



Designation: G 35 – 98

Standard Practice for Determining the Susceptibility of Stainless Steels and Related Nickel-Chromium-Iron Alloys to Stress-Corrosion Cracking in Polythionic Acids¹

This standard is issued under the fixed designation G 35; 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 practice describes procedures for preparing and conducting the polythionic acid test at room temperature, 22 to 25°C (72 to 77°F), to determine the relative susceptibility of stainless steels or other related materials (nickel-chromium-iron alloys) to intergranular stress corrosion cracking.

1.2 This practice can be used to evaluate stainless steels or other materials in the “as received” condition or after being subjected to high-temperature service, 482 to 815°C (900 to 1500°F), for prolonged periods of time.

1.3 This practice can be applied to wrought products, castings, and weld metal of stainless steels or other related materials to be used in environments containing sulfur or sulfides. Other materials capable of being sensitized can also be tested in accordance with this test.

1.4 This practice may be used with a variety of stress corrosion test specimens, surface finishes, and methods of applying stress.

1.5 *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.* For more specific precautionary statements, see Section 7.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens³

G 15 Terminology Relating to Corrosion and Corrosion Testing³

¹ This practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals, and is the direct responsibility of Subcommittee G01.06 on Environmentally Assisted Cracking.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 03.02.

G 30 Practice for Making and Using U-Bend Stress-Corrosion Test Specimens³

3. Summary of Practice

3.1 The stressed specimens are placed in the container along with a sensitized and stressed AISI Type 302 (UNS S30200) or Type 304 (UNS S30400) stainless steel control specimen. A sufficient amount of the previously prepared polythionic acid solution is added to the container to immerse the test specimens. A cover is placed on the container and the test is carried out at room temperature.

4. Significance and Use

4.1 This environment provides a way of evaluating the resistance of stainless steels and related alloys to intergranular stress corrosion cracking. Failure is accelerated by the presence of increasing amounts of intergranular precipitate. Results for the polythionic acid test have not been correlated exactly with those of intergranular corrosion tests. Also, this test may not be relevant to stress corrosion cracking in chlorides or caustic environments.

4.2 The polythionic acid environment may produce areas of shallow intergranular attack in addition to the more localized and deeper cracking mode of attack. Examination of failed specimens is necessary to confirm that failure occurred by cracking rather than mechanical failure of reduced sections.

5. Apparatus

5.1 Any suitable glass or other transparent, inert container can be used to contain the acid solution and stressed specimens during the period of test at room temperature, 22 to 25°C (72 to 77°F). The container should be fitted with a removable top to reduce evaporation and to allow access to the stressed specimen (or specimens) for the periodic inspection.

6. Reagents

6.1 *Purity of Reagents*—The polythionic acid solution shall be prepared using reagent grade sulfurous acid and technical grade hydrogen sulfide; or, distilled water, commercial grade sulfur dioxide, and technical grade hydrogen sulfide.

6.2 *Purity of Water*—Reagent water Type IV (Specification D 1193) shall be used to prepare the test solutions.

6.3 *Wackenroder's or Polythionic Acid Solution (1)*⁴—A slow current of hydrogen sulfide is passed for an hour through a fritted glass tube into a flask containing chilled (0°C, 32°F) 6 % sulfurous acid, after which the liquid is kept in the stoppered flask for 48 h at room temperature. This operation is repeated until the liquid no longer gives off the odor of sulfur dioxide after standing at room temperature for a few hours. Note safety precautions in Section 7.

6.3.1 In an alternative method (2), the polythionic acid solution is prepared by passing a slow current of sulfur dioxide gas through a fritted glass bubbler submerged in a container of distilled water. This is continued until the solution becomes saturated and then the hydrogen sulfide gas is slowly bubbled into the sulfurous acid solution.

6.3.2 The presence of polythionic acids in the solution prepared in accordance with 6.3 or 6.3.1 can be checked by either of the following methods. Polarography (3) can be employed to identify the thionic acids, or the percent of acid present in the solution can be determined by wet techniques (4). The simplest method of checking the solution for polythionic acids is to expose a stressed and sensitized sample of AISI Type 302 stainless steel. The sample should fail by cracking in less than 1 h. Alternatively, Type 304 sheet (0.07% carbon) which has been sensitized and exposed as a U-bend should crack in 1 h (5). Detection of cracks can be facilitated by closing the legs of the U-bend and examining with a 20× binocular microscope.

7. Safety Precautions

7.1 Hydrogen sulfide should be handled with extreme caution. The characteristic odor of hydrogen sulfide cannot always be used as an alarm system because the olfactory nerves become deadened when exposed to a concentration of a few parts per million of this gas. The maximum allowable concentration in the air for an 8-h work day is 10 ppm, which is well above the level detectable by smell. Exposure to high concentrations of hydrogen sulfide can be fatal. For additional information on the toxicity of hydrogen sulfide, consult Ref (6).

7.2 Normal laboratory precautions should be observed when handling the sulfurous or polythionic acid solutions. Hydrogen sulfide should only be used in a hood observing the above precautions.

8. Test Specimens

8.1 Any type of stress corrosion test specimen can be used with this test solution. For a comprehensive discussion of the

various types of test specimens available, see Ref (7), as well as Practices G 1 and G 30, and Terminology G 15.

8.2 The AISI Type 302 control specimens should be sensitized by heating in a furnace for 4 h at 650°C (1200°F) and then allowing to air cool. The AISI Type 304 control specimens should be sensitized by heating in a furnace for 2 h at 677°C (1250°F) and then allowing to air cool.

9. Procedure

9.1 Prepare the polythionic acid test solution as described in 6.3 and 6.3.1.

9.2 Prior to usage, filter the acid solution to remove the excess sulfur and test for the presence of polythionic acids. The simplest method of testing for the acid is to expose a stressed specimen of sensitized AISI Type 302 or Type 304 stainless steel. The specimen should fail by cracking in less than 1 h.

9.3 Place the stressed specimens in the container along with the sensitized and stressed AISI Type 302 or Type 304 stainless steel control sample. Add a sufficient amount of polythionic acid solution to the container to immerse the test specimens. Close the container and carry out the test at room temperature. Record starting time for the test.

10. Report

10.1 Record starting time, type of specimen, stress, and type of exposure. A distinction must be made in the type of exposure; that is, complete immersion, vapor phase exposure, or a combination of immersion and vapor phase. The time required to initiate cracks, the rate of crack growth, and time to failure may be of importance, depending upon the purpose of the test.

10.1.1 Periodic removal of specimens from the solution may be necessary to determine the time when cracks first appear and the rate of crack propagation. Microscopical examination of polished surfaces is required to detect crack initiation. All stressed surfaces should be examined at magnifications up to 20× at the completion of this test. Metallographic examination of exposed surfaces and of polished and etched cross sections at higher magnifications are necessary to establish the type of cracking.

10.1.2 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. Duplicate tests with thicker specimens should be made in cases of doubt.

11. Keywords

11.1 accelerated test; nickel-chromium-iron alloys; polythionic acids; stainless steels; stress-corrosion cracking

⁴ The boldface numbers in parentheses refer to the reports and papers appearing in the list of references at the end of this practice.

REFERENCES

- (1) Debus, H., "Chemical Investigation of Wackenroder's Solution," *Journal of the Chemical Society, London, Transactions*, Vol 53, 1888, p. 278 (paper XXV).
- (2) Samans, C. H., "Stress Corrosion Cracking Susceptibility of Stainless Steels and Nickel-Base Alloys in Polythionic Acids and Acid Copper Sulfate Solution," *Corrosion*, Vol 20, August 1964, pp. 256t-262t.
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- (4) Jay, P. R., "Determination of Polythionic Acids," *Analytical Chemistry*, Vol 25, No. 2, 1953, pp. 288-290.
- (5) Crum, J. R., Adkins, M. E., and Lipscomb, W. G., "Performance of High Nickel Alloys in Refinery and Petrochemical Environments," *Materials Performance*, Vol. 25, No. 7, July, 1986, pp. 27-33.
- (6) *Chemical Safety Data Sheet SD-36*, Manufacturing Chemists Association, 1825 Connecticut Ave., N.W., Washington, DC 20009.
- (7) "Stress Corrosion Testing Methods," *Stress Corrosion Testing, ASTM STP 425*, ASTM Testing Mats. (Out of print. Send orders directly to University Microfilms, Inc., 300 North Zeeb Rd., Ann Arbor, MI 48106.)

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