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Designation: A923 – 23

Standard Test Methods for Detecting Detrimental Intermetallic Phase in Duplex Austenitic/Ferritic Stainless Steels[1]

This standard is issued under the fixed designation A923; 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 The purpose of these test methods is to allow detection of the presence of intermetallic phases in certain duplex stainless steels as listed in Table 1, Table 2, and Table 3 to the extent that toughness or corrosion resistance is affected significantly. These test methods will not necessarily detect losses of toughness or corrosion resistance attributable to other causes. Similar test methods for other duplex stainless steels are described in Test Method A1084, but the procedures described in this standard differ significantly from Test Methods A, B, and C in A1084.

1.2 Duplex (austenitic-ferritic) stainless steels are susceptible to the formation of intermetallic compounds during exposures in the temperature range from approximately 600 to 1750 °F (320 to 955 °C). The speed of these precipitation reactions is a function of composition and thermal or thermomechanical history of each individual piece. The presence of these phases is detrimental to toughness and corrosion resistance.

1.3 Correct heat treatment of duplex stainless steels can eliminate these detrimental phases. Rapid cooling of the product provides the maximum resistance to formation of detrimental phases by subsequent thermal exposures.

1.4 Compliance with the chemical and mechanical requirements for the applicable product specification does not necessarily indicate the absence of detrimental phases in the product.

1.5 These test methods include the following:

1.5.1 *Test Method A*—Sodium Hydroxide Etch Test for Classification of Etch Structures of Duplex Stainless Steels (Sections 3 – 7).

1.5.2 *Test Method B*—Charpy Impact Test for Classification of Structures of Duplex Stainless Steels (Sections 8 – 13).

1.5.3 *Test Method C*—Ferric Chloride Corrosion Test for Classification of Structures of Duplex Stainless Steels (Sections 14 – 20).

1.6 The presence of detrimental intermetallic phases is readily detected in all three tests, provided that a sample of appropriate location and orientation is selected. Because the occurrence of intermetallic phases is a function of temperature and cooling rate, it is essential that the tests be applied to the region of the material experiencing the conditions most likely to promote the formation of an intermetallic phase. In the case of common heat treatment, this region will be that which cooled most slowly. Except for rapidly cooled material, it may be necessary to sample from a location determined to be the most slowly cooled for the material piece to be characterized.

1.7 The tests do not determine the precise nature of the detrimental phase but rather the presence or absence of an intermetallic phase to the extent that it is detrimental to the toughness and corrosion resistance of the material.

1.8 Examples of the correlation of thermal exposures, the occurrence of intermetallic phases, and the degradation of toughness and corrosion resistance are given in Appendix X1 and Appendix X2.

1.9 The values stated in either inch-pound or SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.10 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.11 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

[1] These test methods are under the jurisdiction of ASTM Committee A01 on Steel, Stainless Steel and Related Alloysand are the direct responsibility of Subcommittee A01.14 on Methods of Corrosion Testing.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

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A370 Test Methods and Definitions for Mechanical Testing of Steel Products
A1084 Test Method for Detecting Detrimental Phases in Lean Duplex Austenitic/Ferritic Stainless Steels
G48 Test Methods for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by Use of Ferric Chloride Solution

TEST METHOD A—SODIUM HYDROXIDE ETCH TEST FOR CLASSIFICATION OF ETCH STRUCTURES OF DUPLEX STAINLESS STEELS

3. Scope

3.1 The sodium hydroxide etch test may be used for the acceptance of material but not for rejection. This test method may be used with other evaluation tests to provide a rapid method for identifying those specimens that are free of detrimental intermetallic phases as measured in these other tests.

3.2 The sodium hydroxide etch test may be used to screen specimens intended for testing in Test Method B, Charpy Impact Test for Classification of Structures of Duplex Stainless Steels, and in Test Method C, Ferric Chloride Corrosion Test for Classification of Structures of Duplex Stainless Steels.

3.3 Reference photomicrographs are provided to show classifications of etch structures of a particular stainless steel type that are equivalent to acceptable or to possibly unacceptable performance for each practice. When Test Method A is used as a screening test for Test Method B or Test Method C, specimens having acceptable etch structures need not be subjected to Test Method B or Test Method C.

3.4 Table 1 indicates the applicability and acceptance criteria for Test Method A. When Test Method A is specified as an acceptance test, specimens having other than acceptable etch structures may, at the option of the producer, be tested by Test Method B or Test Method C.

3.5 The steel shall be tested in the final solution heat treated condition or such other conditions as are agreed upon between the producer and the user.

4. Apparatus

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

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

TABLE 1 Applicability and Acceptance Criteria for Test Method A

Grade	Acceptable Etch Structure	Nonacceptable Etch Structure
S82441, S31803, S32205, S32750, S32760, S32550,S32520, J92205, J93404	unaffected structure (Fig. 1, Fig. 2)	possibly affected structure (Fig. 3, Fig. 4)
		affected structure (Fig. 5, Fig. 6) centerline structure (Fig. 7)

4.3 *Variable Resistance* (see Note 1).

4.4 *Cathode*—A cylindrical piece of conductive metal.

4.5 *Large Electric Clamp,* to hold the specimen to be etched.

4.6 *Metallurgical Microscope,* for examination of etched microstructures to 400 to 500 diameters.

4.7 *Electrodes of the Etching Cell*—The specimen to be etched is made the anode, and a cylindrical piece of metal as large as the specimen to be etched is made the cathode.

4.8 *Electrolyte,* sodium hydroxide (NaOH), reagent grade.

NOTE 1—The variable resistance and 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 For mill products, examination shall be made on a longitudinal or transverse section. For cast products, examination shall be made on a separately cast test coupon which was heat treated in the same furnace load as the casting it represents. Unless otherwise specified, selection of the test coupon size shall be at the discretion of the producer. Because high temperature or mechanical deformation associated with particular cutting processes may alter the structure of the steel, the cutting of the test specimen should be by a technique that prevents these effects. Alternatively, after the specimens are cut, any material that may have been affected by high temperature or deformation associated with the cutting should be removed by machining or wet grinding prior to testing.

5.2 For mill products, the specimen should allow for a survey across the full thickness of the section or, in the case of a heavy section, a survey from one surface through the mid-thickness of the section. The specimen shall include the mid-thickness.

5.3 For cast materials, the specimen shall be taken at approximately 1/4T.

5.4 *Polishing*—On all materials, cross-sectional surfaces should be polished to a metallographic finish suitable for examination at 400× after etching. Specimens containing welds should include base metal, weld heat–affected zone, and weld metal. The area to be etched may be prepared by grinding to an 80- or 120-grit finish on a grinding belt or wheel without excessive heating and then by polishing on successively finer emery papers, No. 1, ½, ⅙, ⅔, ⅜, and finer. Other methods of polishing may be acceptable.

5.5 *Etching Solution*—The solution for etching is prepared by adding 40 g of reagent grade sodium hydroxide (NaOH) to 100 g of distilled water.

5.6 *Etching Conditions*—The polished specimen should be etched at approximately 1 to 3 V dc, for 5 to 60 s (see Note 2).

NOTE 2—When etching is performed at 1 to 3 V dc with a platinum cathode for 5 to 60 s, any intermetallic phase is revealed by yellow, then brown, staining, followed by staining of the ferrite.

5.7 *Rinsing*—Following etching, the specimen should be rinsed thoroughly in hot water and in acetone or alcohol, followed by air drying.

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6. Classification of Etch Structures

6.1 The etched surface shall be examined on a metallurgical microscope at 400 to 500×.

6.2 The etched cross-sectional areas should be examined thoroughly by complete traverse of the full sample and across all zones such as weld metal, weld-affected zones, and base metal on specimens containing welds.

6.3 The etch structures are classified into the following types:

6.3.1 *Unaffected Structure* (Fig. 1, Fig. 2)—The ferrite has been etched without revelation of intermetallic phase. The interphase boundaries are smooth.

6.3.2 *Possibly Affected Structure* (Fig. 3, Fig. 4)—The ferrite has been etched with isolated indications of possible intermetallic phase. The interphase boundaries may show a fine waviness.

6.3.3 *Affected Structure* (Fig. 5, Fig. 6)—The indications of an intermetallic phase are readily revealed before or simultaneously with the staining of the ferrite during etching.

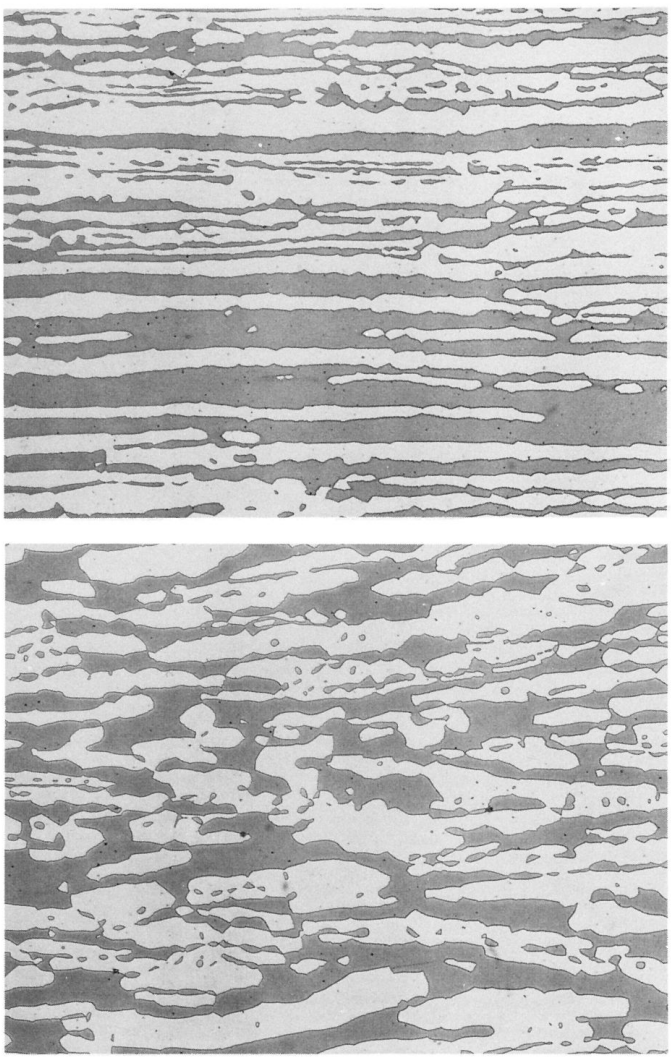

FIG. 1 Unaffected Structure in S31803: (*a*) Longitudinal Section and (*b*) Transverse Section (500× Magnification Before Reproduction)

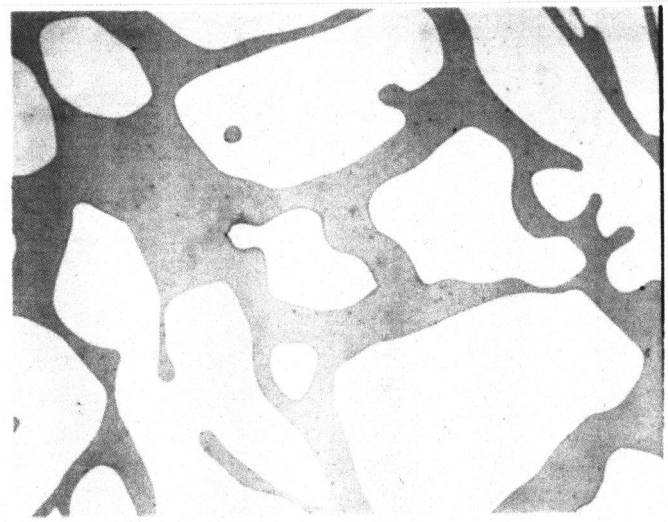

FIG. 2 (a) J92205: Unaffected Structure-Statically Cast (400× Magnification)

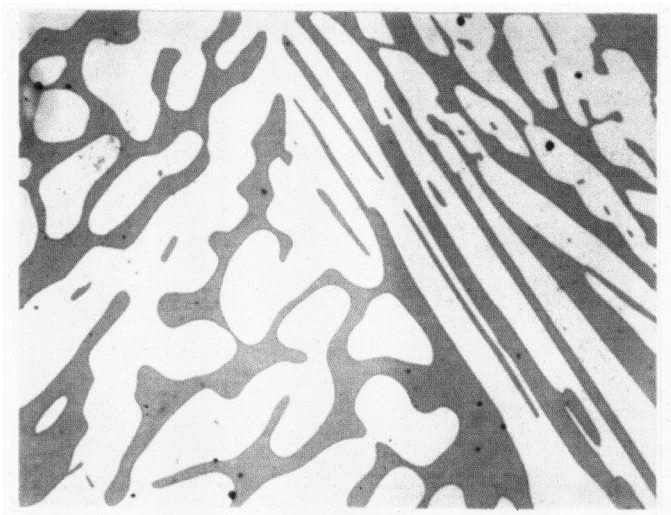

FIG. 2 (b) J92205: Unaffected Structure-Centrifugally Cast (400× Magnification) *(continued)*

6.3.4 *Centerline Structure* (Fig. 7)—An intermetallic phase is observed as a continuous or semi-continuous phase in the mid-thickness region of the product, with or without the affected structure outside of the mid-thickness region, indicative of segregation.

7. Interpretation and Use of the Etch Structure Classifications

7.1 When Test Method A is used as a screening test, the use of these etch structures depends on the test method for which the specimens are being screened. Important characteristics of each of the test methods are described as follows.

7.2 *Test Method B*—The Charpy impact test detects reductions in toughness from that of the optimal composition and processing. Such reductions may be attributable to intermetallic phases or to other causes not necessarily detectable by Test Method A. A Possibly Affected Structure is taken to be

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