



Designation: A923 – 23

# Standard Test Methods for Detecting Detrimental Intermetallic Phase in Duplex Austenitic/Ferritic Stainless Steels<sup>1</sup>

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 ( $\epsilon$ ) 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 thermo-mechanical 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).

<sup>1</sup> These test methods 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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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*:<sup>2</sup>

<sup>2</sup> 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

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).

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, 1/2, 1/6, 2/6, 3/6, 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.

**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)

**6. Classification of Etch Structures**

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

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.

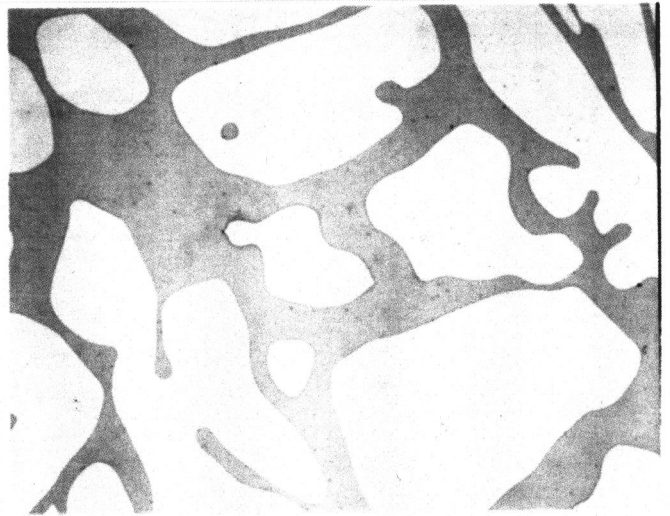


FIG. 2 (a) J92205: Unaffected Structure-Staticly Cast (400x Magnification)

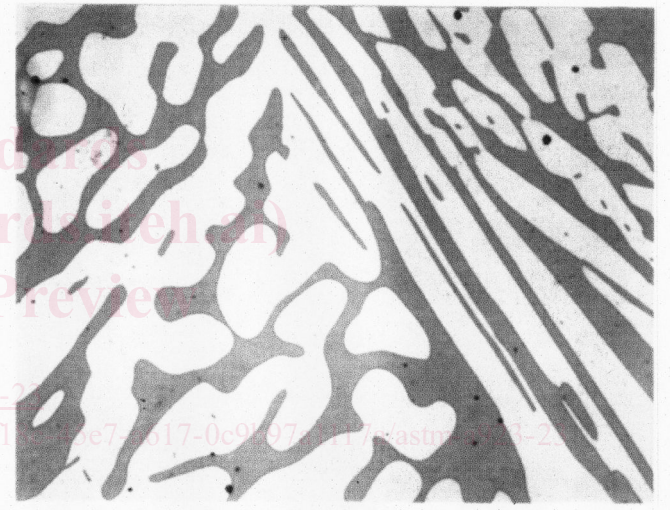


FIG. 2 (b) J92205: Unaffected Structure-Centrifugally Cast (400x Magnification) (continued)

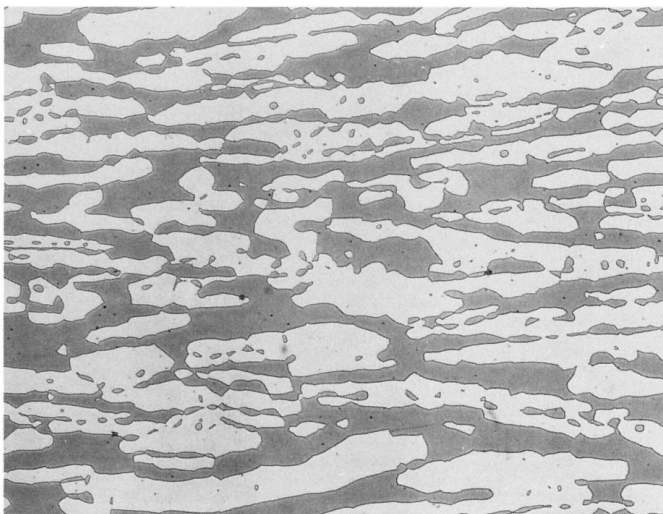
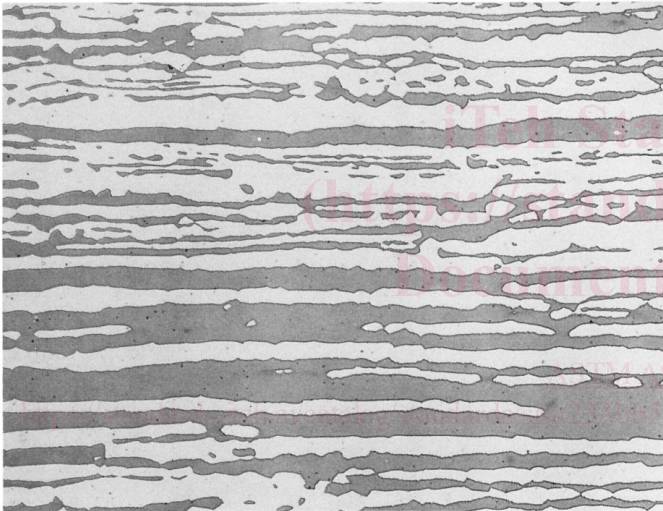


FIG. 1 Unaffected Structure in S31803: (a) Longitudinal Section and (b) Transverse Section (500x Magnification Before Reproduction)

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 likely to be

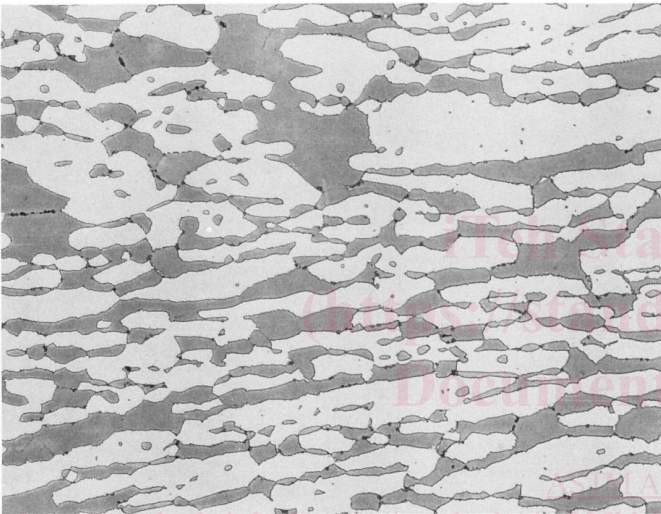
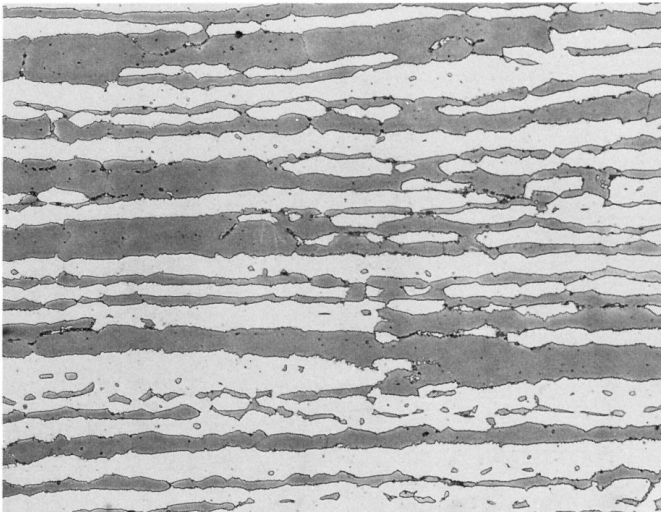


FIG. 3 Possibly Affected Structure in S31803: (a) Longitudinal Section and (b) Transverse Section (500× Magnification Before Reproduction)

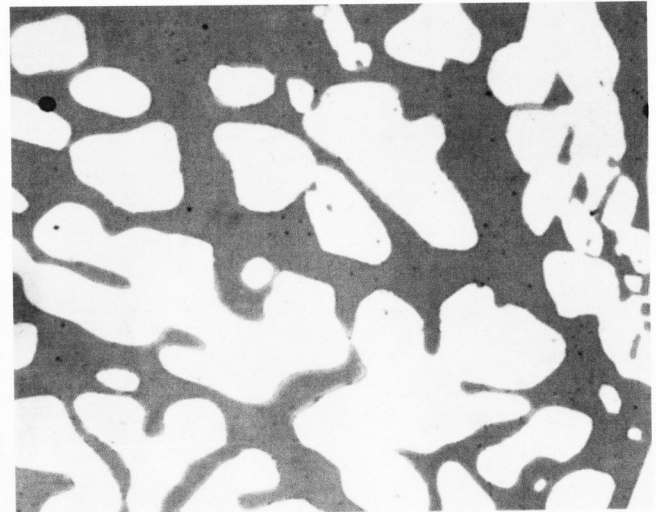


FIG. 4 (a) J92205: Possibly Affected Structure-Statically Cast (400× Magnification)

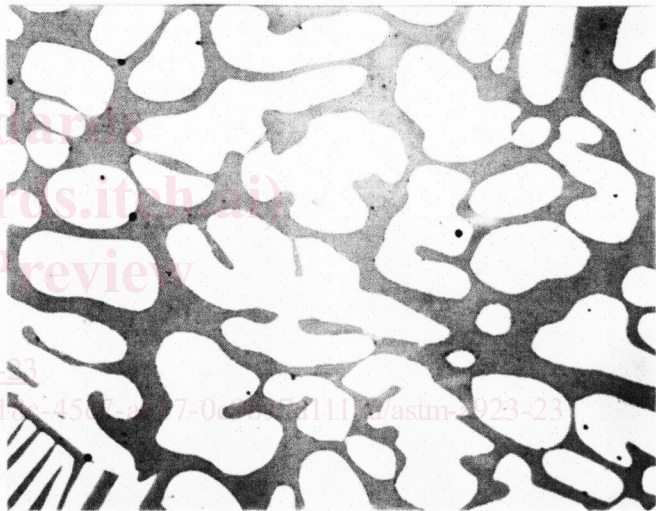


FIG. 4 (b) J92205: Possibly Affected Structure-Centrifugally Cast (400× Magnification) (continued)

associated with a loss of Charpy impact toughness ranging from slight to severe. An Affected Structure is associated with a severe loss of Charpy impact toughness. A Centerline Structure may or may not be detected by a Charpy test, depending on the orientation of the Charpy specimen.

7.3 *Test Method C*—The ferric chloride corrosion test is a 24-h test in 10 % ferric chloride. It will detect a loss of corrosion resistance associated with local depletion of chromium and molybdenum as a result of the precipitation of chromium-rich and possibly molybdenum-rich phases, not limited to intermetallic phases. An Affected Structure is associated with significant weight loss in the corrosion test. A Possibly Affected Structure is likely to be associated with significant weight loss in the corrosion test.

**TEST METHOD B—CHARPY IMPACT TEST FOR CLASSIFICATION OF STRUCTURES OF DUPLEX STAINLESS STEELS**

**8. Scope**

8.1 This test method describes the procedure for conducting the Charpy impact test as a method of detecting the precipitation of detrimental intermetallic phases in duplex stainless steels. The presence or absence of an indication of intermetallic phase in this test is not necessarily a measure of performance of the material in service with regard to any property other than

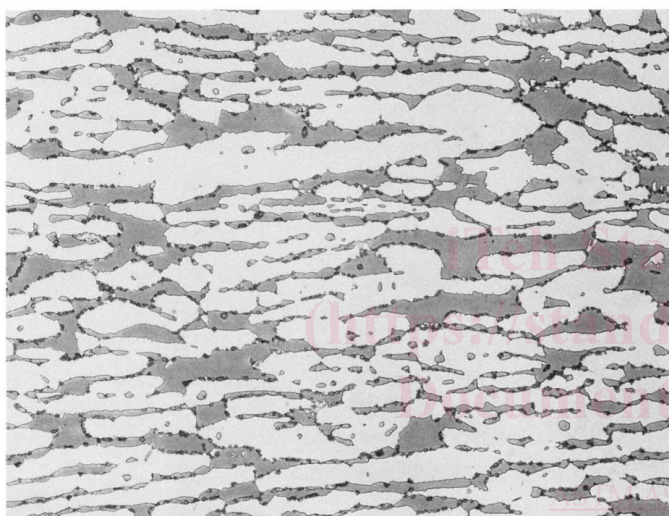
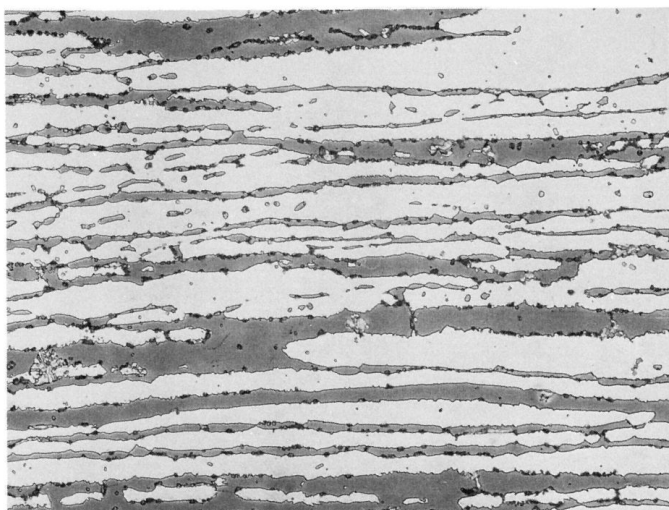


FIG. 5 Affected Structure in 8a S31803: (a) Longitudinal Section and (b) Transverse Section (500× Magnification Before Reproduction)

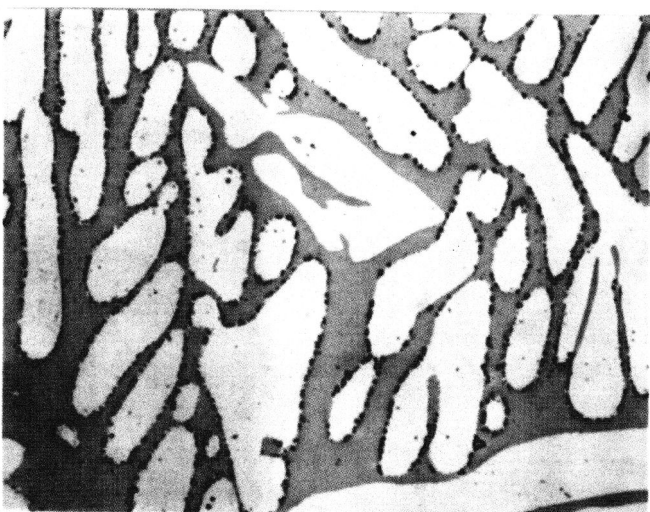


FIG. 6 J92205: Affected Structure (400× Magnification)

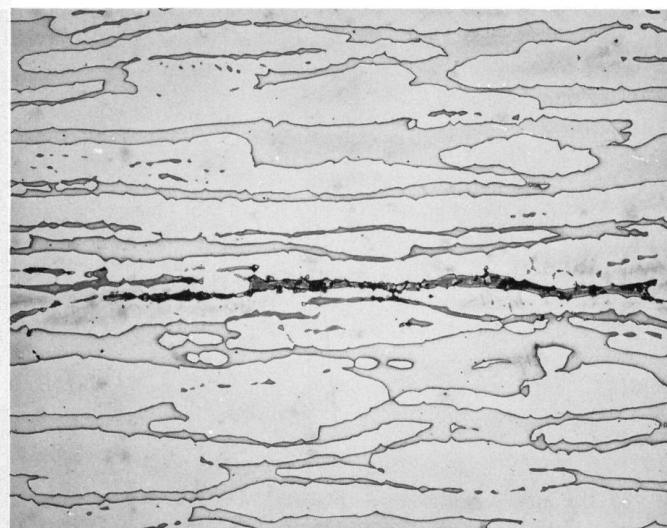


FIG. 7 S31803: Longitudinal Section (500× Magnification Before Reproduction)

that measured directly. The Charpy procedure as here applied is different from that as commonly applied for the determination of toughness and should not be used when characterization of material toughness is the purpose of the testing.

8.2 The Charpy impact test may be used to evaluate mill and cast products, provided that it is possible to obtain a specimen of relevant location and geometry.

8.3 Table 2 indicates the applicability and acceptance criteria for Test Method B.

### 9. Rapid Screening Test

9.1 Before testing by the Charpy impact test, specimens of the steel may be given a rapid screening test in accordance with the procedures of Test Method A, Sodium Hydroxide Etch Test for Classification of Etch Structures of Duplex Stainless Steels. Preparation, etching, and the classification of etch structures are described therein. Specimens having an etch structure described as Unaffected Structure in Test Method A will be essentially free of detrimental effect on impact toughness

TABLE 2 Applicability and Acceptance Criteria for Test Method B

Grade	Condition	Test Temperature	Minimum Impact Energy <sup>A</sup>
S82441	base metal	−40 °F (−40 °C)	40 ft-lb (54 J) <sup>A</sup>
S31803, S32205, J92205	base metal	−40 °F (−40 °C)	40 ft-lb (54 J) <sup>A</sup>
	heat-affected zone	−40 °F (−40 °C)	40 ft-lb (54 J) <sup>A</sup>
	weld metal	−40 °F (−40 °C)	25 ft-lb (34 J) <sup>A</sup>
S32750, S32760	base metal	−40 °F (−40 °C)	<sup>B</sup>
J93404	base metal	−50 °F (−46 °C)	40 ft-lb (54 J) <sup>A</sup>

<sup>A</sup> Energy for a full-size specimen. Required energy for a subsize specimen is reduced in direct proportion to the reduced area of the subsize specimen relative to that of the full-size specimen. Test may be conducted at a temperature below the specified temperature.

<sup>B</sup> The acceptable minimum impact energy shall be agreed upon by seller and purchaser.

related to the formation of intermetallic phase. Other mechanisms for loss of toughness may occur independently but are beyond the scope of this test method. Specimens showing Unaffected Structure in Test Method A are acceptable with respect to the absence of intermetallic phases, without the necessity of performing the Charpy test as described in Test Method B. All specimens having other than Unaffected Structure shall be tested by the Charpy impact test.

**10. Apparatus**

10.1 The test apparatus shall be as described in Test Methods and Definitions A370.

**11. Preparation of Test Specimens**

11.1 The test specimen shall be as described in Test Methods and Definitions A370.

11.2 An impact test for the purpose of detecting intermetallic phases shall consist of a single specimen taken from the product piece or lot to be represented.

11.3 For mill products, the test specimen may be aligned in either the longitudinal or transverse direction (see Note 3). For castings, the test specimen shall be parallel to the side of the test coupon; the notch may be either parallel to or perpendicular to the coupon surface.

NOTE 3—The impact toughness of a transverse specimen from mill products of duplex stainless steels is typically one half to two thirds of that of a longitudinal specimen.

11.4 Subsize specimens may be used for products with thickness less than that of a full-size Charpy specimen. Subsize specimens should be as large as permitted by the product, in quarter-size increments.

11.5 When this test is applied to a welded structure, or to any product having a less than uniform structure, particular attention should be paid to the location of the V-notch. For example, in the heat-affected zone of a weld, the degree of intermetallic formation may vary significantly over short distances as a function of the local thermal cycle. In such cases, the placement of the V-notch may affect the measured result significantly.

**12. Procedure**

12.1 Perform the test in accordance with the procedures described in Test Methods and Definitions A370.

12.2 The test temperature shall be as specified in Table 2 for the grade being evaluated.

**13. Acceptance Values and Retests**

13.1 Unless otherwise specified, the acceptance criteria shall be as given in Table 2.

13.2 If a test specimen shows a value below the specified minimum, one retest of two specimens is permitted. For acceptance, both retest specimens shall show a value at or above the specified minimum value.

13.3 A product that has failed the Charpy impact test may, at the option of the producer, be given a full anneal and retested.

**TEST METHOD C—FERRIC CHLORIDE CORROSION TEST FOR CLASSIFICATION OF STRUCTURES OF DUPLEX STAINLESS STEELS**

**14. Scope**

14.1 This test method describes the procedure for conducting the ferric chloride corrosion test for detecting the presence of detrimental intermetallic phases in duplex stainless steels. The presence or absence of corrosion 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 not associated with the precipitation of intermetallic phases (see Note 4).

14.2 The ferric chloride corrosion test may be used to evaluate mill products, provided that it is possible to obtain a specimen of relevant location and geometry.

14.3 Table 3 indicates the applicability and acceptance criteria for Test Method C.

NOTE 4—Although this test method uses some equipment and procedures similar to those of Test Methods G48, this test method should not be confused with Test Methods G48. This test method does not determine the critical pitting temperature or test for the suitability for use in a particular environment. This test method is designed solely for detection of the precipitation of detrimental intermetallic phases in duplex stainless steels.

**15. Rapid Screening Test**

15.1 Before testing by the ferric chloride corrosion test, specimens of the steel may be given a rapid screening test in accordance with the procedures of Test Method A, Sodium Hydroxide Etch Test for Classification of Etch Structures of Duplex Stainless Steels. Preparation, etching, and the classification of etch structures are described therein. Specimens having an etch structure described as Unaffected Structure in Test Method A will be essentially free of detrimental effects on pitting corrosion resistance as related to the formation of an intermetallic phase. Other mechanisms for loss of pitting resistance may occur independently but are beyond the scope of this test method. Specimens showing Unaffected Structure in Test Method A are acceptable with respect to the absence of intermetallic phases and need not be tested by the ferric chloride corrosion test as described in Test Method C. All specimens having other than Unaffected Structure shall be tested by the ferric chloride corrosion test.

**TABLE 3 Applicability and Acceptance Criteria for Test Method C**

Grade	Condition	Test Temperature	Maximum Acceptable Corrosion Rate Calculated from Weight Loss
S82441	base metal	15 °C (59 °F)	10 mdd
S31803, S32205	base metal	25 °C (77 °F)	10 mdd
S32750	weld metal	22 °C (72 °F)	10 mdd
S32750	base metal	40 °C (104 °F)	10 mdd
S32760	base metal	40 °C (104 °F)	10 mdd
S32550	base metal	40 °C (104 °F)	10 mdd
S32520	base metal	40 °C (104 °F)	10 mdd

**16. Apparatus**

16.1 *Glass Beakers*, 1000 mL, tall-form, or Erlenmeyer flasks, 1000 mL, wide neck, or 50 mm (2-in.) diameter test tubes, or other suitable glass containers.

16.2 *Glass Cradles* (Fig. 8)—The dimensions of the cradle shall be restricted to those that will permit its passage through the test container opening, a diameter of approximately 40 mm (1.6 in.) in the case of the Erlenmeyer flask.

16.3 *Water or Oil Bath*, constant temperature.

**17. Ferric Chloride Test Solution**

17.1 The test solution is prepared by dissolving 100 g of reagent-grade ferric chloride,  $FeCl_3 \cdot 6H_2O$ , in 900 mL of distilled water (approximately 6%  $FeCl_3$  by weight). The solution is filtered through glass wool or filter paper to remove insoluble particles.

17.2 The pH of the test solution shall be adjusted to approximately 1.3 prior to beginning the test by the addition of HCl or NaOH, as required.

**18. Test Specimen**

18.1 Various shapes and sizes of test specimens may be used. For flat products, a specimen should be approximately 25 mm by 50 mm (1 in. by 2 in.) by thickness. The full thickness of the product should be included. In the case of heavier sections, the specimen may be taken in a perpendicular orientation with dimensions of approximately 6 mm by 25 mm (1/4 in. by 1 in.) by thickness. In very heavy sections, the thickness dimension of the specimen may be cut so that one half to two thirds of the product thickness is tested.

18.2 Other product forms may be cut for test specimens convenient for testing, provided that the specimen exposes surfaces representative of the full thickness of the product.

18.3 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 grinding prior to testing.

18.4 For mill products, all surfaces of the specimen should be polished to a uniform finish at least equal to a 120-grit finish, or finer. Wet polishing is preferred. If used, dry polishing should be performed slowly to prevent overheating. Sharp edges of the specimen should be rounded, with care taken to remove all burrs.

18.5 The surface of the specimen shall not be chemically passivated by treatments such as nitric, citric, or phosphoric acid, or pickled by treatments such as nitric hydrofluoric acid, subsequent to grinding the surface.

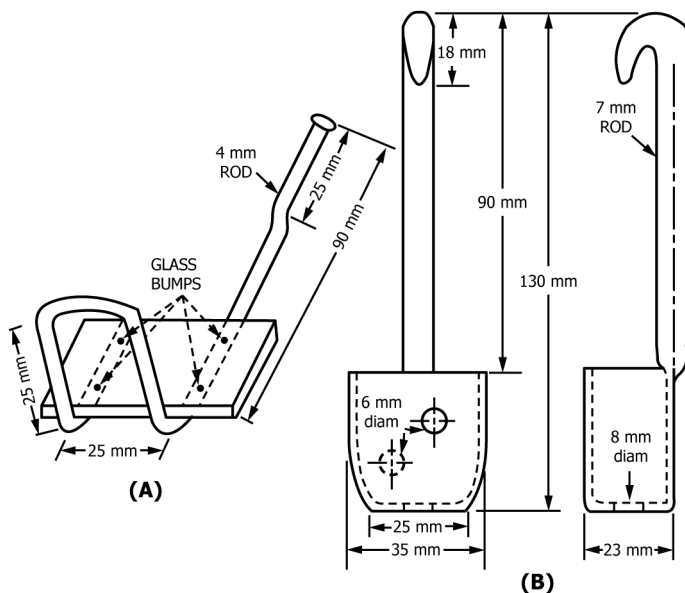
18.6 For other than mill products, testing of a specimen with the surface in the as-fabricated condition may be relevant to the application.

18.7 The dimensions of the specimen are measured, and the total exposed surface area is calculated.

18.8 The specimen should be cleaned with magnesiumoxide paste or equivalent, rinsed well with water, dipped in alcohol or acetone, and air dried. The specimen shall be weighed to the nearest 0.001 g or better. It should be stored in a desiccator until ready for testing.

**19. Procedure**

19.1 Perform the test using ferric-chloride solution with a volume at least the larger of 150 mL or 20 mL/cm<sup>2</sup> (125 mL/in.<sup>2</sup>) of the specimen surface area. Fill the test container with the required volume, transfer to the constant temperature bath, and allow to come to equilibrium at the desired test temperature.



**FIG. 8 Examples of Glass Cradles That Can Be Used to Support the Specimen**