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Slow Strain Rate Test Method for Screening Corrosion-Resistant Alloys for Stress Corrosion Cracking in Sour Oilfield Service

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ABSTRACT

This standard establishes a slow strain rate (SSR) test method for screening corrosion-resistant alloys (CRAs) (i.e., stainless steels and nickel-based alloys) for resistance to stress corrosion cracking (SCC) at elevated temperatures in sour oilfield production environments. The SSR test, which is relatively short in duration, incorporates a slow, dynamic strain applied at a constant extension rate. This results in acceleration of the initiation of cracking in susceptible materials, thereby simulating rather severe conditions.

The standard specifies reagents, test specimen, test equipment, determination of baseline material properties, environmental and mechanical test conditions, test procedure, and analysis and reporting of test results. It is intended for use by used by laboratory investigators for screening CRAs for resistance to SCC in sour oilfield service.

This revision extends the scope of the standard to address the screening of precipitation-hardened nickel-based alloys for resistance to hydrogen induced stress cracking (HISC) using the SSR test method.

KEYWORDS

Slow strain rate (SSR) test, hydrogen sulfide, corrosion-resistant alloy (CRA), stress corrosion cracking (SCC), hydrogen induced stress cracking (HISC), TG 133.

Foreword

Failures of metals exposed to hydrogen sulfide (H_2S)-containing (sour) oilfield production environments have been reported for more than 50 years and have usually occurred in carbon or low-alloy steels.^{1,2} Failures of high-strength steels by brittle cracking (sulfide stress cracking [SSC]) and of lower-strength plate and pipe steels by blistering and hydrogen-induced (stepwise) cracking have also been reported. As a result, engineers and scientists have developed test methods to evaluate steels for resistance to failure by these mechanisms in sour environments.

These and other considerations led to the establishment of NACE Task Group (TG) T-1F-9, "Metallic Materials Testing Techniques for Sulfide Corrosion Cracking," which originally developed NACE Standard TM0177³ in 1977. The task group (now TG 085) has continued to revise that standard.

An additional interest of the original TG T-1F-9 was the application of corrosion-resistant alloys (CRAs), primarily stainless steels and nickel-based alloys, in oilfield production environments. Some of these CRAs have experienced stress corrosion cracking (SCC) when exposed to H_2S , carbon dioxide (CO_2), and brine. Therefore, a standardized method for screening CRAs for use in oilfield production environments is of extreme importance to the entire petroleum industry, and work group TG T-1F-9e (now TG 133) was formed to address this issue.

Several screening methods were considered: autoclave tests with statically stressed specimens, fracture mechanics methods, and the slow strain rate (SSR) test methods. Each has advantages and disadvantages that make the selection of a single test method for standardization difficult. However, the SSR test has emerged as a relatively quick, simple method that can be used for the evaluation of CRAs for resistance to a variety of environmental cracking phenomena, including SCC, hydrogen embrittlement, and liquid metal cracking.^{1,2} The use of SSR test methods, particularly in screening tests, has become more common in many laboratories for evaluation of CRAs for downhole applications.

The SSR test incorporates a slow (compared with conventional tensile tests), dynamic strain applied at a constant extension rate. Extension rates of 2.54×10^{-9} to 2.54×10^{-7} m/s (1.00×10^{-7} to 1.00×10^{-5} in/s) are commonly used. The principal effect of the constant extension rate, in combination with environmental or corrosive attack, is to accelerate the initiation of cracking in susceptible CRAs. Failure is obtained within a few days for commonly used extension rates.

Because of its relatively short test duration, the SSR test has been found useful in evaluating CRAs for resistance to SCC in simulated oilfield production environments at elevated temperatures.^{4,5} By comparison, it has been observed that it may take thousands of hours of exposure time to evaluate CRAs using more conventional statically stressed specimens.^{6,7}

In an SSR test, the test specimen is pulled to failure. One benefit of this method is that the ultimate failure of the test specimen is a positive result. That is, parameters (including reduction in area and plastic strain to failure) and visual observations can always be quantified. These results are usually further quantified by comparison with the results of similar tests performed in an inert environment. Accelerating the crack initiation by this mechanical technique tends to make the SSR test appear to be a rather severe test by being able to fail CRAs under environmental conditions in which no other test method (at reasonable exposure times) can produce failures. Because the exposure time is short and the strain rate is somewhat arbitrary, the results of SSR testing are not intended to be used directly to infer service performance. It is primarily a screening or ranking method that should be used in combination with a more extensive laboratory evaluation involving complementary testing for corrosion and environmental cracking. Service experience should be reviewed before material selection decisions are made.

A round-robin testing program was conducted by former TG T-1F-9 during the early development of this standard to evaluate the variability of SSR test data and the influences of various testing-related parameters. Draft #5 of the proposed test method was used as the basis for the round-robin testing program, and a total of seven companies participated. The results of the round-robin testing program indicated that large deviations in the SSR test data were observed for some conditions. However, with the evaluation of the procedures used by the round-robin participants, several recommendations for changes in SSR test procedures were made. Most of the recommended changes were included in this standard to reduce the amount of deviation in the test results. These changes included:

- (1) Ground surfaces (not turned) and finer surface finish on the test specimen reduced section;
- (2) Additional specifications regarding testing machine compliance;
- (3) Improved calculation technique for reduction in area; and
- (4) References to industry standards containing accepted procedures for autoclave and SSR testing.

Based on the above-mentioned considerations, TG T-1F-9 developed this standard test method incorporating the SSR test to be used by laboratory investigators for screening CRAs for resistance to SCC in sour oilfield service. It was originally developed by TG T-1F-9 in 1998 under the direction of Unit Committee T-1F, "Metallurgy of Oilfield Equipment." It was revised in 2004 and 2011 by TG 133, "Review and Revise as Necessary NACE Standard TM0198-2004," which was administered by Specific Technology Group (STG) 32, "Oil and Gas Production—Metallurgy." It was reaffirmed in 2016 by STG 32 and revised in 2020 by TG 133, now "Slow Strain Rate Test Method for Screening Corrosion-Resistant Alloys for SCC in Sour Oilfield Service." This 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. The terms **shall** and **must** are used to state a requirement, and are considered mandatory. The term **should** is used to state something good and is recommended, but is not considered mandatory. The term **may** is used to state something considered optional.

NACE International Test Method (TM0198-2020)

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Section 1: General

- 1.1** This standard establishes an SSR test method for screening CRAs (i.e., stainless steels and nickel-based alloys) for resistance to SCC at elevated temperatures in sour oilfield production environments. The fact that this test method is a screening method implies that further evaluation or additional experience may be required before materials selection decisions can be made.
- 1.2** This standard specifies reagents, test specimen, test equipment, determination of baseline material properties, environmental and mechanical test conditions, test procedure, and analysis and reporting of test results.
- 1.3** The test procedure can be summarized as follows: an SSR test specimen is exposed to a continuously increasing uniaxial tensile stress imposed by a slow and constant extension rate in the presence of an acidic aqueous environment containing H₂S, CO₂, and brine at an elevated temperature. The ductility parameters (plastic strain to failure and reduction in area) obtained from evaluation of the SSR test specimen along with visual observation of its reduced section and fracture surface morphology are used as indicators of the material's resistance to SCC in the test environment. These results are then compared to the results of a similar test performed in an inert environment to quantify the resistance or susceptibility to SCC in the test environment.
- 1.4** Procedures for SSR testing shall be consistent with those provided in ASTM⁽¹⁾ G129.⁸ Tests involving high pressure or high temperature, or both, shall be performed using procedures consistent with those provided in ASTM G111⁹ and NACE TM0177 Section 7. The only deviations from these procedures shall be those specifically stated in this standard.
- 1.5** In 2020, TG 133 extended the scope of this standard to address the screening of precipitation-hardened nickel-based alloys for Hydrogen Induced Stress Cracking (HISC) resistance using the SSR test method. The new screening method was incorporated as Appendix C (Nonmandatory), in which the appropriate hydrogen charging test provisions are specified. All other provisions of this standard shall apply.
- 1.6** Safety Precautions
- 1.6.1** H₂S is an extremely toxic gas that must be handled with extreme care. (See Appendix A [nonmandatory] for a discussion of safety considerations and toxicity of this gas).
- 1.6.2** Precautions must be taken to protect personnel from the hazards of rapid release of hot gases and fluids and explosion when working with the high-pressure, high-temperature test conditions.
- 1.7** This standard is not intended to include procedures for cyclic SSR testing. However, such procedures are currently under development by NACE TG 544 and are in use in some laboratories.

Section 2: Reagents

- 2.1** Reagent Purity
- 2.1.1** The gases, sodium chloride (NaCl), and solvents shall be reagent or chemically pure grade chemicals. The reasons for this reagent purity are discussed in Appendix B (nonmandatory).
- 2.1.2** The water shall be distilled or deionized and of quality equal to or greater than ASTM Type IV in accordance with ASTM D1193.¹⁰ Tap water shall not be used.
- 2.2** Inert gas shall be used for removal of oxygen. Inert gas shall mean high-purity (≥ 99.998%) nitrogen, argon, or other suitable nonreactive gas.

Section 3: Test Specimen

- 3.1** A uniaxial tensile test specimen shall be used for this test because it provides for a simple stress state and a common basis for comparison of test results.
- 3.2** The SSR test specimen shall be machined from the CRA to be tested in the most appropriate location and orientation relative to the specific evaluation being performed. The material form of the CRA, however, can often place restrictions on the SSR test specimen location and orientation. Furthermore, the location and orientation of the SSR test specimen can affect the test results.

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