

Experimental study on stress corrosion cracking behavior of high-strength steel in acidic marine environment

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ABSTRACT

This study investigates the stress corrosion cracking (SCC) behavior of Nb–Ti microalloyed API 5L X70 high-strength low-alloy (HSLA) steel—widely deployed in marine flexible risers and subsea pipelines—under a simulated acidic sour marine environment (pH 3, 3.5 wt.% NaCl, H₂S saturation). While prior research has examined hydrogen embrittlement (HE) or anodic dissolution (AD) individually in high-strength steels, this work uniquely integrates slow strain rate tensile (SSRT) testing with in-situ potentiodynamic polarization, electrochemical impedance spectroscopy (EIS), and high-resolution fractography to concurrently assess mechanical degradation, electrochemical behavior, and fracture morphology. The results demonstrate pronounced SCC susceptibility, with a substantial reduction in ductility and time to failure in the corrosive environment compared to air. Electrochemical data revealed high corrosion rates, active dissolution, and a low-frequency inductive loop characteristic of hydrogen-related interfacial processes. Fractography confirmed predominantly brittle fracture modes, including quasi-cleavage and transgranular cracking, which directly correlate with the electrochemical signatures. This concurrent analysis provides new mechanistic insight into the synergistic operation of HE—strongly promoted by low pH and H₂S—and AD in Nb–Ti microalloyed steels, offering critical guidance for material selection and SCC mitigation strategies in aggressive marine service.

Keywords: High-strength steel; Stress corrosion cracking; Acidic marine environment; Hydrogen embrittlement; API 5L X70.

1. INTRODUCTION

The utilization of high-strength steels (HSS) in marine engineering applications has seen a substantial increase in recent decades [1]. This trend is driven by the demand for materials that offer enhanced structural efficiency and economic benefits, particularly in the construction of offshore oil and gas platforms, subsea pipelines, naval vessels, and other critical maritime infrastructures. The superior strength-to-weight ratio of HSS allows for the design of lighter structures, which can lead to reduced material consumption, lower transportation and installation costs, and improved load-bearing capacities. Among the various grades of HSS, API 5L X70 steel, a high-strength low-alloy (HSLA) steel, is extensively employed for line pipe applications, especially for the transportation of oil and gas over long distances and in challenging terrains [2]. The economic and operational imperatives pushing for exploration in deeper offshore fields and the construction of higher-pressure pipelines further necessitate the use of materials like X70. However, these demanding service conditions frequently expose the steels to severely corrosive environments, making their long-term integrity and resistance to specific failure modes, such as stress corrosion cracking (SCC), a paramount concern for ensuring operational safety and reliability [3].

SCC is recognized as a particularly insidious and often catastrophic failure mechanism that can affect normally ductile metallic materials [4]. It results from the complex and synergistic interaction of three critical factors: a sustained or fluctuating tensile stress (which can be either applied or residual), a material that is susceptible to SCC, and a specific, often seemingly mild, corrosive environment [5]. Failures due to SCC are frequently characterized by their brittle nature and can occur without significant prior warning, posing substantial risks to structural integrity, personnel safety, and environmental protection.

Acidic marine environments represent a particularly challenging scenario for HSS. These environments are typically characterized by a low pH, a high concentration of chloride ions (Cl^-) inherent to seawater, and, critically for many industrial applications, the potential presence of dissolved hydrogen sulfide (H_2S) [6]. H_2S in marine settings can originate from various sources, including the metabolic activity of sulfate-reducing bacteria (SRB) in anoxic sediments or within biofilms on the steel surface, contamination of seawater by industrial effluents, or directly from the hydrocarbon products being transported. When H_2S is present, the SCC phenomenon is often referred to as sulfide stress cracking (SSC), which is a particularly aggressive form of hydrogen-induced cracking [7, 8]. The combination of these aggressive species creates a potent environment for material degradation. Low pH conditions accelerate general corrosion processes and, more importantly, significantly increase the rate of cathodic hydrogen evolution. Chloride ions are well-known for their ability to break down protective passive films that might otherwise form on the steel surface, leading to localized corrosion such as pitting, which can act as SCC initiation sites [9, 10]. Hydrogen sulfide plays a uniquely detrimental role by not only contributing to the acidity and reacting to form sulfide films but also by acting as a “poison” for the hydrogen recombination reaction ($2\text{H}_{\text{ads}} \rightarrow \text{H}_2$) on the steel surface. This poisoning effect dramatically increases the surface concentration and chemical potential of adsorbed atomic hydrogen (H_{ads}), thereby promoting its absorption into the steel matrix and significantly increasing the susceptibility to hydrogen embrittlement (HE) [11, 12]. Thus, the acidic, H_2S -containing marine environment presents a formidable challenge, creating what can be described as a “perfect storm” for the initiation and propagation of SCC in susceptible HSS.

The mechanisms responsible for SCC in HSS, particularly in complex environments like acidic marine settings, are multifaceted and often involve the interplay of electrochemical and mechanical processes [13]. Two primary mechanisms are widely recognized: anodic dissolution (AD) and HE. AD mechanisms propose that crack propagation occurs due to the localized and preferential dissolution of metal at the crack tip [14]. This process can be initiated at sites of passive film breakdown, such as pits, crevices, or emergent slip steps. A common model is the film rupture-repassivation mechanism, where the protective passive film at the highly stressed crack tip is mechanically ruptured by plastic strain, exposing the bare metal underneath to the corrosive environment [15]. This exposed metal undergoes rapid anodic dissolution before a new passive film can reform. Continuous repetition of this cycle leads to crack advance. Alternatively, SCC can proceed along a pre-existing active path within the material, such as sensitized grain boundaries, specific crystallographic planes, or interfaces between different microstructural phases that are electrochemically less noble than the bulk matrix [16].

HE is another critical mechanism, especially for HSS in environments that promote hydrogen generation and absorption [17]. Atomic hydrogen is produced as a byproduct of cathodic corrosion reactions, such as the reduction of H^+ ions in acidic solutions or water reduction. In the presence of H_2S , the entry of this atomic hydrogen into the steel is significantly facilitated [18]. Once absorbed, hydrogen atoms, being small and mobile, can diffuse through the steel lattice, particularly along grain boundaries or dislocations. They tend to accumulate in regions of high triaxial stress, such as the plastic zone ahead of a crack tip, or at microstructural trapping sites like inclusions, vacancies, or interfaces [19]. The accumulation of hydrogen in these critical regions can lead to a local degradation of the material’s mechanical properties. Several theories describe how hydrogen causes embrittlement, including the hydrogen-enhanced decohesion (HEDE) model, which posits that hydrogen reduces the cohesive strength of the atomic bonds at the crack tip or along specific interfaces, and the hydrogen-enhanced localized plasticity (HELP) model, which suggests that hydrogen enhances dislocation mobility and promotes localized plastic deformation, leading to premature failure [20]. Regardless of the precise sub-mechanism, HE results in a reduction in ductility, fracture toughness, and an increased propensity for brittle-like fracture [21].

In many practical SCC scenarios, particularly in aggressive environments like those containing H_2S , AD and HE do not operate in isolation but rather act synergistically to accelerate crack initiation and propagation [22]. For instance, localized anodic dissolution can create sharp pits or crevices that act as potent stress concentrators [23]. These geometric features not only intensify the local stress state but can also create occluded chemical conditions (e.g., lower pH, higher Cl^- concentration) that further promote hydrogen evolution and absorption [24]. The sharp crack formed by AD then becomes a prime site for hydrogen accumulation and subsequent HE-driven crack extension. Conversely, hydrogen accumulation can influence local electrochemical potentials and dissolution kinetics [25]. This dynamic interplay means that the overall SCC rate can be significantly faster than if either AD or HE were acting alone. The specific environmental conditions (e.g., pH, H_2S partial pressure, potential) and the material’s microstructure and strength level dictate the relative dominance and interaction of these mechanisms. Understanding this synergy is crucial for accurately predicting SCC behavior and developing effective mitigation strategies.

The susceptibility of HSS to SCC is profoundly influenced by a range of environmental and metallurgical parameters, whose effects are often interdependent. Low pH values in the environment directly increase the concentration of H^+ , which are the primary reactants in the cathodic hydrogen evolution reaction. This accelerates the rate of hydrogen generation on the steel surface [26]. Furthermore, acidic conditions can destabilize or prevent the formation of protective passive films, leading to increased general corrosion rates and providing more sites for hydrogen absorption [27].

The concentration of H_2S is a critical factor in sour environments. As previously mentioned, H_2S acts as a potent hydrogen recombination poison, dramatically increasing the amount of atomic hydrogen entering the steel. Additionally, H_2S reacts with iron to form iron sulfide (FeS_x) scales on the steel surface [28]. The properties of these sulfide scales are complex and depend on factors like H_2S concentration, pH, temperature, and exposure time. While some sulfide films might offer a degree of barrier protection, others can be brittle and prone to cracking, or they can create occluded cells that alter the local electrochemical conditions, potentially exacerbating SCC [29].

Cl^- , ubiquitous in marine environments, are well-known for their ability to locally break down passive films on steels, leading to pitting corrosion. These pits can act as significant stress concentrators and preferred sites for the initiation of SCC cracks. Temperature influences virtually all aspects of SCC, including the kinetics of electrochemical reactions (both anodic dissolution and cathodic hydrogen evolution), the diffusion rates of species in solution and of hydrogen within the steel, the solubility of gases like H_2S , and the stability of surface films. The temperature dependence of SCC susceptibility is often non-monotonic, with maximum susceptibility sometimes observed at intermediate temperatures due to a complex balance of these competing factors [30]. From a metallurgical perspective, the strength level of the steel is a key determinant. Generally, higher strength steels exhibit greater susceptibility to HE, as higher stress levels can be sustained, and the microstructures that confer high strength (e.g., martensitic or heavily dislocated structures) may offer more sites for hydrogen trapping or facilitate hydrogen-assisted cracking [31]. Microstructural features such as grain size, the type and distribution of phases (e.g., ferrite, pearlite, bainite, retained austenite), the presence, morphology, and distribution of non-metallic inclusions, and crystallographic texture can all play significant roles.

API 5L X70 steel is a widely recognized and extensively used grade of HSLA steel, primarily specified for the manufacture of line pipes for the transportation of oil and natural gas [32]. Its designation “X70” indicates a specified minimum yield strength (SMYS) of 70,000 psi (approximately 485 MPa) [33]. This steel achieves its favorable combination of high strength, good toughness, and adequate weldability through controlled rolling and microalloying with elements such as niobium, vanadium, and titanium. These elements promote grain refinement and precipitation hardening [34, 35]. The typical microstructure of X70 steel consists of fine-grained ferrite and pearlite, or in some modern thermomechanically controlled processed (TMCP) variants, acicular ferrite or bainitic structures, which contribute to its enhanced mechanical properties. Given its widespread deployment in pipelines that may traverse or operate in marine environments, or carry fluids containing H_2S (sour service), understanding the SCC behavior of API 5L X70 steel under such aggressive conditions is of considerable practical and scientific importance. While the general susceptibility of API 5L X70 steel to SCC in sour environments is well-established, a detailed and integrated understanding of the competing and synergistic failure mechanisms under the combined, highly aggressive conditions of low pH, H_2S saturation, and dynamic loading remains an area of active investigation. Previous studies on microalloyed pipeline steels have often examined the role of Nb or Ti individually—typically linking Nb to grain refinement and Ti to precipitation strengthening or inclusion control—and have reported beneficial effects on hydrogen trapping and resistance to hydrogen-induced damage under varied processing conditions. However, these works are frequently limited by differing steel chemistries, thermomechanical histories, and testing environments, making it difficult to isolate and compare the influence of each element under identical metallurgical and environmental conditions. Systematic research on steels containing both Nb and Ti, subjected to the same controlled rolling and cooling treatments, and tested under the same severe sour marine environment, is scarce. In particular, the combined influence of Nb–Ti microalloying on SCC susceptibility—through their concurrent effects on grain refinement, precipitation of nanoscale carbides, and irreversible hydrogen trapping—has not been explicitly quantified in relation to the simultaneous action of anodic dissolution and hydrogen embrittlement. This study addresses this gap by employing a single heat of Nb–Ti microalloyed API 5L X70 steel, processed under industrially relevant TMCP conditions, and conducting a concurrent mechanical–electrochemical–fractographic evaluation to elucidate how these combined microalloying effects govern SCC behavior in an acidic H_2S -saturated marine environment. By doing so, we seek to provide a clearer and more integrated mechanistic model that explains the synergistic roles of AD and HE in the rapid failure of API 5L X70 steel in a simulated acidic sour marine environment.

The primary objective of the present research is to conduct a comprehensive experimental investigation into the stress corrosion cracking behavior of API 5L X70 high-strength steel when subjected to a simulated acidic marine environment containing dissolved hydrogen sulfide.

To achieve this primary objective, the following secondary objectives were defined:

1. To quantitatively evaluate the SCC susceptibility of API 5L X70 steel using slow strain rate tensile (SSRT) tests under the specified environmental conditions, comparing its performance to that in an inert (air) environment.
2. To characterize the electrochemical corrosion response of the steel in the simulated aggressive environment through potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) measurements, to understand the corrosion kinetics and interfacial processes.
3. To perform detailed fractographic analysis of the fractured SSRT specimens and microstructural examination of crack propagation paths to identify the predominant failure modes, crack initiation sites, and the interaction of cracks with the material's microstructure.
4. To elucidate the synergistic SCC mechanism by directly correlating the electrochemical data (indicative of AD and hydrogen absorption) with the specific brittle fracture features (indicative of HE), thereby clarifying the interplay that governs the degradation of API 5L X70 steel in this specific acidic sour marine environment.

2. MATERIALS AND METHODS

2.1. Material and initial characterization

The material selected for this investigation was API 5L X70 grade seamless pipeline steel, obtained commercially as a section from a pipe with an outer diameter of 610 mm and a wall thickness of 12.7 mm. All tests were conducted on the material in its as-received condition, without any subsequent laboratory heat treatment.

The elemental chemical composition of the supplied API 5L X70 steel was determined using spark optical emission spectrometry. The average composition from multiple measurements is presented in Table 1. The composition is typical for this grade, with low carbon content and microalloying additions for strength and grain refinement.

The baseline mechanical properties of the as-received X70 steel were determined by conducting standard tensile tests in laboratory air at room temperature (25 ± 2 °C) using a universal testing machine, in accordance with ASTM E8/E8M standard procedures. Cylindrical tensile specimens with a gauge diameter of 6 mm and a gauge length of 30 mm were used. Hardness measurements were performed using a Vickers hardness tester with a 10 kgf load (HV10). The average mechanical properties are summarized in Table 2. These properties confirm that the material meets the requirements for API 5L X70 grade.

For initial microstructural characterization, specimens were sectioned from the pipe, mounted in bakelite, and prepared using standard metallographic techniques. This involved grinding with silicon carbide (SiC)

Table 1: Chemical composition of API 5L X70 steel (wt.%).

ELEMENT	C	Mn	Si	P	S	Nb	V	Ti	Al	Fe
wt. %	0.08	1.65	0.30	0.015	0.004	0.042	0.051	0.018	0.033	Balance

Table 2: Mechanical properties of as-received API 5L X70 steel (in air at 25 °C).

PROPERTY	SYMBOL	VALUE
Yield strength (0.2 % offset)	σ_{YS}	492 MPa
Ultimate tensile strength	σ_{UTS}	575 MPa
Total elongation	ϵ_{total}	26.5%
Reduction in area	RA	68.0%
Vickers hardness	HV ₁₀	195

papers up to 1200 grit, followed by polishing with diamond paste suspensions down to 1 μm , and finally etching with a 2% Nital solution (2 mL nitric acid in 98 mL ethanol). The etched microstructures were examined using an optical microscope (OM) and a scanning electron microscope (SEM).

2.2. Specimen preparation for SCC tests

Cylindrical tensile specimens for the SSRT tests were machined from the X70 steel pipe with their tensile axis oriented parallel to the longitudinal direction of the pipe. The specimen geometry was designed according to NACE TM-198 29, featuring a gauge length of 25 mm and a gauge diameter of 4 mm. After machining, the gauge sections of the specimens were carefully ground longitudinally using SiC papers up to 1200 grit to achieve a consistent surface finish and remove any circumferential machining marks that could act as stress concentrators. Prior to each test, the specimens were ultrasonically cleaned in acetone for 10 minutes, rinsed thoroughly with deionized water, and then dried in a stream of warm air.

2.3. Test environment simulation

The simulated acidic marine environment was prepared as follows:

A 3.5 wt.% NaCl solution was made by dissolving analytical grade sodium chloride in deionized water (resistivity $> 18 \text{ M}\Omega\cdot\text{cm}$). This solution was then acidified to a target pH of 3.0 ± 0.1 using analytical grade hydrochloric acid (HCl). The pH of the solution was monitored and adjusted as necessary using a calibrated glass electrode pH meter before and during the tests.

To create sour conditions, the acidified NaCl solution was purged with a certified gas mixture of 5% H_2S balanced with N_2 (by volume) at a flow rate of approximately 150 mL/min. Prior to introducing the $\text{H}_2\text{S}/\text{N}_2$ mixture, the solution was deaerated for at least 2 hours with high-purity N_2 gas to establish an anaerobic environment. The $\text{H}_2\text{S}/\text{N}_2$ purging then commenced at least 1 hour before specimen immersion and was maintained continuously throughout the entire duration of each test. This standard and continuous purging procedure is designed to ensure that the test solution remains saturated with H_2S with respect to its partial pressure in the gas phase, in alignment with practices outlined in NACE TM0177/ISO 15156-2. Verification of environmental stability was achieved by monitoring the open circuit potential (OCP) until a steady state was reached prior to testing. While direct online measurement of the dissolved H_2S concentration is challenging, the theoretical concentration can be calculated using Henry's Law. For a gas phase containing 5% H_2S at a total pressure of 1 atm, the partial pressure of H_2S is 0.05 atm. The concentration of dissolved molecular H_2S ($[\text{H}_2\text{S}]_{\text{aq}}$) at 25 $^\circ\text{C}$ in a 3.5% NaCl solution is estimated to be approximately 4.9 mmol/L (or $\sim 167 \text{ ppm}$) [36]. Given the acidic condition (pH 3), the dissociation of H_2S into HS^- is negligible, thus the total dissolved sulfide concentration is equivalent to the $[\text{H}_2\text{S}]_{\text{aq}}$ value. This concentration represents a severely aggressive sour environment, known to promote high hydrogen uptake and SCC susceptibility.

All experiments were conducted at a constant ambient temperature of $25 \pm 2 \text{ }^\circ\text{C}$, which was monitored throughout the tests. Prior to $\text{H}_2\text{S}/\text{N}_2$ purging, the solution was deaerated by bubbling high-purity N_2 gas for at least 2 hours to minimize the concentration of dissolved oxygen, as H_2S -containing environments relevant to SCC are typically anaerobic.

2.4. SSRT tests

SSRT tests were performed using a computer-controlled, screw-driven universal tensile testing machine equipped with a corrosion-resistant glass environmental cell of 500 mL capacity. This cell allowed for the full immersion of the specimen's gauge length in the test solution while tensile load was applied.

A nominal strain rate of $1.0 \times 10^{-6} \text{ s}^{-1}$ was applied to all specimens during the SSRT tests. This specific rate was carefully selected based on extensive literature, which identifies a critical strain rate window (typically 10^{-5} to 10^{-7} s^{-1}) for revealing maximum SCC susceptibility in high-strength steels [37]. A rate of $1.0 \times 10^{-6} \text{ s}^{-1}$ is slow enough to allow sufficient time for the critical and time-dependent environmental interactions, such as hydrogen absorption and diffusion, to occur ahead of the crack tip [38]. At the same time, it is fast enough to ensure the continuous mechanical rupture of any surface films (e.g., iron sulfides), which constantly exposes fresh, reactive metal to the corrosive environment [39]. This balance prevents crack tip blunting or repassivation that might occur at slower rates, thereby maximizing the conditions for observing embrittlement and ensuring the test's sensitivity.

Before starting the tensile loading, each specimen was exposed to the H_2S -saturated acidic marine solution within the cell for 1 hour at its open circuit potential (OCP) to allow for stabilization of the steel/environment interface. Subsequently, the tensile load was applied at the specified constant strain rate until the specimen fractured. Control SSRT tests were also conducted in laboratory air at the same strain rate and temperature for baseline comparison.

During each test, load, elongation (via crosshead displacement), and time were continuously recorded by the testing machine's data acquisition system. From these raw data, engineering stress-strain curves were plotted. Key mechanical parameters determined included σ_{YS} , σ_{UTS} , ϵ_{total} , plastic elongation ($\epsilon_{plastic}$), RA, and time to failure (TTF).

2.5. Electrochemical measurements

Electrochemical measurements were performed using a Palmsens4 potentiostat/galvanostat. A standard three-electrode electrochemical cell configuration was employed. The X70 steel SSRT specimen (for in-situ measurements) or a separate coupon (10 mm $\times$ 10 mm $\times$ 2 mm, prepared with the same surface finish as SSRT specimens, for ex-situ measurements) served as the working electrode (WE). A saturated calomel electrode (SCE) was used as the reference electrode (RE), positioned close to the WE via a Luggin capillary filled with the test solution to minimize IR drop. A large surface area platinum mesh acted as the counter electrode (CE). For in-situ measurements during SSRT, the environmental cell was designed to accommodate these electrodes without interfering with the mechanical loading.

Potentiodynamic polarization scans were conducted after immersing the WE in the test solution and allowing the OCP to stabilize for 1 hour. The potential was scanned from -300 mV relative to OCP towards the anodic direction up to $+600$ mV relative to OCP, at a scan rate of 0.167 mV/s. From the polarization curves, the corrosion potential (E_{corr}), corrosion current density (i_{corr}) (determined by Tafel extrapolation of the linear portions of the anodic and cathodic branches), and the anodic (β_a) and cathodic (β_c) Tafel slopes were determined.

Electrochemical Impedance Spectroscopy (EIS) measurements were carried out at the OCP after a 1-hour stabilization period. For some experiments, EIS was also performed at selected intervals of strain during the SSRT tests to monitor changes in the electrochemical behavior of the steel under dynamic straining. A sinusoidal AC voltage perturbation of 10 mV (rms) was applied over a frequency range from 100 kHz down to 10 mHz. The impedance data were presented as Nyquist ($-Z''$ vs. Z') and Bode ($|Z|$ and phase angle vs. frequency) plots. The experimental EIS data were analyzed and fitted to appropriate equivalent electrical circuits (EECs) using ZView software (Scribner Associates Inc.) to extract quantitative parameters describing the electrochemical processes at the steel/solution interface. The continuous dynamic straining during SSRT can disrupt passive films, exposing fresh metal, and if the environment is aggressive and the material susceptible, this enhances the conditions for crack initiation and propagation, making SSRT a sensitive technique for evaluating SCC.

2.6. Microstructural and fractographic analysis

Following fracture in the SSRT tests, the primary fracture surfaces of specimens tested in both air and the acidic H_2S -containing marine environment were carefully sectioned, ultrasonically cleaned in ethanol for 5 – 10 minutes to remove loose corrosion products and solution residues, and then dried. These fracture surfaces were subsequently examined in detail using an SEM (JEOL JSM-6490LV) operating at an accelerating voltage of 20 kV. The examinations focused on identifying the overall fracture mode (e.g., ductile, brittle, or mixed-mode), specific micro-fractographic features indicative of SCC (such as quasi-cleavage facets, transgranular or intergranular cracking, river patterns, dimples, secondary cracks), and potential crack initiation sites.

To observe the crack propagation path in relation to the steel's microstructure, longitudinal sections were cut from selected fractured SSRT specimens, particularly those tested in the corrosive environment, ensuring the section included the main fracture surface and regions with secondary cracks. These sections were then mounted, prepared using standard metallographic procedures (grinding with SiC papers up to 1200 grit, polishing with $6\ \mu\text{m}$, $3\ \mu\text{m}$, and $1\ \mu\text{m}$ diamond paste), and etched with 2% Nital. The etched cross-sections were examined using both OM and SEM to determine if crack propagation was predominantly transgranular (through the grains), intergranular (along grain boundaries), or mixed-mode. The interaction of cracks with microstructural features such as ferrite grains, pearlite colonies, and non-metallic inclusions was also carefully documented.¹⁹ The morphology of the fracture surface provides direct evidence of the operating SCC mechanism(s); for example, ductile dimples are characteristic of mechanical overload in air, while brittle features like intergranular facets or transgranular cleavage in the corrosive environment are strong indicators of SCC, with specific features often linked to HE or AD.

3. RESULTS AND DISCUSSION

3.1. Initial microstructure of API 5L X70 steel

The initial microstructure of the as-received API 5L X70 steel was examined to provide a baseline for understanding its behavior during SCC testing. Figure 1 shows a representative optical micrograph of the longitudinal section after etching with 2% Nital. The microstructure predominantly consists of polygonal ferrite (PF) grains,

appearing as the lighter etched phase, with colonies of pearlite (P), the darker etching lamellar constituent of ferrite and cementite (Fe_3C), distributed throughout the ferritic matrix. This ferrite-pearlite microstructure is typical for conventionally processed API 5L X70 grade pipeline steels. The average ferrite grain size, determined using the linear intercept method as per ASTM E112, was found to be $18 \pm 3 \mu\text{m}$. Using the Hall–Petch relation ($\sigma_{\text{ys}} = \sigma_0 + k_y \cdot d^{-1/2}$) with literature values for σ_0 ($\approx 180 \text{ MPa}$) and k_y ($\approx 0.70 \text{ MPa} \cdot \text{m}^{1/2}$) for Nb–Ti microalloyed HSLA steels [40], the calculated grain refinement contribution to yield strength is $\approx 486 \text{ MPa}$. This prediction is in close agreement with the measured σ_{ys} in air (492 MPa), confirming that grain refinement is the dominant strengthening mechanism, supplemented by precipitation hardening from Nb/Ti carbides and carbonitrides.

Further examination using SEM, as shown in Figure 2, provided more detailed insights into the microstructural features. Figure 2(a) offers a higher magnification view of the ferrite grains and pearlite colonies, revealing the lamellar structure of cementite within the pearlite. Figure 2(b) highlights the presence of non-metallic inclusions. These were identified by EDS analysis (not shown) as primarily elongated manganese sulfide (MnS) inclusions, aligned parallel to the pipe’s rolling direction, along with some smaller, more globular aluminosilicate inclusions. To quantitatively support their role in SCC initiation, we conducted a statistical image analysis on high-resolution SEM micrographs over a cumulative area of $\sim 2.0 \text{ mm}^2$. The MnS inclusions exhibited an average length of $7.3 \pm 2.8 \mu\text{m}$, with an aspect ratio of 4.6 ± 1.2 , and an areal density of $220 \pm 15 \text{ inclusions/mm}^2$. Such elongated and densely distributed inclusions are known to act as stress concentrators and hydrogen traps, which can promote localized dissolution and hydrogen uptake. This morphology strongly suggests that MnS inclusions may serve as preferential sites for SCC initiation in X70 steel exposed to acidic sour marine environments. The ferrite-pearlite interfaces can also influence crack paths [41]. Hydrogen trapping in steels is conventionally categorized into reversible traps—such as grain boundaries, dislocations, and lattice defects—and irreversible traps, including secondary phase particles and inclusions. Reversible

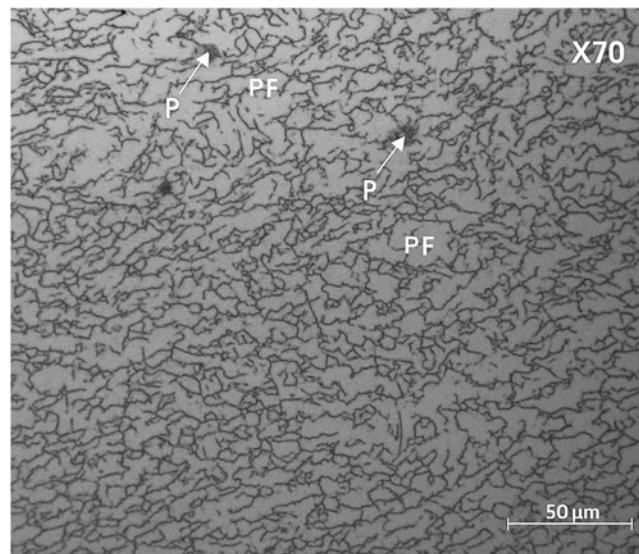


Figure 1: Optical micrograph of as-received API 5L X70 steel (longitudinal section, 2% Nital etch), showing PF and P colonies.

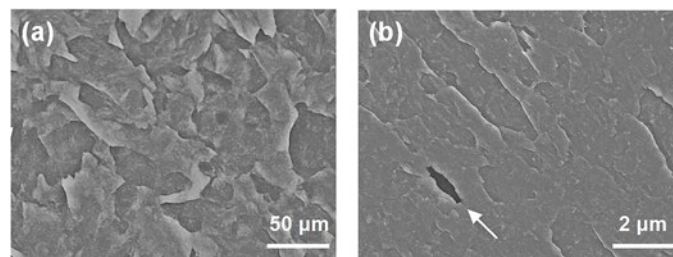


Figure 2: SEM micrographs of as-received API 5L X70 steel (2% Nital etch): (a) showing ferrite grains and pearlite lamellae detail, and (b) showing elongated MnS inclusions (indicated by arrows) within the ferrite matrix.

traps bind hydrogen weakly, allowing desorption under moderate thermal or stress perturbations. In contrast, nanoscale Nb–Ti carbide precipitates serve as irreversible traps due to their high binding energies. In our TEM observations, Nb–Ti carbides are clearly identified within the matrix, and EDS confirms their Nb–Ti rich composition (Figure 3). Nb–Ti carbides are clearly identified within the matrix, and quantitative EDS analysis of representative precipitates confirms their composition as Nb: 14.1 at.%, Ti: 69.1 at.%, C: 26.8 at.%, with trace Fe (< 1 at.%) from the matrix. Literature reports that such nanoscale carbides exhibit elevated hydrogen desorption activation energies and thus function as irreversible traps [42]. Grain boundaries, however, are primarily associated with reversible trapping behavior [43]. Such nanoscale carbides, typically 15–50 nm in diameter, exhibit elevated hydrogen desorption activation energies—reflecting strong irreversible trapping capacity—which reduces the concentration of mobile diffusible hydrogen available to participate in crack propagation in an acidic H_2S marine environment. Consequently, Nb–Ti carbides are effective at permanently immobilizing diffusible hydrogen, reducing the mobile hydrogen concentration responsible for stress corrosion cracking in an acidic marine environment. Future investigations using thermal desorption spectroscopy or atom probe tomography could further quantify trap energetics and hydrogen distribution at the atomic scale.

The observed alignment of MnS inclusions, a common feature in hot-rolled pipeline steels, can introduce anisotropy in mechanical properties and SCC resistance. Such inclusion stringers may provide preferential pathways for crack initiation or early-stage propagation, especially if they coincide with regions of localized stress or hydrogen accumulation. This initial microstructural state forms the basis upon which the subsequent SCC phenomena will develop and manifest.

3.2. SCC susceptibility from SSRT tests

The susceptibility of API 5L X70 steel to SCC in the simulated acidic marine environment was evaluated using SSRT tests. Representative engineering stress-strain curves obtained from tests conducted in laboratory air and in the H_2S -saturated acidic marine solution (pH 3, 3.5% NaCl) at 25 °C and a nominal strain rate of $1.0 \times 10^{-6} \text{ s}^{-1}$ are presented in Figure 4. A summary of the mechanical properties derived from these SSRT tests, along with calculated SCC susceptibility indices, is provided in Table 3.

The data in Figure 4 and Table 3 clearly illustrate the severe detrimental effect of the acidic H_2S -containing marine environment on the tensile properties and fracture resistance of API 5L X70 steel. Compared to the behavior in air, specimens tested in the corrosive solution exhibited a marginal increase in yield strength (σ_{YS} from 492 MPa to 505 MPa), but a significant reduction in ultimate tensile strength (σ_{UTS} from 575 MPa to 530 MPa). More strikingly, the ductility parameters were drastically diminished: total elongation (ϵ_{total}) decreased from 26.5% in air to only 9.8% in the solution, and the percentage RA plummeted from 68.0% to 35.2%. Consequently, the TTF was substantially shortened, from an average of 7.36 hours in air to 2.72 hours in the aggressive environment. These findings are consistent with previous studies on X70 and similar HSLA steels in acidic and/or H_2S -containing media, which report significant embrittlement [44]. The slight elevation in yield strength observed in the corrosive medium, coupled with the dramatic loss of ductility, is a characteristic manifestation of HE, where hydrogen absorbed into the steel lattice can impede dislocation motion or

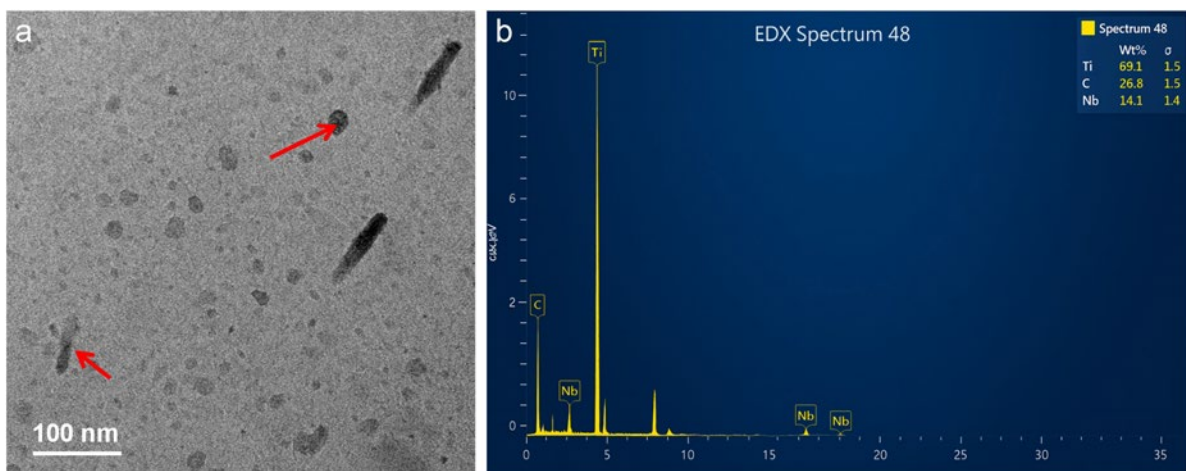


Figure 3: (a) TEM micrograph of API 5L X70 steel showing nanoscale Nb–Ti carbide precipitates (indicated by arrows) distributed within the ferritic matrix. (b) Corresponding EDX spectrum acquired from a representative precipitate.

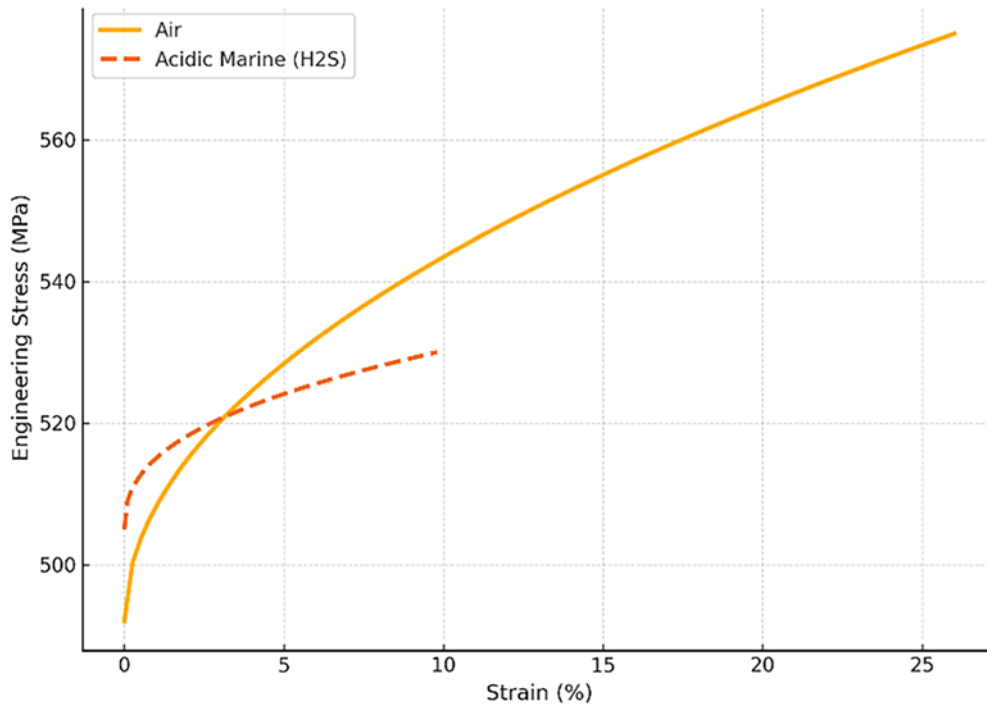


Figure 4: Representative engineering stress-strain curves for API 5L X70 steel from SSRT tests conducted in laboratory air and in the simulated acidic marine environment (pH 3, 3.5% NaCl, H₂S-saturated) at 25 °C and a strain rate of $1.0 \times 10^{-6} \text{ s}^{-1}$.

Table 3: Summary of mechanical properties and SCC susceptibility indices for API 5L X70 steel from SSRT tests.

TEST CONDITION	σ_{YS} (MPa)	σ_{UTS} (MPa)	ϵ_{TOTAL} (%)	$\epsilon_{PLASTIC}$ (%)	RA (%)	TTF (HOURS)	I_{RA}	$I_{\epsilon PLASTIC}$
Air	492 ± 5	575 ± 8	26.5 ± 0.8	24.2 ± 0.8	68.0 ± 1.5	7.36 ± 0.15	1.00	1.00
Acidic Marine (H ₂ S)	505 ± 7	530 ± 10	9.8 ± 0.5	7.5 ± 0.5	35.2 ± 1.2	2.72 ± 0.10	0.52	0.31

promote premature localized fracture initiation, thereby increasing the stress required for macroscopic yielding but severely curtailing the material's capacity for plastic deformation.

The SCC susceptibility was quantified using indices based on the loss of ductility. The reduction in area index (I_{RA}) was calculated to be 0.52, and the plastic elongation index ($I_{\epsilon PLASTIC}$) was 0.31. Both indices are well below 1.0, and significantly lower than the commonly accepted threshold of 0.8 (or 0.75) used to classify a material as highly susceptible to SCC under the given test conditions. This pronounced susceptibility underscores the aggressive nature of the simulated acidic marine environment [45], where the combined effects of low pH (enhancing hydrogen evolution) and the presence of H₂S (acting as a hydrogen recombination poison and promoting hydrogen entry) lead to severe embrittlement of the X70 steel. The continuous straining during the SSRT test ensures that any protective surface films are constantly ruptured, exposing fresh metal surfaces to the aggressive environment and facilitating the electrochemical reactions that drive SCC, particularly hydrogen absorption.

3.3. Electrochemical corrosion behavior

3.3.1. Potentiodynamic polarization

To investigate the general corrosion behavior and electrochemical reactions occurring on the X70 steel surface in the test environment, potentiodynamic polarization measurements were conducted. Figure 5 presents the polarization curve for API 5L X70 steel in the deaerated, H₂S-saturated 3.5% NaCl solution at pH 3 and 25 °C. For comparative purposes, a typical polarization curve for X70 steel in a neutral, aerated 3.5% NaCl solution (without H₂S) is also schematically included, based on literature data. The key electrochemical parameters

derived from the polarization curve in the acidic H_2S environment are summarized in Table 4, along with typical comparative values for a neutral NaCl solution.

In the acidic H_2S -containing solution, the X70 steel exhibited a corrosion potential (E_{corr}) of -685 mV vs. SCE and a high corrosion current density (i_{corr}) of $250 \mu\text{A}/\text{cm}^2$ [46]. This i_{corr} value is significantly higher than what is typically observed for this steel in neutral chloride solutions (e.g., $5\text{--}50 \mu\text{A}/\text{cm}^2$), indicating a substantially accelerated corrosion rate in the acidic sour environment. The polarization curve shows no evidence of a passive region; instead, the anodic branch indicates continuous active dissolution of the steel as the potential increases. This behavior is characteristic of steels in aggressive acidic media where stable protective films cannot form or are rapidly undermined. The cathodic branch is characterized by high current densities and a Tafel slope (β_c) of approximately 130 mV/decade, consistent with the hydrogen evolution reaction ($2H^+ + 2e^- \rightarrow H_2$) being the primary cathodic process, significantly promoted by the low pH. The presence of H_2S is also known to catalyze the hydrogen evolution reaction and, more importantly, to inhibit the recombination of adsorbed hydrogen atoms (H_{ads}) into H_2 gas, thereby increasing the surface concentration of H_{ads} and facilitating its absorption into the steel. This high rate of hydrogen generation and absorption is a critical factor contributing to the observed SCC susceptibility [47, 48].

3.3.2. Electrochemical impedance spectroscopy (EIS)

EIS measurements were performed at the OCP to further probe the electrochemical processes occurring at the steel/solution interface. Figure 6 shows the Nyquist plot, and Figure 7 shows the corresponding Bode plots (impedance modulus $|Z|$ and phase angle vs. frequency) for X70 steel in the acidic H_2S -containing marine solution.

The Nyquist plot is characterized by a depressed capacitive loop at higher and medium frequencies and a distinct inductive loop at lower frequencies. The depressed nature of the capacitive loop suggests a non-ideal capacitive response, often attributed to surface heterogeneity, roughness, or a distribution of relaxation times. This is commonly modeled using a constant phase element (CPE) instead of an ideal capacitor [49].

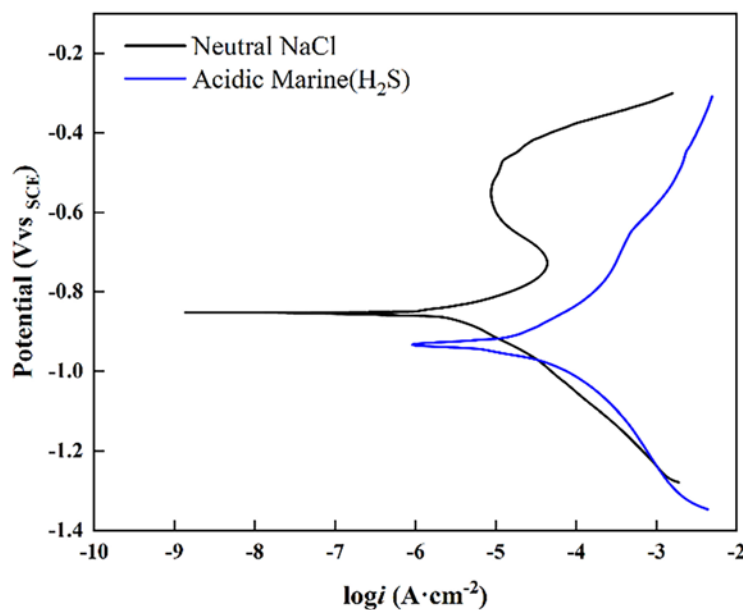


Figure 5: Potentiodynamic polarization curves for API 5L X70 steel: in deaerated, H_2S -saturated 3.5% NaCl solution at pH 3, and schematic representation for X70 steel in neutral, aerated 3.5% NaCl solution. Scan rate: 0.167 mV/s.

Table 4: Electrochemical polarization parameters for API 5L X70 steel.

ENVIRONMENT	E_{corr} (mV vs. SCE)	i_{corr} ($\mu\text{A}/\text{cm}^2$)	β_a (mV/dec)	β_c (mV/dec)
Acidic Marine (pH 3, H_2S -sat. 3.5% NaCl)	-685 ± 10	250 ± 20	75 ± 5	130 ± 8
Neutral Aerated 3.5% NaCl	~ -600 to -750	$\sim 5\text{--}50$	$\sim 60\text{--}100$	$\sim 120\text{--}180$

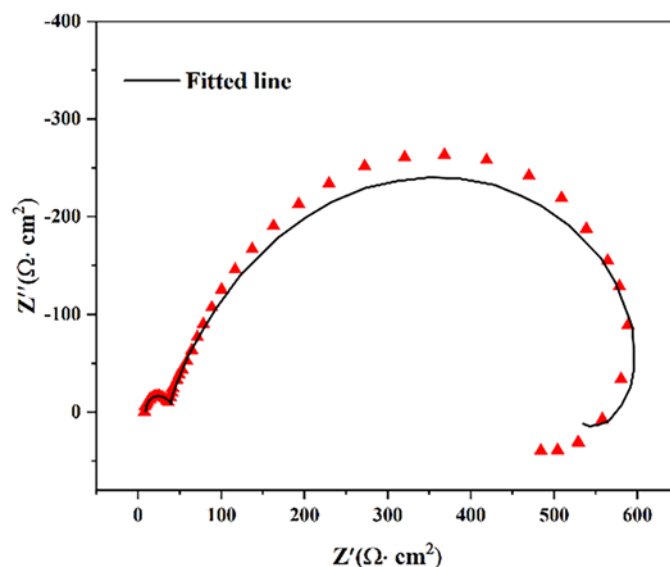


Figure 6: Nyquist plot for API 5L X70 steel in deaerated, H_2S -saturated 3.5% NaCl solution (pH 3) at OCP after 1 hour immersion.

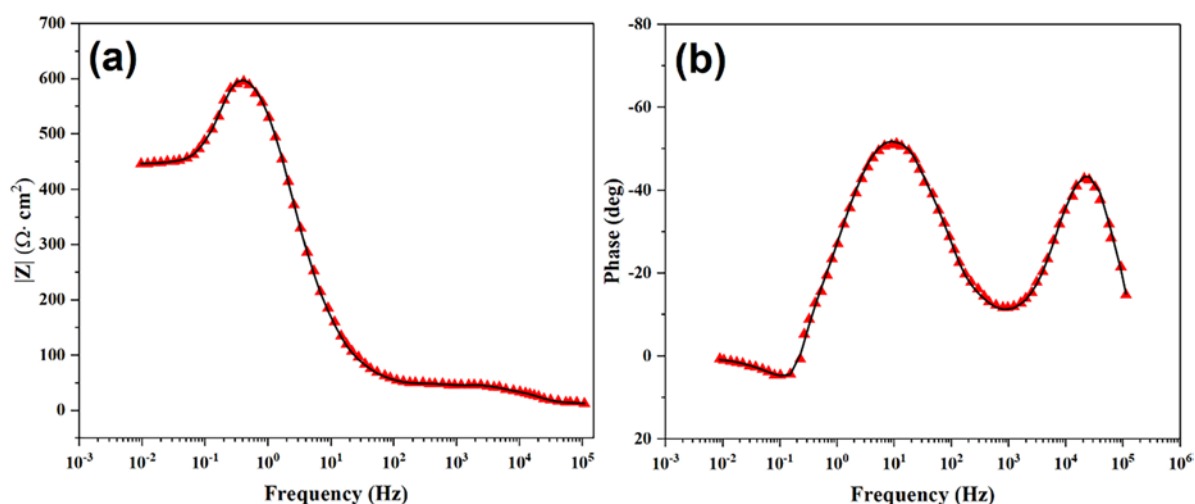


Figure 7: Bode plots for API 5L X70 steel corresponding to Figure 6: (a) Impedance modulus $|Z|$ vs. frequency, and (b) Phase angle vs. frequency.

The inductive loop observed at low frequencies in H_2S -containing acidic solutions is frequently associated with the relaxation of adsorbed intermediate species involved in the corrosion process or hydrogen evolution/adsorption steps, such as adsorbed hydrogen (H_{ads}) or iron-sulfide complexes (e.g., $Fe(HS)_{ads}$). The presence of this inductive feature is a strong indicator of the significant role of hydrogen-related processes at the interface [50].

The Bode plots (Figure 7) provide complementary information. The impedance modulus plot (Figure 7a) shows that the overall impedance is relatively low, consistent with the high corrosion rate observed from polarization data [51]. The phase angle plot (Figure 7b) exhibits a broad peak corresponding to the capacitive loop, with a maximum phase angle significantly less than -90° , confirming the non-ideal capacitive behavior. At the lowest frequencies, the phase angle becomes positive, which is characteristic of the inductive behavior seen in the Nyquist plot [52].

To quantitatively analyze the EIS data, the equivalent electrical circuit (EEC) shown in Figure 8 was used for fitting. This circuit includes the solution resistance (R_s) [53], a constant phase element (CPE_{dl}) representing the double-layer capacitance [54], the charge transfer resistance (R_{ct}) associated with the corrosion reaction, and an inductive element (L) in series with a resistance (R_L) to account for the low-frequency inductive loop [55].

Fitted parameters (example values): $R_s = 2.5 \Omega \cdot \text{cm}^2$, $R_{ct} = 80 \Omega \cdot \text{cm}^2$, $\text{CPE}_{dl} \cdot T = 5.5 \times 10^{-4} \text{ F} \cdot \text{s}^{(\alpha-1)} \cdot \text{cm}^{-2}$, $\text{CPE}_{dl} \cdot P(\alpha) = 0.85$, $L = 350 \text{ H} \cdot \text{cm}^2$, $R_L = 45 \Omega \cdot \text{cm}^2$. The relatively low value of R_{ct} ($80 \Omega \cdot \text{cm}^2$) is indicative of a high corrosion rate, corroborating the potentiodynamic polarization results. The significant inductive component (L and R_L) further supports the hypothesis that adsorption/desorption processes, likely involving hydrogen intermediates, play a crucial role in the overall electrochemical reaction mechanism under these acidic sour conditions [56, 57]. Such EIS features are consistent with environments promoting hydrogen uptake by the steel.

3.4. Fractographic analysis

The fracture surfaces of the API 5L X70 steel specimens tested under SSRT conditions in air and in the acidic H_2S -containing marine environment were examined by SEM to identify the operative failure mechanisms. Figure 9 shows a typical SEM fractograph of a specimen tested in air. The fracture surface exhibits characteristics of a fully ductile failure, with extensive plastic deformation [58]. The central region is dominated by equiaxed dimples, formed by microvoid coalescence (MVC), which is typical for the tensile overload of ductile steels. A distinct cup-and-cone fracture morphology was observed macroscopically [59].

In stark contrast, the fracture surface of the specimen tested in the acidic H_2S -containing marine environment, shown in Figure 10, displays predominantly brittle fracture features, indicative of SCC [60]. Figure 10(a) provides a low-magnification overview, revealing a relatively flat fracture surface with limited macroscopic plastic deformation. At higher magnification (Figure 10(b)), the dominant fracture mode is identified as quasi-cleavage. This is characterized by a complex array of small, poorly defined cleavage-like facets connected by tear ridges, with fine river patterns that are often discontinuous [61]. This morphology is distinct

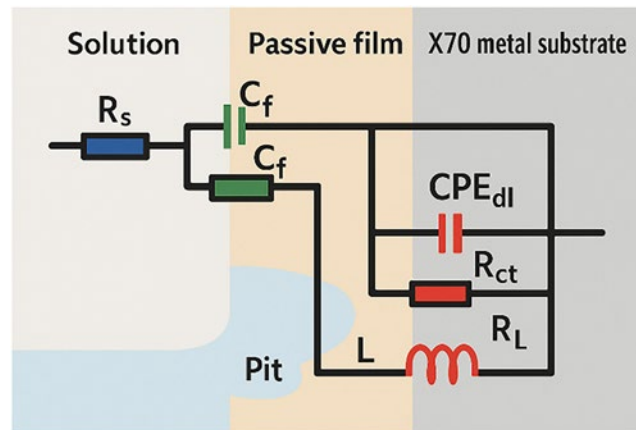


Figure 8: EEC model used for fitting the EIS data obtained in the acidic H_2S -containing environment.

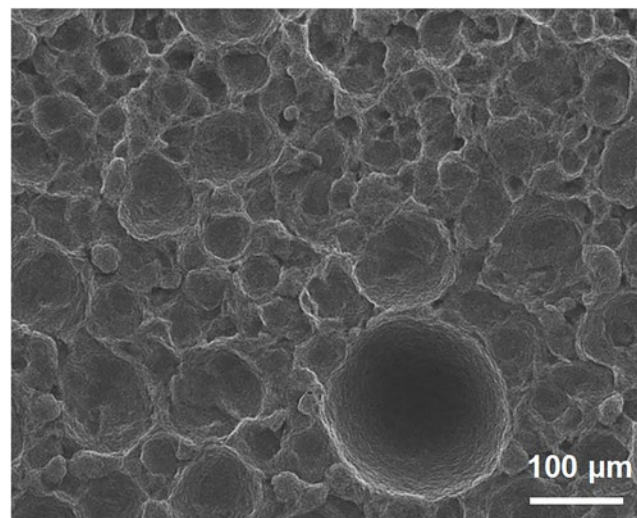


Figure 9: SEM fractograph of API 5L X70 steel specimen tested in air, showing ductile failure with extensive dimple rupture.

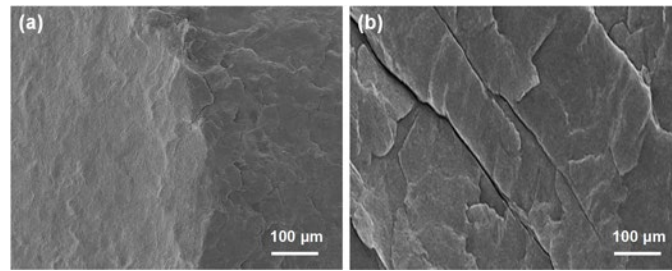


Figure 10: SEM fractographs of API 5L X70 steel specimen tested in the acidic H_2S -containing marine environment: (a) Low magnification overview showing brittle fracture appearance, and (b) Higher magnification revealing quasi-cleavage facets, and transgranular cracking.

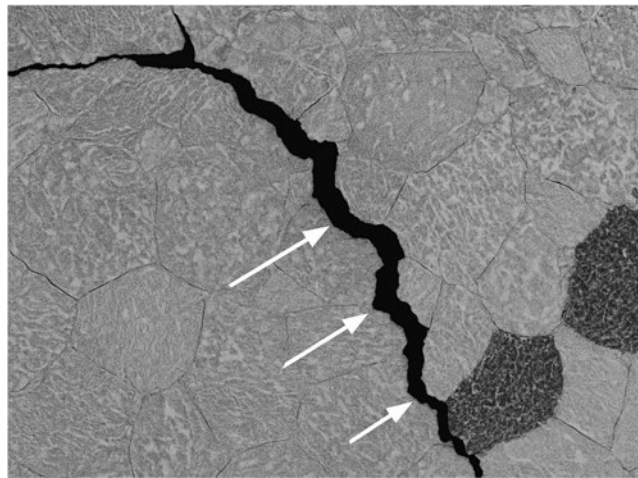


Figure 11: Optical micrograph of a longitudinal cross-section of a fractured SSRT specimen (tested in acidic H_2S -containing marine environment, 2% Nital etch), showing a transgranular secondary crack (indicated by arrows) propagating through ferrite grains.

from true cleavage, which would present as large, flat crystallographic planes. QC is widely recognized as a hallmark of HE in ferritic steels, resulting from hydrogen-assisted localized plastic processes ahead of the crack tip rather than simple crystallographic cleavage [62].

A detailed analysis reveals that the fracture is overwhelmingly transgranular, with cracks propagating directly through the ferrite grains. While some isolated intergranular (IG) facets were observed at grain boundary triple points, they constituted a minor fraction of the fracture surface, suggesting that under these specific environmental and stress conditions, the grain interiors are the preferential path for crack advance [63]. Numerous secondary cracks, oriented roughly perpendicular to the main fracture surface, were also observed across the gauge section near the primary fracture, a common feature in SCC that signifies a high susceptibility to cracking throughout the stressed material. The significant difference in fracture morphology between the air-tested and environment-tested specimens provides compelling evidence of environmentally assisted cracking. The prevalence of QC facets and transgranular cracking are classic fractographic indicators consistent with HE being the primary mechanism of failure in the acidic sour solution [64]. Regions of transgranular cracking are evident, where the crack has propagated through the ferrite grains. Some areas also showed evidence of intergranular facets, suggesting that grain boundaries were also susceptible paths for crack propagation, albeit to a lesser extent than transgranular quasi-cleavage under these specific conditions. Numerous secondary cracks, oriented roughly perpendicular to the main fracture surface, were also observed across the gauge section near the primary fracture, a common feature in SCC. The significant difference in fracture morphology between the air-tested and environment-tested specimens provides compelling evidence of environmentally assisted cracking. The brittle features observed are consistent with HE being a major contributing factor to the failure in the acidic sour solution [65].

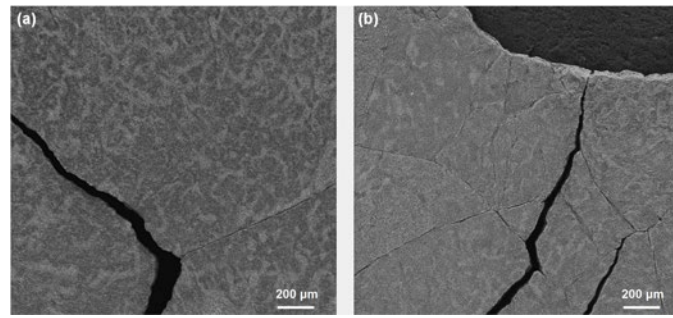


Figure 12: SEM micrographs of longitudinal cross-sections (acidic H_2S -containing marine environment, 2% Nital etch): (a) Detail of a transgranular crack tip, and (b) Multiple transgranular secondary cracks initiating from the specimen surface.

3.5. Crack path analysis

To further understand the SCC mechanism and its interaction with the microstructure, longitudinal cross-sections of fractured SSRT specimens tested in the acidic H_2S -containing marine environment were metallographically prepared and examined. Figure 11 is an optical micrograph showing the path of a secondary crack near the main fracture surface. The crack appears to propagate predominantly in a transgranular manner, cutting through the ferrite grains. There is some evidence of crack branching, which is often observed in SCC. The crack path does not seem to be significantly deflected or arrested by the pearlite colonies, suggesting that under these conditions, the ferrite matrix itself is susceptible to the cracking mechanism [66].

Higher magnification SEM examination of the crack paths, as shown in Figure 12, confirms the predominantly transgranular nature of the SCC cracks. Figure 12(a) shows a crack tip region where the crack is clearly advancing through the body of a ferrite grain [67]. The crack path is relatively straight within individual grains but changes direction as it crosses grain boundaries. Figure 12(b) illustrates multiple secondary cracks initiating from the specimen surface and propagating inwards. These cracks also exhibit a transgranular character. While some minor intergranular segments were occasionally observed, particularly at triple points or along specific grain boundaries, the overwhelming evidence points to transgranular cracking as the primary mode of propagation. This transgranular path, often associated with quasi-cleavage features on the fracture surface, is frequently linked to HE mechanisms where hydrogen affects processes within the grain interior or along specific crystallographic planes [68]. The observation of branched secondary cracks further supports an SCC mechanism rather than simple mechanical overload.

3.6. Elucidation of SCC mechanisms and discussion

As articulated in the introduction, while the general susceptibility of X70 steel in sour environments is known, a key objective of this work was to provide a more integrated mechanistic picture by correlating findings from multiple concurrent analyses. The results presented herein achieve this by establishing a clear and direct link between the electrochemical evidence of high corrosion activity and hydrogen promotion, and the resulting mechanical degradation and brittle fracture morphology [69]. Specifically, the appearance of a low-frequency inductive loop in the EIS data—a feature linked to hydrogen-related surface processes—is shown to directly correspond with a failure mode dominated by quasi-cleavage and transgranular cracking, which are classic hallmarks of HE. This integrated approach allows for a more robust conclusion that the failure is not merely due to AD or HE in isolation, but a potent synergy where aggressive dissolution creates ideal conditions for catastrophic hydrogen-driven crack propagation, a dynamic that this study clearly illustrates. The experimental results obtained from SSRT, electrochemical measurements, fractography, and crack path analysis collectively provide strong evidence for the high susceptibility of API 5L X70 steel to SCC in the simulated acidic (pH 3) marine environment saturated with H_2S . The significant reduction in ductility ($I_{\text{RA}} = 0.52$, $I_{\text{eplastic}} = 0.31$) and time to failure during SSRT tests, coupled with the observed brittle fracture morphologies (quasi-cleavage, transgranular cracking) and high corrosion rates, point towards a complex interplay of failure mechanisms [70].

The electrochemical data (Figure 4, Table 4) revealed a high corrosion current density ($i_{\text{corr}} = 250 \mu\text{A}/\text{cm}^2$) and an active corrosion potential ($E_{\text{corr}} = -685 \text{ mV}_{\text{SCE}}$), indicating aggressive corrosion [71]. The absence of a passive region in the polarization curve suggests continuous active dissolution of the steel surface. The low pH of the solution (pH 3) directly contributes to this by providing a high concentration of H^+ ions, which accelerates both the anodic dissolution of iron ($\text{Fe} \rightarrow \text{Fe}^{2+} + 2\text{e}^-$) and, crucially, the cathodic hydrogen evolution reaction ($2\text{H}^+ + 2\text{e}^- \rightarrow 2\text{H}_{\text{ads}} \rightarrow \text{H}_2$).

The presence of H_2S in the environment, at a high dissolved concentration of approximately 4.9 mmol/L, plays a pivotal role in promoting HE [72]. H_2S is a well-known hydrogen recombination poison; it adsorbs onto the steel surface and inhibits the combination of adsorbed hydrogen atoms (H_{ads}) to form molecular hydrogen (H_2) gas. This significantly increases the surface concentration and chemical potential of H_{ads} , thereby enhancing its rate of absorption into the steel lattice. The EIS results (Figures 5 and 6), particularly the presence of a low-frequency inductive loop, are consistent with processes involving adsorbed hydrogen species and suggest a high surface coverage of H_{ads} , facilitating its entry into the steel. Once inside the steel, hydrogen can diffuse to regions of high stress concentration, such as the area ahead of a crack tip, and induce embrittlement through mechanisms like HEDE or HELP [73]. The prevalence of transgranular quasi-cleavage (QC) facets and corresponding crack paths (Figures 10(b), 12(a))—often containing tear ridges and fine steps indicative of localized slip—are characteristic of hydrogen-enhanced localized plasticity (HELP) in ferritic steels [74]. The trend observed in the hydrogen embrittlement sensitivity index, where lower values correspond to reduced HE susceptibility, can be mechanistically linked to the microstructural features of the Nb–Ti microalloyed X70 steel. The refined ferrite grain size ($18 \pm 3 \mu\text{m}$) produced by TMCP increases the grain boundary area, which provides additional reversible hydrogen traps that slow long-range hydrogen diffusion. More critically, TEM/EDS analysis confirmed a high density of nanoscale Nb–Ti carbides (15–50 nm in diameter, Nb: 14.1 at.%, Ti: 69.1 at.%, C: 26.8 at.%). These precipitates permanently immobilize a portion of absorbed hydrogen, thereby lowering the concentration of mobile diffusible hydrogen that can participate in crack propagation [75]. This combination of irreversible trapping and grain refinement has been shown to suppress HE susceptibility in HSLA steels under sour service conditions, and our results confirm this correlation under the present acidic H_2S marine environment. In this mechanism, hydrogen facilitates dislocation motion ahead of the crack tip, producing transgranular QC fracture before substantial global plasticity develops. In contrast, occasional isolated intergranular facets, generally flat and lacking slip steps (e.g., Figure 12(b)), are consistent with hydrogen-enhanced decohesion (HEDE) [76], where hydrogen weakens atomic bonds along grain boundaries. These observations suggest that under the present acidic H_2S marine environment, HELP is the dominant cracking mechanism, with HEDE contributing locally, particularly at grain boundary triple points. The slight increase in yield strength during SSRT in the corrosive environment (Table 3) can also be attributed to hydrogen interacting with dislocations, leading to a form of hardening but with a catastrophic loss of overall ductility.

While HE appears to be the dominant mechanism responsible for the severe embrittlement and premature fracture, the role of anodic dissolution cannot be entirely discounted, especially in the initiation stages and in modifying the local crack tip environment [77]. The high corrosion rate indicates significant metal loss. Chlorides in the 3.5% NaCl solution can contribute to the breakdown of any transiently formed surface films and promote localized attack, potentially forming pits that act as SCC initiation sites. Although distinct pitting was not the primary focus of the fractography, the general active dissolution at the surface, particularly at microstructural heterogeneities like MnS inclusions (Figure 2b), could create favorable sites for crack nucleation. Furthermore, ongoing dissolution at the crack tip can maintain a sharp crack geometry and, within the occluded crack environment, lead to local chemical changes (e.g., further pH reduction due to metal ion hydrolysis) that could sustain both AD and hydrogen evolution. Thus, a synergistic interaction between AD and HE is likely operative: AD may facilitate crack initiation and maintain an aggressive local environment, while HE, significantly amplified by the H_2S and low pH, drives the rapid propagation of these cracks in a brittle manner.

The microstructure of the X70 steel also influences its SCC behavior. The transgranular nature of the cracking suggests that the ferrite matrix itself is susceptible under these conditions, with cracks propagating across grains rather than being confined to grain boundaries [78]. While MnS inclusions are potential initiation sites, the bulk of the crack propagation appears to be through the ferritic phase. The interaction between diffusing hydrogen and the dislocation substructure within the plastically deformed zone ahead of the crack tip is a key aspect of HE-driven transgranular cracking.

Figure 13 presents a schematic illustration of the proposed synergistic SCC mechanism for API 5L X70 steel in the studied acidic H_2S -containing marine environment. The process begins with general and localized anodic dissolution at the steel surface, potentially exacerbated by chloride ions and microstructural heterogeneities. The low pH and the presence of H_2S lead to a high rate of cathodic hydrogen evolution and a significantly enhanced rate of hydrogen absorption into the steel [79]. This absorbed hydrogen diffuses to regions of high tensile stress, particularly at the tip of any existing or newly formed cracks/notches [68]. The accumulation of hydrogen in these critical zones leads to embrittlement of the local material, reducing its resistance to fracture. Under the applied tensile stress, these hydrogen-embrittled regions fracture, causing the crack to advance. The newly created crack surfaces are then exposed to the aggressive environment, and the cycle of dissolution, hydrogen evolution, absorption, and embrittlement repeats, leading to sustained SCC propagation [80]. The iron

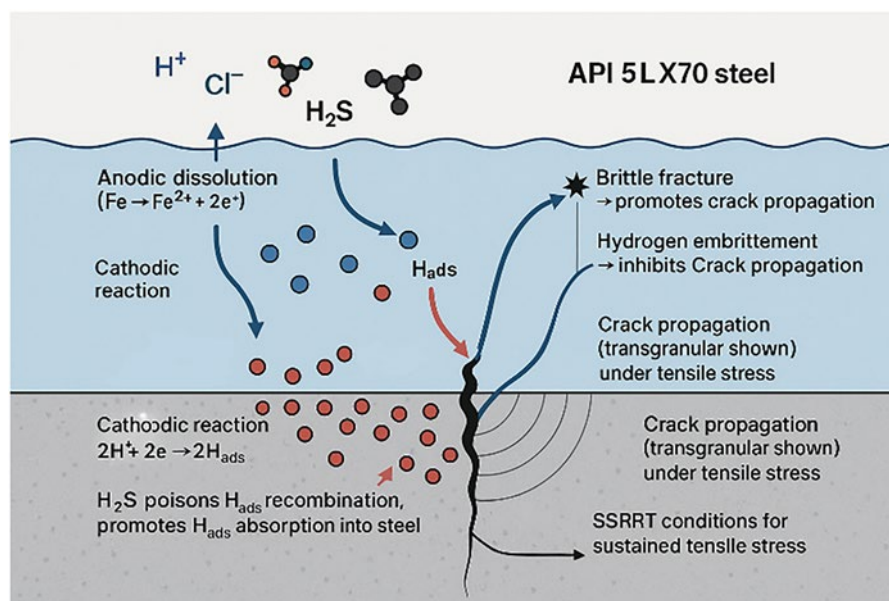


Figure 13: Schematic diagram illustrating the proposed synergistic SCC mechanism for API 5L X70 steel in the acidic H₂S-containing marine environment.

sulfide film formed due to H₂S may also play a role; if brittle, it could crack under strain, exposing fresh metal, or it could influence the local electrochemistry.

While this study was conducted under specific isothermal conditions (25 °C) and at a fixed H₂S partial pressure (0.05 atm), it is crucial to consider how variations in these parameters would impact SCC susceptibility in real-world applications. The influence of temperature on SCC is complex and often non-monotonic. An increase in temperature generally accelerates the kinetics of electrochemical reactions, including both AD and the HE reaction, which would suggest increased SCC susceptibility. Simultaneously, it increases the diffusion rate of hydrogen within the steel, potentially allowing hydrogen to reach critical fracture sites more quickly. However, competing phenomena also exist. The solubility of H₂S in aqueous solutions decreases as temperature rises [81], which could reduce the severity of the environment. Furthermore, the nature, stability, and protectiveness of the iron sulfide (FeS_x) scale that forms on the steel surface are highly dependent on temperature. Consequently, many studies report a peak in SCC susceptibility at an intermediate temperature, often between 60 °C and 90 °C, where the combination of accelerated kinetics and sufficient H₂S availability creates a worst-case scenario [82]. Similarly, the partial pressure of H₂S is a critical parameter that directly governs the severity of the sour environment. According to Henry's Law, the partial pressure of H₂S determines the concentration of dissolved H₂S in the aqueous phase. As demonstrated in this study, H₂S acts as a potent hydrogen recombination poison, and a higher dissolved concentration significantly increases the amount of atomic hydrogen absorbed into the steel, thereby exacerbating the risk of hydrogen embrittlement (HE). Industry standards, such as NACE MR0175/ISO 15156, explicitly define domains of environmental severity for sour service based on the partial pressure of H₂S and the in-situ pH. Generally, an increase in H₂S partial pressure leads to a marked increase in SCC and SSC susceptibility, although the effect may plateau at very high concentrations where the surface becomes saturated with adsorbed hydrogen or the properties of the resulting sulfide film become the rate-limiting factor [83]. Therefore, the severe susceptibility observed in our study at 0.05 atm partial pressure would be expected to become even more pronounced under conditions with higher H₂S partial pressures commonly encountered in some oil and gas fields.

The mechanistic insights from this study, particularly the identification of HE as the dominant failure driver, directly inform the selection of effective corrosion mitigation strategies. The primary goal of any protection system in this environment must be to prevent or drastically reduce the rate of hydrogen absorption into the steel. High-integrity protective coatings (e.g., fusion-bonded epoxy, three-layer polyethylene/polypropylene) serve as the first line of defense by providing a physical barrier between the steel and the aggressive environment. However, at coating defects (holidays), the steel becomes exposed and highly vulnerable. Cathodic protection (CP) is used to protect these areas by lowering the electrochemical potential of the

steel. In H_2S -containing environments, however, the application of CP must be carefully controlled. Overprotection (excessively negative potentials) can dramatically increase the rate of the hydrogen evolution reaction, exacerbating the risk of HE, which our results show is the critical failure mechanism. Therefore, CP potentials must be maintained within a narrow, optimized window as specified by standards like NACE MR0175/ISO 15156. Furthermore, the use of corrosion inhibitors, particularly those formulated for sour service, can offer significant protection. Effective inhibitors function by adsorbing onto the steel surface to form a protective film that hinders both anodic dissolution and the cathodic hydrogen evolution reaction, thereby reducing both general corrosion and hydrogen uptake. The selection of an inhibitor must consider its performance in the presence of H_2S and its ability to prevent hydrogen permeation. A combined approach, integrating high-performance coatings with carefully monitored CP and, where applicable, chemical inhibition, represents the most robust strategy for mitigating SCC in these severe conditions.

The pronounced SCC susceptibility observed for API 5L X70 steel, a material widely used for pipelines, under these simulated acidic sour marine conditions, highlights a significant engineering concern. Accidental exposure to such environments, perhaps due to coating failure in marine sediments with SRB activity, or internal process upsets leading to acidic H_2S contamination, could severely compromise pipeline integrity. This underscores the critical importance of comprehensive environmental assessment, appropriate material selection for particularly aggressive service, and the implementation of robust corrosion protection and monitoring strategies (e.g., coatings, CP, inhibition) in line with industry standards such as NACE MR0175/ISO 15156 for sour service applications. The continuous straining in SSRT creates a worst-case scenario by constantly exposing fresh metal, which might accelerate hydrogen uptake compared to static conditions where more stable sulfide films could potentially modulate hydrogen entry, albeit these films can also be detrimental if they are brittle or spall. This emphasizes the need to understand both the material's inherent susceptibility and the specific exposure conditions.

4. CONCLUSION

This experimental investigation into the SCC behavior of API 5L X70 high-strength steel in a simulated acidic (pH 3) marine environment saturated with H_2S has led to the following principal conclusions:

1. API 5L X70 steel exhibits high susceptibility to SCC when exposed to a simulated acidic (pH 3) marine environment saturated with H_2S , corresponding to a dissolved concentration of ~ 4.9 mmol/L. SSRT tests revealed a significant reduction in ductility, with the reduction in area index (I_{RA}) being 0.52 and the plastic elongation index ($I_{eplastic}$) being 0.31, both indicating severe embrittlement. The time to failure was also drastically reduced compared to tests in air. This finding is consistent with literature on similar steels in H_2S -containing environments.
2. Electrochemical measurements confirmed the aggressive nature of the environment. Potentiodynamic polarization indicated a high corrosion rate ($i_{corr} \approx 250 \mu A/cm^2$) and active dissolution behavior with no passivation. EIS showed low charge transfer resistance and a characteristic low-frequency inductive loop, suggesting that processes related to hydrogen adsorption and absorption are significant at the steel/solution interface.
3. Fractographic analysis by SEM showed a clear transition from ductile failure (microvoid coalescence) in air to predominantly brittle fracture modes in the acidic H_2S environment. The SCC fracture surfaces were characterized by quasi-cleavage facets and transgranular cracking, with evidence of secondary cracking. Crack path analysis confirmed that cracks propagated mainly in a transgranular manner through the ferrite grains.
4. The dominant SCC mechanism for API 5L X70 steel under these conditions is a synergistic effect of HE and AD. The low pH and the presence of H_2S create conditions highly conducive to hydrogen generation and its absorption into the steel, leading to HE. Concurrently, active anodic dissolution contributes to crack initiation and potentially influences the local crack tip chemistry.

The findings of this study underscore the severe degradation that API 5L X70 steel can undergo in acidic marine environments containing H_2S . This has significant implications for the integrity and safety of pipelines and other marine structures fabricated from this material if exposed to such conditions. Further research could focus on the influence of varying H_2S and chloride concentrations, the effect of temperature, the performance of different HSLA steel grades or microstructures, and the efficacy of corrosion inhibitors or CP in mitigating SCC in these aggressive environments.

5. ACKNOWLEDGMENTS

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