



Article

Sulfide Stress Cracking of C-110 Steel in a Sour Environment

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Abstract: The scope of this study includes modeling and experimental investigation of sulfide stress cracking (SSC) of high-strength carbon steel. A model has been developed to predict hydrogen permeation in steel for a given pressure and temperature condition. The model is validated with existing and new laboratory measurements. The experiments were performed using C-110 grade steel specimens. The specimens were aged in 2% (wt.) brine saturated with mixed gas containing CH₄, CO₂, and H₂S. The concentration H₂S was maintained constant (280 ppm) while varying the partial pressure ratio of CO₂ (i.e., the ratio of partial pressure of CO₂ to the total pressure) from 0 to 15%. The changes occurring in the mechanical properties of the specimens were evaluated after exposure to assess material embrittlement and SSC corrosion. Besides this, the cracks developed on the surface of the specimens were examined using an optical microscope. Results show that the hydrogen permeation, and subsequently SSC resistance, of C-110 grade steel were strongly influenced by the Partial Pressure Ratio (PPR) of CO₂ when the PPR was between 0 and 5%. The PPR of CO₂ had a limited impact on the SSC process when it was between 10 and 15 percent.

Keywords: corrosion; sulfide stress cracking; modeling; hydrogen permeation



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1. Introduction

1.1. Overview

In a sour environment, Sulfide Stress Cracking (SSC) corrosion often degrades downhole tubulars by embrittlement which leads to premature failure. This study is aimed at understanding the mechanism of hydrogen diffusion in metals in presence of H₂S, determining the factors that influence the vulnerability of tubulars to SSC corrosion, and formulating a model to predict SSC corrosion susceptibility of metals. Often SSC corrosion studies [1–4] are performed at pressures less than 6 bar. The assessment of SSC corrosion is commonly performed applying the standard NACE test Method A with cylindrical specimens. Besides this, other methods have been developed in the industry to assess the SSC corrosion resistance of metals [5]. Cernocky et al. [6] developed an experimental setup for SSC corrosion testing of a minipipe specimen subjected to triaxial stress loading conditions. The test setup simulated the loading conditions that are similar to the actual wellbores. Results were validated with measurements obtained from full-scale experiments.

The life of oil wells critically depends on casings, which protect the integrity of the wellbore. With increasing depth of wells, casing materials with high strength are required. Even though high-strength materials can handle high pressure and stress levels, they could be susceptible to Sulfide Stress Cracking (SSC) corrosion in a sour environment [7]. This type of corrosion rapidly degrades downhole tubulars by embrittlement, and subsequently cracking that causes early failure. SSC is corrosion that is caused often when stressed tubulars are exposed to an H₂S environment. Because of SSC, tubulars usually fail at stress levels well below their yield stress [7]. As a result, SSC is affected by different environmental and metallurgical factors, including material type, microstructure, pressure, temperature, and the composition and pH of the surrounding solution [7–10].

1.2. Factors Affecting SSC Corrosion Resistance

Effect of Partial Pressure of H_2S and Solution pH: The partial pressure of H_2S and pH of the surrounding solution are the most important factors that affect the SSC corrosion resistance of metals. A recent SSC corrosion study [11] was conducted using the Method A tests. Experiments were performed varying the partial pressure from 0.10 to 50.0 kPa and pH from 3.5 to 5.5. Results demonstrated higher SSC resistance of P-110 carbon steel as compared to high-strength steels (Q-125, Grade 140, and Grade 150 carbon steel). Based on experimental results, a window of applicability plot (Figure 1) was prepared for each steel grade.

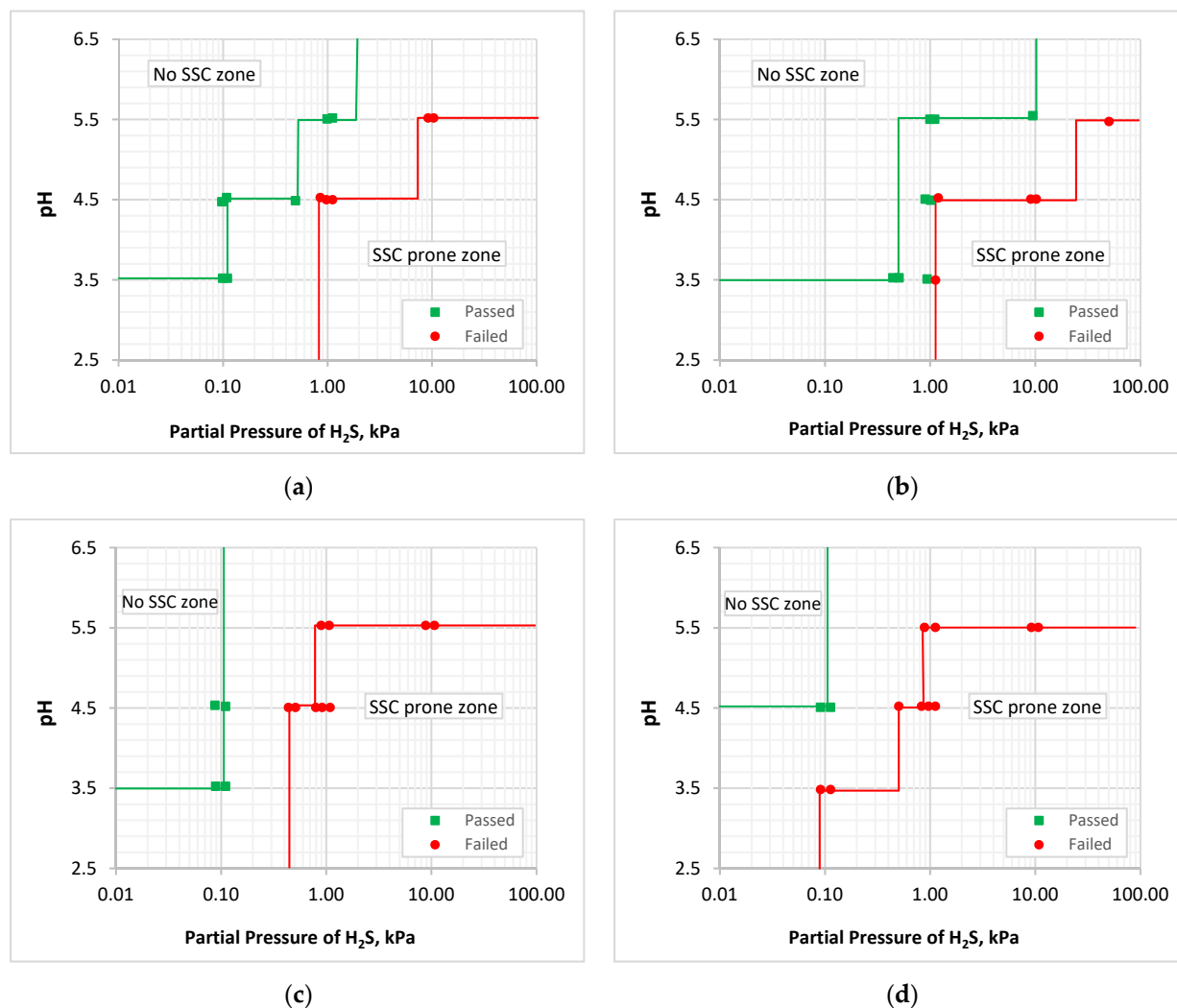


Figure 1. Operability window of different steels: (a) Q-125, (b) P-110, (c) Garde 140, and (d) Grade 150 (Redrawn from 11).

Figure 1a displays the operating conditions for Q-125. The area on the left side of the green line represents the safe conditions (i.e., conditions with low partial pressures and high solution pH) in which the vulnerability of the metal to SSC corrosion is minimal while the area on the right side of the red line shows the unsafe conditions in which the material is highly prone to the SSC corrosion. The area in between the two lines represents less certain conditions in which there is a moderate level of vulnerability to the SSC corrosion. Comparing the vulnerability of different steels, P-110 demonstrated the highest window of applicability following Q-125. The SSC corrosion resistance of P-110 was better than Q-125 when they were tested at the same conditions. Thus, as anticipated, the grade with higher yield strength was more susceptible to SSC corrosion.

A study conducted by Vera and Case [12] examined the effects of pH and temperature on SSC corrosion resistance of high strength API carbon steel (P-110) varying pH (2.5 to 3.5) at room temperature and changing temperature (25 to 95 °C) at a pH of 2.5. Increasing pH and temperature improved the SSC corrosion resistance of the metal. Other studies [7,13–17] reported similar observations indicating improvement of the SSC corrosion resistance of steel with an increase in solution pH and temperature.

Effects of Microstructure and Cold Work: A number of investigations [18,19] were performed to study the impact of microstructure on SSC corrosion. A study [19] conducted on materials with varying martensite and bainitic contents indicated that materials with a high martensite content exhibit a higher SSC corrosion resistance. These findings concur with the observations of an earlier study [18]. A more recent SSC corrosion study [20] performed on API-grade pipeline steel demonstrated the impact of microstructure on SSC corrosion. Due to the presence of hard phase cementite at grain boundaries, the ferrite-bainitic microstructure exhibited a lower SSC corrosion resistance as compared to the acicular ferrite microstructure.

Besides microstructure, cold work and the presence of inclusion clusters influence the SSC corrosion resistance. Increasing inclusion clusters in metals exacerbates their susceptibility towards SSC corrosion [21]. Moreover, acicular ferrite exhibits higher SSC resistance than ferrite degenerated pearlite. These findings are in agreement with the observations of a similar study [20]. Furthermore, cold work considerably influences the SSC corrosion resistance of metals. Cold work is regularly conducted on metals to improve their mechanical characteristics such as yield strength, Young modulus, and hardness. When cold work is performed, the grain structure of the material could get distorted, increasing the number of structural dislocations. This causes the loss of ductility and reduction in SSC corrosion resistance [13]. Often heat treatment such as tempering is applied after cold work to recover the SSC corrosion resistance of metals.

Effects of Alloying Element, Hardness, and Fluid Chemistry: In addition to heat treatment, different alloying elements are introduced into the formulation of metals and alloys to improve their metallurgical characteristics including SSC corrosion resistance. Chromium (Cr), Molybdenum, and Nickel are important alloying elements used for improving SSC corrosion resistance. Chromium lessens the rate of hydrogen diffusion into metals [22]. When Nickel content is less than 1%, it does not affect the SSC corrosion resistance of metals [23,24]. However, when it is more than 2%, the yield strength of metals increases, reducing their SSC corrosion resistance [25]. The addition of Molybdenum significantly enhances the SSC corrosion resistance of steels by decreasing the depassivation pH [26]. Metals with a reduced depassivation pH perform better in severe sour environments.

Hardness is one of the commonly applied criteria for selecting steels used in sour environments. Increasing hardness exacerbates the vulnerability of metals towards SSC corrosion. Even if the Rockwell hardness (HRC) limit of 22 is one of the criteria recommended by NACE, it is not a sufficient precondition to prevent SSC corrosion in sour environments. Predominantly, the SSC threshold stress reduces with hardness (Figure 2), even though there is an abnormal threshold stress trend in the hardness range of 22–24 [27]. The SSC threshold stress is a stress level below which metal vulnerability to failure due to SSC corrosion is minimal in standard corrosive solution. Some case studies [28,29] presented in the literature showed the SSC corrosion failures of soft metals that had a hardness level of less than the specified limit (22 HRC).

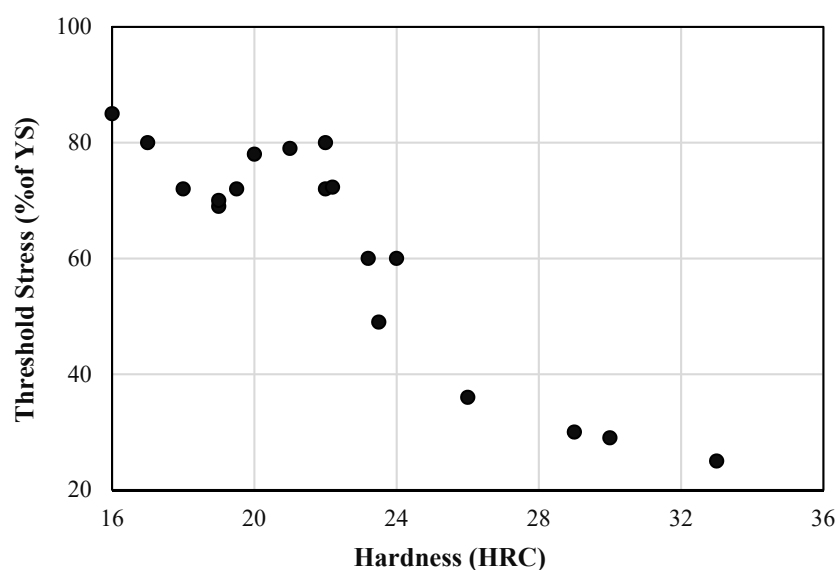


Figure 2. Threshold stress vs. HRC of carbon steel (Redrawn from Ciaraldi [27]).

In addition to the characteristics of metals, the surrounding fluid chemistry is a key factor that affects their SSC corrosion resistance. The presence of a corrosion inhibitor significantly increases the critical stress intensity factor, which represents the minimum stress value required to propagate a crack on SSC corroded metal [30]. The effect of salt content on SSC corrosion resistance of steel is often limited [31].

1.3. Mechanism of SSC Corrosion

SSC corrosion is a hydrogen embrittlement phenomenon resulting from the combined effects of stress and the diffusion of atomic hydrogen into the lattice structure of metals following corrosion by wet H_2S . The requirements for SSC corrosion include (i) tensile stress (imposed by either applied loads or residual stresses); (ii) susceptible material with a structural defect, and (iii) entry of atomic hydrogen through exposure to a corrosive environment containing hydrogen sulfide [32].

SSC corrosion occurs due to the combined effect of a corrosive environment containing hydrogen sulfide and tensile stress, which leads to the formation of cracks when its level exceeds the threshold value. The presence of H_2S promotes the penetration of hydrogen atoms into the lattice structure of the metal. Because of the stretching of the metal, the corrosion protective layer regularly breaks and detaches from the metal surface. As the stretching continues, a small molecular opening develops on the surface of the metal through which hydrogen atoms can diffuse into it. In presence of a corrosive solution, the atoms of the metal get oxidized leaving electrons on the surface of the metal. These electrons that are absorbed by hydrogen ions form free H atoms, which could convert to H_2 molecules. The following chemical reactions describe the process:



Hydrogen sulfide plays a major role in SSC corrosion by impeding the reaction represented by Equation (3). This increases the concentration of hydrogen atoms near the metal surface [33]. Subsequently, the hydrogen atoms enter the metal lattice through openings developed on the surface of the metal due to the stretching of the metal. Consequently, the presence of H_2S and tensile stress promotes the entry or permeation of hydrogen atoms into the metal lattice. The permeated atoms occupy void spaces present in the lattice structure

resulting in the embrittlement and failure of the metal. Moreover, the presence of H_2S facilitates the localized crevice corrosion caused by chloride ions [34].

2. Modeling SSC Corrosion

A new mathematical model has been developed based on an existing model [35] to predict the susceptibility of materials towards SSC corrosion under different environmental conditions. The model predicts hydrogen atom concentration in the steel matrix. The existing model has been improved to extend its range of applicability. The new model predicts H_2S concentration using modified Raoult's law which is applicable for a wide range of pressures (0 to 6.2 MPa) while the existing model predicts the solubility based on Henry's law which is valid for low pressures (less than 0.21 MPa). The new model also accounts for the impacts of salt content and solution pH on SSC corrosion.

2.1. Model Formulation

The SSC corrosion susceptibility model includes three major calculation steps: (i) determination of H_2S and CO_2 concentrations in the surrounding solution using solubility model (Appendix A), (ii) predicting of the surrounding solution pH (Appendix B), and (iii) computation of the hydrogen concentration in steel using hydrogen permeation model (Appendix C). The critical hydrogen concentration in steel is utilized as a threshold condition required for the determination of SSC susceptibility of the metal. Along with the solubility of H_2S , the solubility model predicts CO_2 solubility at a specific pressure and gas-phase composition. The model is appropriate up to 80 °C. The commonly-used API grade steels (Q-125, T-95, and C-110) are not susceptible to SSC corrosion above this temperature [11]. The model is applicable for H_2S and CO_2 partial pressures of up to 6 and 15 MPa, respectively. The details of the model are presented in Appendix A.

2.2. Numerical Procedures

The flowchart presented in Figure 3 displays the calculation steps that are performed to obtain a reliable and unique numerical solution to the SSC corrosion model. The model first (Step 1) determines the solubility of CO_2 in brine solutions at a given CO_2 partial pressure, temperature, and salt concentration applying Appendix A (Equation (A7)). Then, using the concentration of CO_2 obtained from the solubility model, the solution pH is computed as presented in Appendix B (Equation (A25)). Subsequently, the model computes the solubility of H_2S in brine at a given H_2S partial pressure, temperature, and salt concentration as presented in Appendix A (Equation (A6)). Using H_2S concentration calculated in Step 3 and pH calculated from Step 2, the model predicts the concentration of hydrogen atom inside the steel matrix using Appendix C (Equation (A28)). Using yield strength of the metal, the critical hydrogen atom concentration is determined using Equation (A28). Then, comparing the hydrogen atom content of the steel with the critical hydrogen content, the susceptibility of the material towards SSC corrosion is determined.

2.3. Validation of SSC model

The new model has been validated extensively by comparing its predictions with published measurements [11,36,37]. The measurements of Morana and Nice [11] and Skogsberg et al. [38] were obtained by performing the NACE Method A tests on API grade carbon steels (P-110 and Q-125) while varying the pH of the corrosive environment (aqueous solution) and the partial pressure of H_2S in a mixed gas, which was in equilibrium with the solution. The SSC model has been applied to forecast the vulnerability of the materials in these environments. The model predictions are in agreement with published measurements (Figure 4). Some of the specimens which passed the NACE Method A are predicted to be prone to SSC in the given environments. This is because the model provides a conservative estimate since it considers the maximum hydrogen permeation rate as a criterion for SSC failure.

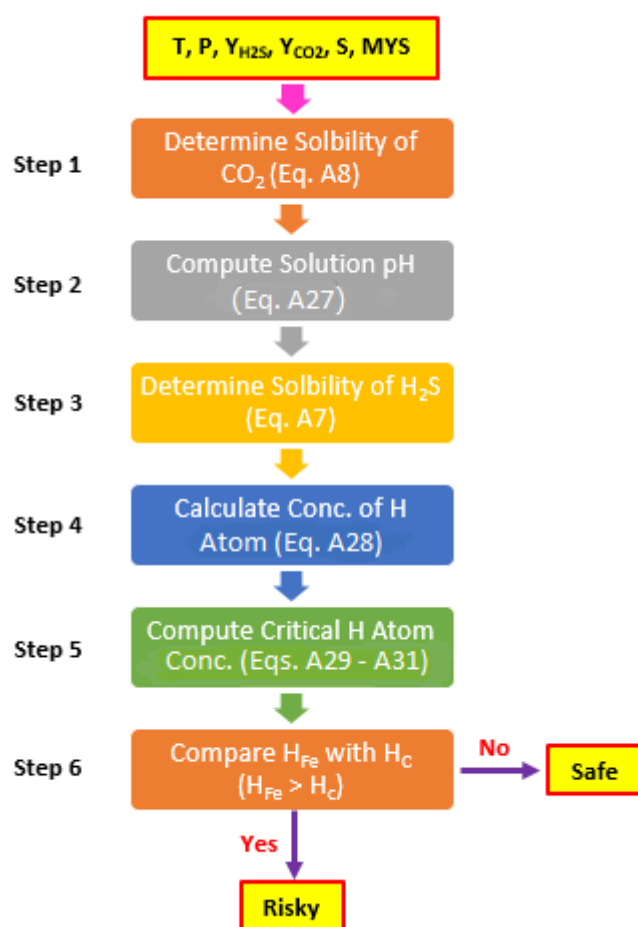


Figure 3. Numerical procedure for SSC corrosion model.

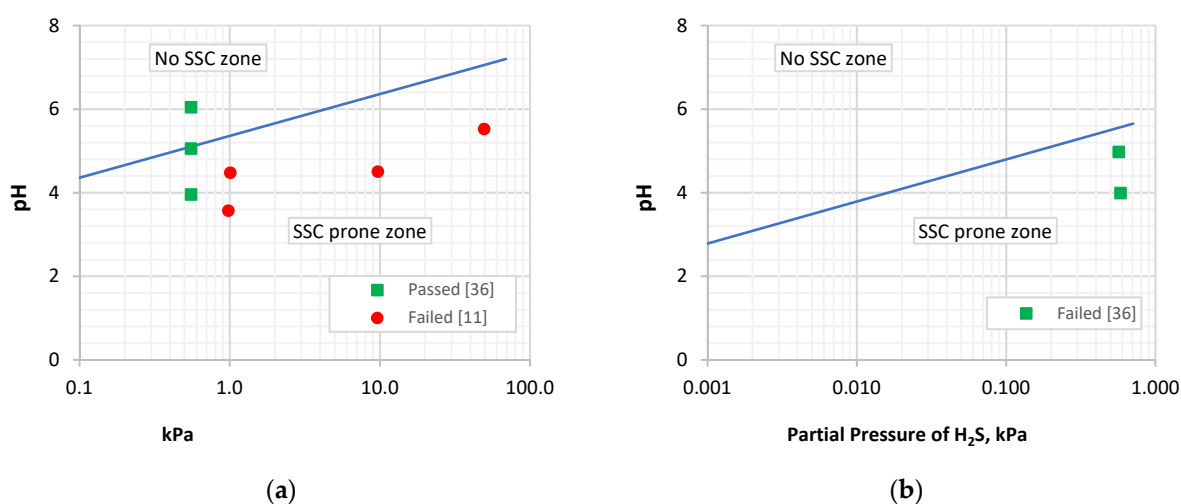


Figure 4. Model predictions and measurements for SSC failure for different API grade carbon steels: (a) P-110, and (b) Q-125.

Furthermore, the SSC corrosion model can be utilized to predict the vulnerability of metal towards SSC-related failures under various environmental conditions having different concentrations of CO_2 and H_2S . Masouri and Zafari [37] presented measurements showing the failure of API grade carbon steel (L-80) under different sour conditions, varying partial pressures of CO_2 and H_2S in the gas phase. Using the experimental conditions and

exposure time, the model is used to predict the susceptibility of the material to SSC failure. The predictions of the model show good agreement with experimental results (Figure 5).

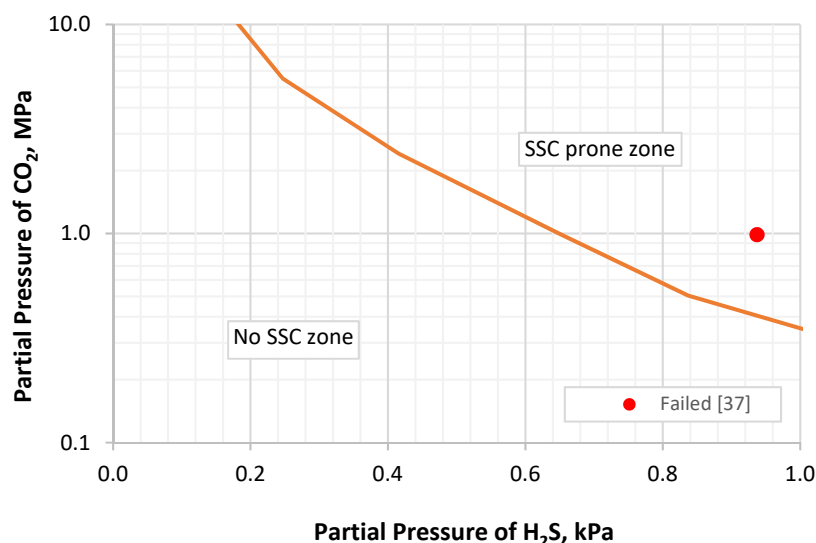


Figure 5. Model predictions and measurements for SSC failure for API L-80 carbon steel.

3. Experimental Study

In addition to modeling, an experimental study was performed to investigate SSC corrosion of API carbon steel. All experiments were conducted at a temperature of 38 °C and high pressure 41.4 MPa (inside the minipipe). Two different experimental setups (tensile testing apparatus and SSC corrosion test setup) were utilized to evaluate the embrittlement of API grade C-110 carbon steel. The Tensile Strength Testing (TST) apparatus and SSC corrosion test setup were used in sequence to evaluate the embrittlement of the material.

3.1. SSC Corrosion Evaluation Method

Minipipe specimens (Figure 6) that were cut from an API casing were used for testing SSC corrosion and the associated embrittlement of the test material. To increase accuracy and minimize machining-induced defects, water-jet cutting and milling machines were utilized in the manufacturing of the specimens. The following test procedure was used to evaluate embrittlement occurring after simultaneously exposing the specimens to the corrosive environment and constant stress load:

- Step 1. To test its fitness for the SSC experiment, the specimen was stretched three times to 80% of its elastic limit using the TST apparatus and examined for cracks and other mechanical defects.
- Step 2. The specimen was thoroughly (inside and outside surfaces) cleaned with methyl ethyl ketone and exposed to a sour environment in the SSC corrosion test setup for one week while being subjected to a stress level of 85% of its yield stress and inner part over-pressurization of 13.8 MPa.
- Step 3. The specimen was recovered from the SSC corrosion setup and strained to failure in the air using the TST apparatus to determine the mechanical properties of corroded specimen material.

Specimen failure during the corrosion test or change in mechanical properties after exposure indicates if the specimen experiences embrittlement.

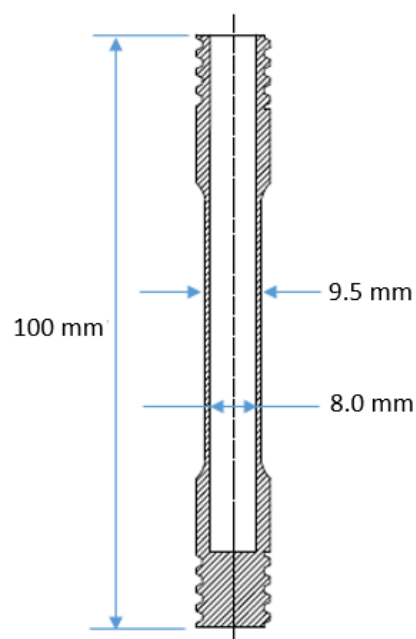


Figure 6. Minipipe specimen.

3.2. TST Apparatus

The TST apparatus (Figure 7) was used to strain specimens to failure. The device consists of: (i) structural frame made of four stud bolts and three rectangular flanges; (ii) double-acting hydraulic cylinder; (iii) two minipipe holders used at the top and bottom; (iv) syringe pump with speed controller; (v) measuring instrumentation (displacement sensor and pressure transmitter); and (vi) data acquisition card. During the experiment, a minipipe specimen was screwed to the top and bottom holders. The top holder was directly attached to a flange. The bottom holder was connected to the piston rod of the hydraulic cylinder using a coupler. The displacement sensor was used to measure the change in specimen length occurring during stretching, which is used to determine the strain. The pressure transmitter was utilized to measure oil pressure during the test. The stress developed in the minipipe during the experiment is calculated using the oil pressure data. According to the ASTM standard, the stress loading rate was maintained at 379 MPa per minute. The stress rate was accurately controlled by a computer equipped with a data acquisition and control system. The computer controlled the rate of stress applied on the minipipe by manipulating the syringe pump speed using a data acquisition system. The air injection line was used to actuate the cylinder for adjusting the piston position during the mounting of the minipipe on the setup.

3.3. SSC Corrosion Test Setup

The SSC corrosion test was conducted by simulating the loading conditions in the field. Hence, the test specimen was subjected to complex loading conditions by applying tensile load and inner part over-pressurization at the same time. Both internal and external parts of the specimen were exposed to the corrosive environment. To perform the experiments, a new test setup has been designed and built. The schematic of the setup is shown in Figure 8. The setup consists of: (i) a jacketed SSC corrosion cell; (ii) two gas chambers (GC1 and GC2) used to store 250 mL gas during the test; (iii) gas injection cylinder needed for boosting the supply gas pressure; (iv) heating system, which circulates heating medium through the jacket of the corrosion cell; (v) pneumatic cylinder to apply a constant tensile load; (vi) measurement and instrumentation system to monitor and recover test pressure and temperature; and (vii) data acquisition system.

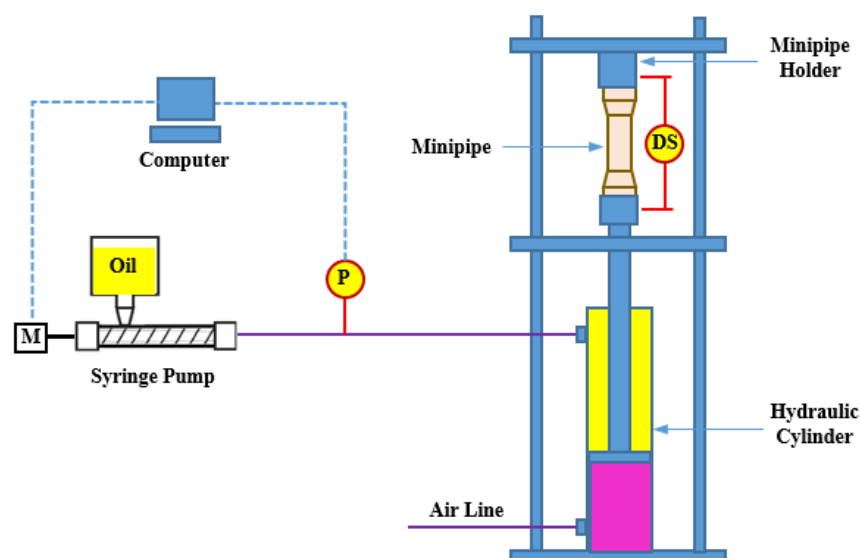


Figure 7. Schematic of TST apparatus.

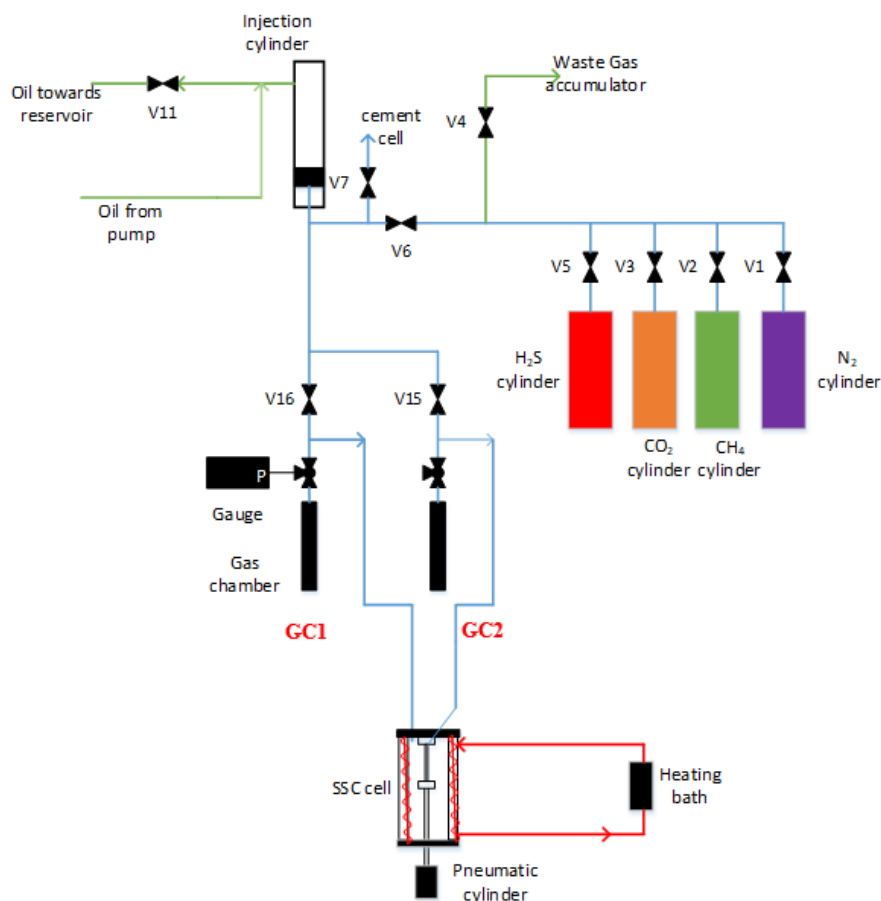


Figure 8. Schematic of SSC corrosion test setup.

During the test, the specimen was first mounted in the SSC corrosion cell. The top side of the specimen was attached to the cell lid. The bottom side of the specimen was connected to the piston rod of the pneumatic cylinder. The inner part of the specimen and the annular space between the cell and specimen were filled with brine (2% NaCl solution). The cell was sealed by tightening the lid. Gas injection lines that were connected to the inner and outer parts of the specimen were attached to the cell. Test gases (methane, carbon dioxide, and hydrogen sulfide) required for the experiment were injected into the

inner and outer parts of the minipipe simultaneously using the injection cylinder. This was performed by opening valves V15 and V16 and injecting the gas. During the injection, the gas chambers which were directly connected to the inside and outside of the minipipe were pressurized. The chambers served as gas accumulators because the volumetric capacities of the minipipe and SSC corrosion cell were insufficient to hold the gas phase in addition to the corrosive solution. The SSC corrosion tests were conducted maintaining the inside and outside of the minipipe at different pressures. The pneumatic cylinder was installed below the SSC cell to apply tensile load on the minipipe. The cylinder rod is directly connected to the bottom side of the specimen.

3.4. Corrosion Test Procedure

A specimen was first stretched three times to 80% of its elastic limit using the TST apparatus and examined for cracks and other mechanical defects. Then, the specimen was scrubbed using methyl-ethyl ketone to remove dirt materials such as oil and grease. After cleaning, the specimen was mounted on the piston rod and filled with brine (2% NaCl solution). Subsequently, the cell was filled with brine. The specimen was lowered into the cell to the appropriate position and the cell lid was assembled. Then, the cell cover was placed and bolted. The gas injection lines were connected to the cell. To deoxygenate the corrosive solution, the inner and outer parts of the specimen were purged with nitrogen at 13.8 MPa for 30 min. After purging, test gases were injected at the desired pressure into the inner and outer parts of the specimen. The inner part of the specimen was maintained at a higher pressure than its outside. During pressurization, both parts of the specimen were first pressurized simultaneously to the desired minipipe external pressure (27.6 MPa). Then, the outer part of the specimen was isolated and more gases were injected into its inner part to increase the pressure to 41.4 MPa. A differential pressure (13.8 MPa) was maintained between the outer and inner parts of the specimen to detect its failure during the test and also to simulate the actual wellbore condition with a certain level of differential pressure load. A tensile load that produces the stress level of 85% of the yield strength of the specimen material was applied using the pneumatic cylinder. The specimen was left in the cell for one week.

Materials that are vulnerable to SSC corrosion are expected to display cracks within one week of exposure [39]; therefore, the one-week time interval was selected for the experiments. Test parameters such as inner and outer specimen pressures, temperature, and pneumatic cylinder pressure were continuously monitored and recorded using the data acquisition system. Test pressures tend to slightly drop because of gas consumption due to the corrosion process and leaks occurring at the inlet connections. Hence, to maintain constant pressure, gas was injected into both sides of the specimen as needed. After seven days of exposure, the cell was depressurized and the specimen was recovered from the cell and examined for cracks.

3.5. Test Matrix

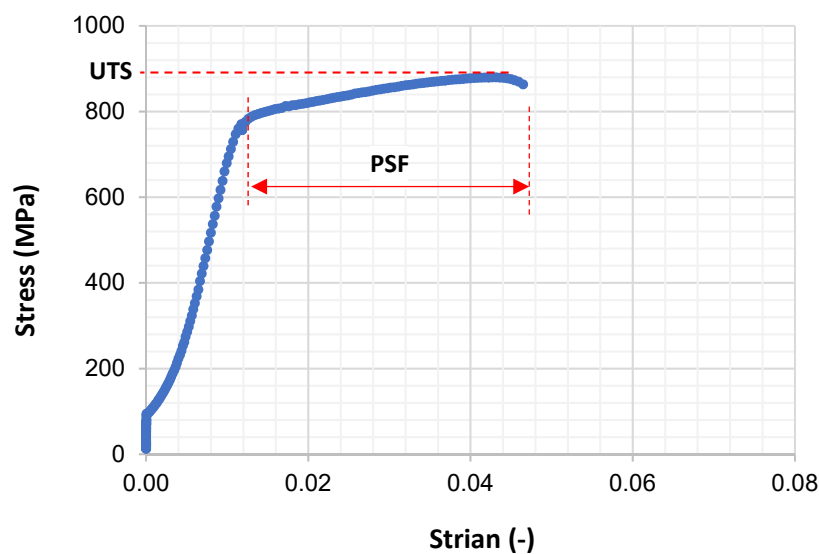
Table 1 shows the test conditions for SSC corrosion experiments. The minipipe specimen used in Test 2 was the baseline, which was not exposed to the corrosive environment. The baseline specimen was tested using the TST machine to obtain reference (uncorroded material) mechanical properties. The rest of the specimens were exposed to different sour conditions as shown in Table 1. All experiments were conducted at constant temperature (38 ± 1 °C) and H₂S concentration (280 ± 20 ppm).

Table 1. Test matrix for SSC experiment.

Test #	Test Duration (Days)	Test Label	Temp. (°C)	Total		Partial Pressure Ratio	
				Pressure, MPa			
				Inside	Outside	CH ₄ (%)	CO ₂ (%)
Test 1	15	Specimen 1	38	41.4	27.6	100	0
Test 2	0	Specimen 2	0	0	0	0	0
Test 3	7	Specimen 3, 6, 8, 10	38	41.4	27.6	89.97	10
Test 4	7	Specimen 4	38	41.4	27.6	99.97	0
Test 5	7	Specimen 5	38	41.4	27.6	94.97	5
Test 6	7	Specimen 7	38	41.4	27.6	84.97	15
Test 7	7	Specimen 9	38	41.4	27.6	97.47	2.5

3.6. TST Test Procedure

Specimens that did not fail during SSC corrosion experiments were stretched to failure using the TST apparatus. For this test, a specimen was mounted on the TST apparatus and stressed gradually at a controlled stress rate until failure. The ultimate tensile strength (UTS) and plastic strain to failure (PSF) were measured (Figure 9). PSF represents the non-elastic part of the total strain before failure [40]. The broken specimen was placed under an optical microscope for examination of the protective layer and the type of failure.

**Figure 9.** Stress vs. strain curve for Specimen 1.

4. Experimental Results and Discussions

4.1. Mechanical Properties

During the investigation, the PPR of CO₂ was varied to examine its effect on SSC susceptibility. UTS and PSF are used to assess the susceptibility of material towards SSC in sour conditions. A number of studies [41–45] demonstrated the use of PSF for the assessment of SSC. Figures 10 and 11 present the UTS and PSF of the specimens as a function of the PPR of CO₂. The UTS and PSF of the specimens showed an exponential reduction with the PPR of CO₂. The relationships are expressed as:

$$\text{UTS} = 784 + 147e^{-0.8X_c} \quad (4)$$

$$\text{PSF} = 1.14 + 3.63e^{-0.2X_c} \quad (5)$$

where x_c represents the PPR of CO₂ in percentage. The correlations are valid for the PPR of CO₂ ranging from 0 to 15%. Test 3 was conducted four times because two of the specimens (Specimens 3 and 10) were broke inside the SSC cell. The UTS and PSF reduced rapidly (exponentially) as the PPR of CO₂ was increased from 0 to 5%. As the PPR of CO₂ was continued to increase above 5%, UTS measurements were stabilized while the PSF data exhibited a gradual reduction. All specimens exposed to the corrosive environment showed a reduction in UTS loss of mechanical strength. This could be explained by considering the effects of hydrogen embrittlement and on the change in mechanical properties (ductility and ultimate tensile strength) of the material. Hydrogen embrittlement is expected to reduce both UTS and PSF [46,47]. These reductions could be because of hydrogen permeation and the associated microstructural changes.

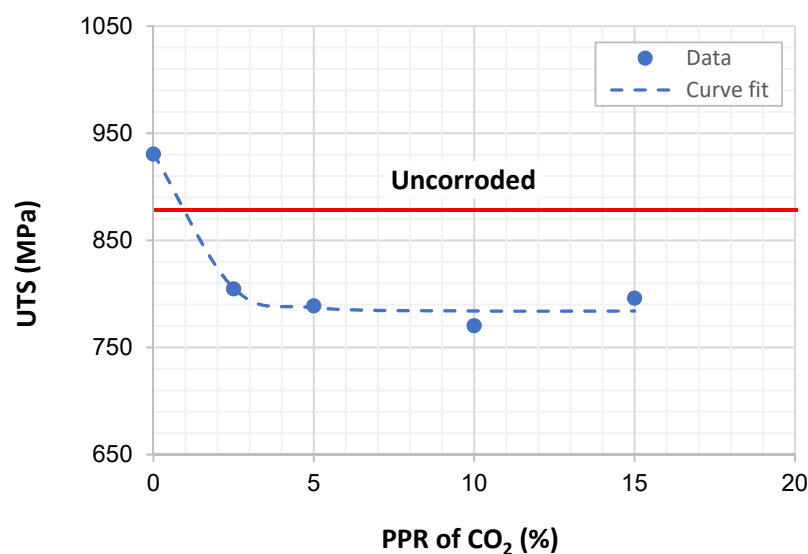


Figure 10. Measured UTS versus PPR of CO₂.

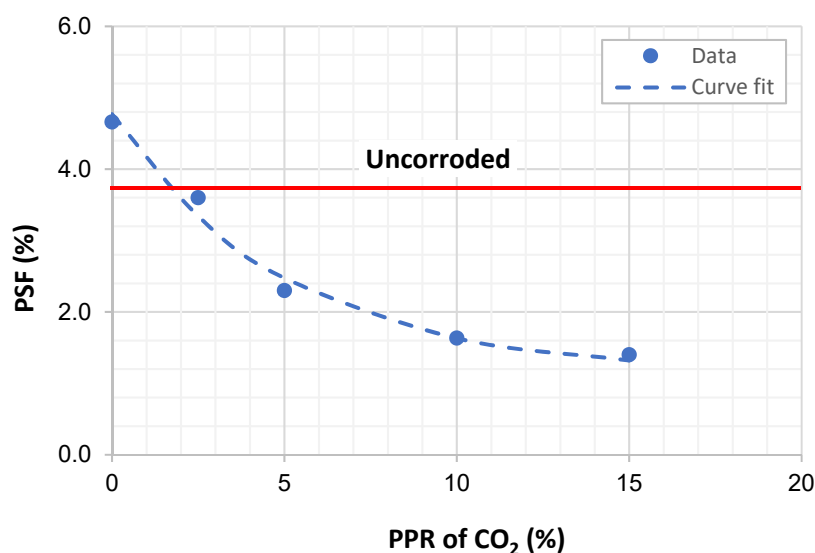


Figure 11. Measured PSF versus PPR of CO₂.

In addition to H₂S, the presence of CO₂ induces embrittlement in high-strength steels [48]. Hence, the presence of these two gases synergized the embrittlement process leading to significant reductions in UTS and PSF when small quantities of these gases (280 ppm of H₂S and 0 to 5% of CO₂) were introduced into the corrosive environment. However, when the PPR of CO₂ was increased above 5%, the reduction in UTS with PPR

vanished. The reduction of PSF with the PPR of CO₂ also diminished gradually. These observations can be explained using the hydrogen permeation model. Figure 12 shows the model predicted atomic hydrogen concentration inside the material as a function of the PPR of CO₂. Experimental data and model show a consistent trend for SSC susceptibility. At high PPRs of CO₂ (greater than 3%), the pH of the corrosive environment is expected to reduce significantly, facilitating the hydrogen permeation process and leading to a high concentration of atomic hydrogen in the metal. As a result, the UTS and PSF of the material reduced sharply with the PPR of CO₂ at low PPRs (between 0 and 5%) and exhibited minor variation at high PPRs (greater than 10%).

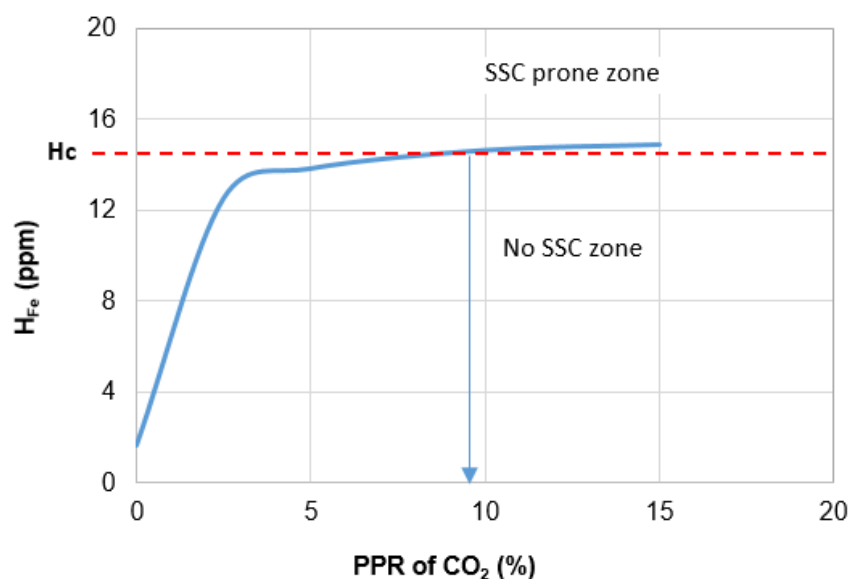


Figure 12. Predicted hydrogen permeation concentration versus PPR of CO₂.

Using the model, the critical hydrogen atom concentration (H_c) for C-110 at 38 °C is 14.7 ppm. It indicates the critical PPR of CO₂ above which the material is expected to fail due to SSC corrosion. Hence, an SSC failure is anticipated when the PPR of the corrosion environment is greater than 9.7%. Five specimens (Specimens 3, 6, 7, 8, and 10) tested above this CO₂ PPR value and 40% of them (Specimens 3 and 10) failed due to SSC. This demonstrates that the model is conservative, as indicated earlier. However, no specimen tested below 9.7% PPR of CO₂ failed due to SSC.

4.2. Analysis of Crack and Scale Characteristics

Ductile and brittle failures have different crack characteristics. Near the crack region, ductile failures display shear deformation and appear fibrous while brittle failures do not exhibit these characteristics [49,50]. Thus, examination of crack characteristics could be useful to assess the embrittlement of a specimen. The micrographs of the specimens (Figures 13 and 14) were taken using a digital microscope after they were strained to failure during tensile strength test or broken in the SSC cell when they were exposed to a corrosive environment while strained to 85% of their yield stress. The crack tips of a specimen (Figure 13a) tested for 15 days without the presence of CO₂ and H₂S showed noticeable necking and fibrous regions. Besides this, the cracks propagated with shear deformation that characterizes ductile failure. The crack tips of the uncorroded specimen (Figure 13b) exhibited similar ductile characteristics. On the other hand, a specimen tested at 10% PPR of CO₂ (Figure 13c) did not exhibit necking or shear deformation around the crack region, indicating the absence of ductile failure. The crack displayed almost no plastic deformation of the material at the crack tip confirming the presence of brittle failure.

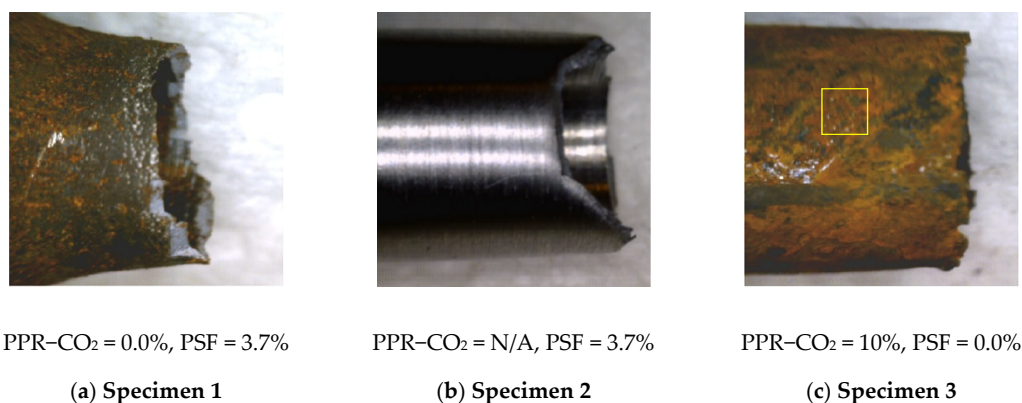


Figure 13. Crack characteristics of Specimens 1, 2, and 3.

The micrographs of Specimens 4, 5, 7, and 9 that were taken after exposure or straining to failure are presented in Figure 14. Specimens 4, 5, and 9, which were tested at low PPRs of CO_2 ($\leq 5\%$), exhibited shear deformation and fibrous regions near the crack tips. Specimen 4, which was tested at 0 PPR of CO_2 displayed the highest plastic deformation before failure. However, Specimen 7, which was corroded at 15% PPR of CO_2 , demonstrated strong brittle behavior with a low PSF value and lack of shear deformation and fibrous regions near the crack tip.

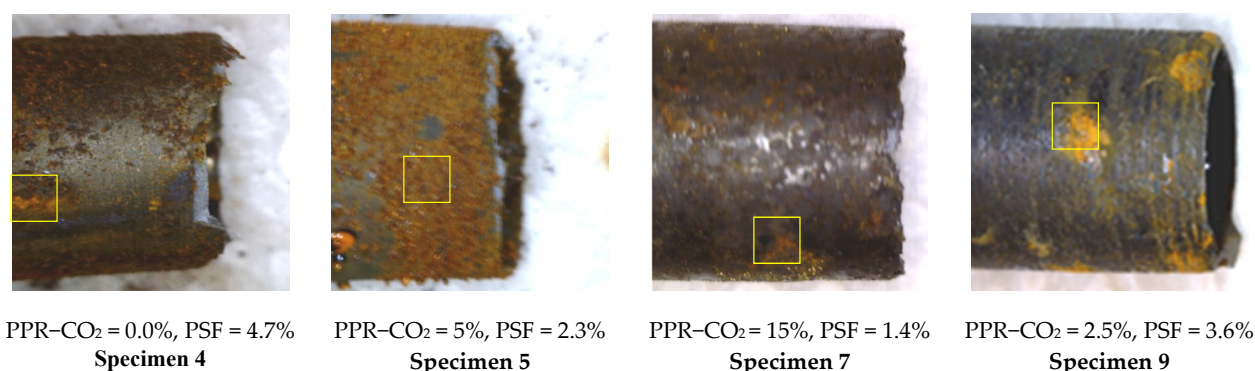


Figure 14. Crack characteristics of Specimens 4, 5, 7, and 9.

The micrographs of the corrosion scales of Specimens 3, 4, 5, 7, and 9 are presented in Figure 15. The images were obtained considering a square area ($2 \text{ mm} \times 2 \text{ mm}$) on the surface of the specimens as shown in Figures 13 and 14. Specimen 3 and 5 were mostly covered with thick flake-like and fragile corrosion scales. Scattered structures that look like fish-eyes were observed on the surface of Specimen 3. Corrosion scales formed on the surface of Specimens 4, 7, and 9 were thin and scattered.

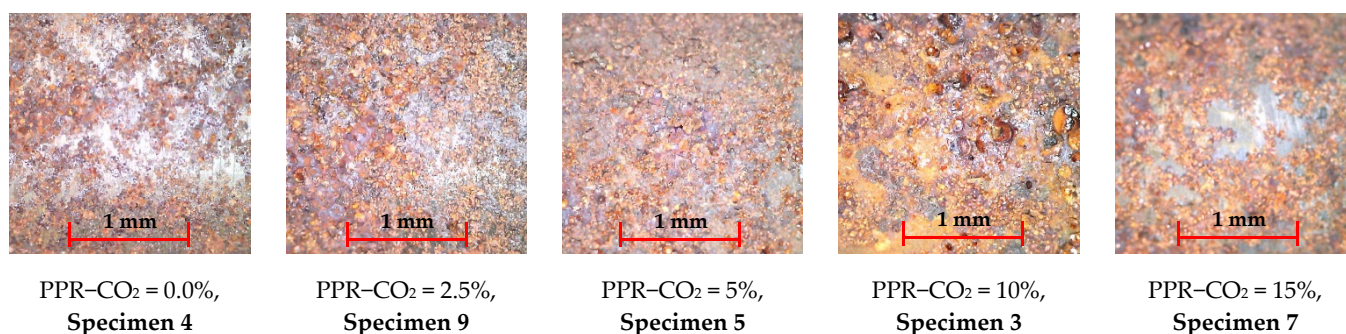


Figure 15. Micrographs of the corrosion scales of Specimens 3, 4, 5, 7, and 9.

5. Conclusions

The following conclusions can be drawn based on experimental and modeling investigations performed in this study:

- Variation in the PPR of CO₂ affected the embrittlement of steel considerably when the PPR was between 1%–5% at H₂S concentration of 280 ppm. When the PPR of CO₂ was between 10 and 15 percent, the deterioration of mechanical properties by the CO₂ content became saturated.
- The trend of hydrogen atom concentration with the PPR of CO₂ predicted by the SSC model is consistent with those of measured UTS and PSF. The existence of CO₂ decreases the pH of the surrounding solution and expedites the SSC process.
- In the absence of CO₂, the pH of the corrosion environment is expected to be close to neutral, and only limited hydrogen ions present in the solution. As a result, experiments did not show noticeable embrittlement of specimens tested at low PPRs of CO₂ in seven days of exposure.
- The SSC model predictions are mostly in agreement with existing and new measurements even though it provides a conservative forecast of failure.

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Nomenclature

a	Activity
c_{CA}	The concentration of carbonic acid, mol/lit
C_H	Hydrogen concentration, ppm
C_{H_2S}	Concentration of H ₂ S (mol/kg)
f	Fugacity
H_c	Critical hydrogen concentration (ppm)
H_{Fe}	Hydrogen concentration in steel (ppm)
K_{a1}	Dissociation constant for acid, (mol/lit)
K_{a2}	Dissociation constant for acid, (mol/lit)
K_{ISSC}	Critical Stress Intensity Factor
ksi	Kilo-pounds per square inch
K_w	Dissociation constant of water
MYS	Minimum yield strength (psi)
P	Pressure (bar)
P_{H_2O}	Vapor pressure of water (bar)
P_c	Critical Pressure (bar)

R	Universal gas constant (m ³ *bar/K/kgmol)
S	Salinity
T	Temperature (°K)
T _c	Critical temperature (°K)
V	Volume
x	Mole fraction in liquid phase
x _c	Partial pressure ratio of CO ₂ in percent
y	Mole fraction ins gas phase
Z	Compressibility factor
Greek Letters	
μ	Chemical potential
β ₁	Parameter for acentric factor
λ	Empirical parameter for calculation of activity coefficient
ε	Empirical parameter for calculation of activity coefficient
ω	Acentric factor
Φ	Fugacity coefficient
γ	Activity coefficient
Subscript	
r	Reduced parameter
c	Critical
Superscript	
v	Vapor phase
l	Liquid phase
Acynoms	
API	American Petroleum Institute
ASTM	American Society for Testing and Materials
BSEE	Bureau of Safety and Environmental Enforcement
HRC	Rockwell Hardness
GC1	Gas Chamber 1
GC2	Gas Chamber 2
MRPPR	Maximum Reduction in Partial Pressure Ratio
MRUTS	Maximum Reduction in Ultimate Tensile Strength
NACE	National Association of Corrosion Engineers
PPR	Partial Pressure Ratio
SSC	Sulfide Stress Cracking
TST	Tensile Strength Testing
UTS	Ultimate Tensile Strength
YS	Yield Stress

Appendix A. Solubility Model

The concentration of H₂S is one of the most vital parameters for predicting SSC corrosion resistance of metals. It provides the equilibrium concentration of H₂S in the liquid phase, which is in the vicinity of the metal. For the surrounding fluid system to be in equilibrium, the following conditions should be satisfied [51]: (i) uniform temperature and pressure in the system, (ii) chemical potential of each component should be the same in both phases present in the system, and (iii) the system is at its minimum Gibbs free energy level. The first condition denotes the thermal and chemical equilibrium criteria. The second condition can be described using fugacity of non-ideal gases as: $f_i^l = f_i^v$, where f is fugacity and i represents the component "i". For ideal gas-liquid mixtures, Raoult's law or Henry's law could be used to predict the solubility of corrosive gases at low pressures. However, these laws provide unrealistic predictions at high pressures (greater than 30 psi). Modified versions of these laws are utilized to predict gas solubility at high pressures. The modified Raoult's law [52] is more accurate than modified Henry's law [53] because it accounts for variation in chemical potential. Under equilibrium conditions, the chemical potential (μ) of

each component should be the same in both phases. Thus: $\mu_i^l = \mu_i^v$. The chemical potential of hydrogen sulfide in liquid can be expressed as:

$$\mu_{H_2S}^l(T, P, x) = \mu_{H_2S}^{lstd}(T, P) + RT \ln a_{H_2S}(T, P, x) \quad (A1)$$

where a_{H_2S} is the activity of H_2S and $\mu_{H_2S}^{lstd}$ is the standard chemical potential of H_2S in the liquid phase. x_{H_2S} is the mole fraction of H_2S in the liquid phase. The activity of H_2S can be expressed as:

$$a_{H_2S} = x_{H_2S} * \gamma_{H_2S} \quad (A2)$$

where γ_{H_2S} and a_{H_2S} are the activity coefficient and activity of H_2S , respectively. Combining Equations (A1) and (A2), the chemical potentials of H_2S in the liquid and gas phases can be expressed as:

$$\mu_{H_2S}^l(T, P, x) = \mu_{H_2S}^{lstd}(T, P) + RT \ln x_{H_2S}(T, P, x) + RT \ln \gamma_{H_2S}(T, P, x). \quad (A3)$$

$$\mu_{H_2S}^v(T, P, y) = \mu_{H_2S}^{vstd}(T, P) + RT \ln f_{H_2S}(T, P, y). \quad (A4)$$

where $\mu_{H_2S}^{vstd}$ and $\mu_{H_2S}^{lstd}$ are the standard chemical potentials of H_2S in gas and liquid phases. f_{H_2S} is the fugacity of H_2S , which is defined as:

$$f_{H_2S} = P * y_{H_2S} * \Phi_{H_2S} \quad (A5)$$

where Φ_{H_2S} is the fugacity coefficient of H_2S .

Combining Equations (A4) and (A5), the following expression for the chemical potential of H_2S in the gas phase can be determined as:

$$\mu_{H_2S}^v(T, P, y) = \mu_{H_2S}^{vstd}(T, P) + RT \ln P * y_{H_2S} + RT \ln \Phi_{H_2S}(T, P, y) \quad (A6)$$

The following formula for determining the solubility of H_2S (x_{H_2S}) in brine can be obtained by equating the liquid and gas phase chemical potentials of H_2S and using the Equations from (A1) to (A6).

$$\ln \left(P * \frac{y_{H_2S}}{x_{H_2S}} \right) = \frac{\left\{ \mu_{H_2S}^{lstd}(T, P) - \mu_{H_2S}^{vstd}(T, P) \right\}}{RT} - \ln \Phi_{H_2S}(T, P, y) + \ln \gamma_{H_2S}(T, P, x) \quad (A7)$$

Applying a similar procedure, an equation for determining the solubility of CO_2 (x_{CO_2}) in brine can be established:

$$\ln \left(P * \frac{y_{CO_2}}{x_{CO_2}} \right) = \frac{\left\{ \mu_{CO_2}^{lstd}(T, P) - \mu_{CO_2}^{vstd}(T, P) \right\}}{RT} - \ln \Phi_{CO_2}(T, P, y) + \ln \gamma_{CO_2}(T, P, x) \quad (A8)$$

Equations (A7) and (A8) can be used to predict the solubility of H_2S or CO_2 at specific pressure and temperature, if the chemical potentials, fugacity coefficient, and activity coefficient are determined using appropriate models presented in Tale [54].

Appendix B. pH Model

In an aqueous solution saturated with mixed gas containing H_2S and CO_2 , pH reduction is dominated more by CO_2 than H_2S (15). Hence, the carbon dioxide solubility model can be applied to predict the pH of aqueous solutions saturated with mixed gas containing H_2S and CO_2 . The equation describing the dissolution of carbon dioxide in water is presented as:



Then, the dissolved gas reacts with water to form carbonic acid.



The first carbonic acid dissociation occurs forming bicarbonate and hydrogen ions.



The second dissociation produces carbonate and additional hydrogen ions.



The primary and secondary dissociation constants for carbonic acid in Equations (A11) and (A12) are expressed as:

$$K_{a1} = \frac{[H^+] * [HCO_3^{-1}]}{[H_2CO_3]} \quad (A13)$$

$$K_{a2} = \frac{[H^+] * [CO_3^{-2}]}{[HCO_3^{-1}]} \quad (A14)$$

Applying the carbonate balance, the initial concentration of carbonic acid in the solution can be expressed as:

$$c_{CA} = [H_2CO_3] + [HCO_3^{-1}] + [CO_3^{-2}] \quad (A15)$$

Combining Equations (A13)–(A15), the initial concentration of carbonic acid is expressed as a function of dissociation constants, carbonate, and hydrogen ion concentrations.

$$c_{CA} = \frac{[H^+]^2 * [CO_3^{-2}]}{K_{a1} * K_{a2}} + \frac{[H^+] * [CO_3^{-2}]}{K_{a2}} + [CO_3^{-2}] \quad (A16)$$

Equation (A16) can be rearranged to express the concentration of carbonate ion:

$$[CO_3^{-2}] = \frac{K_{a1} * K_{a2} * c}{[H^+]^2 + K_{a1} * [H^+] + K_{a1} * K_{a2}} \quad (A17)$$

Similarly, the concentration of bicarbonate ion can be given by:

$$[HCO_3^{-1}] = \frac{K_{a1} * [H^+] * c}{[H^+]^2 + K_{a1} * [H^+] + K_{a1} * K_{a2}} \quad (A18)$$

Applying the concept of electroneutrality (charge balance), the concentrations of hydrogen ions can be determined as:

$$[H^+] = 2[CO_3^{-2}] + [HCO_3^{-1}] + [OH^-] \quad (A19)$$

The following equation for hydrogen ion can be obtained after combining Equations from (A17) to (A19) and rearranging:

$$[H^+] = c_{CA} * \frac{2 * K_{a1} * K_{a2} + K_{a1} * [H^+]}{[H^+]^2 + K_{a1} * [H^+] + K_{a1} * K_{a2}} + [OH^-] \quad (A20)$$

The dissociation constant of water is expressed as: $K_w = [OH^-] * [H^+]$. Inserting this relation into Equation (A20) yields:

$$[H^+] = c_{CA} * \frac{2 * K_{a1} * K_{a2} + K_{a1} * [H^+]}{[H^+]^2 + K_{a1} * [H^+] + K_{a1} * K_{a2}} + \frac{K_w}{[H^+]} \quad (A21)$$

Since the second dissociation constant of carbonic acid is considerably lower than the first dissociation constant, $K_{a2} * [H^+]$ can be inserted on the numerator and denominator side in Equation (A21).

$$[H^+] = c_{CA} * \frac{2 * K_{a1} * K_{a2} + K_{a1} * [H^+] + K_{a2} * [H^+]}{[H^+]^2 + K_{a1} * [H^+] + K_{a1} * K_{a2} + K_{a2} * [H^+]} + \frac{K_w}{[H^+]} \quad (A22)$$

The following expression for hydrogen concentration can be obtained after rearranging Equation (A22):

$$[H^+] = c_{CA} * \left\{ \frac{K_{a1}}{K_{a1} + [H^+]} + \frac{K_{a2}}{K_{a2} + [H^+]} \right\} + \frac{K_w}{[H^+]} \quad (A23)$$

Hydrogen concentration is considerably higher than the first dissociation constant at low solution pH and low temperatures (pH less than 4 and less than 150 °F). Hence Equation (A23) can be simplified as:

$$[H^+] = [K_{a1} * c_{CA} + K_{a2} * c_{CA} + K_w]^{0.5}. \quad (A24)$$

The first and the second dissociation constants of saline water are determined using the following equations [55]:

$$pK_{a1} = -\log(K_{a1}) = \frac{3670.7}{T} - 62.008 + 9.7944 \ln T - 0.0118 * S + 0.000116S^2 \quad (A25)$$

$$pK_{a2} = -\log(K_{a2}) = \frac{1394.7}{T} + 4.777 - 0.0184 * S + 0.000118S^2 \quad (A26)$$

where S is the salinity of water in %, T is temperature. After determining the concentration of hydrogen ions, solution pH is computed as:

$$pH = -\log[H^+] \quad (A27)$$

Appendix C. Hydrogen Permeation Model

The hydrogen permeation model predicts the concentration of hydrogen atoms in the steel matrix for a specific environmental condition with known solution pH and H_2S concentration. Besides, it compares the hydrogen atom concentration in the steel matrix with a predetermined critical/threshold hydrogen atom concentration value to determine the susceptibility of the metal to SSC corrosion in a specific environment. The following empirical equation, developed by Asahi et al. [35], is used to determine hydrogen atom concentration (H_{Fe}) in the steel matrix.

$$H_{Fe} = 13 * (H^+ * C_{H_2S})^{0.26} \quad (A28)$$

where H^+ and C_{H_2S} are the concentrations of hydrogen ions and H_2S in the solution. The empirical coefficient (13) in Equation (A28) depends on the steel type. The effect of temperature on the predictions of Equation (A28) is minimal.

The critical hydrogen atom concentration (H_c) represents the concentration of hydrogen atoms in the steel matrix below which the metal does not fail due to SSC corrosion at a stress level less than or equal to its yield strength. The following equations are developed to determine the critical hydrogen concentration (ppm) at different temperatures for carbon steels:

$$\log H_c = -4.05 * 10^{-3} * MYS + 4.01 \text{ at } 25^\circ C \quad (A29)$$

$$\log H_c = -3.45 * 10^{-3} * MYS + 3.89 \text{ at } 50^\circ C \quad (A30)$$

$$\log H_c = -3.14 * 10^{-3} * MYS + 3.91 \text{ at } 80^\circ C \quad (A31)$$

where MYS is the maximum yield strength in MPa.

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