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Standard Test Method for Evaluating Stress-Corrosion Cracking of Stainless Alloys with Different Nickel Content in Boiling Acidified Sodium Chloride Solution¹

This standard is issued under the fixed designation G123; 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 This test method covers a procedure for conducting stress-corrosion cracking tests in an acidified boiling sodium chloride solution. This test method is performed in 25 % (by *mass*) sodium chloride acidified to pH 1.5 with phosphoric acid. This test method is concerned primarily with the test solution and glassware, although a specific style of U-bend test specimen is suggested.

1.2 This test method is designed to provide better correlation with chemical process industry experience for stainless steels than the more severe boiling magnesium chloride test of Practice G36. Some stainless steels which have provided satisfactory service in many environments readily crack in Practice G36, but have not cracked during interlaboratory testing (see Section 12) using this sodium chloride test method.

1.3 This boiling sodium chloride test method was used in an interlaboratory test program to evaluate wrought stainless steels, including duplex (ferrite-austenite) stainless and an alloy with up to about 33 % nickel. It may also be employed to evaluate these types of materials in the cast or welded conditions.

1.4 This test method detects major effects of composition, heat treatment, microstructure, and stress on the susceptibility of materials to chloride stress-corrosion cracking. Small differences between samples such as heat-to-heat variations of the same grade are not likely to be detected.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 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 applica*bility of regulatory limitations prior to use*. For specific hazard statements, see Section 8.

2. Referenced Documents

- 2.1 ASTM Standards:²
- D1193 Specification for Reagent Water
- E8 Test Methods for Tension Testing of Metallic Materials
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- G15 Terminology Relating to Corrosion and Corrosion Testing (Withdrawn 2010)³
- G16 Guide for Applying Statistics to Analysis of Corrosion Data
- G30 Practice for Making and Using U-Bend Stress-Corrosion Test Specimens
- G36 Practice for Evaluating Stress-Corrosion-Cracking Resistance of Metals and Alloys in a Boiling Magnesium Chloride Solution
- G49 Practice for Preparation and Use of Direct Tension O Stress-Corrosion Test Specimens
- G107 Guide for Formats for Collection and Compilation of Corrosion Data for Metals for Computerized Database Input

3. Terminology

3.1 *Definitions*—For definitions of corrosion-related terms used in this test method, see Terminology G15.

4. Summary of Test Method

4.1 A solution of 25 % sodium chloride (by *mass*) in reagent water is mixed, and the pH is adjusted to 1.5 with phosphoric acid. The solution is boiled and U-bends (or other stressed specimens) are exposed in fresh solution for successive one-week periods.

¹This test method is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.06 on Environmentally Assisted Cracking.

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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.

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

4.2 The test may be continued for as many weeks as necessary, but six weeks (about 1000 h) or less are expected to be sufficient to crack susceptible materials. Longer exposures provide greater assurance of resistance for those materials which do not crack.

4.3 It is recommended that samples of a susceptible material, for example, UNS S30400 or S31600 (Type 304 or Type 316 stainless, respectively), be included as a control when more resistant materials are evaluated.

5. Significance and Use

5.1 This test method is designed to compare alloys and may be used as one method of screening materials prior to service. In general, this test method is more useful for stainless steels than the boiling magnesium chloride test of Practice G36. The boiling magnesium chloride test cracks materials with the nickel levels found in relatively resistant austenitic and duplex stainless steels, thus making comparisons and evaluations for many service environments difficult.

5.2 This test method is intended to simulate cracking in water, especially cooling waters that contain chloride. It is not intended to simulate cracking that occurs at high temperatures (greater than 200° C or 390° F) with chloride or hydroxide.

NOTE 1—The degree of cracking resistance found in full-immersion tests may not be indicative of that for some service conditions comprising exposure to the water-line or in the vapor phase where chlorides may concentrate.

5.3 Correlation with service experience should be obtained when possible. Different chloride environments may rank materials in a different order.

5.4 In interlaboratory testing, this test method cracked annealed UNS S30400 and S31600 but not more resistant materials, such as annealed duplex stainless steels or higher nickel alloys, for example, UNS N08020 (for example 20Cb- 3^4 stainless). These more resistant materials are expected to crack when exposed to Practice G36 as U-bends. Materials which withstand this sodium chloride test for a longer period than UNS S30400 or S31600 may be candidates for more severe service applications.

5.5 The repeatability and reproducibility data from Section 12 and Appendix X1 must be considered prior to use. Interlaboratory variation in results may be expected as occurs with many corrosion tests. Acceptance criteria are not part of this test method and if needed are to be negotiated by the user and the producer.

6. Apparatus

6.1 The glassware used for this test method is shown in Fig. 1 and is as follows:

6.1.1 *Flask*—1000-mL Erlenmeyer flask with a 45/50 ground-glass joint.

6.1.2 *Condenser*, a four-bulb Allihn condenser with a 45/50 ground-glass joint (water-cooled joint suggested), a water jacket at least 20 cm (8 in.) long and a 1 to 2.5 cm (0.4 to



FIG. 1 Apparatus Used for Stress-Corrosion Cracking Test

0.95 in.) long drip tip is used. (Modified Allihn condensers with no drip tip and condensers with longer drip tips may produce different results. These alternate Allihn condenser designs may be used if control samples of susceptible (for example, UNS S31600) and resistant (for example, UNS N08020) materials are included in the study.)

6.1.3 *Hot Plate*, capable of maintaining the solution at its boiling point.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without affecting results.

⁴ 20Cb-3 is a registered trademark of Carpenter Technology Corp., Reading, PA.

⁵ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

7.2 *Purity of Water*—Solutions shall be made with water of purity conforming to at least Type IV reagent water as specified in Specification D1193 (except that for this method limits for chlorides and sodium may be ignored).

7.3 Sodium Chloride (NaCl)—A solution of 25 % NaCl (by mass) acidified to pH 1.5 with phosphoric acid (H_3PO_4) is used. The solution may be prepared by adding 750 g H_2O (750 mL) to 250 g NaCl, and adjusting to pH 1.5 with H_3PO_4 . Varying quantities of solution may be prepared and larger amounts may be stored indefinitely in appropriate glassware. The pH must be determined prior to each use.

8. Hazards

8.1 Normal precautions for handling boiling liquid should be observed.

8.2 All heating or boiling of the NaCl solution should be done in an area where personnel are not likely to accidentally bump the flask. A hooded area is preferred.

8.3 Minimum personal protective equipment for handling boiling sodium chloride should include safety glasses or goggles, face shield, laboratory coat, and rubber gloves. (Warning—U-bends (and other highly stressed test specimens) may be susceptible to high rates of crack propagation and a specimen containing more than one crack may splinter into two or more pieces. This may also occur due to a cracked restraining bolt. Due to the highly stressed condition in a U-bend specimen, these pieces may leave the specimen at high velocity and can be dangerous.)

9. Test Specimens

9.1 U-bends are preferred but other stress corrosion cracking specimens may be used with this test solution. The specimen style chosen should provide sufficient stress to crack less resistant materials (for example, UNS S30400 or S31600) in 1000 h or less). (See Annex A1.) Regardless of the specimen style, it is recommended that UNS S30400 or UNS S31600, or both, be included as controls.

9.2 The test specimen must be thick enough so that the applied stress does not cause mechanical rupture of less resistant materials if the cross section is reduced by pitting or general corrosion.

9.3 The size of alternate specimens (other than those in Annex A1) must allow a solution volume to specimen surface area ratio of at least $5:1 \text{ mL/cm}^2$ (33 mL/in.²).

9.4 A minimum of four replicates (two per flask) is required because of the variability typical in stress-corrosion testing.

9.5 Methods of fabricating U-bend specimens are provided in Annex A1. These procedures are based on Practice G30, but in addition provide a specimen that fits through a 45/50 ground-glass joint. Assurance that the legs are stressed sufficiently by the bolt is also provided.

9.5.1 Other methods of producing U-bends described in Practice G30 may be used; however, during exposure the U-bends must be (1) in the plastic range and (2) stressed to the

maximum applied tensile load experienced during fabrication. The same method must be used to fabricate all the U-bends in a given study.

9.5.2 The bolt, nut, and flat washer must be made of a material resistant to general corrosion, pitting, and stress corrosion cracking in the environment. UNS N10276 (Alloy C-276) is recommended because some other materials (for example, titanium or UNS N06600 [Alloy 600]) may be attacked resulting in an increase in solution pH.

9.5.3 The metallic fastener must be electrically isolated from the specimen by a rigid shoulder washer (that is, zirconia or another material that will not be compressed during the test).

9.5.4 The extended end of the bolt may require cutting to fit into the test vessel.

10. Procedure

10.1 Stress the specimens, examine at 20x, and replace any specimens with cracks or other defects.

Note 2—The direction and intensity of the incident light may affect crack detection during the $20\times$ examination.

10.2 Degrease in a halogen-free solvent or laboratory detergent, rinse as necessary, and dry. It is best practice to stress the specimens immediately before the beginning of the test. Any storage of the specimens should be in a clean enclosure. A desiccant such as silica gel may be used. The specific level of relative humidity is not important for the alloys of interest.

10.3 Place duplicate specimens in each 1000-mL Erlenmeyer flask. Duplicate flasks (four specimens) are necessary to evaluate a given sample of the specific material, material condition, etc. (The specimens may be placed in the flasks after the solution has been added, if convenient.)

10.4 The specimens in each flask must be kept separate and completely submerged. Tight crevices between the stressed (bend) area and any means of specimen support should be avoided. The stressed area should be free from direct contact with heated surfaces. Specimens may be supported on glass rods or tubes or by glass fixtures.

10.5 Drop boiling chips⁶ into the flasks.

10.6 Add 600 mL of 25 % NaCl solution, pH 1.5, to each flask. When each flask contains two U-bends as described in Annex A1, the solution volume to sample surface area ratio is $5:1 \text{ mL/cm}^2$ (33 mL/in.²).

10.7 Place the flasks on a hot plate and insert the condenser. Begin recording the test duration when the solution begins boiling. The boiling point during interlaboratory testing was 106 to 110° C (223 to 230°F).

10.8 After one week remove the flask from the hot plate, determine the final pH of the solution at room temperature, and discard the remaining solution. A final pH over about 2.5

⁶ The sole source of supply of amphoteric alundum granules known to the committee at this time is Hengar Co., Philadelphia, PA. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

suggests that general corrosion or pitting of the specimen or fastening device has occurred. A pH at this level is expected to reduce the test severity and may delay or preclude failures of UNS S31600. More rapid cracking of UNS S31600 appears likely with a final pH of about 2 or less.

10.9 Rinse and dry the specimens. Examine the bend area, legs, and area adjacent to the crevice (at the fastener) at $20 \times$ for cracking. See Note 3. Record location of cracks. Additional exposures or metallographic evaluation may be used to determine if questionable indications are, in fact, stress-corrosion cracks.

Note 3—Any cracking at the fastener is very likely due to residual stresses and more aggressive solution which may be formed in crevices. If crevices are expected in service (due to design of service equipment or deposits), a U-bend specimen employing a crevice on the bend may be evaluated.

10.10 Periodic removal of the specimens from the solution may be necessary during the first week to determine the time when cracks first appear. Removal of the specimens is expected to disturb local corrosion cells and may influence test results. All specimens in a given test program should have the same removal/examination schedule. When the time-to-crack is recorded, the test duration at the previous examination (no cracks) should also be noted.

10.11 Expose for additional one-week periods as necessary. Fresh solution must be used for each exposure and the initial and final pH (at room temperature) must be recorded weekly. See 10.8 regarding the effect of the final pH.

10.12 After the final $20 \times$ examination (following the last test period) remove the fastener and examine the crevice areas at $20 \times$ for cracking.

10.13 A final examination for cracks may be performed by additionally bending the specimens until the ends of the legs touch. This may expose tight cracks which were not previously detected. The additional bending may not be appropriate for materials which are susceptible to hydrogen embrittlement in this environment. Do not re-expose specimens after this additional bending.

10.14 Ruptured specimens should also be examined for evidence of mechanical failure resulting from the action of applied stress on specimens whose cross sections have been reduced by general or pitting corrosion, or both. Such failures usually show evidence of ductility. Repeat tests with thicker specimens should be made in case of doubt.

11. Report

11.1 Report the type of specimen used, method of specimen fabrication, times to cracking (including maximum time without cracks), location of cracks, final pH of each exposure, and details regarding the Allihn condenser drip tip. Note whether or not metallographic techniques or additional bending of the specimen (see 10.13) were employed. Electronic data exchange can be facilitated by using the fields suggested in Appendix X2 (excerpted from Guide G107).

11.2 Data for resistant materials shall be accompanied by data for at least four susceptible control specimens; for example, UNS \$30400 or UNS \$31600.

12. Precision and Bias⁷

12.1 *Precision*—Variability occurred in both repeatability and reproducibility for time-to-fail data developed using UNS S30400 and S31600 in an interlaboratory test program (Appendix X1). Such variability is typical in time-to-fail data for stress-corrosion cracking tests, and is expected to preclude detection of small differences between samples.

12.1.1 Histograms of the time-to-crack for UNS S30400 and S31600 tested in accordance with this test method appear in Appendix X1 along with data from each laboratory. The time-to-crack values in Appendix X1 are not necessarily the maximum or minimum values which could be obtained in other testing.

12.1.2 Every specimen of UNS S30400 and S31600 cracked within the 1000-h interlaboratory test duration while no cracking occurred for more resistant materials, UNS S32550 (Ferralium⁸ Alloy 225) and N08020.

12.1.3 It has been observed in stress-corrosion tests of various metal-alloy systems that the precision is best for tests of specimens that have either a very low probability of stress-corrosion cracking (few, if any, failures in the prescribed test duration) or a high probability (short failure times). The precision is least for groups of test specimens with an intermediate probability. This was observed in the interlaboratory test program. There were no failures of the more resistant materials (UNS S32550 and UNS N08020), generally rapid failure of the least resistant material (UNS S30400, see Fig. X1.3), and greater variation in failure times for the material expected to have intermediate resistance (UNS S31600, see Fig. X1.4).

12.1.4 Reproducibility between laboratories frequently varied more widely than repeatability within each laboratory. Although this variation is substantial, it is within what may be expected for a stress-corrosion cracking test. The effects of interlaboratory variation must be considered if data from multiple laboratories are used.

12.1.5 Analysis of the interlaboratory test data using a log normal distribution appears in Appendix X1.

12.2 *Bias*—The procedure in this test method for measuring time-to-cracking of specimens in acidified sodium chloride solution has no bias because the time-to-cracking is defined only in terms of this test method, and there is no absolute standard for reference. Time-to-cracking is a function of specimen type as well as stress and material composition.

13. Keywords

13.1 boiling acidified sodium chloride (NaCl); glassware; histograms; stress corrosion cracking; U-bend specimens; UNS N08020

⁷ Supporting data (including UNS S30400, S31600, S32550, and N08020) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:G01-1013.

⁸ Ferralium is a registered trademark of Langley Alloys, Ltd. of Slough, United Kingdom.

ANNEX

(Mandatory Information)

A1. SUGGESTED TEST SPECIMEN

A1.1 Using the procedure described in this test method, the U-bend specimen described in A1.2 has produced cracking of UNS S30400 and S31600 in less than 1000 h, without cracking more resistant duplex stainless and higher nickel (for example, about 33 %) materials.

A1.2 Suggested specimen dimensions appear in Fig. A1.1. The specimen differs slightly from those suggested in Practice G30 to allow the completed U-bend to pass through a 45/50 ground-glass joint while being large enough to accommodate ceramic insulators of sufficient size to resist cracking during use.

A1.3 If surface finish is not the subject of the evaluation, prepare the specimens to produce a 120-grit finish or its equivalent with machining techniques.

A1.4 Bend the specimens around a 9.5 mm (0.375-in.) diameter mandrel using an adjustable die similar to that in Fig. A1.2 as follows. (This figure is the same as Fig. 4a in Practice G30.)

A1.4.1 Mark the centerline on the specimen to aid aligning.

A1.4.2 Set the gap in the die at the mandrel diameter plus two times the specimen thickness.

A1.4.3 First, depress the mandrel (hydraulic) until the apex of the U-bend is approximately level with the bottom of the die. Continue stressing until the legs of the U-bend are nearly parallel. Final stressing is preferably done with the fastener.



FIG. A1.1 Suggested U-Bend Specimen Dimensions



NOTE 1—Mandrel has 9.5 mm (0.375-in.) diameter. Dimension Z is mandrel diameter plus two times the specimen thickness.

FIG. A1.2 Suggested Stressing Fixture (fastener inserted while specimen in die)

The specimen may be stressed in the die or it may be removed and restressed outside the die. Partial stressing in the die followed by final stressing outside the die may be optimum.

A1.4.4 Insert the stressing fastener. Use ceramic insulators (zirconia or other non-compressible, corrosion resistant, non-conductive material). Flat washers should be used between the ceramic insulator and fastener to extend the life of the insulator. The bolt, nut, and flat washer must resist corrosion in the NaCl environment. UNS N10276 is recommended for all three items.

A1.4.5 Stress the U-bend so that the legs are parallel, that is, the U-bend is more severely bent than it was due to the die pressure. The inside dimension between the legs will be about 11.4 mm (about 0.450 in.).

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APPENDIXES

(Nonmandatory Information)

X1. SUMMARY OF INTERLABORATORY TEST DATA ANALYSIS

X1.1 Test Method

X1.1.1 Testing in accordance with this test method was performed by seven laboratories and included UNS S30400, S31600, S32550, and N08020. Each laboratory exposed a series of duplicate flasks, each containing two U-bends of each grade. UNS N10276 fasteners were used by most laboratories and the solution was replaced weekly. Examinations were scheduled after 6 h, daily for one week, and weekly for six weeks. Concerns of Practice E691 and Guide G16 were considered as possible and appropriate.

X1.1.2 The pH was recorded before and after each exposure. The initial pH was specified as 1.5. The change in pH during all the one-week periods for all laboratories ranged from +0.6 to +2.8 for UNS S30400 and -0.1 to +2.72 for UNS S31600. Only one of the laboratories experienced a pH increase greater than 0.55 for UNS S31600 and this increase may have been related to attack of the fasteners.

X1.2 Interlaboratory Test Data Analysis Using Histograms

X1.2.1 Fig. X1.1 and Fig. X1.2 present the time-to-crack data for all U-bends of susceptible materials, UNS S30400 and UNS S31600, respectively. The within-laboratory repeatability was often better than the between-laboratory reproducibility. This could occur if different operators had different ability to detect cracks or tended to find cracks in all samples after cracks in one sample were noticed.

X1.2.2 The nature of the data suggest that analysis by histogram is superior to attempting to calculate mean and standard deviation values.

X1.2.3 Fig. X1.3 and Fig. X1.4 present histograms of the time-to-crack data for UNS S30400 and UNS S31600, respectively. All but one of the failures occurred in 28 days or less.



Note 1—Four replicates tested per laboratory. Examinations scheduled after 6 h, daily for one week, and weekly for six weeks.





NOTE 1—Four replicates tested per laboratory. Examinations scheduled after 6 h, daily for one week, and weekly for six weeks.



NUMBER REPLICATES CRACKED IN TIME SHOWN



Note 1—Seven laboratories, each tested four replicates. Examinations scheduled after 6 h, daily for one week, and weekly for six weeks. FIG. X1.3 Histogram of Time-To-Crack Data for UNS S30400 Stainless U-Bends in pH 1.5 NaCl Test

Cracks in one sample of UNS S31600 were found after 42 days (1000 h). An increase in pH due to attack of the UNS N06600 fasteners may have delayed this cracking for at least one week.

X1.2.4 Four U-bends of UNS S32550 and N08020 were exposed by each laboratory. These materials have demonstrated good resistance to stress-corrosion cracking in service and none of the U-bends cracked in the 42-day test.

X1.2.5 Analysis of the interlaboratory test data indicates that materials which withstand this 42-day test without cracks are more resistant to stress-corrosion cracking than UNS S30400 and UNS S31600. This statement is based upon use of the sampling and testing procedures employed in this interlaboratory test program.

X1.3 Interlaboratory Test Data Analysis Using a Log Normal Distribution

X1.3.1 The average time to cracking for the four replicates of each grade tested at each laboratory are plotted in Fig. X1.5.