Designation: A1084 – 15a

Standard Test Method for Detecting Detrimental Phases in Lean Duplex Austenitic/Ferritic Stainless Steels[1]

1. Scope*

1.1 The purpose of this test method is to allow detection of the presence of detrimental chromium-containing phases in selected lean duplex stainless steels to the extent that toughness or corrosion resistance is affected significantly. Such phases can form during manufacture and fabrication of lean duplex products. This test method does not necessarily detect losses of toughness nor corrosion resistance attributable to other causes, nor will it identify the exact type of detrimental phases that caused any loss of toughness or corrosion resistance. The test result is a simple pass/fail statement.

1.2 Lean duplex (austenitic-ferritic) stainless steels are typically duplex stainless steels composed of 30 to 70 % ferrite content with a typical alloy composition having Cr > 17 % and Mo < 1 % and with additions of Nickel, Manganese, Nitrogen and controlled low carbon content as well as other alloying elements. This standard test method applies only to those alloys listed in Table 1. Similar test methods for some higher alloyed duplex stainless steels are described in Test Methods A923, but the procedures described in this standard differ significantly for all three methods from the ones described in Test Methods A923.

1.3 Lean duplex stainless steels are susceptible to the formation of detrimental chromium-containing compounds such as nitrides and carbides and other undesirable phases. Typically this occurs during exposures in the temperature range from approximately 300 to 955°C (570 to 1750ºF) with a maximum susceptibility in the temperature range around 650 to 750°C (1200 to 1385ºF). The speed of these precipitation reactions is a function of composition and the thermal or thermo-mechanical history of each individual piece. The presence of an amount of these phases can be detrimental to toughness and corrosion resistance.

1.4 Because of the low molybdenum content, lean duplex stainless steels only exhibit a minor susceptibility to sigma or other types of molybdenum containing intermetallic phases. Heat treatment, that could lead to formation of small amounts of molybdenum containing intermetallics, would result in a large amount of precipitation of detrimental nitrides or carbides, long before any signs of sigma and similar phases would be observed.

1.5 Correct heat treatment of lean duplex stainless steels can eliminate or reduce the amount and alter the characteristics of these detrimental phases as well as minimizing Cr-depletion in the matrix phase in the immediate vicinity of these phases. Adequately rapid cooling of the product from a suitable annealing temperature provides the maximum resistance to formation of detrimental phases by subsequent thermal exposures. For details of the proper annealing temperature recommendations for the alloy and product in question, the user is referred to the relevant applicable ASTM product specification.

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

1.7 These test methods include the following:

1.7.1 *Test Method A*—Etch Method for detecting the presence of potentially detrimental phases in Lean Duplex Stainless Steels

1.7.2 *Test Method B*—Charpy V-notch Impact Test for determining the presence of detrimental phases in Lean Duplex Stainless Steels.

1.7.3 *Test Method C*—Inhibited Ferric Chloride Corrosion Test for determining the presence of detrimental phases in Lean Duplex Stainless Steels.

1.7.4 Examples of the correlation of thermal exposures, the occurrence of detrimental phases, and the degradation of toughness and corrosion resistance are given in Appendix X2, Appendix X3, and the References.

1.8 Guidelines for the required data needed for subcommittee A01.14 to consider listing a lean duplex stainless steel in this standard test method are given in Annex A1.

1.9 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to other units that are provided for information only and are not considered standard.

---

[1] This test method is under the jurisdiction of ASTM Committee A01 on Steel, Stainless Steel and Related Alloys and is the direct responsibility of Subcommittee A01.14 on Methods of Corrosion Testing.

Current edition approved Sept. 1, 2015. Published September 2015. Originally approved in 2013. Last previous edition approved in 2015 as A1084 – 15. DOI: 10.1520/A1084 – 15A.

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

TABLE 1 List of Lean Duplex Grades Covered by This Standard

Grades
UNS S32101
UNS S32304
UNS S32202
UNS S82011

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A370 Test Methods and Definitions for Mechanical Testing of Steel Products
A923 Test Methods for Detecting Detrimental Intermetallic Phase in Duplex Austenitic/Ferritic Stainless Steels
A1084 Test Method for Detecting Detrimental Phases in Lean Duplex Austenitic/Ferritic Stainless Steels
E6 Terminology Relating to Methods of Mechanical Testing
E23 Test Methods for Notched Bar Impact Testing of Metallic Materials
G15 Terminology Relating to Corrosion and Corrosion Testing (Withdrawn 2010)[3]
G48 Test Methods for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by Use of Ferric Chloride Solution

3. Terminology

3.1 *Definitions:*

3.1.1 The terminology used herein, if not specifically defined otherwise, shall be in accordance with Terminology E6 and G15. Definitions provided herein and not given in Terminology E6 or in G15 are limited only to this standard.

4. Significance and Use

4.1 Test Method A shall only be used to supplement the results of Test Methods B and C. It shall not be used as a rejection criterion, nor shall it be used as an acceptance criterion. Test Methods B and C are intended to be the procedures giving the acceptance criteria for this standard.

4.2 Test Method A can reveal potentially detrimental phases in the metallographic structure. As the precipitated detrimental phases can be very small, this test demands high proficiency from the metallographer, especially for thinner material.

4.3 The presence of detrimental phases is readily detected by Test Methods B and C provided that a sample of appropriate location and orientation is selected.

4.4 The tests do not determine the precise nature of the detrimental phase but rather the presence or absence to the extent that the normally expected toughness and corrosion resistance of the material are significantly affected.

4.5 This standard covers testing of samples taken from coil, coil- and plate mill plate, sheet, tubing, piping, bar and deformed bar, though some of these products might not be suitable for testing according to Method B (see Test Method B for further details). Other product forms have thus far not been sufficiently tested and documented to be an integral part of this standard, though the standard does not prohibit testing of these product forms according to the three test methods. For these other product forms, this standard gives only limited and non-exhaustive guidance as to interpretation of result and associated acceptance criteria.

4.6 Testing on product forms outside the present scope of this standard shall be agreed between purchaser and supplier.

5. Sampling, Test Specimens, and Test Units

5.1 *Sampling:*

5.1.1 Because the occurrence of detrimental 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 detrimental phases. In the case of common heat treatment, this region can be that which cooled most slowly or undergoes extremely rapid cooling.

5.1.2 For practical purposes, it is considered sufficient that the sampling location for flat mill products be from a location that is at least twice the material thickness from the as-heated edges.

5.1.3 Purchaser and supplier may agree on more detailed rules regarding the sampling location.

5.1.4 The number of samples as well as frequency of sampling shall be agreed between purchaser and supplier of the material.

5.2 *Test Specimens and Test Units:*

5.2.1 Details of test specimen and test unit requirements are listed together with each of the Test Methods A, B and C.

TEST METHOD A—ETCH METHOD FOR EVALUATION OF THE PRESENCE OF POTENTIALLY DETRIMENTAL PHASES IN LEAN DUPLEX STAINLESS STEELS

6. Introduction

6.1 The etch test in this standard shall only be used for exploratory purposes. The reason for this is the small size of the detrimental phases typically occurring in lean duplex stainless steels and the difficulty in achieving a fully reproducible etch structure, which depends on factors such as specimen size and geometry, etching current and potential, composition of the lean duplex as well as the amount and type of detrimental phases present. The test method contained in this standard is, however, the best known metallographic procedure to show the appearance and approximate amount of detrimental phases in a lean duplex stainless steel.

---

[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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

6.2 As there is no formal test result from the metallographic etch method, the actual test method is attached to this standard as Appendix X1.

TEST METHOD B—CHARPY V-NOTCH IMPACT TEST FOR DETERMINATION OF THE PRESENCE OF DETRIMENTAL PHASES IN LEAN DUPLEX STAINLESS STEELS

7. Scope

7.1 This test method describes the procedure for conducting the Charpy V-notch impact test as a method of detecting the precipitation of detrimental phases in lean duplex stainless steels. The presence or absence of an indication of a detrimental phase in this test is not necessarily a measure of performance of the material in service with regard to any property other than that measured directly. The Charpy V-notch procedure as applied here is different from that commonly applied for the determination of toughness and shall not be used when characterization of material toughness is the purpose of the testing.

8. Significance and Use (Test Method B)

8.1 The Charpy V-notch impact test may be used to evaluate mill products, provided that it is possible to obtain a specimen of the proper size from a relevant location.

8.2 Charpy V-notch impact toughness of a material is affected by factors other than the presence and absence of detrimental phases. These factors are known to include different compositions, even when the material is in fully annealed condition; small and otherwise acceptable variations in austenite/ferrite balance; and the lamellar distance between phases. Testing transverse and longitudinal test specimens from mill products can also give different absolute levels of impact toughness.

8.3 Table 2 indicates the applicability and acceptance criteria for Test Method B. These acceptance criteria have been shown to allow for the natural variation of impact toughness in sound material tested in the transverse direction on plate and in the longitudinal direction on bar and deformed bar, while still being able to identify whether detrimental amounts of undesirable phases are present.

TABLE 2 Applicability and Acceptance Criteria for Test Method B

Grade	Sampling Location	Test Temperature	Minimum Impact Energy[A]
S32101	base metal	Room temperature[B]	70 J (50 ft-lb)
S32304	base metal	Room temperature[B]	100 J (75 ft-lb)
S32202	base metal	Room temperature[B]	70 J (50 ft-lb)
S82011	base metal	Room temperature[B]	70 J (50 ft-lb)

[A] Energy for a full-size specimen tested in transverse direction for flat rolled products and tested in the longitudinal direction for bar products. Required energy for a sub-size specimen is discussed further in subsection 10.1.3 and Note 2.

[B] In this standard, room temperature is defined as the temperature range 23 ± 5ºC (73 ± 9ºF).

8.4 Acceptance criteria for Test Method B for other products including mill welded pipe, weldments and weld metal are not presently covered by this standard, though purchaser and supplier may agree upon an acceptance criteria (see Note 1). Note that the results of weldment testing will depend on the filler metal or weld deposit chemistry.

8.5 Acceptance criteria of sub-size specimens are not covered by this standard, though purchaser and supplier may agree upon a proper conversion factor of the given acceptance criteria in Table 2. Conversion factors generally vary by product type and dimensions of product for which the sub-size specimen sampling is needed (see Note 2).

NOTE 1—As no data has been presented to subcommittee A01.14 for welded mill products or other products, no recommendation can be given as to the acceptance criteria for these products. Any acceptance criteria and other details of the test should be supported with data from a pre-qualification test in line with the minimum requirements of Annex A1 in this standard.

NOTE 2—As stated in Test Methods and Definitions A370, Appendix A5.3.3 and Test Methods E23, Appendix X1.3, there is no general correlation between impact values obtained with specimens of different size or shape. However, limited correlations may be established for specification purposes on the basis of special studies of particular materials and particular specimens. It is commonly seen that the conversion factor is set directly proportional to the ratio between standard and sub-size specimen fracture surface area or a percentage thereof, though whether this is an acceptable way forward to still be able to identify the presence or absence of detrimental phases needs to be documented.

9. Apparatus

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

10. Test Specimens

10.1 *General Requirements (All Products):*

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

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

10.1.3 Provided purchaser and supplier have agreed upon a proper acceptance criterion, sub-size specimens may be used for products with thickness less than that of full-size Charpy V-notch specimen. Required energy for sub-size specimens shall be established and agreed upon based on the specific product type and geometry in question.

10.2 *Flat Products (Sheet, Coil, Plate):*

10.2.1 The specimen shall be prepared in the transverse direction. The notch shall be perpendicular to the major rolled surface.

10.3 *Non-deformed bar products:*

10.3.1 The specimen shall be prepared in the longitudinal direction.

10.4 *Deformed Bar Products:*

10.4.1 The specimen shall be prepared in the longitudinal direction.

10.5 *Other Products Including Mill Pipe:*

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

10.5.2 Following the guidelines of Test Methods A370, the specimen preparation method shall be agreed between purchaser and supplier.

11. Procedure

11.1 Perform the test for a single specimen in accordance with the procedures described in Test Methods and Definitions A370.

11.2 The test temperature shall be as specified in Table 2 or lower (see also subsection 12.3) for the grade being evaluated.

12. Acceptance Values and Retests

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

12.2 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.

12.3 Testing at lower temperatures than indicated in Table 2 is permissible and if the obtained impact energy is higher than the acceptance criteria indicated in Table 2 then the sample is approved in accordance with Test Method B of this test method. Failure at a lower test temperature than indicated in Table 2 does not imply that the sample has failed Test Method B, but only that the test shall not be counted as a proper test and a new test shall be performed at the temperature specified in Table 2.

12.4 A product that has failed the Charpy V-notch impact test may be given a full anneal and retested at the option of the supplier.

TEST METHOD C—INHIBITED FERRIC CHLORIDE CORROSION TEST FOR DETERMINATION OF THE PRESENCE OF DETRIMENTAL PHASES IN LEAN DUPLEX STAINLESS STEELS

13. Scope

13.1 This test method describes the procedure for conducting an inhibited ferric chloride corrosion test for detecting the presence of detrimental phases in lean duplex stainless steels. The presence or absence of corrosion attack in this test is not necessarily a measure of 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 detrimental phases (see Note 3).

13.2 The test method uses a ferric chloride solution inhibited by addition of sodium nitrate, since a standard ferric chloride solution is too aggressive to give valuable results for lean duplex stainless steels.

NOTE 3—Although this test method uses some equipment and procedures similar to those of Test Methods G48, this test method shall 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 phases in lean duplex stainless steels. The inhibited solution might not give the same ranking as the uninhibited solution, when comparing a range of stainless steels, but it can reveal the presence of deleterious phases in the lean duplex stainless steels listed in Table 1.

14. Significance and Use (Test Method C)

14.1 The inhibited ferric chloride corrosion test may be used to evaluate mill products as well as fabricated products, provided that it is possible to obtain a specimen from a relevant location and having the proper geometry.

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

14.3 Acceptance criteria for weldment and weld metal shall be agreed upon between purchaser and supplier of the product in question prior to testing. Results obtained from testing of weldments also depend on the filler metal and weld deposit chemistry.

15. Apparatus

15.1 *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.

15.2 *Glass Cradles* (Fig. 1)—The dimensions of the cradle shall be restricted to those that permit its passage through the test container opening, a diameter of approximately 40 mm (1.6 in.) in the case of the Erlenmeyer flask.

15.3 *Constant Temperature Device*—Water or Oil bath or other device that ensures constant temperature of solution and specimen.

16. Inhibited Ferric Chloride Test Solution

16.1 Dissolve 55.1 g of reagent-grade ferric chloride, $FeCl_3 \cdot 6H_2O$, and 6.6 g of reagent-grade sodium nitrate, $NaNO_3$, in 600 mL of distilled water (approximately 5% $FeCl_3$ and 1% $NaNO_3$ by weight). Filter the solution through glass wool or filter paper to remove insoluble particles.

17. Test Specimen

17.1 *General Requirements (All Products):*

17.1.1 Various shapes and sizes of test specimens may be used.

TABLE 3 Applicability and Acceptance Criteria for Test Method C

Grade	Sample Location	Test Temperature	Maximum Acceptable Corrosion Rate Calculated from Weight Loss
S32101	base metal	25°C (77°F)	10 mdd[A]
S32304	base metal	25°C (77°F)	10 mdd[A]
S32202	base metal	25°C (77°F)	10 mdd[A]
S82011	base metal	25°C (77°F)	10 mdd[A]

[A] For a definition of "mdd", see Note 4.

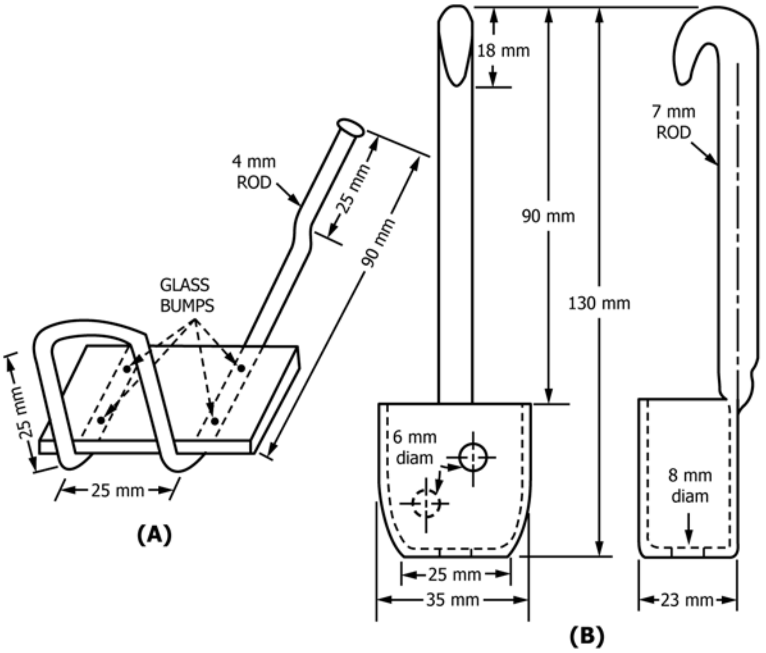

FIG. 1 Examples of Glass Cradles That Can Be Used to Support Specimen

17.1.2 After the specimens are cut, any material that might have been affected by high temperature or deformation associated with the cutting shall be removed by machining or polishing prior to testing. This procedure shall include rounding of sharp edges with care taken to remove all burrs.

17.1.3 For all polishing procedures, wet polishing is preferred. If used, dry polishing shall be performed slowly to prevent overheating.

17.1.4 All polishing shall be done to a 120-grit finish or finer.

17.1.5 For other than mill products, testing of a specimen with the surface in the as-fabricated condition can be relevant. However, if oxide scales from heat treatment or weldments are visibly present, then they shall be removed by polishing prior to testing as the oxide scale is likely give rise to a weight loss higher than 10 mdd despite the fact that no actual attack on the metallic part of the specimen is occurring.

17.2 *Flat Products (Coil, Plate, Sheet):*

17.2.1 The test specimen shall be approximately 25 by 50 mm (1 by 2 in.) by thickness. The full thickness of the product shall be included if practical. In the case of thicker sections, the specimen shall be taken in a perpendicular orientation so that the thickness of the product becomes the longest dimension of the specimen resulting in approximate specimen dimensions of 6 by 25 mm (¼ by 1 in.) by product thickness. In very thick sections, the thickness dimension of the specimen shall be cut so that one-half to two-thirds of the product thickness is tested resulting in approximate specimen dimensions of 6 by 25 mm (¼ by 1 in.) by one-half to two-thirds product thickness.

17.2.2 All surfaces shall be polished to a uniform finish.

17.3 *Non-Deformed Bar Products:*

17.3.1 All surfaces of the specimen shall be polished to a uniform finish.

17.4 *Mill Tube and Pipe Products:*

17.4.1 The ID side shall be left as is, whereas cut and OD surfaces shall be polished to a uniform finish.

17.5 *Mill deformed bar products:*

17.5.1 For practical reasons, the default specimen preparation shall be to let the deformed surface be as is, whereas cut surfaces shall be polished to a uniform finish.

17.5.2 If agreed between purchaser and seller, the deformed surface may be machined and polished to a 120-grit finish, or finer.

17.6 *Other Product Forms:*

17.6.1 Test specimens shall be cut into test specimens convenient for testing, provided that the specimen exposes surfaces representative of the full thickness of the product.

17.6.2 All surfaces of the specimen shall be polished to a uniform finish.

17.7 *Test Specimen Preparation*—After cutting and polishing:

17.7.1 Subsequent to polishing of the required surfaces, the surfaces of the specimen shall not be chemically passivated by any treatments such as nitric, citric, or phosphoric acid, or pickled by treatments such as nitric/hydrofluoric acid mixture or other pickling acids.

17.7.2 Measure the dimensions of the specimen and calculate the total exposed surface area.

17.7.3 Clean the specimen with magnesium oxide paste or equivalent. Rinse the specimen well with water followed by a dip in alcohol or acetone. Finally dry the specimen in air. The specimen shall be weighed to a precision of 0.001 g or better. Store the specimen in a desiccator until ready for testing.

18. Procedure

18.1 Perform the test using the nitrate-inhibited ferric-chloride solution with a volume at least the larger of 150 mL or

20 mL/cm^2 (125 mL/in.^{2}) of the specimen surface area. Fill the test container with the required volume of solution, transfer to the constant temperature bath, and allow it to come to equilibrium at the desired test temperature.

18.2 Unless otherwise specified, test temperatures shall be used as indicated for each type of steel in Table 3, maintained within an accuracy of ±1°C (2°F) during the test.

18.3 Place the specimen in the glass cradle and immerse in the test solution once the temperature has been established. Maintain the test temperature throughout the test. Cover the test container with a watch glass during the test period. Unless otherwise specified, the test period shall be 24 h.

18.4 At the end of the 24 h test period, remove the specimen from the solution, rinse with water, scrub with a soft bristle brush under running water to remove corrosion products, dip in acetone or alcohol, and dry in air. Ultrasonic cleaning is a permitted alternative when there are corrosion products that are difficult to remove.

18.5 Weigh the specimen to a precision of 0.001 g or better and reserve for examination.

19. Acceptance Values

19.1 The corrosion rate is calculated in accordance with the weight loss and total surface area (see Note 4). The calculated corrosion rate shall not exceed the indicated maximum value (see Note 5) for the type of stainless steel in question as indicated in Table 3.

NOTE 4—The corrosion rate is calculated in accordance with the following:

corrosion rate (mdd) = weight loss (mg) / [specimen area (dm^2) × time (days)]

NOTE 5—It is probable that corrosion occurs in the form of pitting. The calculation of a uniform corrosion rate is an inappropriate method of expressing the rate of pitting corrosion. However, in this case, the calculation of a corrosion rate is used primarily to normalize the weight loss for the variety of specimen sizes and shapes permitted.

19.2 If the specimen shows a corrosion rate in excess of the maximum allowed valued as found in Table 3, one retest on two new specimens from the same product is permitted. No retest specimen shall exhibit a corrosion rate in excess of the maximum allowed value.

19.3 Testing temperatures higher than indicated in Table 3 are permissible. If such a test has a corrosion rate less than that specified in Table 3, then the sample is approved in accordance with Test Method C of this specification. Failure at a test temperature higher than indicated in Table 3 does not imply that the sample has failed test Method C, but only that the test shall not be counted as a proper test and shall be redone at the temperature specified in Table 3. The same test specimen shall not be reused for testing at a lower temperature.

19.4 At the option of the producer, a product that has failed the inhibited ferric-chloride corrosion test may be given a full anneal and retested as if it had not been tested before.

19.5 When testing materials in the as-fabricated condition, weight loss can occur with no visible pitting. The cause can be surface inhomogeneities or contamination. In such cases a retest shall be performed after removing the entire original surface and finishing the surface by grinding to a 120-grit finish.

20. Report for Test Methods A, B and C

20.1 *Test Method A*—No test result is reported for this test method. But the specimen size, the etching parameters used and the magnification used should be recorded together with at least one representative image of the etched microstructure for documentation.

20.2 *Test Method B*—Report the test result as being either passed or failed based on the recorded impact toughness relative to the specified acceptance criteria. For informational purposes, record the test temperature and the measured impact toughness.

20.3 *Test Method C*—Report the test result as being either passed or failed based on the recorded corrosion rate relative to the specified acceptance criteria. For failed specimens, record the general position (what surface, edge, or both) of the corroded area; otherwise, report the failed specimen having a "non-visible" attack. For informational purposes, record the test temperature, duration of the test, the measured weight loss and the calculated corrosion rate in "mdd."

21. Precision and Bias for Test Method A, B and C

21.1 *Precision and Bias*—No information is presented about either the precision or bias of Test Method A1084 for the microstructure evaluation, nor for the pass/fail results of impact toughness and corrosion testing in Test Methods A, B and C since the test results are non-quantitative.

22. Keywords

22.1 acceptance testings; detrimental phases; duplex stainless steels; etch methods; ferric chloride testings; impact toughness testings; inhibited ferric chlorides; lean duplexes; microstructure evaluations; secondary phases; stainless steels

ANNEX

(Mandatory Information)

A1. GUIDELINES REGARDING REQUIREMENTS FOR INCORPORATING NEW ALLOYS AND PRODUCTS

A1.1 When introducing new alloys or products to be incorporated into this standard the following minimum requirements shall be considered by the responsible subcommittee.

A1.2 Any new alloy submitted for incorporation shall be consistent with the overall description of the term "lean duplex alloy" as described in subsection 1.2 of this standard and shall be identified specifically with its UNS number in Table 1.

A1.3 Appropriate maximum or minimum test temperatures and associated acceptance criteria shall be supplied for both Test Methods B and C respectively together with information on the test specimen preparation used.

A1.4 Examples of microstructural evaluation shall be presented to the subcommittee to validate that the existing examples of microstructure shown in Appendix X2 are relevant for the new alloy/product. If it is found that there is a significant difference in the microstructural appearance, then supportive examples should at the discretion of subcommittee A01.14 be incorporated in to Appendix X2.

A1.5 Test data and microstructure photographs shall be presented that illustrate the ability of the suggested test temperature and acceptance criteria to differentiate between "good" and "bad" material.

A1.6 The product forms covered by the suggested new alloy and test criteria shall be described in the application and if necessary incorporated into the proposed changes of the standard.

APPENDIXES

(Nonmandatory Information)

X1. TEST METHOD A—ETCH METHOD FOR EVALUATION OF THE PRESENCE OF POTENTIALLY DETRIMENTAL PHASES IN LEAN DUPLEX STAINLESS STEELS

X1.1. Scope

X1.1.1 The etch test in this standard shall only be used for exploratory purposes. The detrimental phases in lean duplex stainless steels are typically small in size, and their presence can therefore be difficult to identify. However, the etchant enlarges the appearance of these precipitates. The appearance of the etched microstructure depends strongly on the size and shape of the specimen, as well as on the exact current and potential applied. Anomalous effects around the edges of the specimen can be caused by current density variations during etching, therefore the light optical microscopy examination shall predominantly be done in the central regions of examined specimens.

X1.1.2 Examples of photomicrographs are provided in Appendix X2, which show structures that have failed and passed Test Methods B and C.

X1.2. Significance and Use (Test Method A)

X1.2.1 The detrimental phases in lean duplex stainless steels can be of such small size that standard light optical microscopy can have problems in detecting them especially from a quantitative or semi-quantitative point of view.

X1.2.2 Careful comparison of different heat treatment histories on similar samples evaluated in the same laboratory and by the same operator has shown expected differences in the apparent amount of detrimental phases.

X1.2.3 The recommended etching technique in this standard shall be seen as an exploratory test and shall be supplemented with testing according to Methods B or C.

X1.2.4 Test Method A is well suited for validation and interpretation of the results from Methods B and C.

X1.2.5 Characterization of the microstructure evaluation according to Method A is most meaningful when reference material of the same material at a similar size, which has already been evaluated by Methods B or C, is available for comparison (see also Note X1.1 and Appendix X2 and Appendix X3).

X1.2.6 Any attempt to characterize the microstructure is best facilitated by comparison with similar samples that have been heat treated for different lengths of time in the sensitive temperature range to create different levels of detrimental phases.

NOTE X1.1—ASTM subcommittee A01.14 has in November 2012 initiated an ILS activity (ILS #0909) that amongst other issues intends to further evaluate whether sufficiently precise etching specifications can lead to a microstructure characterization that is reliable for screening purposes such as Test Methods A923 presently uses for higher alloyed duplex stainless steels.

X1.3. Apparatus

X1.3.1 *Source of Direct Current*—Battery, generator, or rectifier capable of supplying approximately 15 V and 20 A.

X1.3.2 *Variable Resistance* (see Note X1.2).

X1.3.3 *Ammeter*—Range from 0 to 30 A (see Note X1.2).

X1.3.4 *Cathode*—A cylindrical piece of conductive metal.

X1.3.5 *Electric Clamp,* suitable to hold the specimen to be etched.

X1.3.6 *Metallurgical Microscope,* for examination of etched microstructures at up to 1000× magnification.

X1.3.7 *Electrodes for 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.

NOTE X1.2—The variable resistance and ammeter are placed in the circuit to measure and control the current on the specimen to be etched.

X1.4 Electrolytes

X1.4.1 Oxalic Acid ($C_2O_4H_2$), reagent grade.

X1.5 Preparation of Test Specimens

X1.5.1 For mill products, examination shall be made on either a longitudinal or transverse section. 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 can alter the structure of the steel, the cutting of the test specimen shall be by a technique that prevents such alterations. Alternatively, after the specimens are cut, any material that might have been affected by high temperature or deformation associated with the cutting shall be removed by machining or wet grinding prior to testing.

X1.5.2 For mill products, the specimen shall 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.

X1.5.3 *Polishing*—On all materials, cross-sectional surfaces shall be polished to a metallographic finish suitable for examination at 400× and 1000× after etching. Specimens containing welds shall 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 abrasive paper. Other methods of polishing may be acceptable.

X1.5.4 *Etching Solution*—The solution for etching is prepared by adding 10 g of reagent grade oxalic acid ($C_2O_4H_2$) to 90 ml of distilled water.

X1.5.5 *Etching Conditions*—The polished specimen shall be etched at approximately 6 to 7 V dc, for 3 to 20 s (see also Note X1.3).

X1.5.6 *Rinsing*—Following etching, the specimen shall be rinsed thoroughly in hot water and in acetone or alcohol, followed by air drying.

NOTE X1.3—The current density normally varies from ~3 to 30 mA/mm^2 depending on the shape of the etched cross-section and the degree of sensitization.

X1.6 Evaluation of Test Specimens

X1.6.1 The specimen shall be examined in a light optical microscope at an appropriate magnification of 400 to 1000×. Material with a gauge thickness less than 3 mm (0.12 in.) shall be examined at 1000×. Examples of microstructures are shown in Appendix X2 for thick and thin gauge UNS S32101 material and for thick gauge UNS S32304 material.

X2. MICROSTRUCTURAL EVALUATION OF ETCHED STRUCTURES

X2.1 The appearance of the etched microstructure can vary considerably depending on the exact etching technique used by individual operators. Apart from an experienced operator, the best way to achieve consistent and meaningful results with the etch method in Method A, is to have reference material of the same material at a similar size, which has already been evaluated by Methods B or C, available for comparison.

X2.2 Further, the microstructure evaluation is best facilitated by comparison with different relevant samples that have been heat treated for different lengths of time in the sensitive temperature range to create different levels of detrimental phases. Below are examples of etch structures from specimens that have passed or failed Test Method B or C, or both.

X2.3 *Microstructures That Passed Test Method B or C, or Both*:

X2.3.1 Fig. X2.1, Fig. X2.2 and Fig. X2.5 show near perfect solution annealed structures. No precipitates or only very small traces of precipitates can be seen in the microstructure. Fig. X2.3, Fig. X2.4 and Fig. X2.6 show a structure as can be typically found after proper solution annealing. In the microstructure a small amount of detrimental phases can be seen. Both samples passed with good margin when tested according to Test Method B or C, or both.

X2.3.2 Figs. X2.7-X2.9 show microstructures that appear to contain higher amounts of detrimental phases that still passed the acceptance criteria in Methods B or C, or both. The samples were heat treated to promote formation of detrimental phases. The holding time is specific for the sample and different holding times for samples of different alloy or size or geometry can give different results with regard to occurrence of detrimental phases.

X2.4 *Microstructures that failed Test Method B or C, or both*:

X2.4.1 Figs. X2.10-X2.12 show microstructures that are of samples that have failed the acceptance criteria in Methods B or C, or both. The samples were heat treated to promote formation of detrimental phases. The holding time is specific for the sample and different holding times for samples of different alloy or size or geometry can give different results with regard to occurrence of detrimental phases.

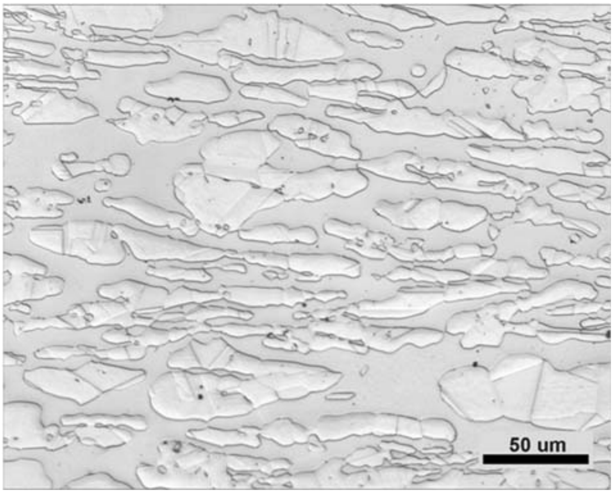

FIG. X2.1 UNS S32101, thickness 10 mm, (500× magnification), Solution annealed 1050°C + WQ, Method B result: Passed – 187 J @ room temperature, Method C result: Passed – 1.01 mdd @ 45°C, 5012 mdd @ 50°C

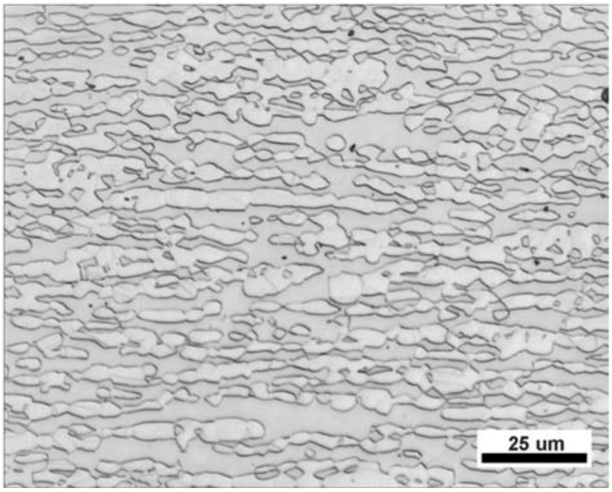

FIG. X2.2 UNS S32101, thickness 1.5 mm, (1000× magnification), Solution annealed 1050°C WQ, Method B result: not tested, Method C result: Passed – 0.0 mdd @ 50°C, 4968 mdd @ 55°C

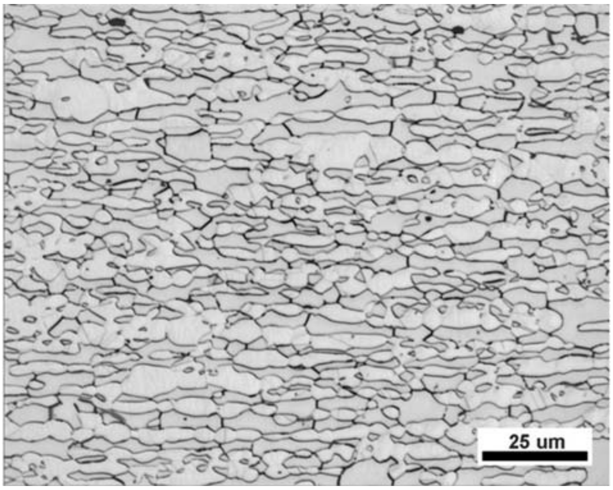

FIG. X2.3 UNS S32101 thickness 1.5 mm, (1000× magnification), Solution annealed 1050°C + 1 min 700°C + WQ, Method B result: not tested, Method C result: Passed – 0.19 mdd @ 35°C, 1971 mdd @ 40°C

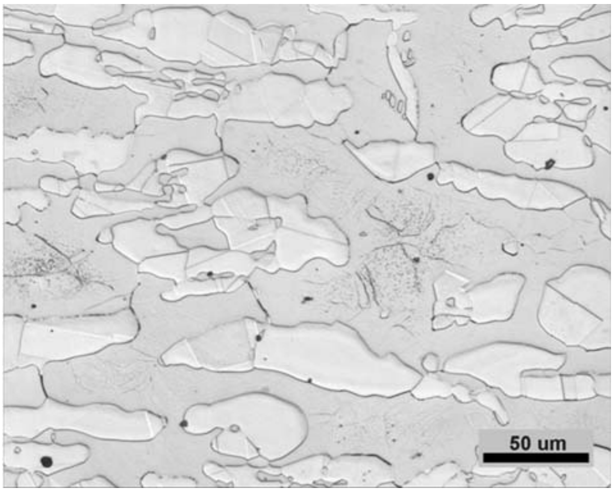

FIG. X2.4 UNS S32101 thickness 30 mm, (500× magnification), Solution annealed 1050°C + 2 min 700°C + WQ. Method B result: Passed – 105J @ room temperature, Method C result: Passed – 0.29 mdd @ 40°C, 3981 mdd @ 45°C

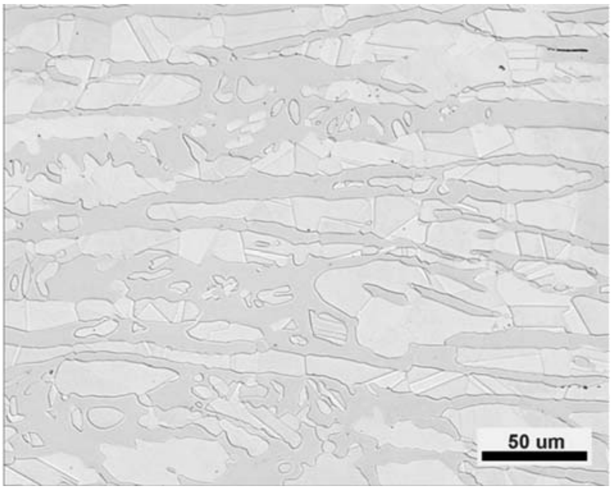

FIG. X2.5 UNS S32304, thickness 30 mm, (500× magnification), Solution annealed 1050°C + WQ, 595 Method B result: Passed – 288 J @ -10°C, Method C result: Passed – 1.57 mdd @ 45°C, 4760 mdd 50°C

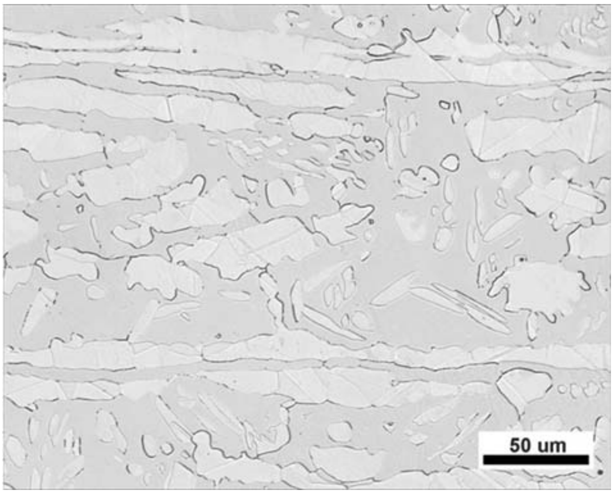

FIG. X2.6 UNS S2304, thickness 30 mm, (500× magnification), Solution annealed 1050°C + 7min 700°C + WQ, Method B result: Passed – 266J @ -10°C, Method C result: Passed – 1.85 mdd @ 30°C, 2521 mdd @ 35°C

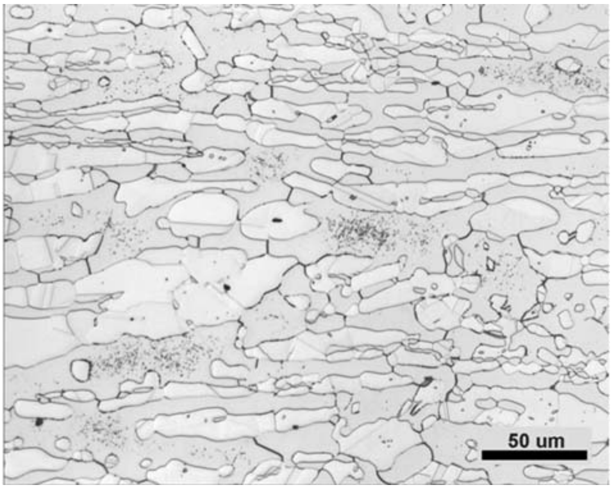

FIG. X2.7 UNS S32101 thickness 10 mm, (500× magnification), Solution annealed 1050°C + 7min 700°C + WQ. Method B result: Passed – 87 J @ room temperature, Method C result: Passed – 0.47 mdd @ 25°C, 803 mdd @ 30°C

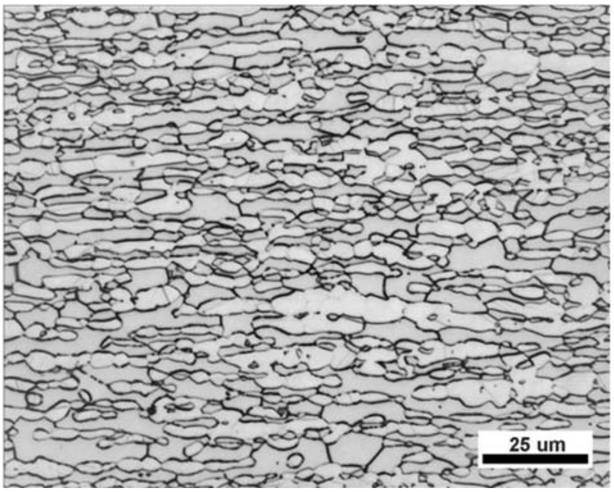

FIG. X2.8 UNS S32101 thickness 1.5 mm, (1000× magnification), Solution annealed 1050°C + 4min 700°C + WQ, Method B result: not tested, Method C result: Passed – 1.11 mdd @ 30°C, 3909 mdd @ 35°C

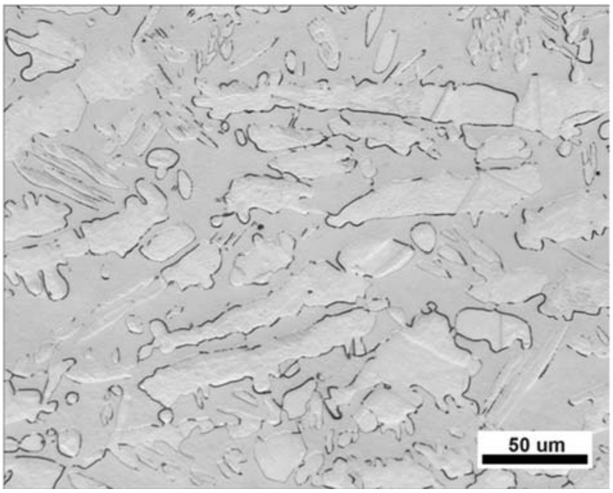

FIG. X2.9 UNS S2304, thickness 30 mm, (500× magnification), Solution annealed 1050°C + 1h 700°C + WQ, Method B result: Passed – 118 J @ -10°C, Method C result: Passed – 0.15 mdd @ 25°C, 1717 mdd @ 30°C

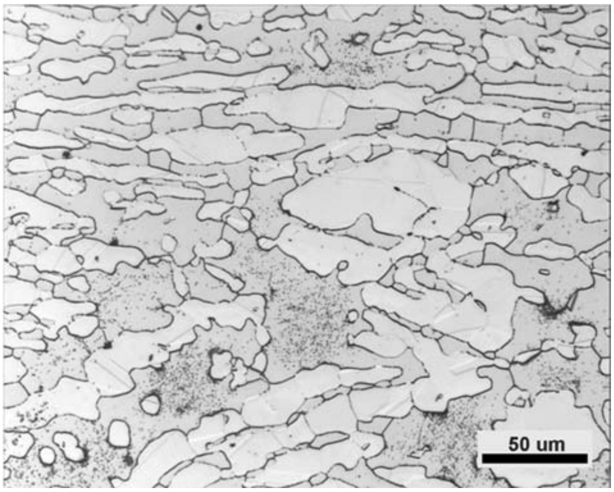

FIG. X2.10 UNS S32101 thickness 10 mm, (500× magnification), Solution annealed 1050°C + 7min 700°C + WQ, Method B result: Failed – 60 J @ room temperature, Method C result: Failed – 0.80 mdd @ 10°C, 275 mdd @ 15°C

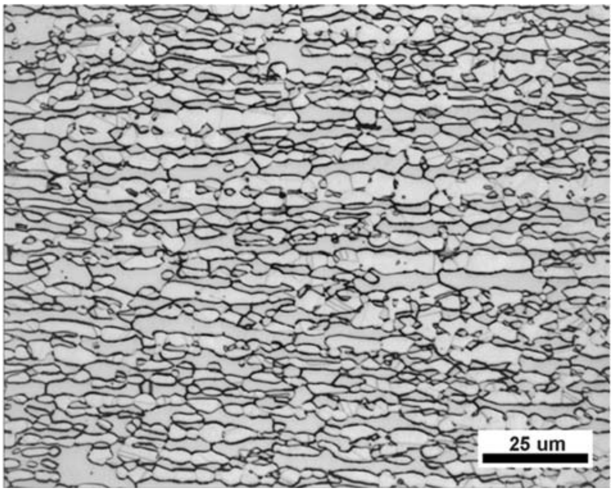

FIG. X2.11 UNS S32101 thickness 1.5 mm, (1000× magnification), Solution annealed 1050°C + 9min 700°C + WQ, Method B result: Not tested, Method C result: Failed – 0.74 mdd @ 15°C, 1430 mdd @ 20°C

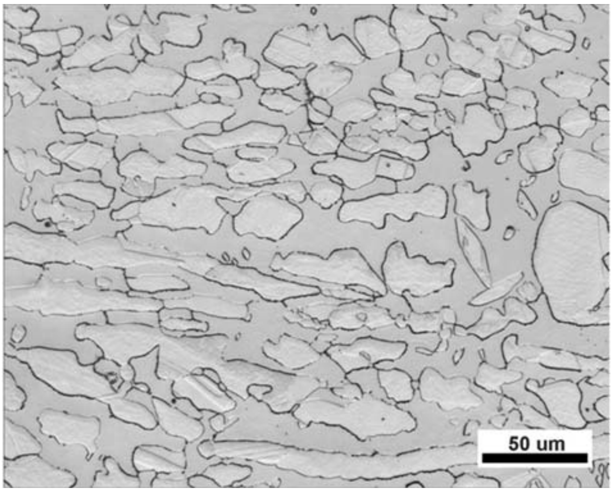

FIG. X2.12 UNS S32304, thickness 30 mm, (500× magnification), Solution annealed 1050°C + 10h 700°C + WQ, Method B result: Not tested, Method C result: Failed – 0.0 mdd 15°C, 331 mdd @ 20°C

X3. EXAMPLES OF THE EFFECT OF THERMAL EXPOSURE ON TOUGHNESS AND CORROSION RESISTANCE

X3.1 Detailed results for the testing of UNS S32101 and UNS S32304 using the three test methods in this standard have been published in the listed references (**1**, **2**, **3**, **4**)[4] and these references are summarized in the below graphic illustrations.

X3.2 The below illustrations supplement the data presented in Appendix X2 as documentation for the appropriateness of the listed acceptance criteria in Methods B and C.

X3.3 Fig. X3.1 shows the highest observed temperature with measured corrosion rate less than 10 mdd (blue circle symbols and left axis). The corresponding measured impact toughness (red square symbols and right axis) at room temperature—all as a function of different sensitization times at 700ºC. The material tested was UNS S32101 from two plates (12 and 30 mm thickness). Examples of the associated microstructures from the indicated test conditions are also inserted.

X3.4 Fig. X3.2 shows similar data as discussed in Fig. X3.1, but results are for the product types sheet and tube. No data for impact toughness are given as this type of measurement requires thicker material.

X3.5 Fig. X3.3 shows similar data for thick plates of UNS S32304. The propensity for the structure of UNS S32304 to deteriorate at the sensitization temperature (700ºC), is clearly less than that of UNS S32101—nonetheless, the method shows a lowering of the measured properties for UNS S32304 and the methods is capable of detecting an unwanted level of detrimental phases in the microstructure of UNS S32304.

X3.6 Fig. X3.4 shows data for UNS S32101 bar (rebar) products. The complexity of the rolling geometries (rolling is

---

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

performed "from all sides") in combination with the impact toughness being measured—by de facto standard and often the only practical way—in the longitudinal direction of the rolling direction, the impact toughness levels measured are generally higher than those measured transverse to the rolling direction on a flat plate or sheet product. This leads to an uncertainty regarding a proper "cut-off impact toughness level, since not only material but also geometry and specific rolling details affects the choice of a proper limit. There are no similar issues that result from the complex rolling geometries in the corrosion test of Method C, which therefore should be regarded as "first choice" when testing bar material.

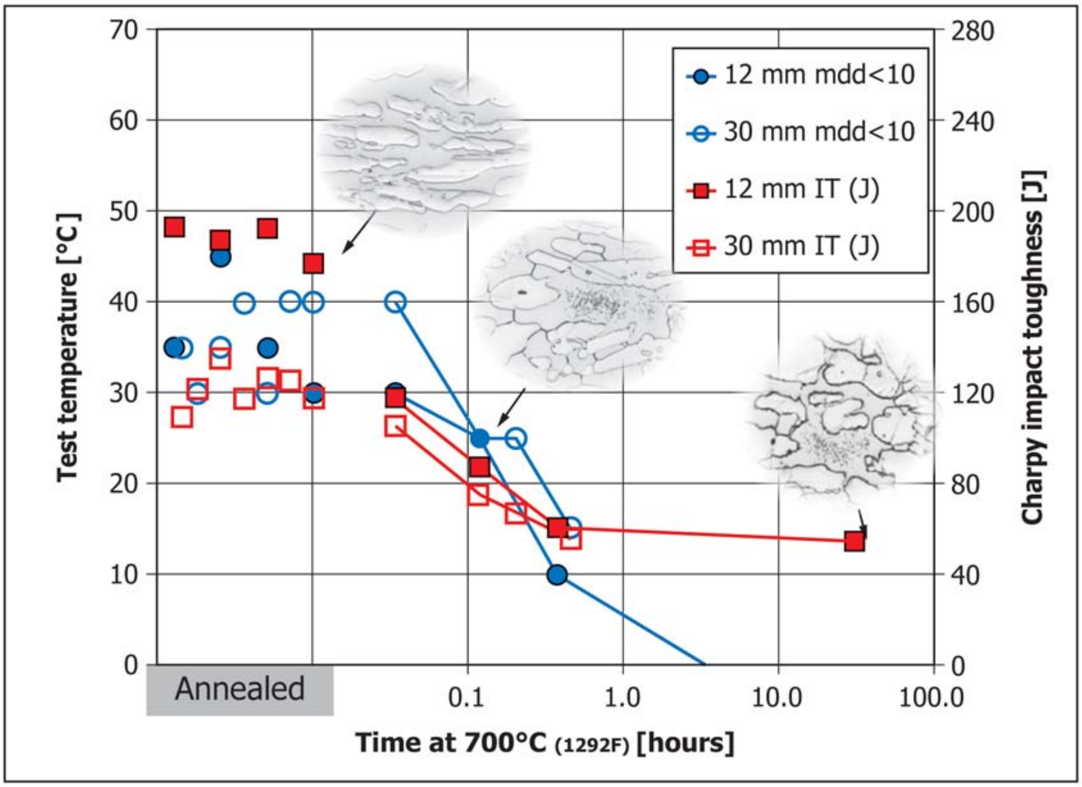

FIG. X3.1 Test results for various heat treatment times using the methodology of Test Methods A, B and C performed on UNS S32101 plate of two thickness: 12 mm and 30 mm

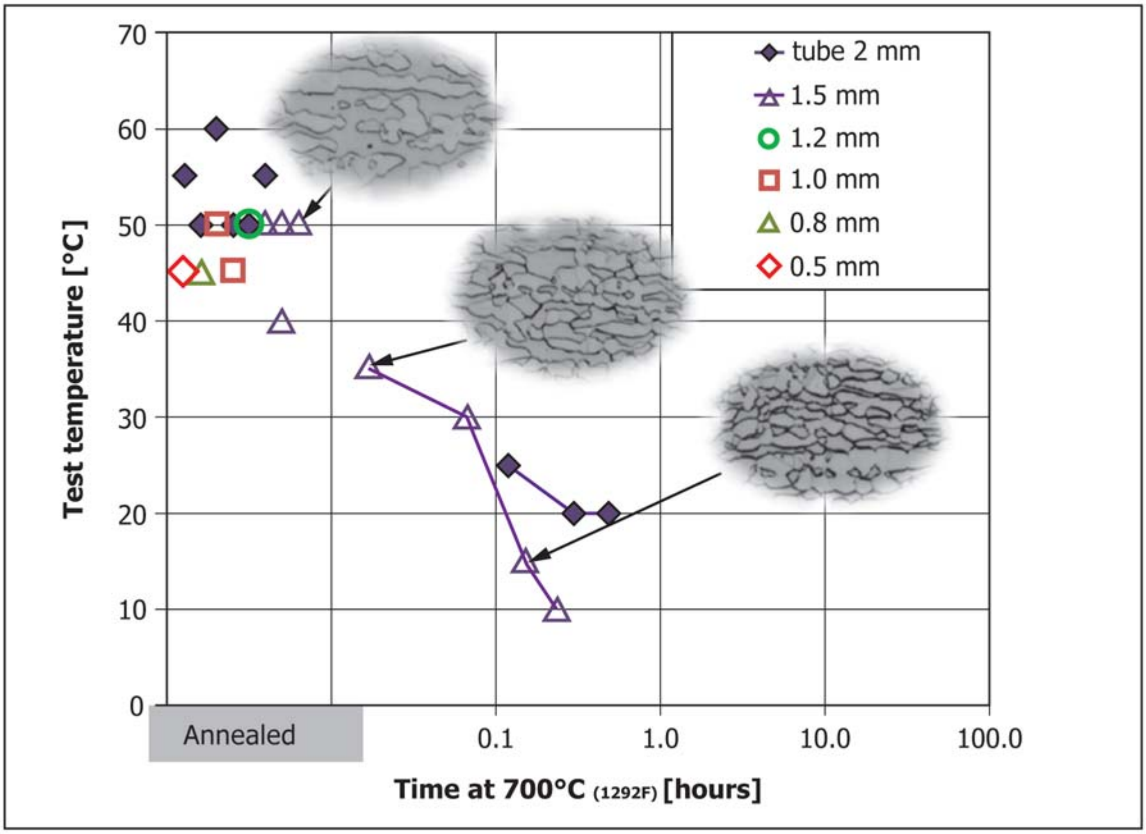

FIG. X3.2 Test results for various heat treatment times using the methodology of Test Methods A and C performed on UNS S32101 sheet and welded tube of various (wall) thicknesses: 0.5 mm to 2 mm. Impact toughness measurement is not possible for such low thicknesses

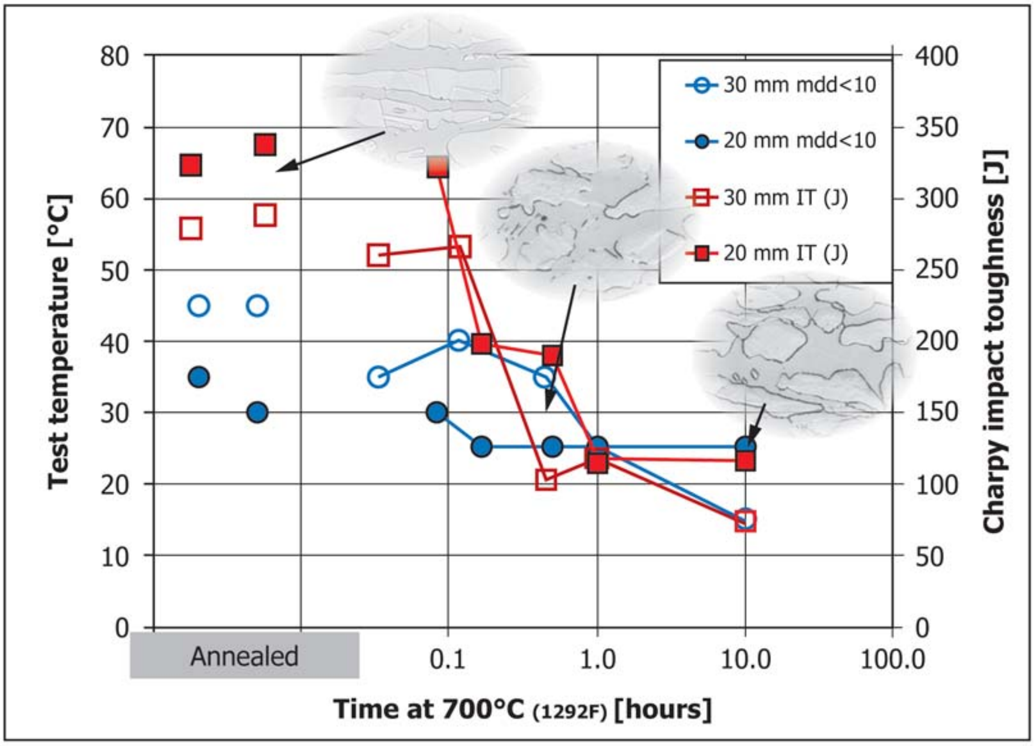

FIG. X3.3 Test results for various heat treatment times using the methodology of Test Methods A, B and C performed on UNS S32304 plate of thicknesses: 20 mm and 30 mm

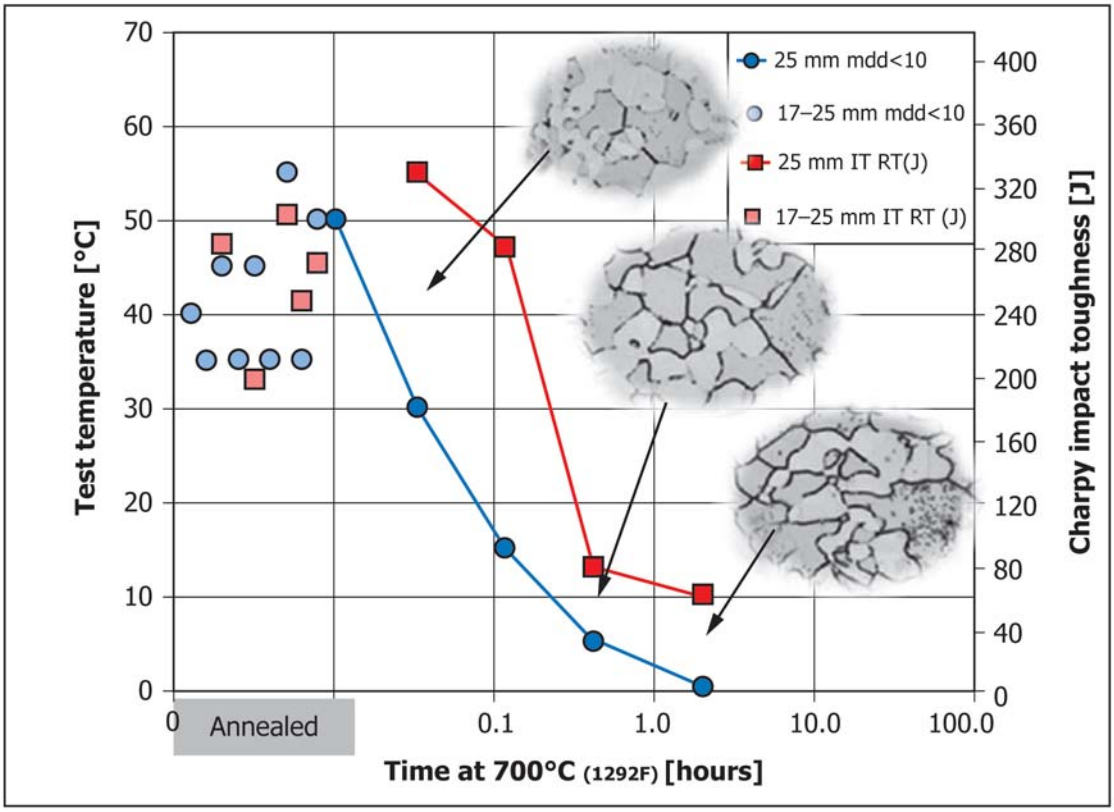

FIG. X3.4 Test results for various heat treatment times using the methodology of Test Method A, B and C performed on UNS S32101 bar of thicknesses: 17 mm and 25 mm

REFERENCES

(1) H. Liu, P. Johansson and M. Liljas, "Structural Evolution of LDX 2101® (EN 1.4162) during Isothermal Ageing at 600-850ºC," *Proc. 6th European Stainless Steel Conference*, Helsinki (2008), pp. 555-560.

(2) J. D. Fritz, P-E. Arnvig, J. Y. Jonsson, R. Pettersson and S Randström, "Evaluation of Possible Test Methods for Qualifying Lean Duplex Stainless Steel," *Proc Stainless Steel World*, Houston, Texas (2010).

(3) J. Y. Jonsson, C. Canderyd and R. Pettersson, "Optimization of a Qualification Test Method for Lean Duplex Stainless Steels," Paper 28 presented at 7th European Stainless Steel Science and Market Conference, 21-23 Sept 2011.

(4) R. Pettersson, J. Y. Jonsson, C. Canderyd and P-E Arnvig, "Qualification Testing of Lean Duplex Stainless Steels for the Process Industry," Paper # 24698 NACE Corrosion 2012, Salt Lake City, Utah (2012).

(5) P. Boillot, R. Bergeron, J. Peultier, K. Wiegers and T. Ladwein, "Investigations on Standard Corrosion Test for Quality control of Lean Duplex Stainless Steel," *Proc 8th Duplex Stainless Steels Conference*, Beaune (2010).

SUMMARY OF CHANGES

Committee A01 has identified the location of selected changes to this standard since the last issue (A1084 – 15) that may impact the use of this standard. (Approved Sept. 1, 2015.)

(1) New grade S82011 was added to Tables 1-3.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard 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.

This 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 this standard or for additional standards and should be addressed to ASTM International 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, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/