Effect of hydrogen-induced plasticity on the stress corrosion cracking of X70 pipeline steel in simulated soil environments

Z.Y. Liu^{a,b1}, X. Z. Wang^a, C. W. Du^{a,b}, J. K. Li^{a,b}, X.G. Li^{a,b,c2}

^a*Corrosion and Protection Center, University of Science and Technology Beijing, Beijing, 100083, China*

^b*Key Laboratory of Corrosion and Protection of Ministry of Education, Beijing, 100083, China*

^c*Ningbo Institute of Material Technology & Engineering, Chinese Academy of Sciences, Ningbo 315201, Zhejiang, China.*

Abstract

The susceptibility to stress corrosion cracking (SCC) of X70 pipeline steel under cathodic protection in near-neutral pH and acidic solutions was investigated by slow-strain-rate tensile test, circumferential-notch tensile (CNT) test, and three-point-bending (TPB) test. Results confirmed the existence of a hydrogen-induced plasticity (HIP) effect within a particular range of cathodic potentials. HIP effect lowered the SCC risk of X70 steel by releasing stress concentration at crack-initiation spots and then decreasing the stress intensity. Crack-growth behavior examined by CNT and TPB tests proved the existence of an HIP effect.

Key words: Pipeline steel; Hydrogen induced plasticity; Stress corrosion cracking; Soil environments

¹ Corresponding authors:

Tel: +86-10-62333931; Fax: +86-10-62334005. E-mail addresses: liuzhiyong7804@ustb.edu.cn (Z.Y. Liu).

² Corresponding authors:

Tel: +86-10-62333931; Fax: +86-10-62334005. E-mail addresses: lixiaogang99@ustb.edu.cn (X.G. Li).

1. Introduction

The stress corrosion cracking (SCC) of pipelines in soil environments have posed significant problems to the petroleum industry over the past decades [1-3]. Two types of SCC, namely, high pH SCC [4-5] ($\text{pH} > 9.0$) and near-neutral pH [6-10] (at a pH around 6.5), have been identified since the first case of SCC found in the USA. High-pH SCC for pipelines follows the anodic dissolution (AD) mechanism [4,7], whereas low-pH SCC involves the combined effect of hydrogen and non-steady electrochemical effect on dissolution of the steel [6-10]. Parkins et al. [10] concluded that near-neutral pH SCC results from the cooperation of anodic dissolution and the effect of hydrogen in the steel. Beavers [11] pointed out that SCC is initiated by anodic dissolution (AD), and its propagation is driven by hydrogen-induced cracking (HIC). Cheng et al. [12-13] quantified the contributions of stress, hydrogen, and their synergism to dissolution of steel at the crack tip, i.e., hydrogen-induced dissolution, in near-neutral pH solution. Hydrogen evolution is responsible for SCC of pipeline steels. However, Lu et al. [1,9] reported that hydrogen has limited effect on active dissolution of pipeline steel in near-neutral pH underground water. These results imply that the relationship between the effect of hydrogen and anodic dissolution has not been fully identified. More recently, Liu et al. [14-19] have extensively studied the SCC mechanism of pipe steels, including X70, X80, and X100 steels, in soil environments. They demonstrated that the different electrochemical statuses between the crack tip and the crack wall give rise to a combined effect of hydrogen. Moreover, both high-pH SCC and near-neutral pH SCC are strongly related to cathodic potential

(CP). The SCC of pipeline steels is under the combined effect of AD and HE. When the applied CP is about between $-730 \text{ mV}_{\text{SCE}}$ and $-920 \text{ mV}_{\text{SCE}}$ in near neutral solution. The SCC susceptibility increases first and then decreases with the negative shift of CP. However, SCC susceptibility increases again when the applied potential is more negative than $-920 \text{ mV}_{\text{SCE}}$ [14]. This finding introduces a big challenge for the security of pipelines on protecting the steel sufficiently and lowering the risk of SCC at the same time. On one hand, hydrogen evolution is intensified when CP-on, which is believed as one of the main reasons of SCC. On the other hand, hydrogen may have an important effect in metal matrix, i.e., hydrogen-induced plasticity (HIP) [20–22] other than hydrogen embrittlement, on mechanical and SCC properties. HIP decreases the stress intensity at the crack tip and lowers the SCC risk of pipeline steels by releasing the stress concentration at SCC initiation spots and enlarging deformation zone in front of the crack tip, thereby providing a possibility to optimize the CP condition. However, the detailed mechanism of HIP and its relationship with SCC under CP remain unclear and need further investigation.

In this work, slow-strain-rate tensile (SSRT) tests were performed on API X70 pipeline steel to confirm an effective CP range of HIP with low SCC susceptibility. The mechanism of HIP was investigated by circumferential-notch tensile (CNT) test and three-point-bending (TPB) test.

2. Experimental

All samples used in this work were made of X70 pipeline steel supplied by

Baosteel Co., Ltd., China, with the following chemical composition (wt.%): 0.065 C, 0.23 Si, 1.57 Mn, 0.056 Nb, 0.20 Ni, 0.18 Cr, 0.22 Cu, 0.027 Al, 0.015 Ti, 0.028 V, 0.0020 S, and 0.0019 P and Fe balance.

The solutions used include a near-neutral pH solution, i.e., NS4 solution, to simulate the electrolyte trapped under disbonded coating in Canada [18] and an acidic solution with $\text{pH} \approx 4$ to simulate an acidic soil located in southeast China [23]. The chemical contents of these two solutions are listed in Table 1. Both solutions were produced from analytical grade reagents and ultra-pure water. Prior to the tests, the NS4 solution was purged with 5% CO_2 balanced with N_2 gas for 1 h to achieve an anaerobic and near-neutral pH condition, with a pH of approximately 6.3. The pH of the acidic solution was adjusted to 4.0 by adding acetic acid; pure nitrogen gas was purged to the solution to remove oxygen before tests. The gas flows of the two solutions were maintained throughout the tests.

The SSRT test was performed at a strain rate of $1 \times 10^{-6} \text{ s}^{-1}$ in air and in the two testing solutions at various CPs through a WDML-30KN Materials Test System. Two types of SSRT samples were used: those for the test in NS4 were cut with the tensile direction parallel to the rolling direction, while those for examination in the pH 4-solution were machined with the loading direction of 45° to the rolling direction. The tensile specimens were ground sequentially to 1500 grit emery papers, with the grinding direction parallel to the applied stress.

The specimen was pre-immersed in the solution under the designed cathodic potential or open circuit potential for 24 h before the SSRT test. Each test was

repeated three times to ensure the reproducibility of the results.^[24] After the SSRT tests, the specimens were immersed into a descaled solution (500 mL HCl + 500 mL H₂O + 3.5 g (CH₂)₆N₄) in an ultrasonic container for 3 min, degreased with acetone, and dried with cool air. Surface morphologies were observed by scanning electron microscopy (SEM).

The crack growth property of the X70 steel was characterized by CNT and TPB tests. The dimensions of the CNT and TPB specimens are shown in **Fig. 1**. The compact tensile specimen was immersed in NS4 solution and loaded a stress of 0.625 R ratio cyclically with the frequency of 0.002 Hz. The maximum stress was controlled to achieve a maximum stress intensity factor ($K_{\max}$) within the range of 77 MPa√m to 104 MPa√m, and the stress intensity factor (ΔK) ranged from 29 MPa√m to 39 MPa√m. The TPB specimen was loaded at a rate of 0.002 mm·min⁻¹ in the pH 4 acidic synthetic soil solution. The loading cycle and test time were systematically documented by a computer. Crack growth was observed and monitored using a microscope equipped with a charge-coupled device camera system as described previously [25]; the crack growth length was measured every few hours.

Potentiodynamic polarization curves were measured in an electrochemical three-electrode cell, with the X70 steel specimen as working electrode, saturated calomel electrode (SCE) as reference electrode, and platinum plate as counter electrode.

All tests were performed at ambient temperature (about 22 °C).

3. Results

The stress–strain curves of the X70 steel measured in air and different testing solutions at $-850 \text{ mV}_{\text{SCE}}$ are shown in **Fig. 2**. The elongation of the steel decreased in both NS4 and acidic solutions compared with that obtained in air, indicating that the X70 steel is susceptible to SCC in these two solutions.

Fig. 3 shows the stress–strain curves of the X70 steel in the NS4 and acidic solutions under various polarizing potentials. The elongation and ultimate tensile strength of the steel exhibited complex dependence on the CPs. To investigate the SCC susceptibility of steel in soil solutions, the ratio of reduction in elongation I_η and the ratio of reduction in area I_ψ were calculated by **Eqs. (1) and (2)**:

$$I_\eta = \left(1 - \frac{\eta_E}{\eta_0}\right) \times 100\% \quad (1)$$

$$I_\psi = \left(1 - \frac{\psi_E}{\psi_0}\right) \times 100\% \quad (2)$$

where η_0 and η_E are the reduction-in-elongation in air and in the solution at various potentials, respectively, and ψ_0 and ψ_E are the reduction-in-area measured in air and in the solution at various potentials, respectively. **Fig. 4** and **5** show the calculated I_η and I_ψ values. Larger I_η and I_ψ values correspond with higher susceptibility to SCC. **Fig. 4** shows that I_η decreases first with the negative shift of potential in the range of $-750 \text{ mV}_{\text{SCE}}$ to $-930 \text{ mV}_{\text{SCE}}$, and then increases as the applied potential negatively shifts further. Therefore, a particular CP range exists, in which the specimens display lower SCC susceptibility, compared with the results at more negative or positive potential conditions. **Fig. 5** shows that I_ψ increases with the negative shift of potential in the range of $-730 \text{ mV}_{\text{SCE}}$ to $-890 \text{ mV}_{\text{SCE}}$, then decreases significantly in the

potential range of $-890 \text{ mV}_{\text{SCE}}$ to $-920 \text{ mV}_{\text{SCE}}$, and finally increases again when the applied potential is more negative than $-920 \text{ mV}_{\text{SCE}}$. This finding exhibits the similar changing rules in the pH 4 acidic solution with slight differences in potential transfer range. This phenomenon is related to the electrochemical behavior of the steel in these two solutions under cathodic potential. According to the non-equilibrium electrochemical theory proposed in our previous work ^[14, 15], anodic dissolution could occur on the polarized steel under the applied potential between two null-current potentials on the polarization curves with slow and fast scanning rates. Therefore, a peak potential is observed under which the steel exhibits the maximum SCC susceptibility. In addition, the difference of the peak potential in the two kinds of solutions is attributed to the discrepancy of the potential range under which both anodic dissolution and hydrogen evolution occur ^[14, 15].

To characterize the relationship of SCC initiation with applied potentials, the micro-cracks on the side surface of the SSRT samples were observed by SEM and shown in **Fig. 6**. The SCC initiation density is strongly related to the potential conditions. Large-scale, discontinuous, small cracks were observed for the X70 steel in pH 4 solution at $-750 \text{ mV}_{\text{SCE}}$. A certain number of cracks connected together in the central areas with the application of $-850 \text{ mV}_{\text{SCE}}$ CP; hence, longer cracks were detected. However, visible cracks became scarce and only slight SCC occurred on the side surface with further negative shift of CP to $-950 \text{ mV}_{\text{SCE}}$, indicating that the steel exhibited better SCC resistance at $-950 \text{ mV}_{\text{SCE}}$. The statistics of crack densities are shown in **Fig. 7**, wherein the largest crack appeared at $-750 \text{ mV}_{\text{SCE}}$, whereas became

smaller at $-850 \text{ mV}_{\text{SCE}}$ because of crack connection. The crack number sharply dropped to $30/\text{cm}^2$, which was approximately 25% of that at $-750 \text{ mV}_{\text{SCE}}$, when the potential negatively shifted to $-950 \text{ mV}_{\text{SCE}}$.

The SCC crack depth on the fracture surface is observed and the maximum crack depth is measured. **Fig. 8** shows the method to measure the maximum crack depth, while the statistic results of the maximum crack depth at different potentials are shown in **Fig. 9**. The maximum crack depth results are consistent with the results of I_{ψ} in **Fig. 5**. In the pH 4 acidic solution, the maximum crack depth significantly increased when the potential shifted negatively from $-680 \text{ mV}_{\text{SCE}}$ to $-980 \text{ mV}_{\text{SCE}}$; a sharp drop was observed at $-1050 \text{ mV}_{\text{SCE}}$. When the applied potential shifted further to $-1200 \text{ mV}_{\text{SCE}}$, the maximum crack depth increased again. For the specimen in the NS4 solution, the change rule of maximum crack depth exhibited a similar pattern to that in the pH 4 solution, except for the transfer potential at which the maximum crack depth started to decrease.

Given that hydrogen evolution is the primary cathodic reaction that occurs in anaerobic NS4 and acidic solutions [14], the cathodic current density has a strong relationship with hydrogen concentration. The hydrogen content in the steel has a significant effect on the SCC process. Thus, both SCC susceptibility index and cathodic current density must be considered to accurately discuss the role of hydrogen inside the steel on the SCC process, as shown in **Fig. 10**. A critical amount of hydrogen was observed; however, the exact amount cannot be determined, which affects the index, as marked with a dotted line in the figure. When the hydrogen

content inside steel is insufficient, hydrogen indirectly affects SCC susceptibility, while other influencing factors are supposed to be working as well. When the hydrogen content reaches above the threshold value, a HIC or HE effect will show up^[18]. Given that the hydrogen evolution is enhanced with the cathodic current density, the HE effect is believed to dominate the SCC growth and dissolution would accelerate the cracking process.

Fig. 11 shows the curves of the crack growth length increase with increased cycle number (loading frequency $f=0.002$ Hz) or test time at varying applied potentials. Crack growth was faster at -1200 mV_{SCE} in both tests than that at -850 mV_{SCE} and OCP. However, the results at -850 mV_{SCE} and OCP were different, indicating that loading modes (cycle loading and continuous loading) have certain effect on growth behavior. For CNT specimen under cycle loading, the crack growth is much slower at -850 mV_{SCE} than that at OCP, while the opposite result is obtained for the TPB specimen under continuous loading.

4. Discussion

4.1 Analysis of the electrochemical reactions for pipeline steels in simulated soil environments and its relationship with the SCC process.

The anodic reaction of the X70 steel in the testing solutions is the oxidation of iron [26].

The cathodic reaction of the steel includes reduction of H^+ , HCO_3^- , H_2CO_3 , and H_2O :

The corrosion product, a layer of porous $\text{Fe}(\text{OH})_2$, is formed on the steel surface with the increase in anodic and cathodic reactions:

Compared with the cathodic reactions in the NS4 solution, the hydrogen evolution process in the pH 4 acidic solution is accelerated based on the fact that the hydrogen ion concentration is much higher, as shown in **Fig. 10**. In the SCC process, fresh matrix surface at crack tip is continuously generated because of tensile stress on the specimen, resulting in an unstable or non-equilibrium electrochemical status at the crack tip. Meanwhile, the crack wall is covered with a corrosion product layer or equilibrium electric double layer, under which electrochemical reactions take place in a quasi-equilibrium state. The anodic dissolution reaction (3) and the cathodic hydrogen evolution reactions (4), (5), and (6) at the crack tip are promoted. As a result, the different electrochemical states between the crack tip and crack wall introduce a combined effect of hydrogen embrittlement and anodic dissolution on the occurrence of SCC, which leads to high SCC susceptibility when the applied potential is between OCP and $-950 \text{ mV}_{\text{SCE}}$ [14].

4.2 HIP and its effect on the SCC process

Two SCC susceptibility factors, namely, ratio of reduction-in-elongation I_η and

ratio of reduction-in-area I_ψ , were widely used to characterize the SCC susceptibility of the pipeline steels. Generally, I_ψ , which reflects the sensitivity of SCC at the stage of crack propagation is mainly affected by cracks and defects inside the steel. Thus, maximum crack depth statistic results in **Fig. 9** and the side surface crack initiation behavior shown in **Fig. 6** are consistent with the results of I_ψ in **Fig. 5** because they are all influenced by the same material factors. Meanwhile, I_η , which mainly characterizes the dislocation and deformation of a specimen, is influenced by hydrogen content inside the steel [27].

The CP applied to the operating pipelines will protect the steel from corrosion with the presence of coating defects by penetrating the coating and reaching the steel surface. However, anodic dissolution will still occur and contribute to the SCC process even when the pipeline is under cathodic protection [14], which challenges the cathodic protection effectiveness. This work demonstrates an effective CP range of HIP with low SCC susceptibility for pipeline steels. As shown in **Figs. 4 and 10**, an HIP effect is confirmed in the potential range between $-680 \text{ mV}_{\text{SCE}}$ and $-930 \text{ mV}_{\text{SCE}}$. Within this potential range, the HIP effect postpones the initiation process of SCC and lowers the growth rate of cracks to slow down the propagation process by releasing the stress concentration at the crack initiation regions and enlarging the deformation zone in front of the crack tip. In other words, HIP effect within this potential range lowers the SCC risk of the pipeline steels.

The results of the crack growth test also display the influence of HIP effect on SCC behavior. For the compact tensile specimen under non-continuous loading mode,

crack opening angle cyclically opens and contracts and the hydrogen at the crack tip could loosen the load on the specimen and decrease the stress concentration, consequently prohibiting crack growth when the hydrogen content inside the steel is below the critical amount. However, for the TPB specimen under continuous loading, crack opening angle continuously increases and fresh crack tip shows up continuously because of the stress effect; hence, the cracks grow continuously. For the cycle loading specimen at CP of -850 mV, crack grows much slower than that under OCP. More hydrogen will be generated as the specimen is under more negative CP than that at OCP. A hydrogen embrittlement effect, which could accelerate crack growth, is expected to appear. However, a HIP phenomenon may also play a role during this SCC process. HIP releases the stress concentration around the crack tip, decreases the stress intensity of SCC, and then prohibits crack growth to some extent, consistent with the results in **Fig. 4**. Therefore, the crack growth of the CNT specimen is much slower at -850 mV_{SCE} than that at OCP, while the result obtained by TPB specimen under continuous loading is opposite, as seen in **Fig.11**.

5. Conclusion

A high SCC susceptibility potential range (owing to the non-steady electrochemical effects) and an HIP potential range exist between OCP and -950 mV_{SCE} CPs for pipeline steel under CP. The HIP phenomenon postpones the initiation and the propagation of SCC when the applied potential is within the high SCC susceptibility potential range. This positive effect of HIP would reduce the risk

of SCC in the corresponding potential ranges.

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Figure caption:

Fig.1. Schematic of the dimensions of compact tensile specimen (a) and three-point-bending specimen (b), units: mm.

Fig.2. Stress-strain curves of X70 steel measured in air and in testing solutions at $-850 \text{ mV}_{\text{SCE}}$.

Fig.3. Stress-strain curves of X70 steel under cathodic protection in NS4 solution (a) and pH=4 solution (b) under various cathodic polarization potentials.

Fig.4. Effect of cathodic potential on the ratio of reduction-in-elongation (I_η) in NS4 solution and pH=4 solution.

Fig.5. Effect of cathodic potential on the ratio of reduction-in-area (I_ψ) in NS4 solution and pH=4 solution.

Fig.6. Location of micro-cracks on side surfaces of X70 steels pH=4 acidic solution at different applied potentials: (a)-750mV; (b)-850mV; (c) -950mV.

Fig.7. Statistics of crack densities on side surfaces of X70 samples in pH=4 acidic solution at different applied potentials.

Fig.8. SEM images of fractographies for measuring the maximum crack depth.

Fig.9. Statistic results of maximum crack depth at different potentials.

Fig.10. Dependence of reduction-in-area and corrosion current density obtained in NS4 solution (a) and pH=4 solution (b) on the applied cathodic potentials.

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Fig.6. Morphology (a, c, and e) and location (b, d and f) of the micro-cracks on the side surface of the X70 pipeline steel fractured under applied potentials of -750 mV (a and b), - 850 mV (c and d), and -950 mV (e and f).

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Fig.11. Crack growth behavior of pipeline steel in simulated soil environment, (a) CNT specimen under cycle loading in NS4 solution; (b) TPB specimen under continuous loading in a pH=4 solution.

Table caption

Table 1. Chemical composition of the NS4 solution and the pH=4 solution.

Solution	Chemical composition (g/L)							
	CaCl ₂ ·2H ₂ O	MgSO ₄ ·7H ₂ O	NaHCO ₃	KCl	KNO ₃	NaCl	Na ₂ SO ₄	CH ₃ COOH
Near-neutral pH solution	0.181	0.131	0.122	0.483	-	-	-	-
Acidic solution	0.294	0.394	0.302	-	0.586	0.936	0.284	Adjust pH to 4.0