

The Influence of Strain Rate and Specimen Size on Assessment of SSC Resistance by Novel NTSSRT Method

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ABSTRACT

Sulfide stress cracking (SSC) has been a concern for the use of high strength low alloy (HSLA) steel in oil and gas industry. A novel methodology named notch tensile slow strain rate test (NTSSRT) is used to obtain the threshold stress intensity factor K_{ISSC} to quantify SSC resistance of HSLA carbon steel. In this study we evaluate the effect of the strain rate and specimen gage diameter, on K_{ISSC} assessed by NTSSRT method. The testing is performed in environment with different H_2S concentrations at pH of 3.5. At the same H_2S concentration, the K_{ISSC} value decreases with a decline in strain rate due to the longer immersion time at slower strain rate. For 40%-notched specimen (notch diameter/gage diameter = 0.6), K_{ISSC} is independent on the specimen gage diameter when the gage diameter is larger than 0.125 in.

Key words: H_2S , K_{ISSC} , SSC, strain rate, specimen geometry

INTRODUCTION

High strength low alloy (HSLA) steels are preferred for oil and gas pipelines due to their outstanding mechanical properties^{1, 2}. Sulfide stress cracking (SSC) has been a major problem for the application of HSLA carbon steel because of the wet H_2S environment which commonly presents in oil and gas industry³⁻⁵. Several techniques are applied to the study of SSC of steels, including constant load test with smooth specimens and DCB testing^{6, 7}. However, the long test duration of these techniques (up to 720 hours) may lead to a change of environmental parameters during testing, which affect the reliability of the testing results. To increase the reproducibility and reliability, the notched slow strain rate testing is applied to the study of the environmentally induced cracking (EIC) first reported by Caballero L. et.al.⁸. The use of notched specimens favors the localization of the environmental effect in the notch tip.

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Therefore, a relationship between environmentally variable and local strain rate at the crack tip can be developed to characterize the EIC susceptibility⁹. However, the local strain rate in the vicinity of crack tip is difficult to determine for brittle materials or for the specimens with high susceptibility to hydrogen embrittlement. A novel notched tensile slow strain rate testing (NTSSRT) proposed by Case et. al,^{3, 10, 11} is applied to evaluate the SSC resistance of HSLA carbon steel, where hydrogen embrittlement tends to happen because of the accelerative effect of H₂S on hydrogen uptake¹². The threshold stress intensity factor K_{ISSC} is used in this novel NTSSRT method to evaluate the SSC resistance. Compared with the commonly used DCB method, the novel NTSSRT method is more time-effective methodology (less than 48 hours).

For the NTSSRT method, theoretically speaking, the effects of strain rate and specimen size on K_{ISSC} should be similar with the effects of arm displacement and specimen size for DCB test. Because the amount of bulk hydrogen in specimen is the main driving force for SSC crack propagation, and the absorbed hydrogen concentration is a time-dependent parameter¹³. Higher strain rate may shorten the test duration, hence decrease the concentration of bulk hydrogen in steel, K_{ISSC} increases consequently^{14, 15}. Besides, small specimens are hydrogenated more rapidly than large specimens¹⁶. However, for NTSSRT method, the evolutions of K_{ISSC} value with the strain rate and specimen size have not been experimentally studied yet. As important experimental parameters, the strain rate and specimen size should be well-defined for the application of NTSSRT method. Therefore, the objective of this study is to evaluate the influences of strain rate and specimen gage diameter on K_{ISSC} value by NTSSRT test under wet-H₂S environment. The morphology of the fracture surface is studied by SEM to have a better understanding about the fracture mechanism.

EXPERIMENTAL PROCEDURE

Experimental design

Following the objectives of the present study an experimental design (Table 1) is developed to evaluate the individual effects applied extension rate and specimen geometry on SSC resistance of the HSLA carbon steel C110 by NTSSRT method.

The independent variables in the study of the strain rate effect are the H₂S concentration in the gas phase and the strain rate of specimen, keeping constant the specimen size. In the case of the evaluation of the effect of specimen size, the independent variables are the H₂S concentration and the specimen gage diameter whereas the strain rate is kept constant. The dependent variables in our study are K_{ISSC} value from NTSSRT and fracture surface morphology detected by SEM.

Table 1: The sequence of experimental conditions.

	H ₂ S mole% gas	Deflection Rate in/s	Gage Diameter in	Notch %
Effect of Strain Rate	55 & 90	$10^{-7} - 10^{-5}$	0.25	40
Effect of Specimen Size	55 & 90	10^{-6}	0.125 – 0.25	40

Material

The steel studied in this work is oil country tubular good (OCTG) grade HSLA carbon steel C110 with a yield strength of 111.68 ksi (770 MPa), a ultimate tensile strength of 122.70 ksi (846 MPa), and a hardness of 27 HRC¹⁷. The chemical composition of C110 is shown in Table 2.

Table 2: Chemical Composition of OCTG Grade HSLA Carbon Steel C110 in wt%.

C	Mn	Si	Mo	Cr	Ni	P	S	Fe
0.27	0.30	0.24	0.80	0.90	0.05	0.017	0.002	Bal.

According to a previous study, when the notch depth is greater than 40%, the NTSSRT methodology loses the selectivity because of strain hardening and notch strengthening¹⁰. Therefore, the specimen with a notch depth of 40%, notch root radius of 0.005 in and the notch angle of 60° as required by ASTM standard E1820 (2020)¹⁸, and gage diameter of 0.25 in is used in NTSSRT testing. The specimen geometry is as shown in Figure 1.

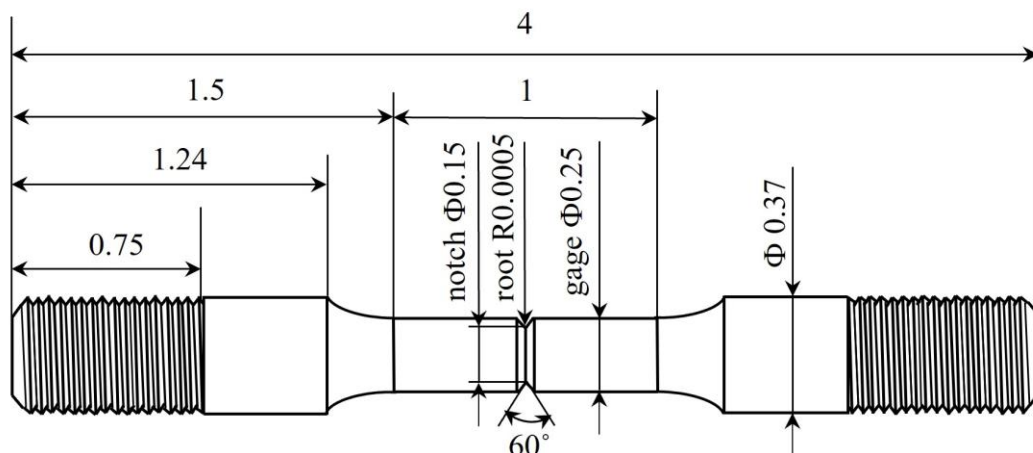


Figure 1: 40%-notched NTSSRT specimen (unit: inch).

Testing Solution

To study the effects of applied extension rate and specimen geometry on SSC resistance, NTSSRT testing is conducted in solution with pH of 3.5, 1 wt% NaCl, and different H₂S concentrations. The solution pH is adjusted by sodium acetate and acetic acid buffer solution (CH₃COONa + CH₃COOH). H₂S is generated by chemical reaction between Na₂S and HCl following Equation (1). A specific concentration of H₂S in gas phase is achieved by controlling the addition of Na₂S and HCl according to the thermodynamical speciation calculation results presented in Table 3. To make sure the correct H₂S concentrations are achieved during the testing, the H₂S concentration in gas phase is verified by H₂S tubes, and the H₂S concentration in solution is checked by inductively-couple plasma emission spectroscopy.



Table 3: The composition of solution with different H₂S concentrations (mol% in gas phase).

	55 mol%	90 mol%
NaCl (g/L)	10	10
CH ₃ COONa (g/L)	4	4
Na ₂ S•9H ₂ O	21.67	116.60
CH ₃ COOH (mL/L)	30	30
HCl (mL/L)	15.67	44.00

Notched Tensile Slow Strain Rate Testing

As mentioned before, NTSSRT method is applied to assess the effects of applied extension rate and specimen geometry on SSC resistance of HSLA carbon steel C110. The NTSSRT test procedure follows the SSR test methodologies described in the standard NACE TM0198 (2011)¹⁹. The cell used for experiments is exhibited in Figure 2.

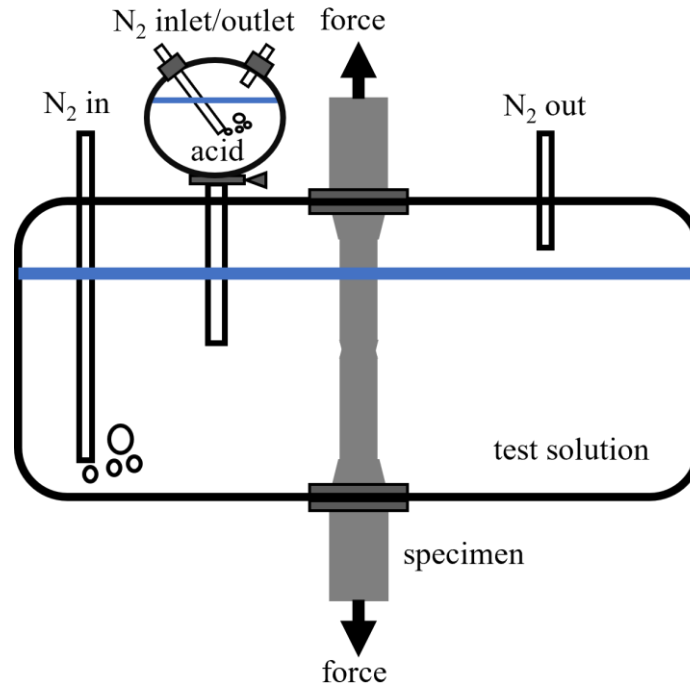


Figure 2: The cell used in NTSSRT experiments.

Before testing, the solution is de-aerated by N_2 for 1 hour. After deaeration, different concentrations of H_2S are generated in the cell through mixing acid and testing solution shown by Table 3. A preload process is conducted with a deflection rate of 10^{-5} in/s (strain rate $10^{-5} s^{-1}$) and a target load (100 lbs.). After achieving the target load, the load is kept at 100 lbs. for 1 hour to allow the adsorbed hydrogen to move in steel. After the preloading, the specimens with different gage diameters and notch sizes are stretched at the constant deflection rates of 10^{-5} , 10^{-6} , and 10^{-7} in/s (strain rate 10^{-5} , 10^{-6} , and $10^{-7} s^{-1}$) until the specimen failure occurs.

Our previous work shows that when the environmental H_2S concentration is higher than 1 mol%, the K_{ISSC} value can be calculated from the NTSSRT results by Equation (2), which assume the fracture mechanism are the linear elastic fracture¹¹.

$$K_{ISSC} = \sigma_{max} D^{\frac{1}{2}} \left[2 \left(\frac{d}{D} \right)^4 \left(1 - \frac{d}{D} \right) + 0.364 \frac{D}{d} \left(1 - \frac{d}{D} \right)^2 \right]^{\frac{1}{2}} \quad (2)$$

where D and d , are the diameter at gage and notch, σ_{max} is the maximum load at notch.

Microscopic Observation

The morphology of the fracture surface of specimen tested under different H_2S concentrations is studied by a scanning electronic microscopy (JCM-7000 NeoScope Benchtop SEM). The area percentage R_b of brittle region is calculated from the SEM image of the fracture surface by Equation (3).

$$R_b = \frac{A_b}{A_f} \times 100\% \quad (3)$$

where A_b is the area of brittle region, and A_f is the area of the total fracture surface.

RESULTS

Effect of The Strain Rate on SSC Resistance

Stress-strain Curves at Different Strain Rate

The strain rate is an important processing parameter as mentioned. The effect of the strain rate on SSC resistance is studied by NTSSRT method on 40%-notched specimen with a gage diameter of 0.25 in in environment with a pH of 3.5 and H₂S concentration of 55 mol% and 90 mol%. The stress-strain curves obtained from NTSSRT testing are shown in Figure 3.

Specimens tested at 55 mol% show more elastoplastic behavior than 90 mol% H₂S. Since at same strain rate, the plastic part of the stress-strain curves at 55 mol% H₂S are larger than these at 90 mol% H₂S. This is consistent with the enhanced hydrogen uptake which is accelerated by adsorbed H₂S layer, more hydrogen is absorbed into steel, steel becomes more brittle due to hydrogen embrittlement.¹²

As it can be also observed in Figure 3, at the same H₂S concentration, both the ultimate tensile stress (UTS) and final strain decrease with reducing strain rate at same H₂S concentration. Specimen tested under slower strain rate experience longer immersion time in wet-H₂S environment.¹⁵ For instance, at 90 mol% H₂S, specimen takes 22 min to break at strain rate of 10^{-5} s^{-1} , while specimen tested by 10^{-7} s^{-1} strain rate needs 18 hours. Therefore, at slower strain rate, hydrogen has enough time to be transmitted to and accumulated at crack tip, which increase the susceptibility to hydrogen embrittlement.

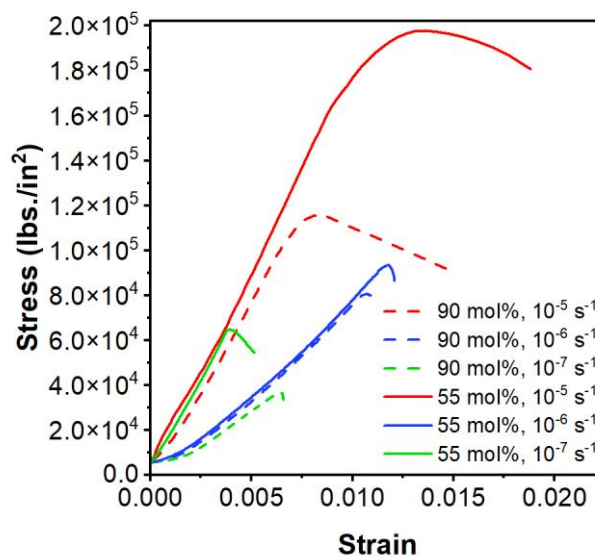


Figure 3: Stress-strain curves of 40%-notched specimens under different H₂S concentrations (solid lines: 55 mol% H₂S, dash lines: 90 mol% H₂S) and strain rate (10^{-5} , 10^{-6} , and 10^{-7} s^{-1})

Assessment of The Effects of Strain Rate on Threshold Stress Intensity (K_{ISSC})

The SSC resistance of HSLA carbon steel C110 is characterized by K_{ISSC} value in this study. Since the H₂S concentrations are higher than 1 mol%, based on the NTSSRT methodology, the K_{ISSC} value is determined from Figure 3 by Equation (2). The results are shown in Figure 4. From Figure 4, at the same H₂S concentration, K_{ISSC} value increases with an increase in strain rate. Under environment with 55 mol% H₂S concentration, K_{ISSC} increases from 14.15 ksi in^{0.5} (15.56 MPa m^{0.5}) at strain rate of 10^{-7} s^{-1} to 43.66 ksi in^{0.5} (48.02 MPa m^{0.5}) at strain rate of 10^{-5} s^{-1} . At environment with a H₂S concentration of