Designation: A763 – 14

StandardPractices for
Detecting Susceptibility to Intergranular Attack in Ferritic Stainless Steels[1]

This standard is issued under the fixed designation A763; 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.

1. Scope*

1.1 These practices cover the following four tests:

1.1.1 *Practice W*—Oxalic acid etch test for detecting susceptibility to intergranular attack in stabilized ferritic stainless steels by classification of the etching structures (see Sections 3 – 10).

1.1.2 *Practice X*—Ferric sulfate-sulfuric acid test for detecting susceptibility to intergranular attack in ferritic stainless steels (Sections 11 – 16).

1.1.3 *Practice Y*—Copper-copper sulfate-50 % sulfuric acid test for detecting susceptibility to intergranular attack in ferritic stainless steels (Sections 17 – 22).

1.1.4 *Practice Z*—Copper-copper sulfate-16 % sulfuric acid test for detecting susceptibility to intergranular attack in ferritic stainless steels (Sections 23 – 29).

1.2 The following factors govern the application of these practices (**1-6**)[2]:

1.2.1 Practice W, oxalic acid test, is a rapid method of identifying, by simple electrolytic etching, those specimens of certain ferritic alloys that are not susceptible to intergranular corrosion associated with chromium carbide precipitation. Practice W is used as a screening test to avoid the necessity, for acceptable specimens, of more extensive testing required by Practices X, Y, and Z. See Table 1 for a listing of alloys for which Practice W is appropriate.

1.2.2 Practices X, Y, and Z can be used to detect the susceptibility of certain ferritic alloys to intergranular attack associated with the precipitation of chromium carbides or nitrides.

1.2.3 Practices W, X, Y, and Z can also be used to evaluate the effect of heat treatment or of fusion welding on susceptibility to intergranular corrosion.

1.2.4 Table 2 lists the identification ferritic stainless steels for which data on the application of at least one of the standard practices is available.

1.2.5 Some stabilized ferritic stainless steels may show high rates when tested by Practice X because of metallurgical factors not associated with chromium carbide or nitride precipitation. This possibility must be considered in selecting the test method. Combinations of alloys and test methods for which successful experience is available are shown in Table 1. Application of these standard tests to the other ferritic stainless steels will be by specific agreement between producer and user.

1.3 Depending on the test and alloy, evaluations may be accomplished by weight loss determination, microscopical examination, or bend test (Sections 30 and 31). The choices are listed in Table 1.

1.4 *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.* For specific safety precautionary statements, see 3.2.5, Section 7, 13.1, and 19.1.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

A370 Test Methods and Definitions for Mechanical Testing of Steel Products

3. Apparatus

3.1 *Apparatus for Practice W, Oxalic Acid Etch Test:*

3.1.1 *Source of DC*—Battery, generator, or rectifier capable of supplying 15 V and 20 A.

3.1.2 *Ammeter,* range 0 to 30 A.

3.1.3 *Variable Resistance,* for control of specimen current.

3.1.4 *Cathode*—One-litre stainless steel beaker or suitable piece of stainless steel.

3.1.5 *Electric Clamp,* to hold etched specimen.

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[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 appended to these practices.

[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.

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

TABLE 1 Methods for Evaluating Ferritic Stainless Steels for Susceptibility to Intergranular Corrosion

Alloy	Time of Test, h	Evaluation Criteria		
		Weight Loss	Microscopical Examination	Bend Test
PRACTICE W—OXALIC ACID ETCH TEST				
439	0.025	NA	A[A]	NA
18Cr-2Mo	0.025	NA	A[A]	NA
XM27	0.025	NA	A[A]	NA
XM33	0.025	NA	A[A]	NA
26-3-3	0.025	NA	A[A]	NA
PRACTICE X—FERRIC SULFATE - SULFURIC ACID TEST				
430	24	A[B,C]	A	NA
446	72	A[C]	A	NA
XM27	120	A[D]	A[C]	NA
29Cr-4Mo	120	NA[E]	A[C]	NA
29Cr-4Mo-2Ni	120	NA	A[C]	NA
PRACTICE Y—COPPER-COPPER SULFATE - 50% SULFURIC ACID TEST				
446	96	A[C]	A	NA
XM27	120	A[D]	A[C]	NA
XM33	120	A[D]	A[C]	NA
26–3–3	120	A[D]	A[C]	NA
29-4C	120	A[D]	A[C]	NA
29Cr-4Mo	120	NA	A[C]	NA
29Cr-4Mo-2Ni	120	NA	A[C]	NA
PRACTICE Z—COPPER-COPPER SULFATE - 16% SULFURIC ACID TEST				
430	24	NA	NA	no fissures
434	24	NA	NA	no fissures
436	24	NA	NA	no fissures
439	24	NA	NA	no fissures
18Cr-2Mo	24	NA	NA	no fissures

[A] Polished surface examined at 250 to 500× with a metallurgical microscope (see 3.1.6). All other microscopical examinations are of the corroded surface under 40× binocular examination (see Section 27).

[B] A = Applicable.

[C] Preferred criterion, these criteria are the most sensitive for the particular combination of alloy and test.

[D] Weight loss measurements can be used to detect severely sensitized material, but they are not very sensitive for alloys noted with this superscript and may not detect slight or moderate sensitization.

[E] NA = Not applicable.

TABLE 2 Steels for Which Test Results are Available

UNS Designation	Alloy	Practice(s)
S43000	430[A]	X, Z
S43400	434[A]	Z
S43600	436[A]	Z
S43035	XM8	Z
S44400	18Cr-2Mo	W, Z
S44600	446[A]	X, Y
S44626	XM33	W, Y
S44627	XM27	W, X, Y
S44660	26–3–3	Y
S44700	29Cr-4Mo	X, Y
S44735	29-4C	Y
S44800	29Cr-4Mo-2NI	X, Y

[A] Types 430, 434, 436, and 446 are nonstabilized grades that are generally not used in the as-welded or sensitized condition in other than mildly corrosive environments. In the annealed condition, they are not subject to intergranular corrosion. For any studies of IGA on Types 430, 434, 436, or 446, the indicated test methods are suggested.

3.1.6 *Metallurgical Microscope,* for examination of etched structures at 250 to 500×.

3.1.7 *Electrodes*—The specimen is made the anode and the beaker or other piece of stainless steel the cathode.

3.1.8 *Electrolyte*—Oxalic acid ($H_2C_2O_4{\cdot}2H_2O$) reagent grade, 10 weight % solution.

3.2 *Aparatus Common to Practices X, Y, and Z*—Suplementary requirements are noted as required.

3.2.1 The apparatus used is shown in Fig. 1.

NOTE 1—No substitution for this equipment may be used. The cold-finger type of condenser with standard Erlenmeyer flasks may not be used.

3.2.2 *Allihn or Soxhlet Condenser,* four-bulb (minimum) with a 45/50 ground-glass joint. Overall length shall be about 330 mm (13 in.), with condensing section 241 mm (9½ in.).

3.2.3 *Erlenmeyer Flask,* 1-L with a 45/50 ground-glass joint. The ground-glass opening is somewhat over 38 mm (1½ in.) wide.

3.2.4 *Glass Cradles* (Note 2), can be supplied by a glass blowing shop. The size of the cradles should be such that they can pass through the ground-glass joint of the Erlenmeyer flask. They should have three or four holes in them to increase circulation of the test solution around the specimen.

NOTE 2—Other equivalent means of specimen support such as glass hooks or stirrups may also be used.

3.2.5 *Boiling Chips,* must be used to prevent bumping. 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.[4]

3.2.6 *Silicone Grease,* is recommended for the ground-glass joint.

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[4] Amphoteric alundum granules, Hengar Granules, from the Hengar Company, Philadelphia, PA have been found satisfactory for this purpose.

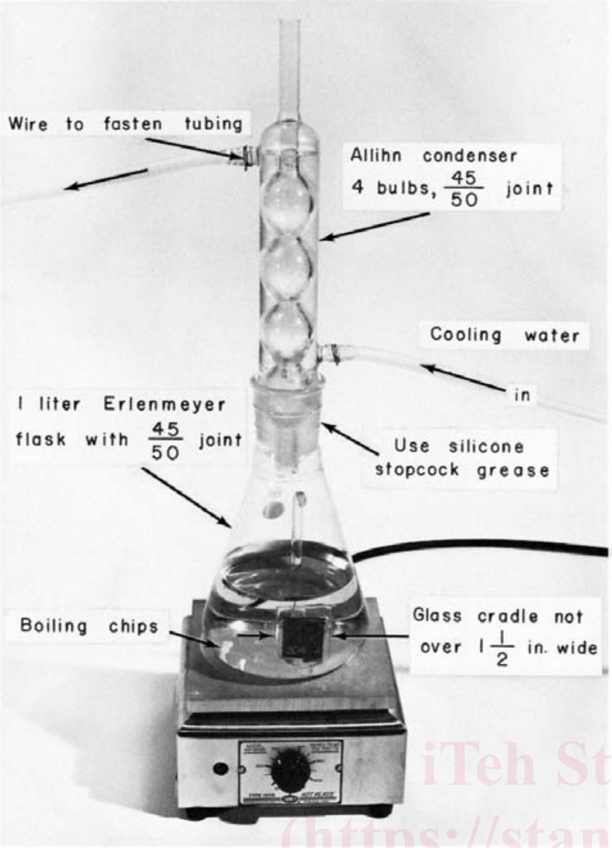

FIG. 1 Test Apparatus

3.2.7 *Electrically Heated Hot Plate,* or other device to provide heat for continuous boiling of the solution.

4. Preparation of Test Specimens

4.1 The preparation of test specimens is common among Practices X, Y, and Z. Additional requirements are noted where necessary.

4.2 A specimen having a total surface area of 5 to 20 cm^2 is recommended for Practices X, Y, and Z. As-welded specimens should be cut so that no more than 13 mm ½ (in.) width of unaffected base metal is included on either side of the weld and heat-affected zone.

4.3 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 prepared 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 when carbonaceous lubricants are employed) it may be possible by heavy grinding or machining to remove the carburized layer completely. Such treatment of test specimens is not permissible, except in tests undertaken to demonstrate such surface effects.

4.4 *Sensitization of Test Specimens:*

4.4.1 Specimens from material that is going to be used in the as-received condition without additional welding or heat treatment may be tested in the as-received condition without any sensitizing treatment.

4.4.2 Specimens from material that is going to be welded or heat treated should be welded or heat treated in as nearly the same manner as the material will experience in service.

4.4.3 The specific sensitizing or welding treatment, or both, should be agreed upon between the supplier and the purchaser.

4.5 For Practice W, a cross section of the sample including material at both surfaces and a cross section of any weld and its heat affected zones should be prepared. If the sample is too thick, multiple specimens should be used. Grind the cross section on wet or dry 80 or 120-grit abrasive paper followed by successively finer papers until a number 400 or 3/0 finish is obtained. Avoid excessive heat when dry-grinding.

4.6 For Practices X, Y, and Z, all surfaces of the specimen including edges should be ground on wet or dry 80 or 120-grit abrasive paper. Avoid excessive heat when dry-grinding. Do not use sand- or grit-blasting. All traces of oxide scale formed during heat treatment must be removed. To avoid scale entrapment, stamp specimens for identification after heat treatment and grinding.

4.7 Degrease and dry the sample using suitable nonchlorinated agents.

PRACTICE W—OXALIC ACID ETCH TEST FOR DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK BY CLASSIFICATION OF MICROSTRUCTURE FOR SCREENING OF CERTAIN FERRITIC STAINLESS STEELS

5. Scope

5.1 The oxalic acid etch test is intended and may be used for screening of certain ferritic stainless steels to precede or preclude the need for corrosion testing as described in Practices X, Y, or Z. Specimens with unacceptable microstructures should be subjected to Practices X, Y, or Z to better determine their susceptibility to intergranular attack. See Table 1 for a listing of alloys for which Practice W is appropriate.

6. Etching Conditions

6.1 The polished specimens should be etched at 1 A/cm^2 for 1.5 min. This may be accomplished with the apparatus prescribed in 3.1 by adjusting the variable resistance until the ammeter reading in amperes equals the immersed specimen area in square centimetres. Immersion of the specimen-holding clamp in the etching solution should be avoided.

7. Etching Precautions

7.1 Etching should be carried out under a ventilating hood. Gas evolved at the electrodes with entrained oxalic acid is poisonous and irritating. The temperature of the etching solution, which increases during etching, should be kept below 50°C by using two beakers of acid, one of which may be cooled while the other is in use.

8. Rinsing Prior to Examination

8.1 Following etching, the specimen should be rinsed in hot water then acetone or alcohol to avoid oxalic acid crystallization on the etched surface during forced air-drying.

9. Examination

9.1 Examine etched specimens on a metallurgical microscope at 250 to 500× as appropriate for classification of etched microstructure type as defined in Section 10.

10. Classification of Etched Structures

10.1 Acceptable structures indicating resistance to chromium carbide-type intergranular attack:

10.1.1 *Step Structure*—Steps only between grains—no ditches at grain boundaries (see Fig. 2).

10.1.2 *Dual Structure*—Some ditches at grain boundaries in addition to steps, but no single grain completely surrounded by ditches (see Fig. 3).

10.2 Unacceptable structures requiring additional testing (Practices X, Y, or Z):

10.2.1 *Ditch Structure*—One or more grains completely surrounded by ditches (see Fig. 4).

PRACTICE X—FERRIC SULFATE-SULFURIC ACID TEST FOR DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN FERRITIC STAINLESS STEELS

11. Scope

11.1 This practice describes the procedure for conducting the boiling ferric sulfate-sulfuric acid test which measures the susceptibility of ferritic stainless steels to intergranular attack. This test detects susceptibility to intergranular attack associated with the precipitation of chromium carbides and nitrides in stabilized and unstabilized ferric stainless steels. It may also detect the presence of chi or sigma phase in these steels. The test will not differentiate between intergranular attack resulting from carbides and that due to intermetallic phases. The ferric sulfate-sulfuric acid solution may also selectively attack titanium carbides and nitrides in stabilized steels. The alloys on which the test has been successfully applied are shown in Table 1.

11.2 This test may be used to evaluate the susceptibility of as-received material to intergranular corrosion caused by chromium carbide or nitride precipitation. It may be applied to wrought products and weld metal.

11.3 This procedure may be used on ferritic stainless steels after an appropriate sensitizing heat treatment or welding procedure as agreed upon between the supplier and the purchaser.

12. Apparatus

12.1 The basic apparatus is described in Section 3. Also needed are:

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

12.1.2 For microscopical examination, a microscope with magnification to at least 40×.

13. Ferric Sulfate-Sulfuric Acid Test Solution

13.1 Prepare 600 mL of test solution as follows. (**Warning—** Protect the eyes and use rubber gloves and apron for handling acid. Place the test flask under a hood.)

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

13.1.2 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 at 250-mL graduate. Add the acid slowly to the water in the Erlenmeyer flask to avoid boiling by the heat evolved.

NOTE 3—Loss of vapor results in concentration of the acid.

13.1.3 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.

13.1.4 Drop boiling chips into the flask.

13.1.5 Lubricate the ground-glass joint with silicone grease.

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

13.1.7 Boil the solution until all the ferric sulfate is dissolved.

14. Preparation of Test Specimens

14.1 Prepare test specimens as described in Section 4.

15. Procedure

15.1 When weight loss is to be determined, measure the sample prior to final cleaning and then weigh.

15.1.1 Measure the sample including the inner surfaces of any holes, and calculate the total exposed surface area.

15.1.2 Degrease and dry the sample using suitable nonchlorinated agents, and then weigh to the nearest 0.001 g.

15.2 Place the specimen in a glass cradle and immerse in boiling solution.

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

15.4 Continue immersion of the specimen for the time shown in Table 1, then remove the specimen, rinse in water and acetone, and dry. Times for steels not listed in Table 1 are subject to agreement between the supplier and the purchaser.

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

15.6 No intermediate weighings are usually necessary. The tests can be run without interruption for the time specified in

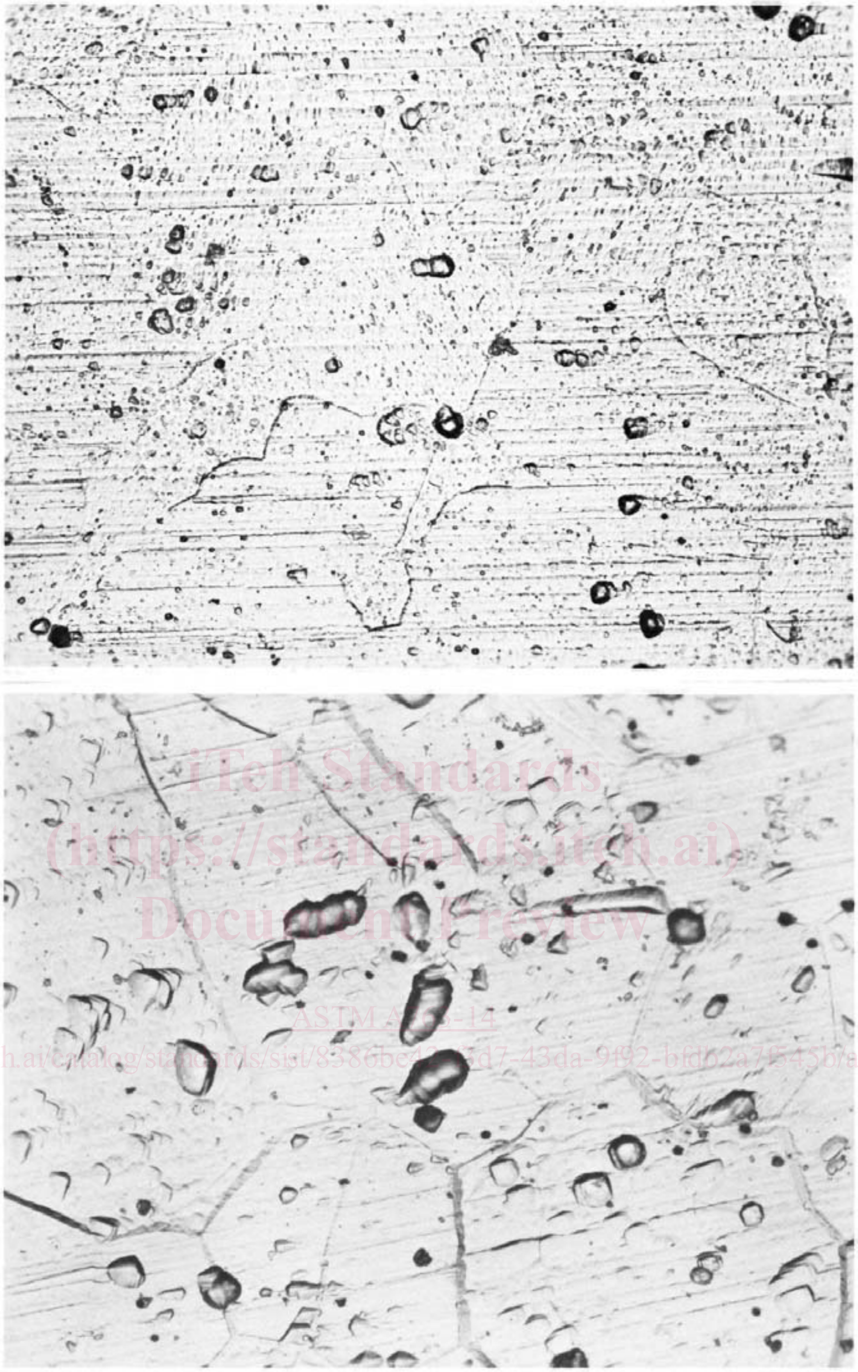

FIG. 2 Acceptable Structures Practice W—Oxalic-Acid Etch Test Steps Between Grains No Ditching

Table 1. However, if preliminary results are desired, the specimen can be removed at any time for weighing.

15.7 No changes in solution are necessary during the test period.

15.8 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.)

15.9 Testing of a single specimen in a flask is preferred. However, several specimens may be tested simultaneously. The number is limited only by the number of glass cradles that can be fitted into the flask (usually three or four). Each sample must be in a separate cradle so that the samples do not touch.

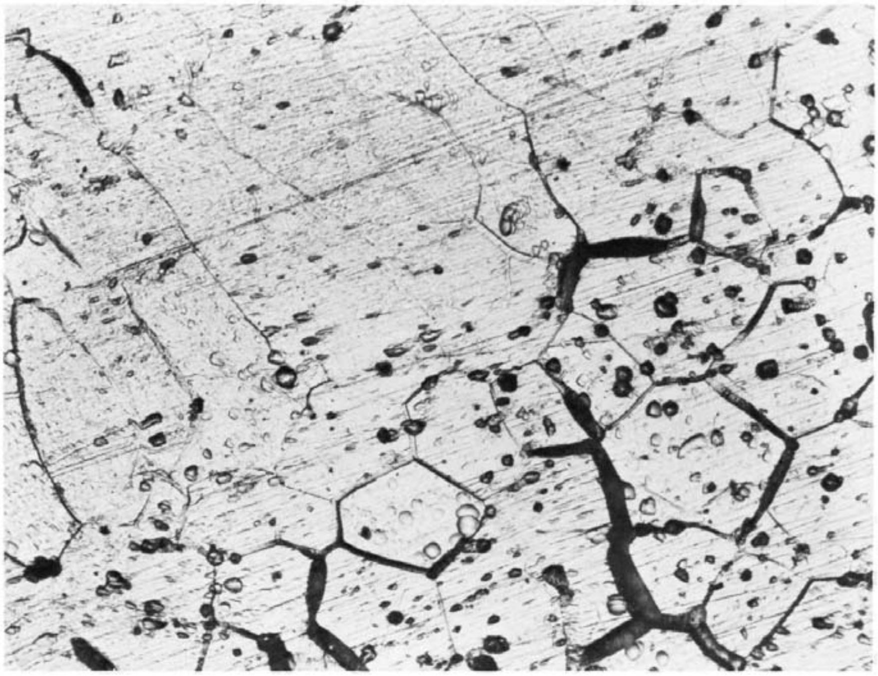

FIG. 3 Acceptable Structure Practice W—Oxalic Acid Etch Test Dual Structure—Some Ditches But No Single Grain Completely Surrounded

15.10 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.

16. Evaluation

16.1 Depending on the agreement between the supplier and the purchaser, the results of the test may be evaluated by weight loss or microscopical examination as indicated in Table 1. (See Sections 30 and 31.)

PRACTICE Y—COPPER-COPPER SULFATE-50 % SULFURIC ACID TEST FOR DETERMINING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN FERRITIC STAINLESS STEELS

17. Scope

17.1 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. This test detects susceptibility to intergranular attack associated with the precipitation of chromium carbides or nitrides in unstabilized and stabilized ferritic stainless steels.

17.2 This test may be used to evaluate the susceptibility of as-received material to intergranular corrosion caused by chromium carbide or nitride precipitation. It may also be used to evaluate the resistance of high purity or stabilized grades to sensitization to intergranular attack caused by welding or heat treatments. It may be applied to wrought products.

17.3 This test should not be used to detect susceptibility to intergranular attack resulting from the formation or presence of chi phase, sigma phase, or titanium carbides or nitrides. For detecting susceptibility to environments known to cause intergranular attack due to these phases use Practice X.

18. Apparatus

18.1 The basic apparatus is described in Section 3. Also needed are:

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

18.1.2 For microscopical examination, a microscope with magnification to at least 40×.

18.1.3 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.

19. Copper-Copper Sulfate-50 % Sulfuric Acid Test Solution

19.1 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.)

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

19.1.2 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 evolved.

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