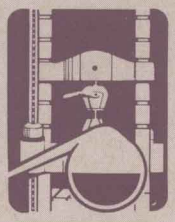
1988 ANNUAL BOOK OF ASTM STANDARDS



SECTION 3

Metals Test Methods and Analytical Procedures

VOLUME 03.02 Wear and Erosion; Metal Corrosion

Revision issued annually

1988 ANNUAL BOOK OF ASTM STANDARDS

SECTION 3

Metals Test Methods and Analytical Procedures

VOLUME

03.02 Wear and Erosion; Metal Corrosion

Includes standards of the following committees:

G-1 on Corrosion of Metals
G-2 on Wear and Erosion

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Foreword

ASTM

ASTM, founded in 1898, is a scientific and technical organization formed for "the development of standards on characteristics and performance of materials, products, systems, and services; and the promotion of related knowledge." It is the world's largest source of voluntary consensus standards.

The Society operates through 140 main technical committees with 2040 subcommittees. These committees function in prescribed fields under regulations that ensure balanced representation among producers, users, general interest, and consumer participants.

The Society currently has 30,000 active members, of whom approximately 19,150 serve as technical experts on committees, representing 91,390 units of participation.

Membership in the Society is open to all concerned with the fields in which ASTM is active. A membership application may be found at the back of this volume. Additional information may be obtained from Member, Committee, and Customer Services, ASTM, 1916 Race St., Philadelphia, PA 19103.

1988 Annual Book of ASTM Standards

The 1988 *Annual Book of ASTM Standards* consists of 67 volumes, divided among 16 sections, of which this volume is one. It contains formally approved ASTM standard classifications, guides, practices, specifications, test methods, and terminology and related material such as proposals. These terms are defined as follows in the Regulations Governing ASTM Technical Committees:

Categories:

standard—as used in ASTM, a document that has been developed and established within the consensus principles of the Society and that meets the approval requirements of ASTM procedures and regulations.

Discussion—The term "standard" serves in ASTM as an adjective in the title of documents, such as test methods or specifications, to connote specified consensus and approval. The various types of standard documents are based on the needs and usages as prescribed by the technical committees of the Society.

proposal—a document that has been approved by the sponsoring committee for publication for information and comment prior to its consideration for adoption as a standard.

Discussion—Complete balloting procedures are not required for proposals.

emergency standard—a document published by the Society to meet a demand for more rapid issuance of a specific standard document.

Discussion—The Executive Subcommittee of the sponsoring committee must recommend the publishing of an emergency standard and the Committee on Standards must concur in the recommendation. Emergency standards are not full consensus documents because they are not submitted to Society ballot.

Types:

The various types of ASTM documents are to provide a flexibility of form, communication, and usage for both the technical committees and the myriad users of ASTM documents. The type of ASTM document that is developed and titled is based on the technical content and intended use, not on the degree of consensus achieved. The three categories of ASTM documents (standard, emergency standard, and proposal) can be of the following forms and types:

classification—a systematic arrangement or division of materials, products, systems, or services into groups based on similar characteristics such as origin, composition, properties, or use.

guide—a series of options or instructions that do not recommend a specific course of action.

Discussion—Whereas a practice prescribes a general usage principle, a guide only suggests an approach. The purpose of a guide is to offer guidance, based on a consensus of viewpoints, but not to establish a fixed procedure. A guide is intended to increase the awareness of the user to available techniques in a given subject area and to provide information from which subsequent evaluation and standardization can be derived.

practice—a definitive procedure for performing one or more specific operations or functions that does not produce a test result. (Compare *test method.*)

Discussion—A practice is not a downgraded test method. Examples of practices include procedures for conducting interlaboratory testing programs or other statistical procedures; for writing statements on sampling or precision and accuracy; and for selection, preparation, application, inspection, necessary precautions for use or disposal, installation, maintenance, and operation of testing equipment.

specification—a precise statement of a set of requirements to be satisfied by a material, product, system, or service that indicates the procedures for determining whether each of the requirements is satisfied.

Discussion—It is desirable to express the requirements numerically in terms of appropriate units together with their limits.

terminology—a document comprising definitions of terms; descriptions of terms; explanations of symbols, abbreviations, or acronyms.

test method—a definitive procedure for the identification, measurement, and evaluation of one or more qualities, characteristics, or properties of a material, product, system, or service that produces a test result. (Compare *practice.*)

A new edition of the Book of Standards is issued annually. Each volume contains all actions approved by the Society at least six months before the issue date. New and revised standards approved by the Society between the annual appearances of any given volume are made available as separate copies. The 1988 edition of the Book of Standards comprises approximately 55,000 pages and includes over 8000 ASTM standards.

Purpose and Use of ASTM Standards

An ASTM standard represents a common viewpoint of those parties concerned with its provisions, namely, producers, users, consumers, and general interest groups. It is intended to aid industry, government agencies, and the general public. The use of an ASTM standard is purely voluntary. It is recognized that, for certain work or in certain regions, ASTM standard specifications may be either more or less restrictive than needed. The existence of an ASTM standard does not preclude anyone from manufacturing, marketing, or purchasing products, or using products, processes, or procedures not conforming to the standard. Because ASTM standards are subject to periodic review and revision, those who use them are cautioned to obtain the latest revision.

Consideration of Comments on ASTM Standards

An ASTM standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of any standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Using the Annual Book of ASTM Standards

The standards are assembled in each volume in alphanumeric sequence of their ASTM designation numbers except for Volumes 11.01, 11.02, and 05.04, which are assembled by subject matter. Volume 06.03 is assembled first by committee, then in alphanumeric sequence. Each volume has a table of contents, listing the standards in alphanumeric sequence by ASTM designation; and a list by subjects, categorizing the standards according to subject. A subject index of the standards in each volume appears at the back of each volume.

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Each ASTM standard is available as a separate copy from ASTM. Special quantity prices and discounts for members can be obtained from Customer Services. When ordering, provide the ASTM standard designation and year of issue, title, quantity desired, and shipping instructions.

Obsolete Editions

This new edition of the *Annual Book of ASTM Standards* makes last year's edition obsolete. Each volume of the *Annual Book of ASTM Standards* is published annually because of additions of new standards and significant revisions in existing standards. On the average, about 30 % of each volume is new or revised. For practical purposes, therefore, it is not wise to use obsolete volumes. However, for teaching purposes, these outdated volumes might be useful.

Precautionary Caveat

In January 1983, the Board of Directors approved the inclusion of the following precautionary caveat:

This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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.

Inclusion of the caveat is required in test methods, specifications (where test methods are detailed other than by reference), practices, and guides. Implementation of the caveat will be phased in as new, revised, and reapproved standards are approved by the Society.

Disclaimer

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in these standards. Users of these standards are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

ANNUAL BOOK OF ASTM STANDARDS

Listed by Section and Volume

ANNUAL BOOK OF ASTM STANDARDS

Listed by Subjects

Contents

1988 ANNUAL BOOK OF ASTM STANDARDS, VOLUME 03.02

STANDARDS RELATING TO METALS—WEAR AND EROSION; METAL CORROSION

A complete Subject Index begins on p. 413

Listed below are those standards included in this book and those standards that appeared previously that have been discontinued within the past five years. Since the standards in this book are arranged in alphanumeric sequence, no page numbers are given in this contents.

In the serial designations prefixed to the following titles, the number following the dash indicates the year of original issue as tentative or of adoption as standard or, in the case of revision, the year of last revision. Thus, standards adopted or revised during the year 1988 have as their final number, 88. A letter following this number indicates more than one revision during that year, that is, 88a indicates the second revision in 1988, 88b the third revision, etc. Tentatives are identified by the letter T. Standards that have been reapproved without change are indicated by the year of last reapproval in parentheses as part of the designation number, for example, (1988). A superscript epsilon indicates an editorial change since the last revision or reapproval—$^{\epsilon 1}$ for the first change,$^{\epsilon 2}$ for the second change, etc.

§ Approved for use by agencies of the Department of Defense and, if indicated on the standard, replaces corresponding Federal or military document. Consult the *DoD Index of Specifications and Standards* for the specific year of issue which has been adopted by the Department of Defense.

† Although this standard has been officially withdrawn from Society approval, a brief description is included for information only.

CONTENTS

RELATED MATERIAL

List by Subjects

1988 ANNUAL BOOK OF ASTM STANDARDS, VOLUME 03.02

METALS—WEAR AND EROSION; METAL CORROSION

A complete Subject Index begins on p. 413

Since the standards in this book are arranged in alphanumeric sequence, no page numbers are given in this list by subjects.

The standards listed in italics are related documents included for information only and do not appear in this volume.

CORROSION

Methods for:

B 287 – 74 (1980)$^{\epsilon 1}$	Acetic Acid-Salt Spray (Fog) Testing (Discontinued 1988†)
§G 43 – 75 (1980)	Acidified Synthetic Sea Water (Fog) Testing (Discontinued 1988†)
D 807 – 82	Assessing the Tendency of Industrial Boiler Waters to Cause Embrittlement (USBM Embrittlement Detector Method)
G 87 – 84	Conducting Moist SO_2 Tests
G 4 – 84	Conducting Corrosion Coupon Tests in Plant Equipment
G 57 – 78 (1984)$^{\epsilon 1}$	Field Measurement of Soil Resistivity Using the Wenner Four-Electrode Method
§B 117 – 85$^{\epsilon 1}$	Salt Spray (Fog) Testing
B 76 – 81	*Accelerated Life Test of Nickel-Chromium and Nickel-Chromium-Iron Alloys for Electrical Heating (see Vol 03.04)*
B 368 – 85	*Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test) (see Vol 02.05)*
B 380 – 85	*Corrosion Testing of Decorative Electrodeposited Coatings by the Corrodkote Procedure (see Vol 02.05)*
C 692 – 77	*Evaluating the Influence of Wicking-Type Thermal Insulations on the Stress Corrosion Cracking Tendency of Austenitic Stainless Steel (see Vol 04.06)*
B 651 – 83	*Measurement of Corrosion Sites in Nickel Plus Chromium or Copper Plus Nickel Plus Chromium Electroplated Surfaces with the Double-Beam Interference Microscope (see Vol 02.05)*
F 483 – 87	*Total Immersion Corrosion Test for Aircraft Maintenance Chemicals (see Vol 15.03)*

Test Methods for:

G 60 – 86	Conducting Cyclic Humidity Tests
G 61 – 86	Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrison Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys
D 2776 – 79	Corrosivity of Water in the Absence of Heat Transfer (Electrical Methods)
D 2688 – 83	Corrosivity of Water in the Absence of Heat Transfer (Weight Loss Methods)
G 41 – 85	Cracking Susceptibility of Metals Exposed Under Stress to a Hot Salt Environment, Determining
§G 34 – 86	Exfoliation Corrosion Susceptibility in 2XXX and 7XXX Series Aluminum Alloys (EXCO Test)
G 51 – 77 (1984)	pH of Soil for Use in Corrosion Testing
G 48 – 76 (1980)$^{\epsilon 1}$	Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by the Use of Ferric Chloride Solution
F 746 – 87$^{\epsilon 1}$	Pitting or Crevice Corrosion of Metallic Surgical Implant Materials
G 28 – 85	Susceptibility to Intergranular Attack in Wrought, Nickel-Rich, Chromium-Bearing Alloys, Detecting
G 67 – 86	Susceptibility to Intergranular Corrosion of 5XXX Series Aluminum Alloys by Mass Loss After Exposure to Nitric Acid (NAMLT Test), Determining the
§G 47 – 79 (1984)$^{\epsilon 1}$	Susceptibility to Stress-Corrosion Cracking of High-Strength Aluminum Alloy Products, Determining
G 37 – 85	Use of Mattsson's Solution of pH 7.2 to Evaluate the Stress-Corrosion Cracking Susceptibility of Copper-Zinc Alloys
G 66 – 86	Visual Assessment of Exfoliation Corrosion Susceptibility of 5XXX Series Aluminum Alloys (Asset Test)
F 482 – 84	*Corrosion of Aircraft Metals by Total Immersion Maintenance Chemicals (see Vol 15.03)*
B 627 – 84	*Electrolytic Corrosion Testing (EC Test)(see Vol 02.05)*
C 876 – 87	*Half Cell Potentials of Reinforcing Steel in Concrete (see Vol 04.02)*

Practices for:

G 44 – 88	Alternate Immersion Stress Corrosion Testing in 3.5 % Sodium Chloride Solution
G 16 – 71 (1984)	Applying Statistics to Analysis of Corrosion Data
G 2 – 88	Corrosion Testing of Products of Zirconium, Hafnium, and Their Alloys in Water at 680°F or in Steam at 750°F
G 2M – 88	Corrosion Testing of Products of Zirconium, Hafnium, and Their Alloys in Water at 633°K or in Steam at 673°K [Metric]

† Although this standard has been officially withdrawn from Society approval, a brief description is included for information only.

§ Approved for use by agencies of the Department of Defense and, if indicated on the standard, replaces corresponding Federal or military document. Consult the *DoD Index of Specifications and Standards* for the specific year of issue which has been adopted by the Department of Defense.

Designation: A 262 – 86

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.

These practices have been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 These practices cover the following six 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 D*—Nitric-Hydrofluoric Acid Test for Detecting Susceptibility to Intergranular Attack in Molybdenum-Bearing Austenitic Stainless Steels (Sections 22 to 28, inclusive), and

1.1.5 *Practice E*—Copper-Copper Sulfate-Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections 29 to 38, inclusive).

1.1.6 *Practice F*—Copper-Copper Sulfate-50 % Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Molybdenum-Bearing Cast Austenitic Stainless Steels (Sections 39 to 45, 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 which 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, the nitric acid test, and the nitric-hydrofluoric 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 non-acceptable.

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

1.6 For AISI Grades 316, 316L, 317, and 317L only, the nitric-hydrofluoric acid test may be used to provide test results in 4 h.

1.7 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 which might be subject to end grain corrosion in nitric acid service.

1.8 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.

1.9 Extensive test results on various types of stainless steels evaluated by these practices have been published in Ref **(10)**.[2]

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

1.11 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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

[1] These practices are under the jurisdiction of ASTM Committee A-1 on Steel, Stainless Steel and Related Alloys and are the direct responsibility of Subcommittee A01.14 on Methods of Corrosion Testing.

Current edition approved Aug. 29, 1986. Published September 1986. Originally published as A 262 – 43 T. Last previous edition A 262 – 85a.

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

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: 316, 316L, 317, 317L	AISI: 304, 304L, 316, 316L, 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)	Nitric-Hydrofluoric Acid Test (4 h at 70°C)	Copper-Copper Sulfate-Sulfuric Acid Test (24 h in boiling solution)	Copper-Copper Sulfate-50 % Sulfuric Acid Testing Boiling Solution
Chromium carbide in: 304, 304L, CF-3, CF-8 Chromium carbide and sigma phase in:[D] 316, 316L, 317, 317L, 321, 347, CF-3M, CF-8M End-grain in: all grades	Chromium carbide in: 304, 304L, 316, 316L, 317, 317L, CF-3, CF-8 Chromium carbide and sigma phase in: 321, CF-3M, CF-8M[E]	Chromium carbide in: 316, 316L, 316LN, 316N, 317, 317L	Chromium carbide in: 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.

precautionary statements are given in 5.6, 11.1.1, 11.1.9, 25.1.4.1, and 42.1.)

2. Referenced Document

2.1 *ASTM Standard:*

A 370 Methods and Definitions for Mechanical Testing of Steel Products[3]

PRACTICE A—OXALIC ACID ETCH TEST FOR CLASSIFICATION OF ETCH STRUCTURES OF AUSTENITIC STAINLESS STEELS (1)

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 which 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 D—Nitric-Hydrofluoric 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 1200 to 1250°F (650 to 675°C), 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 1250°F.

[3] *Annual Book of ASTM Standards*, Vols 01.01 to 01.05.

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 D—Nitric Hydrofluoric Acid Test	316, 316L, 317, 317L	...
Practice E—Copper-Copper Sulfate-16 % Sulfuric Acid Test	304, 304L, 316, 316L, 317, 317L, 321, 347	...
Practice F—Copper-Copper Sulfate-50 % Sulfuric Acid Test	...	CF-8M, CF-3M

4. Apparatus

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

4.2 *Ammeter*—Range 0 to 30 A (Note 1).

4.3 *Variable Resistance* (Note 1).

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

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

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

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 1 by 1 in. (25 by 25 mm).

5.2 The intent is to test a specimen representing as nearly as possible the surface of the material as it will be used in service. Therefore the preferred sample is a cross section including the surface to be exposed in service. Only such surface finishing should be performed as is required to remove foreign material and obtain a standard, uniform finish as described in 5.3. For very heavy sections, specimens should be machined to represent the appropriate surface while maintaining reasonable specimen size for convenient testing. Ordinarily, removal of more material than necessary will have little influence on the test results. However, in the special case of surface carburization (sometimes encountered, for instance, in tubing or castings when lubricants or binders containing carbonaceous materials are employed) it may be possible by heavy grinding or machining to completely remove the carburized surface. Such treatment of test specimens is not permissible, except in tests undertaken to demonstrate such effects.

5.3 *Polishing*—On all types of materials, cross sectional surfaces should be polished for etching and microscopical examination. Specimens containing welds should include base plate, weld heat-affected zone, and weld metal. Scale should be removed from the area to be etched, by grinding to an 80 or 120-grit finish on a grinding belt or wheel without excessive heating, and then polishing on successively finer emery papers, No. 1, ½, ⅓, ⅖, 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 ($H_2C_2O_4$;pd $2H_2O$) 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.

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 com-