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An Evaluation of the Ripple-Slow Strain Rate (R-SSR) Technique for Environmentally Assisted Cracking (EAC) Qualification of Modified 13Cr-110 Stainless Steel

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ABSTRACT

The ripple-slow strain rate (R-SSR) technique is an alternative screening test to the conventional-SSR (C-SSR) method. Both methods assess susceptibility to environmentally assisted cracking (EAC) of corrosion resistant alloys (CRAs) and involve loading a NACE TM0198-compliant tensile specimen to 100% AYS (0.2% proof stress of actual yield strength) at a slowly applied extension rate. While the C-SSR specimen is monotonically strained to failure, a R-SSR tensile specimen is typically loaded to 100% AYS and subsequently cycled between 80% and 100% AYS until failure or for up to 250 cycles or 1 week of exposure.

This paper will provide a comprehensive review of the current understanding of the R-SSR technique. There are only a limited number of publications discussing the utility of the R-SSR technique, because running a R-SSR test is more complicated than a C-SSR test, and the added phenomenological/mechanistic value of performing a R-SSR test is unclear. On the one hand, cycling loading may be more representative of certain in-field service conditions. On the other hand, there is evidence to suggest that cyclic loading facilitates film rupture during the tensile loading part of the cycle and repassivation of passive and/or protective film during load reversal, which may influence crack propagation. R-SSR may also be a better approach for martensitic stainless steels than C-SSR where cumulative strain in the conventional test may be detrimental to material performance. Regardless of the motivation, there is not a clearly defined and universally recognized acceptance criteria for assessing EAC resistance/susceptibility by R-SSR.

A pilot research program was initiated to better understand the value of results generated by R-SSR in comparison to C-SSR. For this study, mod13Cr-110 alloy was used to evaluate and benchmark the two techniques. The overall goal of the project was to provide guidance on when to use R-SSR and how to interpret the test results.

Keywords: *Slow Strain Rate, Ripple SSR, Cyclic SSR, EAC, Sour Service Qualification, Mod13Cr-110*

INTRODUCTION

Since the 1960s, the slow strain rate (SSR) technique, also known as constant strain rate test or constant extension rate test (CERT), has been used extensively to evaluate the environmentally assisted cracking (EAC) susceptibility of corrosion resistant alloys (CRAs) [1-3]. The purpose of the test is to determine whether the mechanical properties of a ductile CRA, typically evaluated in air, deteriorates in the presence of a corrosive environment under a continuously increasing uniaxial tensile stress. The SSR testing procedures are well established and are described in the following standards: NACE TM0198-2020 [4], ASTM G129 [5] and ISO 7539 Part 7 [6]. SSR testing has been widely used to evaluate stress corrosion cracking (SCC) threats for oil and gas sour service, buried natural gas pipelines, ethanol transportation, nuclear and fossil fuel power, and low pressure turbines applications [2,3].

Conventional-SSR (C-SSR) testing incorporates a slow, dynamic strain applied at a constant extension rate. Typically, NACE TM0198-compliant tensile specimens are monotonically strained to failure in C-SSR tests. C-SSR is often used as a screening method to evaluate CRA susceptibility to a specific environment relative to its behavior in air. Following an environmental C-SSR test, several ductility measurements are made (e.g., reduction in area, elongation, and reduction of plastic strain) in addition to time to failure. Collectively, these ductility parameters are often expressed as a ratio of the material's behavior in an environment relative to its performance in air. C-SSR testing remains popular as a single test can be completed generally within one to three days depending on the strain rate.

Some researchers [7] have argued that, while the C-SSR technique is relatively easy to perform and provides a rapid assessment of performance in an aggressive environment, the method is far too aggressive/conservative as specimens are strained to failure. For oil and gas applications, materials are rarely designed to be loaded above yield. Therefore, the concern is making material selection and/or procurement choices based on mechanical behavior within the plastic region (of a load-displacement curve) may be too conservative and may unnecessarily limit a user's available material selection options.

During the 1990s, the ripple-SSR (R-SSR) technique, also known as rippled SSR, or cyclic SSR, was proposed as an alternative screening test to the C-SSR [7]. Instead of pulling a specimen to failure, in a R-SSR test the tensile specimen is merely loaded to 100% of actual yield strength (AYS) and subsequently cycled between 80 and 100% AYS until failure or for a specified duration (typically up to 250 cycles or 1 week of exposure). It has been argued that this level of cyclic loading is more representative of certain field conditions. R-SSR is also considered as a hybrid testing methodology in between rapid strain-to-failure C-SSR and long-term constant load methods for fitness for purpose in terms of testing severity.

Currently, there is an effort to prepare a standard R-SSR test method within NACE. In an effort to investigate the utility of the R-SSR methodology, a research investigation was launched to compare the performance of modified 13Cr-110 by both C-SSR and R-SSR methodologies under mildly sour service conditions previously known to induce/resist EAC [8].

APPLICATIONS OF RIPPLE SSR TEST

R-SSR technique has been used to qualify CRAs [7-10] in the oil and gas industry. R-SSR is frequently used to simulate field cyclic straining/stressing in the lab and/or selected to support failure analysis investigations [11-14]. Advantages of the R-SSR method include: 1) Cycling loading may be more representative of certain in-field service conditions; and 2) Cyclic loading may facilitate film rupture events during the tensile loading part of the cycle and repassivation of passive and/or protective film during load reversal, which may influence crack initiation and crack propagation.

Compared to C-SSR, however, the number of publications discussing the R-SSR technique is relatively few. Performing a single R-SSR test takes far longer to complete and is more complicated to administer

than a C-SSR test. Unlike performing a C-SSR test, the apparent AYS of material, measured directly on SSR frame used to evaluate the material, is required in advance of testing in order to actually define the loading/unloading stress limits during cycling. Lastly, the added phenomenological/mechanistic value of performing a R-SSR test, instead of a C-SSR, remains unclear.

Qualification of CRAs by R-SSR Technology

Nisbet *et al.* [7] developed the R-SSR technique as a methodology to evaluate CRAs for oil and gas industrial applications. R-SSR was considered mechanically less aggressive than the standard C-SSR test and more representative of service loading. It involves straining to some preselected level (often yield) and then cyclically stressing the specimen between certain predetermined limits, avoiding the excessive plastic strain imposed during a C-SSR test. According to Nisbet *et al.* [7], during a R-SSR test, the tensile specimen was strained at 1×10^{-6} 1/s up to the 0.2% proof stress, and then was cycled between 100% and 80% of the 0.2% proof stress with a loading strain rate of 1×10^{-6} 1/s and an unloading strain rate of 5×10^{-6} 1/s until failure occurred or for a set period of 1 week.

Wilms and coworkers [9] adopted R-SSR test to assess 17Cr-125 alloy suitability for a slightly sour high-pressure high temperature (HTHP) gas field located in the North Sea containing 0.039 bara (0.57 psia) pH_2S and 38,000 ppm Cl^- . 17Cr-125 SSR tensile specimens were strained at a loading rate of 1×10^{-6} 1/s and unloading strain rate of 5×10^{-6} 1/s, cycling between 80% and 100% of 0.2% proof stress for 1 week or until failure, whichever came first. R-SSR tests were performed at 20 °C, 60 °C and 185 °C. At 20 °C, the tensile ruptured after 3 cycles, whereas the tensile ruptured after 13 cycles at 60 °C. At 185 °C, 133 cycles were completed within 1 week. Therefore, 17Cr-125 showed the highest susceptibility to cracking at 20 °C, implying that sulfide stress cracking (SSC) is the most critical failure mode for this alloy.

In contrast, Al-Harthi and his colleagues [10] used R-SSR to qualify 316L alloy (UNS 31603) as a lower cost alternative to Alloy 825 and 22Cr/25Cr duplex stainless steels for a Middle East project. R-SSR tests were performed using 316L tensile specimens at a loading strain rate of 1×10^{-6} 1/s and unloading strain rate of 5×10^{-6} 1/s, between 80% and 100% AYS. The R-SSR test failed at 1 bar pH_2S , 60 °C and 230,000 mg/Kg NaCl, indicating that the service limit “ pH_2S 1 bar up to 60 °C with any chloride concentration and pH” defined in ANSI/NACE MR0175/ISO 15156-2015:3, Table A.2, for UNS 31603 is too optimistic. However, specimens passed the R-SSR test at a lower NaCl content, 132,000 mg/Kg. R-SSR test results indicated that 316L could not be used under the expected operating conditions.

While R-SSR test was successfully utilized to assess EAC susceptibility of CRAs in the above studies, neither study reported complementary C-SSR tests. It is unclear whether a different material recommendation would have been made based on the results by C-SSR.

Simulation of Field Cyclic Loading Conditions

R-SSR has also been used to simulate the effect of field pressure fluctuation in an underground carbon steel pipeline. Wang *et al.* [11] simulated field fluctuation by cyclically loading tensile specimens prior to corrosion exposure. The objective was to understand the effect of cycling on SSC initiation of pipeline steels exposed to a near-neutral pH solution. The authors found that the cyclic loading prior to pulling the SSR specimen to failure increased cracking susceptibility.

Griffiths and coauthors [12] utilized the cyclic SSR test to evaluate super 13Cr parent and weld specimens at stress levels corresponding to 0.2% and 2% plastic strain. Cycling was applied to simulate pipe movement expected in the field. The authors were interested in the cumulative effect of plastic strain on the resistance to hydrogen embrittlement under cathodic protection for 2 weeks. Griffiths *et al.* found that the majority of the cumulative plastic deformation occurred in the first cycle of the cyclic SSR testing, and more over, no cracking was observed during the cyclic SSR testing. Based on the testing observations, the authors concluded that little additional value was gained from conducting cyclic SSR testing for work-hardened CRAs when evaluated for their system [12].

Gui *et al.* [13] also performed R-SSR tests on 2507 DSS (UNS S32750) tensile under anodic polarization to study its cracking mechanism. R-SSR testing results supported the convention that stress corrosion cracking (SCC) of CRAs may occur above the repassivation potential.

Dean *et al.* [14] performed cyclic straining on 304L, 2205 DSS and Alloy 625 tensile specimens to simulate the cracking failure that had occurred in a hydrogen plant. The C-SSR test did not reveal cracking of 304L, whereas cracking was detected by R-SSR. In this instance, the failure mechanism was considered to be corrosion-fatigue dependent.

EXPERIMENTAL

Material

In this study, the 110 ksi grade of modified 13Cr martensitic stainless steel tubing (mod 13Cr MSS) with a “13Cr-5Ni-2Mo” nominal chemistry was used for machining specimens. The “13Cr-5Ni-2Mo” alloy is commonly used in oilfield applications that require high yield strength, and where CO₂ corrosion is a potential concern. Table 1 shows the chemical composition of the mod 13Cr-110 tubing used in this work. Mechanically, the mill certificate reported the material had an AYS of 134.8 ksi, tensile strength of 138.9 ksi, percent elongation of 22.7 %, and a hardness of 31 HRC.

Specimens Machining

SSR tensile specimens were prepared in accordance with the design tolerances specified in NACE TM0198-2020 and is schematically shown in Figure 1.

Test Matrix

Table 2 shows test matrix in this study. A total of fourteen (14) specimens were evaluated by performing complementary C-SSR and R-SSR tests per environmental condition. For this study, three pH₂S levels were considered, 0, 0.15, and 1.5 psia, in the presence of CO₂ and at a total pressure of 2,000 psig. 1.5 psia pH₂S is the sour service limit for martensitic stainless steels defined by ANSI/NACE MR0175/ISO 15156-3:2015. In addition, tests were performed at two different temperatures 75 °F and 200 °F for screening susceptibility to sulfide stress cracking (SSC) at low temperature and stress corrosion cracking (SCC) at high temperature, respectively.

Equipment

All tests were performed in SSR autoclaves as the autoclaves themselves constitute part of the specimen alignment apparatus. The autoclaves were fabricated from Alloy C-276 with an internal capacity of 500 ml. They were equipped with sliding chevron Teflon and PEEK seals in the top and bottom bulkheads through which sliding Xylan-coated SM2535 pull-rods are inserted into the autoclave. The pull-rods were coated with a fluoropolymer coating masking the pull-rods and minimizing galvanic effects between the SSR tensile and the frame. All SSR equipment and testing procedures were performed in compliance with NACE TM0198-2016 and all SSR equipment was calibrated prior to testing.

Test Procedure

SSR Frame Setup and Speed Calibration

All tests at the same temperature were performed on a single SSR frame, to maintain the same mechanical compliance. The SSR frame was calibrated to a constant displacement rate of 4×10^{-6} in/s using a nickel-based alloy specimen. After the engineering stress-strain curve was generated for the calibration specimen, the change in displacement (or distance) of the linear variable differential transformer (LVDT) with time was reviewed. The stress-independent strain values collected within the plastic region was then used to calculate an equivalent constant displacement (strain) rate of 4×10^{-6} 1/s per frame.

Tensile Preparation

Prior to testing, the gauge section diameter and the total length of each SSR tensile specimen was measured to the nearest 0.001 inch. Afterwards, the specimen was cleaned with toluene/acetone and subsequently dried in ultra-high purity (UHP) nitrogen.

The cleaned specimen was then inserted into the SSR autoclave, and later subsequently sealed. A pressure check was performed to ensure the autoclave was sealed properly at 1.5 times the test pressure. After a successful pressure check, each tensile specimen was threaded into two Xylan-coated pull-rods. In addition, the surface area of each pull-rod was wrapped with Teflon tape to further minimize galvanic effects to prevent inadvertent exposure of the rods to the internal autoclave test environment.

As a final check, the electrical resistance between the two pull-rods, and the resistance between each pull-rod and the autoclave body, were individually measured to ensure electrical isolation of the tensile specimen using a multimeter.

C-SSR Test

The specimens used in all C-SSR tests were strained to failure at a constant extension rate of 4×10^{-6} 1/s. Prior to environmental C-SSR tests, at each test temperature, an air test was conducted as a baseline to assess changes in ductility. In addition, a baseline pressurized nitrogen (N₂) SSR test was performed in a brine-free autoclave with pressurized nitrogen gas at 2000 psig at each test temperature for determination of apparent yield strength at the 0.2% offset at both 75 °F (AYS₇₅) and 200 °F (AYS₂₀₀).

For each environmental SSR test, the autoclave was pressure checked and deaerated with UHP N₂ gas. Afterwards, 375 ± 5 ml of the deaerated environment-specific brine solution, having a composition shown in Table 2, was drawn into the autoclave under a UHP nitrogen blanket. After solution transfer, the solution was purged with UHP nitrogen for an additional 30 minutes at a nitrogen flow rate of 500 ml/min, which also flushed the headspace gas with the UHP nitrogen. Later, the autoclave headspace was evacuated by a vacuum pump and the required inventories of H₂S and CO₂ (based on thermodynamic model calculations) were quantitatively loaded into the autoclave for environmental testing. The autoclave was then brought to the test temperature and pressurized to $2,000 \pm 50$ psig with UHP nitrogen. For each test, the specimen was pulled to failure at an applied strain rate of 4×10^{-6} 1/s.

Table 2 lists a summary of the C-SSR tests performed in this study.

R-SSR Test

The autoclave was loaded and charged with a specific environment composition as listed in Table 2 and followed the same procedure for loading of a C-SSR test as described above. The yield strength (0.2% offset) (AYS) evaluated by CSSR tests at each environment shown in Table 3 were used for determining the stress level used in R-SSR tests at each test condition.

Once the temperature and total pressure were stabilized in the autoclave, R-SSR testing commenced. The tensile specimen was initially loaded toward an upper stress level corresponding to the apparent 100% AYS previously determined by C-SSR at a strain rate of 1×10^{-6} 1/s. Upon reaching the upper stress level, the specimen was unloaded at a strain rate of 5×10^{-6} 1/s to 80% AYS. Cycling between 100% AYS and 80% AYS continued until the test ran for 250 cycles or 10 days or until failure, whichever came first.

After completion of the R-SSR test, each tensile specimen was inspected visually and by microscope. Any tensile specimen that remained intact was later pulled to failure in air (at atmospheric pressure) at a strain rate of 4×10^{-6} 1/s at the same temperature evaluated during the R-SSR test. Straining the environmental R-SSR tensile specimens to failure in air allowed for the assessment of ductile properties.

Table 2 lists a summary of the R-SSR tests performed in this study.