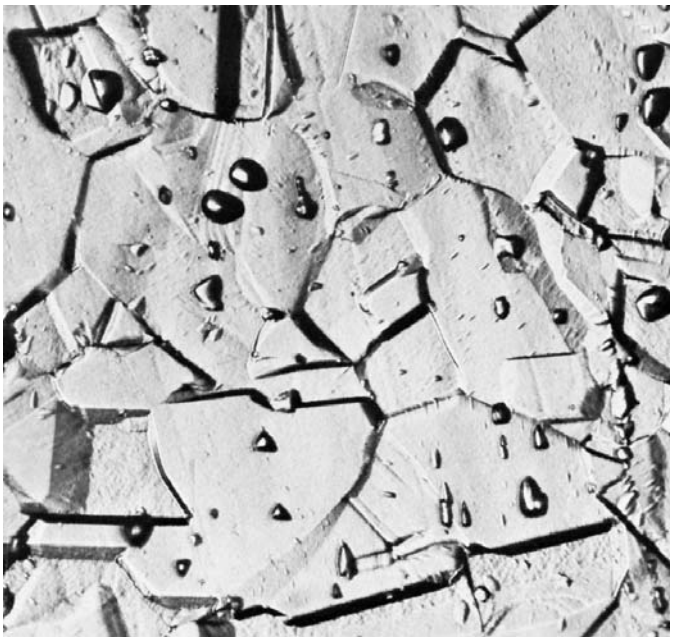


FIG. 1 Step Structure (500×) (Steps between grains, no ditches at grain boundaries)

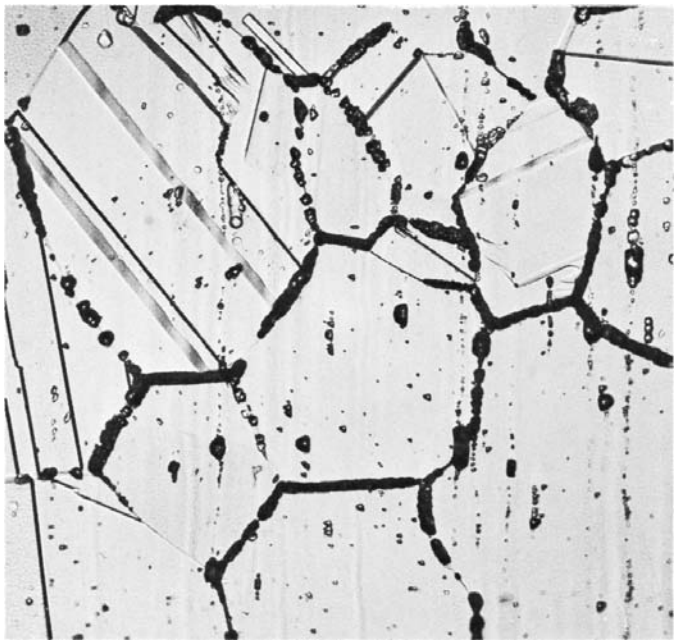


FIG. 2 Dual Structure (250×) (Some ditches at grain boundaries in addition to steps, but no one grain completely surrounded)

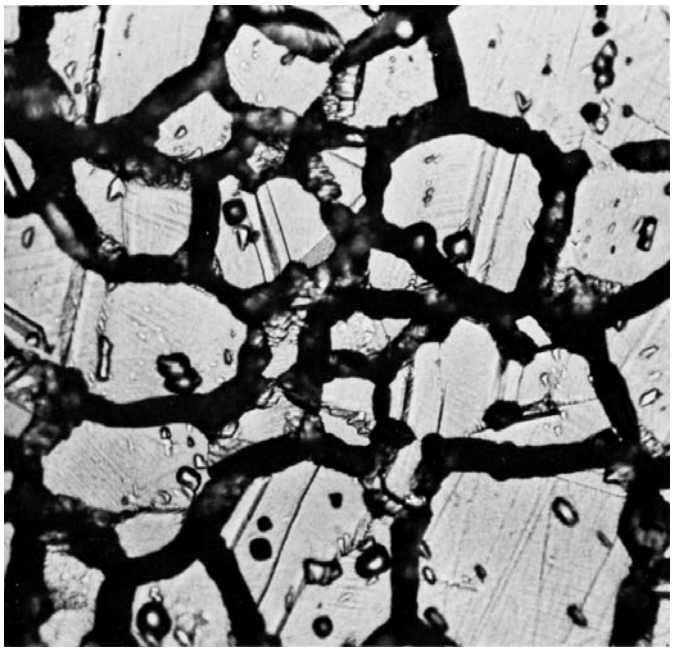


FIG. 3 Ditch Structure (500×) (One or more grains completely surrounded by ditches)

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

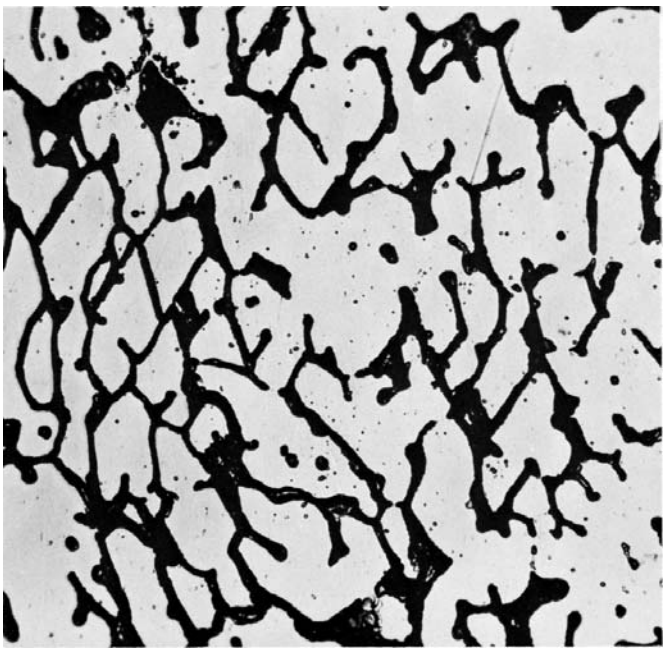
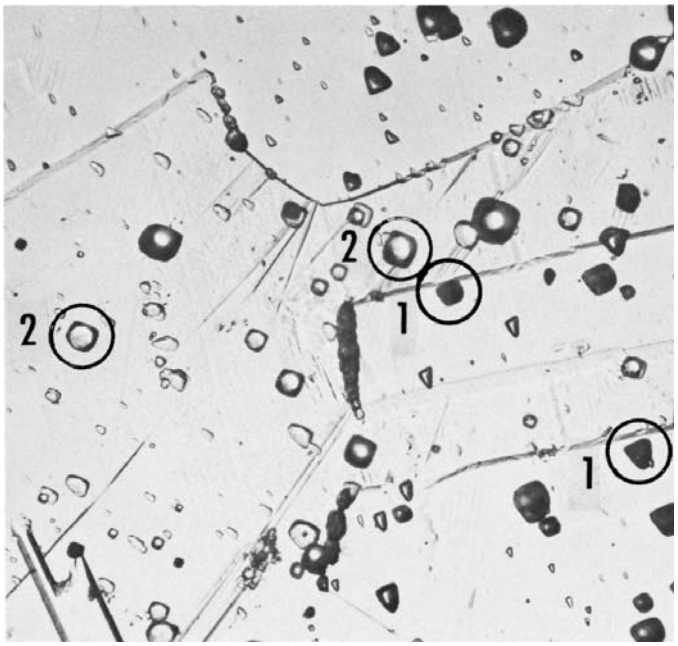


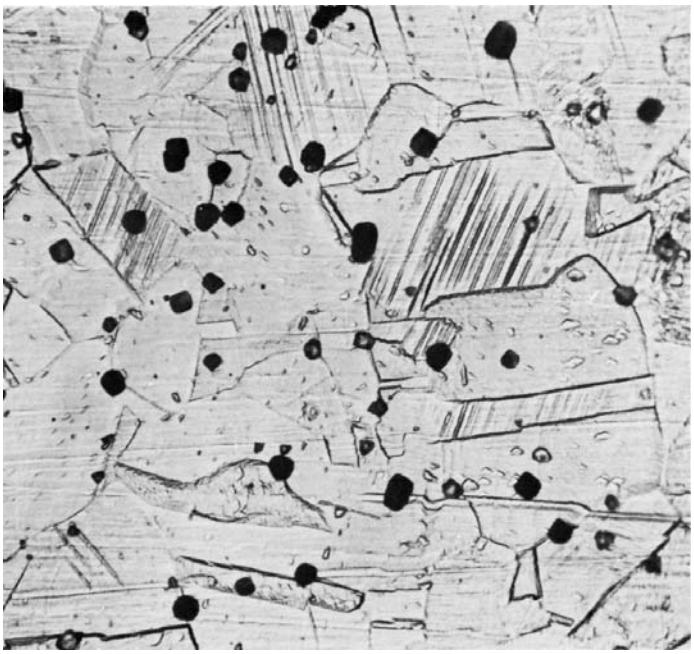
FIG. 5 Interdendritic Ditches (250×) (Observed in castings and welds. Deep interconnected ditches)

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



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))



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×)

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.

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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 15-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

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.

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.

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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.

10.1.1 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, 9½ in. (241 mm).

10.1.2 A 1-L Erlenmeyer flask with a 45/50 ground glass joint. The ground glass opening is somewhat over 38 mm (1½ in.) wide.

10.1.3 The glass cradle (Note 5) 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 (1½ in.), and the front-to-back distance must be such that the cradle will fit the 34-mm (1⅓-in.) diameter opening. It should have three or four holes to increase circulation of the testing solution around the specimen.

NOTE 5—Other equivalent means of specimen support, such as glass hooks or stirrups, may also be used.

10.1.4 Boiling chips must be used to prevent bumping.

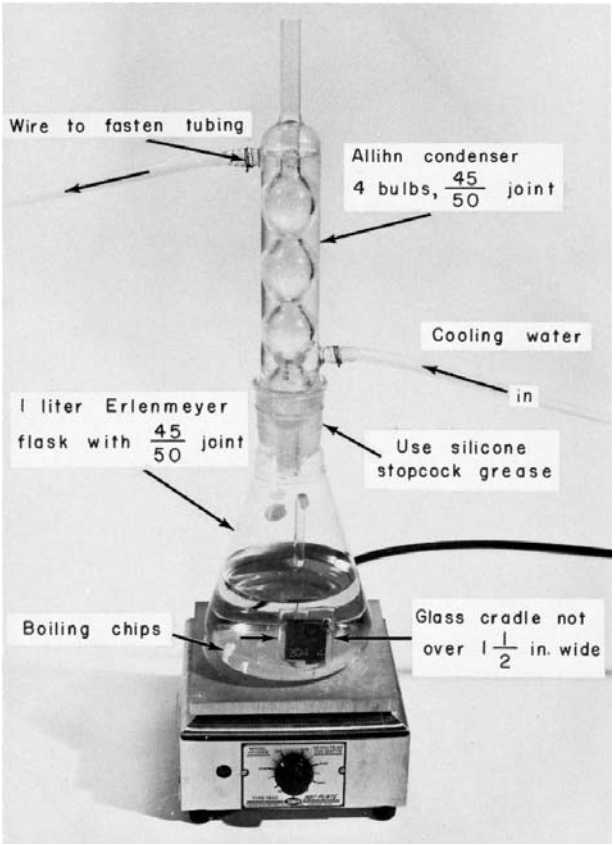


FIG. 8 Apparatus for Ferric Sulfate–Sulfuric Acid Test

10.1.5 A silicone grease[5] is recommended for the ground glass joint.

10.1.6 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.

10.1.7 A device such as an electrically heated hot plate that provides heat for continuous boiling of the solution.

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

NOTE 6—No substitutions for this equipment may be used. The cold-finger type of condenser with standard Erlenmeyer flasks may not be used.

11. Ferric Sulfate–Sulfuric Acid Test Solution

11.1 Prepare 600 mL of 50 % (49.4 to 50.9 %) solution as follows:

11.1.1 **Warning:**Protect the eyes and use rubber gloves for handling acid. Place the test flask under a hood.

11.1.2 First, measure 400.0 mL of distilled water in a 500-mL graduate and pour into the Erlenmeyer flask.

11.1.3 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. Add the acid slowly to the water in the Erlenmeyer flask to avoid boiling by the heat evolved.

NOTE 7—Loss of vapor results in concentration of the acid.

11.1.4 Weigh 25 g of reagent-grade ferric sulfate (contains about 75 % $Fe_2(SO_4)_3$) and add to the sulfuric acid solution. A trip balance may be used.

11.1.5 Drop boiling chips into the flask.

11.1.6 Lubricate ground glass joint with silicone grease.

11.1.7 Cover flask with condenser and circulate cooling water.

11.1.8 Boil solution until all ferric sulfate is dissolved (see Note 7).

11.1.9 **Warning:** It has been reported that violent boiling resulting in acid spills can occur. It is important to ensure that the concentration of acid does not become more concentrated and that an adequate number of boiling chips (which are resistant to attack by the test solution) are present.[6]

12. Preparation of Test Specimens

12.1 A specimen having a total surface area of 5 to 20 cm^2 is recommended. Specimens containing welds should be cut so that no more than 13-mm (½-in.) width of base metal is included on either side of the weld.

12.2 The intent is to test a specimen representing as nearly as possible the surface of the material as used in service. Only such surface finishing should be performed as is required to remove foreign material and obtain a standard, uniform finish

[5] Dow Corning Stopcock Grease has been found satisfactory for this purpose.

[6] The sole source of supply of the apparatus known to the committee at this time is Amphoteric alundum granules, Hengar Granules, from the Hengar Co., Philadelphia, PA. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

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as specified. For very heavy sections, specimens should be machined to represent the appropriate surface while maintaining reasonable specimen size for convenience in 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 remove the carburized surface completely. Such treatment of test specimens is not permissible, except in tests undertaken to demonstrate such surface effects.

12.3 When specimens are cut by shearing, the sheared edges should be refinished by machining or grinding prior to testing.

12.4 Any surfaces of the specimen, including sheared edges, that are to be refinished should be done by using No. 80 or 120 grit abrasive paper. If dry abrasive paper is used, polish slowly to avoid overheating. Sand blasting should not be used.

12.5 All traces of oxide scale formed during heat treatments must be thoroughly removed. Any scale that cannot be removed by grinding (for example, in stamped numbers) must be removed by immersing the specimen in concentrated nitric acid at about 93°C (200°F). (Residual oxide scale causes galvanic action and consequent activation in the test solution.)

12.6 The specimen should be measured, including the inner surfaces of any holes, and the total exposed area calculated.

12.7 The specimen should then be degreased and dried using suitable nonchlorinated agents, such as soap and acetone, and then weighed to the nearest 0.001 g.

13. Procedure

13.1 Place specimen in glass cradle and immerse in boiling solution.

13.2 Mark liquid level on flask with wax crayon to provide a check on vapor loss, which would result in concentration of the acid. If there is an appreciable change in the level, the test must be repeated with fresh solution and a reground specimen.

13.3 Continue immersion of the specimen for a total of 120 h, then remove specimen, rinse in water and acetone, and dry.

13.4 Weigh specimen and subtract weight from original weight.

13.5 No intermediate weighings are usually necessary. The tests can be run without interruption for 120 h. However, if preliminary results are desired, the specimen can be removed at any time for weighing.

13.6 No changes in solution are necessary during the 120-h test periods.

13.7 Additional ferric sulfate inhibitor may have to be added during the test if the corrosion rate is extraordinarily high as evidenced by a change in the color of the solution. More ferric sulfate must be added if the total weight loss of all specimens exceeds 2 g. (During the test, ferric sulfate is consumed at a rate of 10 g for each 1 g of dissolved stainless steel.)

13.8 Several specimens may be tested simultaneously. The number (3 or 4) is limited only by the number of glass cradles that can be fitted into the flask.

14. Calculation and Report

14.1 The effect of the acid solution on the material shall be measured by determining the loss of weight of the specimen. The corrosion rates should be reported as millimetres of penetration per month (Note 8), calculated as follows:

$$\text{Millimetre per month} = (7290 \times W)/(A \times t \times d) \quad (1)$$

where:

t = time of exposure, h,
A = area, cm^2,
W = weight loss, g, and
d = density, g/cm^3

for chromium-nickel steels, $d = 7.9$ g/cm^3
for chromium-nickel-molybdenum steels, $d = 8.00$ g/cm^3

NOTE 8—Conversion factors to other commonly used units for corrosion rates are as follows:
Millimetres per month × 0.0^4= inches per month
Millimetres per month × 0.4^7= inches per year
Millimetres per month × 12 = millimetres per year
Millimetres per month × 472 = mils per year
Millimetres per month × 1000 × density/3 = milligrams per square decimetre per day
Millimetres per month × 1.39 × density = grams per square metre per hour

PRACTICE C—NITRIC ACID TEST FOR DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEELS

15. Scope

15.1 This practice describes the procedure for conducting the boiling nitric acid test (**4**) as employed to measure the relative susceptibility of austenitic 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; in particular, it does not provide a basis for predicting resistance to forms of corrosion other than intergranular, such as general corrosion, pitting, or stress-corrosion cracking.

15.2 The boiling nitric acid test may be used to evaluate the heat treatment accorded "as-received" material. It is also sometimes used to check the effectiveness of stabilizing elements and of reductions in carbon content in preventing susceptibility to rapid intergranular attack.

NOTE 9—Intergranular attack in nitric acid is associated with one or more of the following: (*1*) intergranular precipitation of chromium carbides, (*2*) sigma or transition phases in molybdenum-bearing grades, and (*3*) sigma phase constituents in stabilized grades. The boiling nitric acid test should not be used for extra-low–carbon molybdenum-bearing grades unless the material tested is to be used in nitric acid service. See Practice A, Oxalic Acid Etching Test, for information on the most appropriate of the several test methods available for the evaluation of specific grades of stainless steel.

15.3 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 used for this sensitizing treatment determines the maximum permissible corrosion rate

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in the nitric acid test. The most commonly used sensitizing treatment is 1 h at 675°C (1250°F).

15.4 This practice may be applied to wrought products (including tubes), castings, and weld metal of the various grades of stainless steel (Note 9).

16. Rapid Screening Test

16.1 Before testing in the nitric acid test, specimens of certain grades of stainless steel as given in 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. The use of the etch structure evaluations in connection with the nitric acid test is specified in Table 4.

16.1.1 Corrosion test specimens having acceptable etch structures in the oxalic acid etch test will be essentially free of intergranular attack in the nitric acid test; such specimens are acceptable without testing in the nitric acid test. All specimens having nonacceptable etch structures must be tested in the nitric acid test.

17. Apparatus

17.1 *Container*—A 1-L Erlenmeyer flask equipped with a cold finger-type condenser, as illustrated in Fig. 9, is recommended.

NOTE 10—Two other types of containers have been employed in the past and may be used if agreed upon between the supplier and purchaser. One of these consists of a 1-L Erlenmeyer flask with a ground glass joint and equipped with a 762-mm (30-in.) reflux condenser; it has been shown that results obtained with a reflux condenser tend to be somewhat higher than with the cold finger-type condenser due to greater vapor loss. The second type of container is the so-called multi-sample testing apparatus (**5**) which was designed to permit the testing of a large number of specimens simultaneously by providing for replacement of the acid in contact with the specimens several times per hour with redistilled acid. Because of the lesser accumulation of corrosion products in the testing solution, the rates obtained with the multi-sample tester are consistently lower than those obtained with the conventional apparatus; the differences are small on properly annealed or stabilized material, which will show low rates in both types of test but can be very large for sensitized specimens. For research purposes or where results are to be compared directly, it is essential that the same type of apparatus be used for all tests.

17.2 *Specimen Supports*—Glass hooks, stirrups, or cradles for supporting the specimens in the flask fully immersed at all times during the test and so designed that specimens tested in the same container do not come in contact with each other.

TABLE 4 Use of Etch Structure Classification from Oxalic Acid Etch Test with Nitric Acid Test

NOTE 1—AISI 316, 316L, 317, 317L, 347, and 321 cannot be screened because these steels may contain sigma phase not visible in the etch structure. This may cause rapid intergranular attack in the nitric acid test.

Grade	Acceptable Etch Structures	Nonacceptable Etch Structures [A]
AISI 304	Step, dual, end grain I	Ditch, end grain II
AISI 304L	Step, dual, end grain I	Ditch, end grain II
ACI CF-8	Step, dual, isolated ferrite pools	Ditch, interdendritic ditches
ACI CF-3	Step, dual, isolated ferrite pools	Ditch, interdendritic ditches

[A] Specimens having these structures must be tested in the nitric acid test.

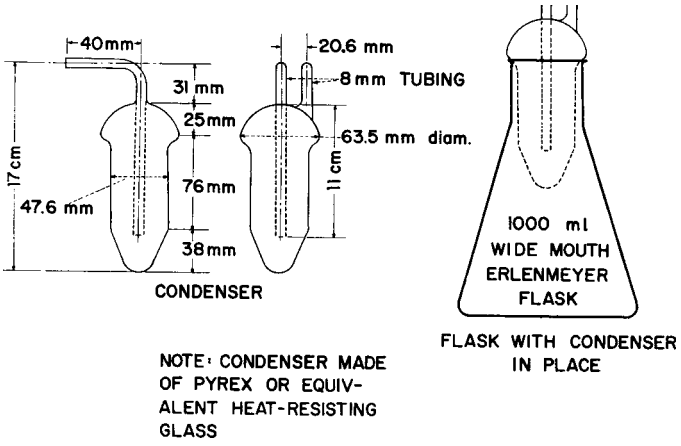


FIG. 9 Flask and Condenser for Nitric Acid Test

17.3 *Heater*—A means for heating the test solutions and of keeping them boiling throughout the test period. An electrically heated hot plate is satisfactory for this purpose.

17.4 *Balance*—An analytical balance capable of weighing to at least the nearest 0.001 g.

18. Nitric Acid Test Solution

18.1 The test solution shall be 65 ± 0.2 weight % as nitric acid determined by analysis. This solution may be prepared by adding distilled water to concentrated nitric acid (reagent grade HNO_3, sp gr 1.42) (Note 11) at the rate of 108 mL of distilled water per litre of concentrated nitric acid.

NOTE 11—The nitric acid used should conform to the recommended specifications for analytical reagent chemicals of the American Chemical Society (**6**) as follows:

Nonvolatile matter, max, %	0.0005
Sulfate (SO_4), max, %	0.0002
Arsenic, max, %	0.000003
Chlorine, max, % about	0.00007
Heavy metals, max, % about lead and iron	0.0005 0.0001

In addition, the fluorine content shall not exceed 0.0001 %, and phosphate (PO_4) shall not exceed 0.00002 %.

19. Preparation of Test Specimens

19.1 The size and shape of the specimen must be considered with respect to available facilities for accurate weighing and the volume of test solution to be used. Normally, the maximum convenient weight of specimen is about 100 g. Specimens containing welds should be cut so that no more than 13 mm (½ in.) width of base metal is included on either side of the weld. Furthermore, in the case of bar, wire, and tubular products, the proportion of the total area represented by the exposed cross section may influence the results. Cross-sectional areas in these products may be subject to end grain attack in nitric acid. The proportion of end grain in the specimen should therefore be kept low unless such surface is actually to be exposed in service involving nitric acid. When specimens of such products are being tested in research investigations, the ratio of the cross-sectional area exposed to the total area should be kept constant from test to test. For inspection tests, specimens cut from bars, wires, or tubes should be proportioned so that the

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areas of the exposed cross sections shall not exceed half the total exposed area of the specimen.

19.2 Special heat treatment of specimens prior to testing or the use of specimens that contain a weld may be specified.

19.3 The intent is to test a specimen representing as nearly as possible the surface of the material as used in service. Only such surface finishing should be performed as is required to remove foreign material and obtain a standard uniform finish as specified. For very heavy sections, specimens should be machined to represent the appropriate surface while maintaining reasonable specimen size for convenience in 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 remove the carburized surface completely. Such treatment of test specimens is not permissible except in tests undertaken to demonstrate such surface effects.

19.4 When specimens are cut by shearing, the sheared edges should be refinished by machining or grinding prior to testing.

19.5 Any surfaces of the specimen, including sheared edges, that are to be refinished should be done by using No. 80 or 120 grit abrasive paper. If dry abrasive paper is used, polish slowly to avoid overheating. Sandblasting should not be used.

19.6 The specimen should be measured, including the inner surfaces of any holes, and the total exposed area calculated.

19.7 The specimen should then be degreased and dried using suitable nonchlorinated agents, such as soap and acetone (Note 12), and then weighed to the nearest 0.001 g (see 17.4).

NOTE 12—The cleaning treatment described may be supplemented by immersing the specimen in nitric acid (for example, 20 weight % at 49 to 60°C (120 to 140°F)) for 20 min, followed by rinsing, drying, and weighing. In the case of small-diameter tubular specimens which cannot be conveniently resurfaced on the inside, it is desirable to include in the preparation an immersion in boiling nitric acid (65 %) for 2 to 4 h using the same apparatus as for the actual test. The purpose of these treatments is to remove any surface contamination that may not be accomplished by the regular cleaning method and which may increase the apparent weight loss of the specimen during the early part of the test.

19.8 It is common practice to test only one specimen of each material or lot of material, as defined by those using the test for specification purposes. However, the use of at least two specimens for check purposes is recommended.

20. Procedure

20.1 Use a sufficient quantity of the nitric acid test solution to cover the specimens and to provide a volume of at least 20 mL/cm^2 (125 mL/in.^{2}) of specimen surface. Normally, a volume of about 600 cm^3 is used.

20.2 The best practice is to use a separate container for each test specimen.

NOTE 13—For routine evaluations, it is acceptable to test as many as three specimens in the same container provided that they all are of the same grade and all show satisfactory resistance to corrosion. If more than one of the specimens tested in the same container fail to pass the test, it is necessary to retest all specimens in separate containers, since excessive corrosion of one specimen may result in accelerated corrosion of the other specimens tested with it. Excessive corrosion may often be detected by changes in the color of the test solution, and it may be appropriate to provide separate containers for such specimens without waiting until the end of the test period. A record should be made showing which specimens were tested together.

NOTE 14—If the multi-sample testing apparatus (see Note 10) is employed, a large number of specimens may be tested in the large container provided.

20.3 After the specimens have been placed in the acid in the container, pass cooling water through the condenser and bring the acid to a boil on the hot plate and then keep boiling throughout the test period (Note 15). After each test period, rinse the specimens with water and treat by scrubbing with rubber or a nylon brush under running water to remove any adhering corrosion products, after which they should be dried and weighed. Drying may be facilitated, if desired, by dipping the specimens in acetone after they are scrubbed.

NOTE 15—Care should be taken to prevent contamination of the testing solution, especially by fluorides, either before or during the test. Experience has shown that the presence of even small amounts of hydrofluoric acid will increase the corrosion rate in the nitric acid test. It is not permissible, for example, to conduct nitric-hydrofluoric acid tests in the same hood with nitric acid tests.

20.4 For most consistent results, the test should consist of five boiling periods of 48 h each (Note 16) with a fresh test solution being used in each period.

NOTE 16—For specification purposes, those experienced in the use of the test may, by mutual agreement, shorten the standard test to three 48-h boiling periods. However, if with this shorter test procedure the rate of attack in the third period should exceed that in either the first or second periods to some previously agreed-upon extent, then the test should be continued for a total of five periods. As an alternative, when the test is being used for inspection prior to approval of steel for shipment, a procedure may be agreed upon by the purchaser and the manufacturer whereby the material will be released for shipment following satisfactory performance in three 48-h boiling periods with final acceptance being dependent upon satisfactory performance in the longer test of five 48-h boiling periods. Also, by mutual agreement, a combination of one 48-h period and two 96-h periods (not necessarily in that order) instead of five 48-h test periods may be acceptable for routine evaluations.

21. Calculation and Report

21.1 *Calculation*—The effect of the acid on the material shall be measured by determining the loss of weight of the specimen after each test period and for the total of the test periods. Such weight-loss determinations should be made with the accuracy prescribed in 17.4. The corrosion rates are usually reported as millimetres per month (Note 17), calculated in the following rate of corrosion equation:

$$\text{Millimetres per month} = (7290 \times W)/(A \times d \times t) \quad (2)$$

where:

t = time of exposure, h,
A = total surface area, cm^2,
W = weight loss, g, and
d = density of the sample, g/cm^3.

NOTE 17—Conversion factors to other commonly used units for corrosion rates are as follows:

Millimetres per month × 0.04 = inches per month
Millimetres per month × 0.47 = inches per year
Millimetres per month × 12 = millimetres per year
Millimetres per month × 472 = mils per year
Millimetres per month × 1000 × density/3 = milligrams per square

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decimetre per day
Millimetres per month × 1.39 × density = grams per square metre per hours

21.2 *Report*—Results should be reported for the individual periods, as well as the average for the three or five test periods.

PRACTICE E—COPPER–COPPER SULFATE–16 % SULFURIC ACID TEST FOR DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEELS 7,8

22. Scope

22.1 This practice describes the procedure by which the copper–copper sulfate–16 % sulfuric acid test is conducted to determine the susceptibility of austenitic stainless steels to intergranular attack. The presence or absence of intergranular corrosion in this test is not necessarily a measure of the performance of the material in other corrosive media. The test does not provide a basis for predicting resistance to other forms of corrosion, such as general corrosion, pitting, or stress-corrosion cracking.

22.2 The copper–copper sulfate–16 % sulfuric acid test indicates susceptibility to intergranular attack associated with the precipitation of chromium-rich carbides. It does not detect susceptibility associated with sigma phase. This test may be used to evaluate the heat treatment accorded as-received material. It may also be used to evaluate the effectiveness of stabilizing element additions (Cb, Ti, and so forth) and reductions in carbon content to aid in resisting intergranular attack.

22.3 All wrought products and weld material of austenitic stainless steels can be evaluated by this test.

23. Rapid Screening Test

23.1 Before testing in the copper–copper sulfate–16 % sulfuric acid test, specimens of certain grades of stainless steel (see Table 5) may be given a rapid screening test in accordance with the procedures given in Practice A (Sections 3 through 7). Preparation, etching, and the classification of etch structures are described therein. The use of etch-structure evaluations in connection with the copper–copper sulfate–16 % sulfuric acid test is specified in Table 5.

TABLE 5 Use of Etch Structure Classifications from the Oxalic Acid Etch Test with the Copper-Copper Sulfate-16 % Sulfuric Acid Test

Grade	Acceptable Etch Structures	Nonacceptable Etch Structures [A]
AISI 201	Step, dual, end grain I and II	Ditch
AISI 202	Step, dual, end grain I and II	Ditch
AISI 301	Step, dual, end grain I and II	Ditch
AISI 304	Step, dual, end grain I and II	Ditch
AISI 304L	Step, dual, end grain I and II	Ditch
AISI 304H	Step, dual, end grain I and II	Ditch
AISI 316	Step, dual, end grain I and II	Ditch
AISI 316L	Step, dual, end grain I and II	Ditch
AISI 316H	Step, dual, end grain I and II	Ditch
AISI 317	Step, dual, end grain I and II	Ditch
AISI 317L	Step, dual, end grain I and II	Ditch
AISI 321	Step, dual, end grain I and II	Ditch
AISI 347	Step, dual, end grain I and II	Ditch

[A] Specimens having these structures must be tested in the copper–copper sulfate–16 % sulfuric acid test.

23.1.1 Corrosion test specimens having acceptable etch structures in the oxalic acid etch test will be essentially free of intergranular attack in the copper–copper sulfate–16 % sulfuric acid test. Such specimens are acceptable without testing in the copper–copper sulfate–16 % sulfuric acid test. All specimens having nonacceptable etch structures must be tested in the copper–copper sulfate–16 % sulfuric acid test.

24. Summary of Practice

24.1 A suitable sample of an austenitic stainless steel, embedded in copper shot or grindings, is exposed to boiling acidified copper sulfate solution for 15 h. After exposure in the boiling solution, the specimen is bent. Intergranular cracking or crazing is evidence of susceptibility.

24.2 *Alternative Testing Procedures:*

24.2.1 Unless prohibited by the purchaser in the purchase order, the supplier is permitted to meet the requirements of Practice E by performing a test in accordance with ISO 3651–2, Method A, provided that the testing period shall be a minimum of 15 h. When a sensitization treatment is required, sensitization heat treatment T1 [700°C ± 10°C (1292°F ± 18°F), 30 minutes, water quench] shall be used unless the supplier and purchaser shall agree upon preparation of welded test pieces to be tested in the as-welded condition.

24.2.2 When this alternative test procedure is used, it shall be noted on the test report.

25. Apparatus

25.1 A 1-L glass Erlenmeyer flask with a ground 45/50 glass joint and four-bulb (minimum) Allihn condenser with 45/50 ground glass joint (as in 10.1.1 and 10.1.2 and Fig. 8) are required. A silicone grease is recommended for the ground glass joint.

25.2 *Specimen Supports*—An open glass cradle capable of supporting the specimens and copper shot or grindings in the flask is recommended.

NOTE 18—It may be necessary to embed large specimens, such as from heavy bar stock, in copper shot on the bottom of the test flask. A copper cradle may also be used.

25.3 *Heat Source*—Any gas or electrically heated hot plate may be utilized for heating the test solution and keeping it boiling throughout the test period.

26. Acidified Copper Sulfate Test Solution

26.1 Dissolve 100 g of copper sulfate ($CuSO_4 \cdot 5H_2O$) in 700 mL of distilled water, add 100 mL of sulfuric acid (H_2SO_4, cp, sp gr 1.84), and dilute to 1000 mL with distilled water.

NOTE 19—The solution will contain approximately 6 weight % of anhydrous $CuSO_4$ and 16 weight % of H_2SO_4.

27. Copper Addition

27.1 Electrolytic grade copper shot or grindings may be used. Shot is preferred for its ease of handling before and after the test.

27.2 A sufficient quantity of copper shot or grindings is to be used to cover all surfaces of the specimen whether it is in a

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vented glass cradle or embedded in a layer of copper shot on the bottom of the test flask.

27.3 The amount of copper used, assuming an excess of metallic copper is present, is not critical. The effective galvanic coupling between copper and the test specimen may have importance **(9)**.

27.4 The copper shot or grindings may be reused if they are cleaned in warm tap water after each test.

28. Specimen Preparation

28.1 The size of the sample submitted for test and the area from which it is to be taken (end or middle of coil, midway surface and center, and so forth) is generally specified in the agreement between the purchaser and the seller. The testing apparatus dictates the final size and shape of the test specimen. The specimen configuration should permit easy entrance and removal through the neck of the test container.

28.1.1 Table 6 may be used as a guide to determine acceptable specimen sizes. There may be restrictions placed on specimen size by the testing apparatus.

28.1.2 Specimens obtained by shearing should have the sheared edges machined or ground off prior to testing. Care should be taken when grinding to avoid overheating or "burning." A "squared" edge is desirable.

28.2 Any scale on the specimens should be removed mechanically unless a particular surface finish is to be evaluated. Chemical removal of scale is permissible when this is the case. Mechanical removal of scale should be accomplished with 120-grit iron-free aluminum oxide abrasive.

28.2.1 Each specimen should be degreased using a cleaning solvent such as acetone, alcohol, ether, or a vapor degreaser prior to being tested.

28.3 All austenitic material in the "as-received" (mill-annealed) condition should be capable of meeting this test.

TABLE 6 Sizes of Test Specimens

Type of Material	Size of Test Specimen
Wrought wire or rod:	
Up to 6 mm (¼ in.) in diameter, incl	Full diameter by 75 mm (3 in.) (min) long
Over 6 mm (¼ in.) in diameter	Cylindrical segment 6 mm (¼ in.) thick by 25 mm (1 in.) (max) wide by 75 to 125 mm (3 to 5 in.) long [A]
Wrought sheet, strip, plates, or flat rolled products:	
Up to 5 mm (3⁄16 in.) thick, incl	Full thickness by 9 to 25 mm (⅜ to 1 in.) wide by 75 mm (3 in.) (min) long
Over 5 mm (3⁄16 in.) thick	5 to 13 mm (3⁄16 to ½ in.) thick by 9 to 25 mm (⅜ to 1 in.) wide by 75 mm (3 in.) (min) long [B]
Tubing:	
Up to 38 mm (1½ in.) in diameter, incl	Full ring, 25 mm (1 in.) wide [C]
Over 38 mm (1½ in.) in diameter	A circumferential segment 75 mm (3 in.) (min) long cut from a 25 mm (1-in.) wide ring [D]

[A] When bending such specimens, the curved surface shall be on the outside of the bend.

[B] One surface shall be an original surface of the material under test and it shall be on the outside of the bend. Cold-rolled strip or sheets may be tested in the thickness supplied.

[C] Ring sections are not flattened or subjected to any mechanical work before they are subjected to the test solution.

[D] Specimens from welded tubes over 38 mm (1½ in.) in diameter shall be taken with the weld on the axis of the bend.

28.3.1 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 most commonly used sensitizing treatment is 1 h at 675°C. Care should be taken to avoid carburizing or nitriding the specimens. The heat treating is best carried out in air or neutral salt.

NOTE 20—The sensitizing treatment 675°C is performed to check the effectiveness of stabilized and 0.03 % maximum carbon materials in resisting carbide precipitation, hence, intergranular attack.

29. Test Conditions

29.1 The volume of acidified copper sulfate test solution used should be sufficient to completely immerse the specimens and provide a minimum of 8 mL/cm^2 (50 mL/in.^{2}) of specimen surface area.

29.1.1 As many as three specimens can be tested in the same container. It is ideal to have all the specimens in one flask to be of the same grade, but it is not absolutely necessary. The solution volume-to-sample area ratio is to be maintained.

29.1.2 The test specimen(s) should be immersed in ambient test solution, which is then brought to a boil and maintained boiling throughout the test period. Begin timing the test period when the solution reaches the boiling point.

NOTE 21—Measures should be taken to minimize bumping of the solution when glass cradles are used to support specimens. A small amount of copper shot (8 to 10 pieces) on the bottom of the flask will conveniently serve this purpose.

29.1.3 The time of the test shall be a minimum of 15 h, unless a longer time is agreed upon between the purchaser and the producer. If not 15 h, the test time shall be specified on the test report. Fresh test solution would not be needed if the test were to run 48 or even 72 h. (If any adherent copper remains on the specimen, it may be removed by a brief immersion in concentrated nitric acid at room temperature.)

NOTE 22—Results in the literature indicate that this test is more sensitive if it is run for longer times **(10), (11).**

30. Bend Test

30.1 The test specimen shall be bent through 180° and over a diameter equal to the thickness of the specimen being bent (see Fig. 10). In no case shall the specimen be bent over a smaller radius or through a greater angle than that specified in the product specification. In cases of material having low ductility, such as severely cold worked material, a 180° bend may prove impractical. Determine the maximum angle of bend without causing cracks in such material by bending an untested specimen of the same configuration as the specimen to be tested.

30.1.1 Duplicate specimens shall be obtained from sheet material so that both sides of the rolled samples may be bent through a 180° bend. This will assure detection of intergranular attack resulting from carburization of one surface of sheet material during the final stages of rolling.

NOTE 23—Identify the duplicate specimen in such a manner as to ensure both surfaces of the sheet material being tested are subjected to the tension side of the 180° bends.

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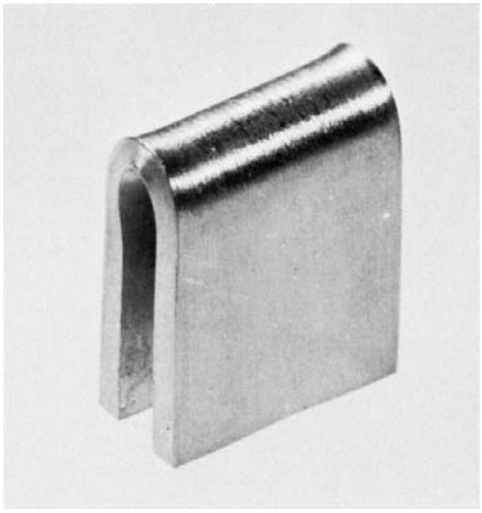


FIG. 10 A Bent Copper–Copper Sulfate–Sulfuric Acid Test Specimen

30.1.2 Samples machined from round sections or cast material shall have the curved or original surface on the outside of the bend.

30.1.3 The specimens are generally bent by holding in a vise and starting the bend with a hammer. It is generally completed by bringing the two ends together in the vise. Heavy specimens may require bending in a fixture of suitable design. An air or hydraulic press may also be used for bending the specimens.

30.1.4 Tubular products should be flattened in accordance with the flattening test, prescribed in Test Methods and Definitions A 370.

30.1.5 When agreed upon between the purchaser and the producer, the following shall apply to austenitic stainless steel plates 4.76 mm (0.1875 in.) and thicker:

30.1.5.1 Samples shall be prepared according to Table 6.

30.1.5.2 The radius of bend shall be two times the sample thickness, and the bend axis shall be perpendicular to the direction of rolling.

30.1.5.3 Welds on material 4.76 mm (0.1875 in.) and thicker shall have the above bend radius, and the weld-base metal interface shall be located approximately in the centerline of the bend.

30.1.5.4 Face, root, or side bend tests may be performed, and the type of bend test shall be agreed upon between the purchaser and the producer. The bend radius shall not be less than that required for mechanical testing in the appropriate material specification (for base metal) or in ASME Code Section IX (for welds).

31. Evaluation

31.1 The bent specimen shall be examined under low (5 to 20×) magnification (see Fig. 11). The appearance of fissures or cracks indicates the presence of intergranular attack (see Fig. 12).

31.1.1 When an evaluation is questionable (see Fig. 13), the presence or absence of intergranular attack shall be determined by the metallographic examination of the outer radius of a longitudinal section of the bend specimen at a magnification of 100 to 250×.

NOTE 24—Cracking that originates at the edge of the specimen should be disregarded. The appearance of deformation lines, wrinkles, or "orange

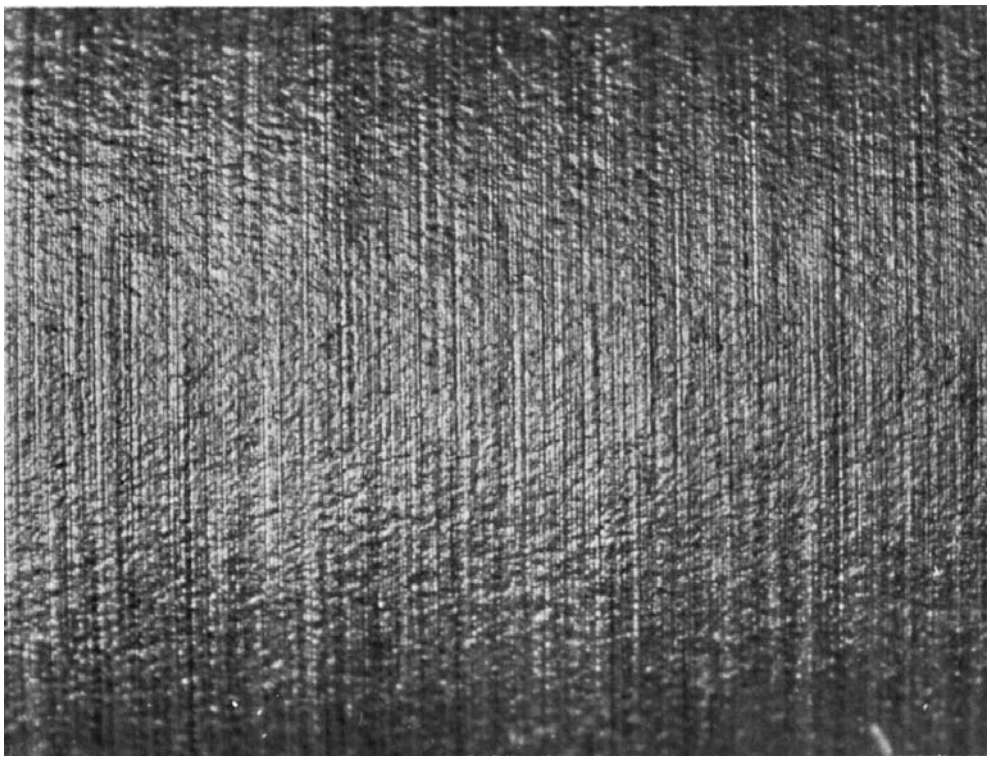


FIG. 11 Passing Test Specimen—View of the Bent Area (20× magnification before reproduction)

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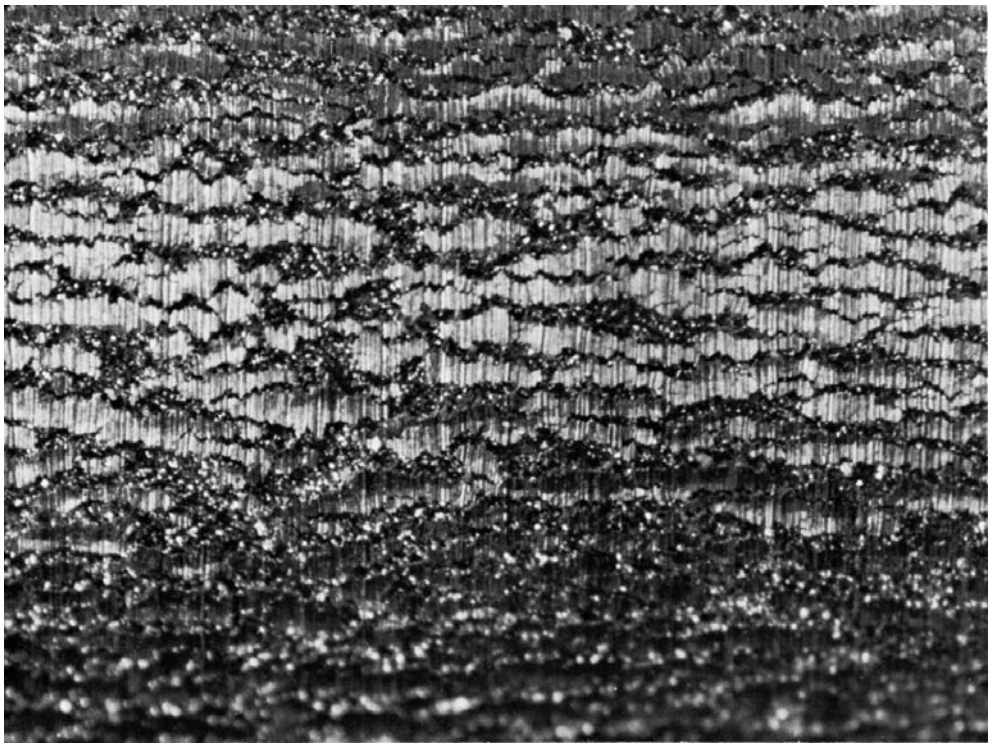


FIG. 12 Failing Test Specimen (Note the many intergranular fissures. Bent area at 20× magnification before reproduction.)

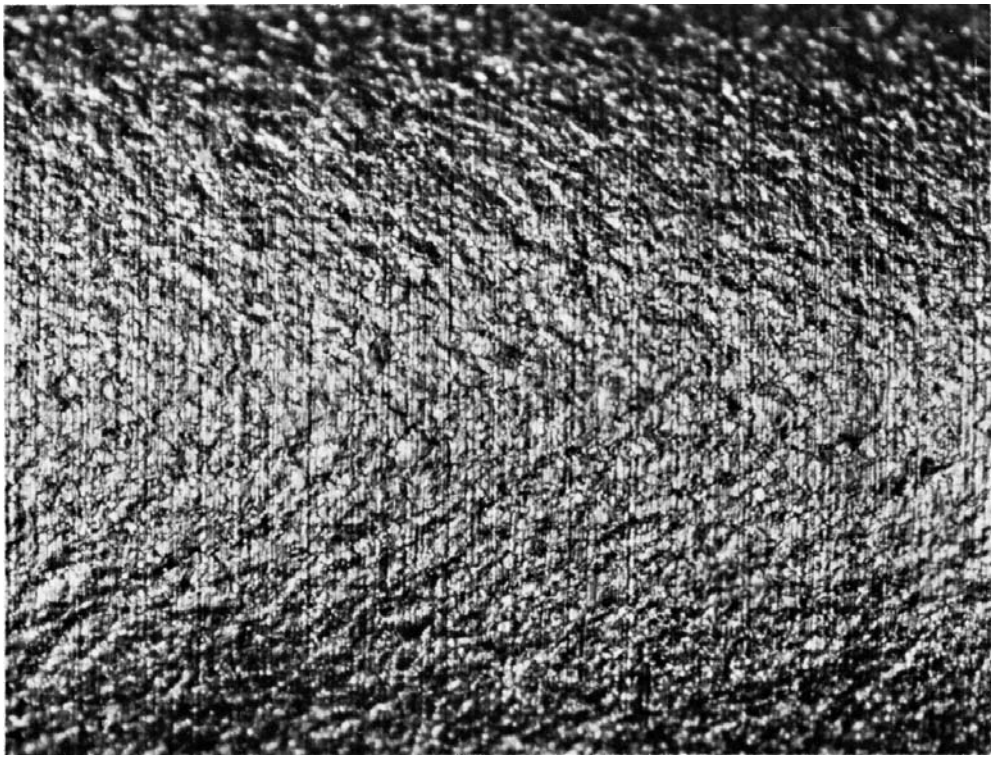


FIG. 13 Doubtful Test Result (Note the traces of intergranular fissures and "orange-peel" surface. Bent area at 20× magnification before reproduction.)

peel" on the surface, without accompanying cracks or fissures, should be disregarded also.

NOTE 25—Cracks suspected as arising through poor ductility may be investigated by bending a similar specimen that was not exposed to the boiling test solution. A visual comparison between these specimens should assist in interpretation.

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PRACTICE F—COPPER–COPPER SULFATE–50 % SULFURIC ACID TEST FOR DETERMINING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEELS

32. Scope

32.1 This practice describes the procedure for conducting the boiling copper–copper 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.

32.2 This test may be used to evaluate the susceptibility of as received material to intergranular corrosion caused by chromium carbide precipitation. It may also be used to evaluate the resistance of extra-low–carbon grades to sensitization to intergranular attack caused by welding or heat treatments.

32.3 This test should not be used to detect susceptibility to intergranular attack resulting from the formation of sigma phase. For detecting susceptibility to environments known to cause intergranular attack due to sigma use Practices B or C.

33. Rapid Screening Test

33.1 Before testing in copper–copper sulfate–50 % 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 copper–copper sulfate–50 % sulfuric acid test is specified in Table 7.

33.1.1 Corrosion test specimens having acceptable etch structures in the oxalic acid etch test will be essentially free of intergranular attack in the copper–copper sulfate–50 % sulfuric acid test. Such specimens are acceptable without testing in the copper–copper sulfate–50 % sulfuric acid test. All specimens having nonacceptable etch structures must be tested in the copper–copper sulfate–50 % sulfuric acid test.

34. Apparatus

34.1 The basic apparatus is described in Section 10. Also needed are the following:

34.1.1 For weight loss determination, an analytical balance capable of weighing to the nearest 0.001 g.

34.1.2 A piece of copper metal about 3.2 by 19 by 38 mm (⅛ by ¾ by 1½ in.) with a bright, clean finish. An equivalent area of copper shot or chips may be used. The copper should be washed and degreased before use. A rinse in 5 % H_2SO_4 will clean corrosion products from the copper.

TABLE 7 Use of Etch Structure Classifications from the Oxalic Acid Etch Test With the Copper-Copper Sulfate-50 % Sulfuric Acid Test

Grade	Acceptable Etch Structures	Nonacceptable Etch Structures
CF-3M	Step, dual, isolated ferrite	Ditch, interdendritic ditches
CF-8M	Step, dual, isolated ferrite	Ditch, interdendritic ditches

35. Copper–Copper Sulfate–50 % Sulfuric Acid Test Solution

35.1 Prepare 600 mL of test solution as follows. **Warning:** Protect the eyes and face by face shield and use rubber gloves and apron when handling acid. Place flask under hood.

35.1.1 First, measure 400.0 mL of distilled water in a 500-mL graduate and pour into the Erlenmeyer flask.

35.1.2 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 weight % in a 250 mL graduate. Add the acid slowly to the water in the Erlenmeyer flask to avoid boiling by the heat received.

35.1.3 Weigh 72 g of reagent grade copper sulfate ($CuSO_4 \cdot 5\ H_2O$) and add to the sulfuric acid solution. A trip balance may be used.

35.1.4 Place the copper piece into one glass cradle and put it into the flask.

35.1.5 Drop boiling chips into the flask.[6]

35.1.6 Lubricate the ground-glass joint with silicone grease.

35.1.7 Cover the flask with the condenser and circulate cooling water.

35.1.8 Heat the solution slowly until all of the copper sulfate is dissolved.

36. Preparation of Test Specimens

36.1 Prepare test specimens as described in Section 12.

36.2 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 copper–copper sulfate–50 % sulfuric acid test. The most commonly used sensitizing treatment is 1 h at 675°C.

37. Procedure

37.1 Place the specimen in another glass cradle and immerse in boiling solution.

37.2 Mark the liquid level on the flask with wax crayon to provide a check on vapor loss, which would result in concentration of the acid. If there is an appreciable change in the level, repeat the test with fresh solution and a reground specimen.

37.3 Continue immersion of the specimen for 120 h, then remove the specimen, rinse in water and acetone, and dry. If any adherent copper remains on the specimen, it may be removed by a brief immersion in concentrated nitric acid at room temperature.

37.4 For weight loss determination, weigh the specimen and subtract the weight from the original weight.

37.5 No intermediate weighings are usually necessary. The tests can be run without interruption. However, if preliminary results are desired, the specimen can be removed at any time for weighing.

37.6 No changes in solution are necessary during the test.

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38. Calculation and Report

38.1 The effect of the acid solution on the material shall be measured by determining the loss of weight of the specimen. The corrosion rate should be reported as millimetres of penetration per month (Note 8) calculated as follows:

$$\text{Millimetres per month} = (7290 \times w)/(A \times t \times d) \quad (3)$$

where:

t = time or exposure, h,
A = area, cm^2,
w = weight loss, g, and
d = density, g/cm^3.

For chromium-nickel-molybdenum steels, d = 8.00 g/cm^3.

39. Keywords

39.1 austenitic stainless steel; copper sulfate; corrosion testing; etch structures; ferric sulfate; intergranular corrosion; nitric acid; oxalic acid

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