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Standard
Test Method

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

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Foreword

Failures of metals exposed to hydrogen sulfide (H_2S)-containing (sour) oilfield production environments have been reported for more than 45 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 T-1F-9 on Metallic Materials Testing Techniques for Sulfide Corrosion Cracking, which developed NACE Standard TM0177[3] in 1977. The task group (now Task Group 085) has continued to revise that standard.

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

Several screening methods were considered by the task group: autoclave tests with statically stressed specimens, fracture mechanics methods, and the slow strain rate (SSR) test technique. Each has advantages and disadvantages that make the selection of a single test method for standardization difficult. However, over the past several years, the SSR test has emerged as a relatively quick, simple method that can be used for the evaluation of metals and alloys 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 CRA evaluation 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 2.5×10^{-9} to 2.5×10^{-7} m/s (1.0×10^{-7} to 1.0×10^{-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 materials. By doing so, the SSR acts in much the same way as a notch or precrack in statically stressed environmental cracking tests. 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 stainless steels and nickel-based alloys 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 these materials using more conventional statically stressed specimens.[6,7]

In a 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 elongation) and visual observations can always be quantified. These results are usually further quantified by comparison with the results of similar tests conducted 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 materials 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 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. A review of service experience should be conducted before material selection decisions are made.

A round-robin testing program was conducted by former NACE Task Group 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 this round-robin program and a total of seven companies participated. The results of this program indicated that large deviations in the SSR test data were observed for some conditions. However, upon 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 in an effort 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 gauge section.

(2) Additional specifications regarding testing machine compliance.

(3) Improved calculation technique for reduction in area.

(4) References to industry standards containing accepted procedures for autoclave and SSR testing.

Based on the above-mentioned considerations, Task Group T-1F-9 developed this standard test method incorporating the SSR test to be used by laboratory investigators for screening CRAs for SCC in sour oilfield service. This NACE standard was originally developed by Task Group T-1F-9 in 1998 under the direction of Unit Committee T-1F on Metallurgy of Oilfield Equipment. It was revised in 2004 by Task Group (TG) 133 on Slow Strain Rate Test Method. TG 133 is administered by Specific Technology Group (STG) 32 on Oil and Gas Production—Metallurgy and sponsored by STG 62 on Science and Engineering Applications and Methods of Corrosion Monitoring and Measurement. 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, 4th ed., Paragraph 7.4.1.9. *Shall* and *must* are used to state mandatory requirements. The *term* should is used to state something 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 establishes a SSR test method for screening CRA materials (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: A test specimen is exposed to a continuously increasing uniaxial tensile stress imposed via a slow and constant extension rate in the presence of an acidic aqueous environment containing H_2S, CO_2, and brine at an elevated temperature. The ductility parameters (plastic elongation and reduction in area) obtained from evaluation of the test specimen along with visual observation of its gauge 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 with the results from a similar test conducted 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) G 129.[8] Tests involving high pressure and/or high temperature shall be performed with procedures consistent with those provided in ASTM G 111.[9] The only deviations from these procedures shall be those specifically stated in this standard.

1.5 Safety Precautions

1.5.1 H_2S is an extremely toxic gas that must be handled with extreme care. (See Appendix A for a discussion of safety considerations and toxicity of this gas.)

1.5.2 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.

1.6 This standard is not intended to include procedures for cyclic SSR testing. However, such procedures are currently under development 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.

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 D 1193.[10] Tap water shall not be used.

2.2 Inert gas shall be used for removal of oxygen. Inert gas shall mean high-purity 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.

3.1.1 The test specimen shall be machined from the material to be tested in the most appropriate location and orientation relative to the specific evaluation being performed. The material form, however, can often place restrictions on the test specimen location and orientation. Furthermore, the location and orientation of the test specimen can affect the test results.

3.1.2 The test specimen shall be fabricated whenever possible in accordance with the configuration of the standard test specimen given in Figure 1. The length of the test specimen has not been specified to accommodate the following two common test configurations: <u>Configuration 1</u>—the test specimen is entirely enclosed in the test vessel with metal grips (pull rods) passing through the ends of the test vessel;

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