NACE INTERNATIONAL

NACE Standard TM0177-2005
Item No. 21212

Standard
Test Method

Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking and Stress Corrosion Cracking in H_2S Environments

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Revised 2005-12-03
Revised 1996
Revised 1990
Revised 1986
Approved in 1977
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ISBN 1-57590-036-X

Foreword

This standard addresses the testing of metals for resistance to cracking failure under the combined action of tensile stress and corrosion in aqueous environments containing hydrogen sulfide (H_2S). This phenomenon is generally termed sulfide stress cracking (SSC) when operating at room temperature and stress corrosion cracking (SCC) when operating at higher temperatures. In recognition of the variation with temperature and with different materials this phenomenon is herein called environmental cracking (EC). For the purposes of this standard, EC includes only SSC, SCC, and hydrogen stress cracking (HSC).

The primary purpose of this standard is to facilitate conformity in testing so that data from different sources can be compared on a common basis. Consequently, this standard aids the evaluation and selection of all types of metals and alloys, regardless of their form or application, for service in H_2S environments. This standard contains methods for testing metals using tensile, bent-beam, C-ring, and double-cantilever-beam (DCB) test specimens. Certain ASTM[(1)] standard test methods have been listed as references for supplementary tests, creating a comprehensive test method standard. In addition, the four-point bent-beam test method is also referenced as a supplementary test.[1,2] This standard is intended for use by laboratory and materials personnel to facilitate conformity in testing.

SSC of metals exposed to oilfield environments containing H_2S was recognized as a materials failure problem by 1952. Laboratory data and field experience have demonstrated that even extremely low concentrations of H_2S may be sufficient to lead to SSC failure of susceptible materials. In some cases H_2S can act synergistically with chlorides to produce corrosion and cracking (SSC and other mode) failures. However, laboratory and operating experiences have also indicated to materials engineers the optimum selection and specification of materials having minimum susceptibility to SSC. This standard covers test methods for SSC (at room temperature) and SCC (at elevated temperature), but other failure modes (e.g., hydrogen blistering, hydrogen-induced cracking [HIC], chloride stress corrosion cracking [SCC], pitting corrosion, and mass-loss corrosion) must also be considered when selecting materials for use in sour (H_2S-containing) environments.

The need for better understanding of the variables involved in EC of metals in oilfield environments and better correlation of data has become apparent for several reasons. New design requirements by the oil and gas production industries call for higher-strength materials that, in general, are more susceptible to EC than lower-strength alloys. These design requirements have resulted in extensive development programs to obtain more resistant alloys and/or better heat treatments. At the same time, users in the petroleum refining and synthetic fuels industries are pushing present materials much closer to their mechanical limits.

Room-temperature (SSC) failures in some alloys generally are believed to result from hydrogen embrittlement (HE). When hydrogen is cathodically evolved on the surface of a metal (as by corrosion or cathodic charging), the presence of H_2S (and other compounds, such as those containing cyanides and arsenic) tends to cause hydrogen atoms to enter the metal rather than to form hydrogen molecules that cannot enter the metal. In the metal, hydrogen atoms diffuse to regions of high triaxial tensile stress or to some microstructural configurations where they become trapped and decrease the ductility of the metal. Although there are several kinds of cracking damage that can occur in metals, delayed brittle fracture of metals resulting from the combined action of corrosion in an aqueous sulfide environment and tensile stresses (failure may occur at stresses far below the yield stress) is the phenomenon known as SSC.

In some cases, however, failure may be the result of localized anodic corrosion processes that may or may not involve hydrogen. In such instances failure is the result of anodic stress corrosion cracking (SCC). Such failures have historically been termed SSC even though their cause may not be hydrogen.

(1) ASTM International (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959.

This standard was originally published in 1977 by NACE International Task Group T-1F-9, a component of Unit Committee T-1F on Metallurgy of Oilfield Equipment. The standard was revised in 1986, 1990, and 1996 by Task Group T-1F-9. It was revised in 2005 by Task Group (TG) 085 on Sulfide Corrosion Cracking: Metallic Materials Testing Techniques. TG 085 is administered by Specific Technology Group (STG) 32 on Oil and Gas Production—Metallurgy and is sponsored by STG 62 on Corrosion Monitoring and Measurement—Science and Engineering Applications. The standard is issued by NACE under the auspices of STG 32.

In NACE standards, the terms *shall, must, should*, and *may* are used in accordance with the definitions of these terms in the *NACE Publications Style Manual*, 4th ed., Paragraph 7.4.1.9. *Shall* and *must* are used to state mandatory requirements. The term *should* is used to state something considered good and is recommended but is not mandatory. The term *may* is used to state something considered optional.

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Contents

Section 1: General

1.1 This standard covers the testing of metals subjected to tensile stresses for resistance to cracking failure in low-pH aqueous environments containing H_2S. Carbon and low-alloy steels are commonly tested for EC resistance at room temperature where SSC susceptibility is typically high. For other types of alloys the correlation of EC susceptibility with temperature is more complicated.

1.2 This standard describes the reagents, test specimens, and equipment to use, discusses base material and test specimen properties, and specifies the test procedures to follow. This standard describes four test methods:

Method A—Standard Tensile Test
Method B—Standard Bent-Beam Test
Method C—Standard C-Ring Test
Method D—Standard Double-Cantilever-Beam (DCB) Test

Sections 1 through 7 of this standard give general comments that apply to all four test methods. Sections 8 through 11 indicate the test method to follow for each type of test specimen. General guidelines to help to determine the aptness of each test method are given at the beginning of each test method description (Sections 8 through 11). Reporting of the test results is also discussed.

1.3 Metals can be tested for resistance to EC at temperatures and pressures that are either ambient (atmospheric) or elevated.

1.3.1 For testing at ambient conditions, the test procedures can be summarized as follows: Stressed test specimens are immersed in acidified aqueous environments containing H_2S. Applied loads at convenient increments can be used to obtain EC data.

1.3.2 For testing at temperatures higher than 27°C (80°F), at either atmospheric or elevated pressure, Section 7 describes an alternative test technique. All methods (A, B, C, and D) are adaptable to this technique.

1.4 This standard may be used for release or acceptance testing to ensure that the product meets a certain minimum level of EC resistance as prescribed in API(2) Specification 5CT,[3] ISO(3) 11960,[4] or as prescribed by the user or purchaser. This standard may also provide a quantitative measure of the product's EC resistance for research or informational purposes. This rating may be based on:

Method A	The highest no-failure stress in 720 hours.
Method B	The statistically based critical stress factor (S_c) for a 50% probability of failure in 720 hours.
Method C	The highest no-failure stress in 720 hours.
Method D	The average K_{ISSC} (threshold stress intensity factor for SSC) for valid tests of replicate test specimens.

1.5 Safety Precautions: H_2S is an extremely toxic gas that must be handled with care. (See Appendix A.)

Section 2: EC Testing Variability

2.1 Interpretation of stress corrosion test results is a difficult task. The test methods contained in this standard are severe, with accelerated tests making the evaluation of the data extremely difficult. In testing the reproducibility of the test methods among different laboratories, several undesirable side effects (frequent with many accelerated tests) that must be noted include:

2.1.1 The test environment may cause failure by HIC and hydrogen blistering. This is especially true for lower-strength steels not usually subject to SSC. HIC may be detected by visual and metallographic observations. Blistering is normally visible on the test specimen surface. (For further information regarding this phenomenon, see NACE Standard TM0284.[5])

2.1.2 The test environment may corrode some alloys that normally do not corrode in actual field service and thereby induce EC failures in alloys that ordinarily do not fail by EC. This problem is especially acute with the martensitic and precipitation-hardened stainless steels.

2.2 Furthermore, other aspects to be considered in the selection of test method(s) include:

2.2.1 Material anisotropy affecting mechanical properties and EC susceptibility can be an important parameter. The fracture path in the test specimen should match what is anticipated in the actual component.

(2) American Petroleum Institute (API), 1220 L St. NW, Washington, DC 20005.
(3) International Organization for Standardization (ISO), 1 rue de Varembé, Case postale 56, CH-1211 Geneva 20, Switzerland.