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Hydrogen in stress corrosion cracking of X-70 pipeline steels in near-neutral pH solutions

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The effect of hydrogen on the stress corrosion cracking (SCC) for X-70A and X-70B pipeline steels in near-neutral pH environments was studied by monotonic tensile tests on slow strain rate tests (SSRT) machine and cyclic tests on SSRT machine and fatigue machine. The results showed that SCC intensity increased at high cathodic protection potential. Hydrogen charged at high pressure enhanced SCC sensitivity. Cyclic tests under elastic strain condition could not take the specimens to fracture and the hydrogen content was very low. However, when the load was raised to produce plastic deformation, the specimens failed within limited time and there was brittle fracture appeared on the fracture surface. Moreover, the hydrogen content was higher than that under elastic strain condition. The SCC mechanism of X-70A and X-70B pipeline steels in near-neutral pH environments was related to hydrogen accumulation induced by plastic strain. © *2006 Springer Science + Business Media, Inc.*

1. Introduction

Hydrogen embrittlement (HE) of structural materials is a serious problem that has received more and more attention for the past seventy years. The ubiquity of the sources of hydrogen-corrosion in aqueous solutions, absorption into pipelines carrying humid and contaminated hydrocarbons, and contaminants in the melting processes contributes to the importance of the problem. The degradation is manifested in such diverse ways as the catastrophic cracking of high-strength steels, a contribution to stress corrosion cracking (SCC) of ferritic stainless steels, failure in nuclear reactors of zirconium alloy tubing by hydride formation, and the failure of the pearlitic rail steels because of residual hydrogen [1]. Particularly in the past several decades, there has been a great amount of research on hydrogen problem, among which hydrogen in iron is an important problem and is intensively studied system [2–6] since severe failure cases can be caused by HE of a steel. Hydrogen induced degradation of carbon and low alloy steels can have the different types of manifestations, which are blisters on the surface of the material, internal cracks parallel to the rolled surface of the material, and cracks perpendicular to the direction of the external tensile stress. Compared with high-strength steels, the low-carbon steels are less susceptible to HE and need larger contents of hydrogen to affect their mechanical properties significantly. The low-carbon steels, like the pipeline steel in oil and gas industries, contact a great variety of aggressive, hydrogen-bearing media, and suffer slow degradation of their mechanical properties and utility. In deep oil wells, hydrogen plays an important role in the environmental failure of pipe, as it promotes the development of cracks through HE [7]. For SCC in near-neutral pH solution on external pipeline surface, Parkins reported that both dissolution and the ingress of hydrogen into the steel involved in the crack propagation of pipeline steel, and the hydrogen facilitated crack growth by promoting reduced ductility [8]. The effect of hydrogen on SCC of pipeline steels in the near-neutral pH solutions was studied using samples with coating disbondments at slow strain rate tests (SSRT) and secondary ion mass spectrometry (SIMS) by Gu [9]. Hydrogen was found to diffuse into the steels around the crack tip during the SCC process, and facilitated the dissolution rate of the steel and increased SCC intensity. Gu proposed hydrogen-facilitated anodic dissolution mechanism of SCC in dilute near-neutral pH solution. Beavers [10] suggested that the mechanism of near-neutral pH SCC was different for crack initiation and propagation. It was possible that the crack was likely to initiate by corrosion fatigue mechanism and then propagated by HE mechanism. How the hydrogen enters the pipeline steel is worth studying. China has built a

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TABLE I. The chemical compositions of X-70A and X-70B line pipe steels (wt%)

Material	C	Mn	Si	S	P	Cr	Ni	Ca	Al	Cu	Ti	Nb	V	Mo	N
X-70A	0.046	1.58	0.23	0.0015	0.012	0.025	0.17	0.003	0.032	0.25	0.010	0.066	0.028	0.23	0.0033
X-70B	0.060	1.59	0.18	0.003	0.007	0.007	0.24	0.001	0.025	0.013	0.020	0.060	0.052	0.23	0.005

gigantic pipeline project, West-East gas transmission project by the end of 2004. So it is necessary to study the effect of hydrogen on SCC in near-neutral pH solutions for the pipeline steels used in this gigantic project. It has been shown that high-pressure hydrogen can seriously degrade the mechanical properties of many of the commonly used engineering alloys [11], in which the crack growth rate is raised by 2 to 4 orders of magnitude. Therefore, in this paper, hydrogen charged at high pressure is used to investigate the influence of hydrogen on SCC of pipeline steels in near-neutral pH environments.

2. Experimental procedures

2.1. Specimen

Materials were from X-70A and X-70B line pipe steels respectively. The X-70A line pipe was spiral-welded, and the angle between weld and longitudinal axis was 30°. Chemical compositions of both steels were listed in Table I. The carbon equivalents in chemical composition on sensitivity of welding crack for the X-70A steel and X-70B steel were 0.39 and 0.40%, respectively.

The cylindrical, waisted tensile specimens with a 12.50-mm gauge length and 2.50-mm gauge diameter were cut from X-70A line pipe and X-70B steel plate, respectively, with gauge length along the pipe longitudinal direction (L) for the X-70A pipe (X-70A L) and parallel to the rolling direction for X-70B steel plate (X-70B T) to study SCC using SSRT machine. The specimens with 10 mm long, 6 mm wide and 5 mm thick gauge length were used in cyclic loading tests on fatigue machine. Specimens were finished longitudinally to 1200 grit with aluminum oxide waterproof abrasive paper, degreased in acetone and ethanol, and masked (except for the gauge length) before testing.

2.2. Solution

The solutions employed were a NS4 solution and an actual soil solution. The NS4 solution consisted of 483 mg/l $NaHCO_3$, 122 mg/l KCl, 137 mg/l $CaCl_2$ and 131 mg/l $MgSO_4 7\cdot H_2O$. The soil was from Xinjiang Uygur Autonomous Region, where West-East natural gas transmission project started. The composition of this soil was 0.0011% $NO_3{}^-$, 0.0145% Cl^-, 0.1172% $SO_4{}^{2-}$, 0.0064% $HCO_3{}^-$, 0.0422% Ca^{2+}, 0.0013% Mg^{2+}, 0.0016% K^+, 0.0195% Na^+, 19.2% water, 0.05% organic matter, 0.002% nitrogen in total, with 0.2038% calculated salt in total, collected from 1,150 mm below the surface of earth in Xinjiang Uygur Autonomous Region. The conductance of lixivium was 0.562 $m\Omega^{-1}$/cm 25°C, while the conductance of the mud was 1.33 $m\Omega^{-1}$/cm 25°C. The soil was placed in a nylon/polyester bag and suspended in a container. The weight ratio of soil to distilled water was 1:5. The soil and distilled water were allowed to equilibrate at room temperature for a week prior to test.

2.3. SCC tests

SCC tests were performed using SSRT and cyclic loading tests at high stress ratio (the ratio of minimum stress to maximum stress) and low frequency conditions. The specimens were contained in airtight cells that were closed with rubber stoppers through which the ends of the specimens protruded for gripping. The test cell contained a probe to an external saturated calomel electrode (SCE), inlet and outlet through which the solution could be circulated via conduct and a small pump. Another container contained facilities for bubbling mixture gas, 5% CO_2 + 95% N_2, through solution. All of the circulated system was anaerobic. Specimens were subjected to conventional, monotonic SSRT in air as an inert environment and a soil solution at the strain rates of 1E-5/s and 2E-6/s at room temperature. Some specimens were precharged under the circumstance of high-pressure hydrogen with 10.1325 MPa and 300°C for 24 h before monotonic SSRT were conducted. For the cyclic loading tests, the specimens were preloaded in air up to about 95 percent of the ultimate tensile strength, unloaded, and then placed in the solution and cyclically strained up to this load at the strain rate of 2E-6/s for those conducted on SSRT machine. As to the cyclic loading tests performed on fatigue machine, balanced triangle waveform load was used with the frequency of 2.319×10^{-4} Hz and the stress ratio of 0.1. Upon the completion of the tests, one part of the fractured specimens were kept in liquid nitrogen in order to measure hydrogen content of the specimens using RH-404 hydrogen measurement instrument, in which the absorbed hydrogen in specimens was measured by the hot extraction technique. So the absorbed hydrogen here referred to the total hydrogen content in the material, i.e., diffusible and trapped hydrogen. The other part of the fractured specimens was examined by XL30 environmental scanning electron microscopy (ESEM) from FEI Company.

3. Results

Fig. 1 showed the ratio of fracture energy in the solution divided by that in air (R) changed with applied potentials for X-70A L and X-70B T specimens strained at 2×10^{-6}/s in a soil solution purged with 5% CO_2 + 95% N_2.

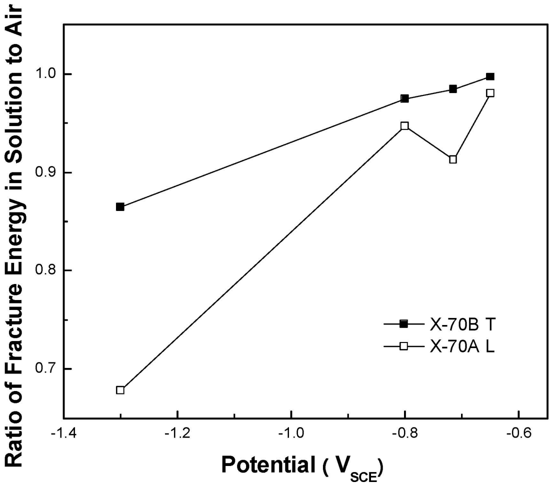

Figure 1 The ratio of fracture energy in solution to that in air for X-70A L and X-70B T specimens strained at 2×10^{-6}/s in a soil solution purged with 5% CO_2 + 95% N_2 at different applied potentials.

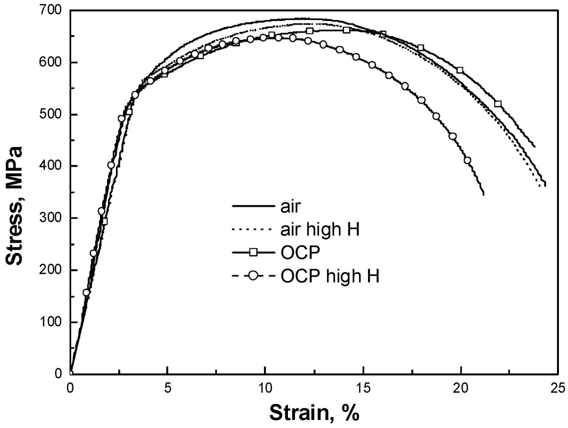

Figure 3 The effect of hydrogen precharging on the stress versus strain curves for X-70A L specimens strained at 1×10^{-5}/s in air and in a soil solution purged with 5% CO_2 + 95% N_2 at OCP.

For X-70A L specimens, *R* increased when the electrochemical potential was anodic, and decreased sharply in the cathodic region. As shown in Fig. 2, at OCP, there were some cracks appeared on the specimen's surface, and at −1.3 V_{SCE}, more cracks on specimen's surface were observed. For X-70B T , *R* decreased as the potential was lowered from −0.65 V_{SCE} to −1.3 V_{SCE}. Moreover, the *R* value was larger than X-70A L steel, and it was evident that there were much fewer cracks appeared on the side surface, indicating a greater propensity for X-70A L than the other at similar experimental conditions.

Fig. 3 illustrated the stress-strain curves for X-70A L specimens strained at 1×10^{-5}/s, where some of the specimens were precharged at high pressure hydrogen condition before SSRT. When the specimens were strained in air, the maximum stress and the strain to failure for the precharged sample only reduced a little, compared to the one without hydrogen precharging (Fig. 3). There were only dimple features on fracture surface of both the precharged one and the non-precharged. When the specimen was strained in the soil solution purged with 5% CO_2 + 95% N_2 at OCP, the maximum stress, 661.8 MPa, and the strain to failure, 23.7% for the specimen without hydrogen precharging decreased a little compared to that in air, but decreased a lot for the sample with hydrogen precharging, where the strain to failure and the maximum stress were 21.1% and 647.3 MPa respectively, as shown in Fig. 3.

For the specimens strained in air, the hydrogen contents in the specimens without and with hydrogen precharging were 0.44 and 2.9 ppm respectively. When the specimens were strained in the actual soil solution purged with 5% CO_2 + 95% N_2 at OCP, the hydrogen contents in the specimens without and with hydrogen precharging changed to 1.9 and 9.8 ppm respectively (Fig. 4). There were some quasi-cleavage features on the fracture surface for the specimen strained at 1×10^{-5}/s in the actual soil solution purged with 5% CO_2 + 95% N_2 at OCP with hydrogen precharging (Fig. 5), whereas only dimple feature for the specimen without hydrogen precharging. When the X-70B T specimens were strained at 1×10^{-5}/s in an actual soil solution purged with 5% CO_2 + 95% N_2 at OCP, the results were similar to those for X-70A L specimens. Large reduction of area was obtained for the specimen with hydrogen precharging (Fig. 6a), and the hydrogen content was also very high, 29 ppm (Fig. 6b), whereas the hydrogen content

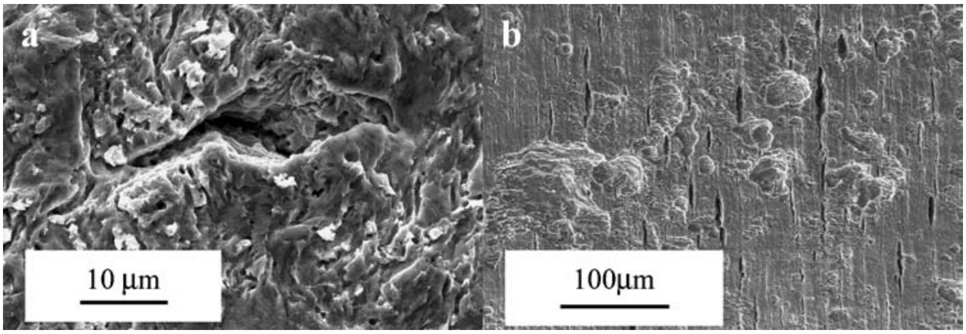

Figure 2 ESEM micrograph of the specimens' surface for X-70A L specimens strained at 2×10^{-6}/s in a soil solution purged with 5% CO_2 + 95% N_2 at: (a) OCP; (b) –1.3 V_{SCE}.

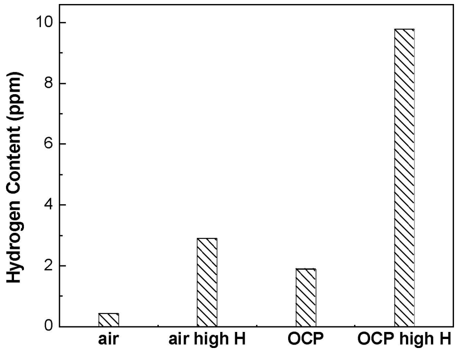

Figure 4 Hydrogen contents for X-70A L tested at 1×10^{-5}/s in air and the actual soil solution purged with 5% CO_2 + 95% N_2 at OCP with and without hydrogen precharging.

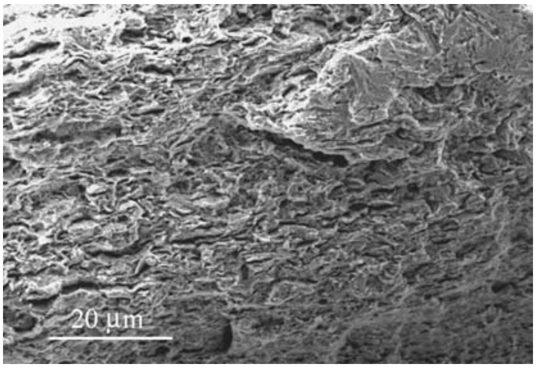

Figure 5 Fractograph of X-70A L strained at 1×10^{-5}/s in an actual soil solution purged with 5% CO_2 + 95% N_2 at OCP with hydrogen precharging before SSRT.

was only 1.5 ppm for the sample at OCP without hydrogen precharging.

Fig. 7 showed the cyclic tests conducted on SSRT machine. The specimens were firstly loaded in air along the line AB until it yielded and then continued to load up to 95% percent of ultimate tensile strength along BC, and unloaded to zero, along CD. Subsequently, the specimens were loaded in the soil solution purged with 5% CO_2 + 95% N_2 at OCP, and cyclically loaded up to the same load

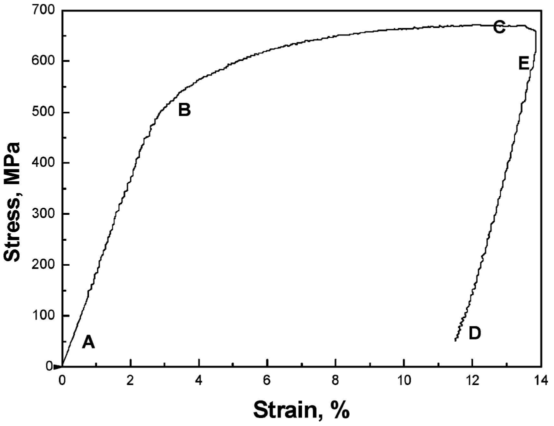

Figure 7 Cyclic test on SSRT machine for X-70A L strained at 2×10^{-6}/s in air until the stress reached 95% maximum stress, then unloaded, followed by cyclically strained in an actual soil solution purged with 5% CO_2 + 95% N_2 at OCP.

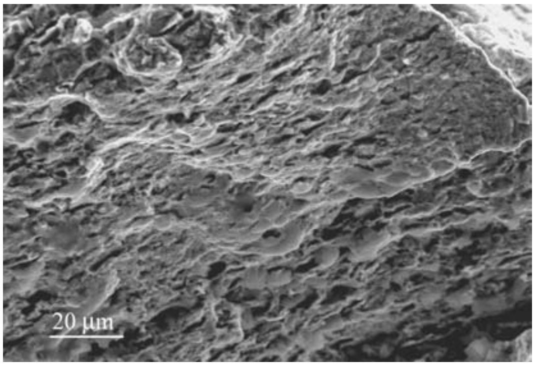

Figure 8 Brittle fracture feature obtained for X-70A L after cyclically strained at 2×10^{-6}/s in an actual soil solution purged with 5% CO_2 + 95% N_2 at OCP.

at the same strain rate of 2×10^{-6}/s. The specimen did not fail after cyclically loaded for 143.9 h (ten cycles), but the time to failure of the normal SSRT at 2×10^{-6}/s was 29.0 h. After 10 cycles, the sample was loaded continually until fractured, and it only took a few hours to break the specimen. There were some brittle fracture features on the fracture surface as shown in Fig. 8.

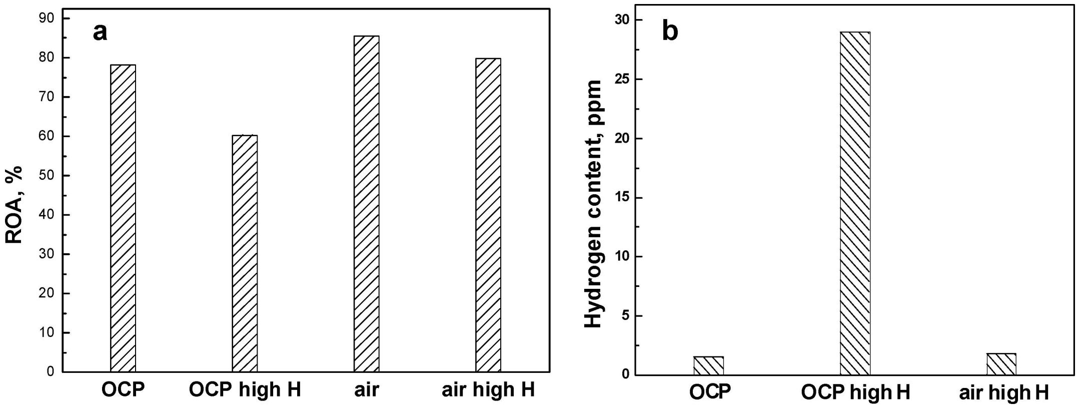

Figure 6 Reduction of area (a); hydrogen content (b) for X-70B T strained at 1×10^{-5}/s in an actual soil solution purged with 5% CO_2 + 95% N_2 at OCP and in air with and without hydrogen precharging before SSRT.

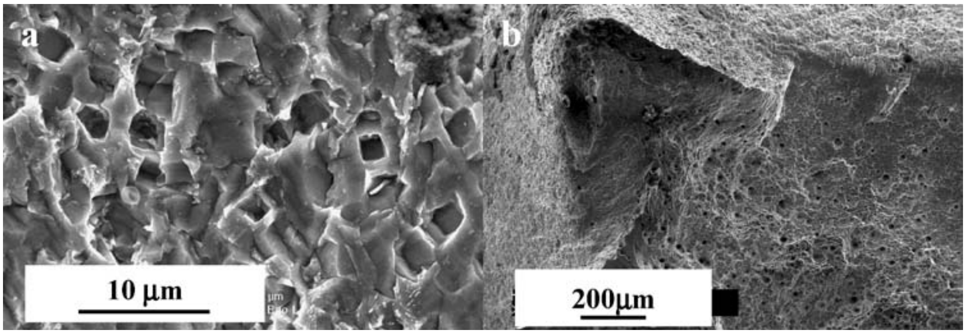

Figure 9 ESEM observation of (a) the specimen surface (loaded at elastic strain) and (b) fracture surface (loaded at plastic strain) for X-70A L in a NS4 solution purged with 5% CO_2 + 95% N_2 at OCP.

When the cyclic loading tests were conducted on fatigue machine, the specimens were also pre-loaded in air up to 95% ultimate tensile strength, and then unloaded. The pre-loaded sample was cyclically loaded up to the same stress, 95% ultimate tensile strength, in a NS4 solution purged with 5% CO_2 + 95% N_2 at OCP at the same parameters as in air. After 400 cycles, the test was stopped and then hydrogen content was measured. There was about 0.86 ppm hydrogen. The ESEM observation of the specimen surface showed that there was not any crack on the side surface but etching and pitting as shown in Fig. 9a. Another specimen was loaded until 400 cycles and then the maximum load was raised to 97% ultimate tensile strength and continued to load up to this stress. 59 more cycles later, the specimen ruptured. The hydrogen content of this specimen was about 4.63 ppm. The fractograph showed that there were some areas with quasi-cleavage features, the crack initiated near the outside of the specimen and then propagated toward the central part of the sample where there were large amounts of dimple (Fig. 9b). One sample without loading was immersed in NS4 solution for about ten days, and the hydrogen content was only 0.46 ppm.

4. Discussion

Fig. 1 showed that high values of *R* were obtained at anodic potential region and near OCP, indicative of low SCC sensitivity for the two steels. It was confirmed by the ESEM observation that the few cracks on specimen surface were found in Fig. 2a at OCP condition. When the potential was lowered, a substantial decrease in *R* (Fig. 1) and more surface cracks were observed, especially at the potential of $-1.3\ V_{SCE}$ (Fig. 2b), suggesting that SCC susceptibility increased as the electrochemical potential decreased, which was related to hydrogen. In order to prove the influence of hydrogen on SCC in near-neutral pH environments, the specimens were precharged with hydrogen under the circumstance of 10.1325 MPa and 300°C for 24 h before tests were conducted. The iron was bcc structure at ambient temperature, and hydrogen in iron was assumed to occupy either octahedral or tetrahedral sites. Although the hydrogen site in iron was not clear, evidence from other bcc materials favored the tetrahedral site [1]. However, hydrogen in iron also chose octahedral site [1]. Iron had large interstice, so if the specimens with hydrogen precharging were conducted at a low strain rate, hydrogen would escape. It was also found in this study that the content of hydrogen in this steel with bcc structure would decrease by about 65% after the specimen with hydrogen precharging was placed in air for about ten hours. So the strain rate of 1×10^{-5}/s in SSRT for the hydrogen precharging specimens was employed. For X-70A L specimen with hydrogen precharging strained at 1×10^{-5}/s in an actual soil solution purged with 5% CO_2 + 95% N_2 at OCP, there was significant decrease of elongation (Fig. 3). There were some areas with quasi-cleavage brittle fracture on the fractograph (Fig. 5). For the specimens with hydrogen precharging, the hydrogen content of 9.8 ppm in solution was much more than 2.9 ppm in air, whilst there were just 1.9 ppm in solution and 0.44 ppm in air for the specimens without hydrogen precharging (Fig. 4). Fig. 3 also showed that hydrogen precharging treatment had no big influence on the mechanical properties for the specimens strained in air. However, hydrogen did affect SCC sensitivity of the steels in near-neutral pH solution. For X-70B T specimens strained in a soil solution at OCP, there was significant reduction of ROA after hydrogen precharging, as shown in Fig. 6a, and the hydrogen content, 29 ppm, was much higher than 1.5 ppm for the sample strained in a soil solution without hydrogen precharging. The results showed that high hydrogen content increased SCC sensitivity for X-70 steel in near neutral pH solutions.

The SCC of pipeline steels did occur near OCP in near-neutral pH solutions, especially in oil and gas pipelines in the field, where it was impossible to apply large cathodic protection potential. Even if large cathodic protection potential was imposed, the environment would change so that near-neutral pH SCC could not occur, as proposed by Parkins [12]. In laboratory, near-neutral pH SCC could still occur at large cathodic protection potential if the mixture CO_2/N_2 gas can be bubbled through solution continuously. In this case, the cracking was engendered by hydrogen induced cracking [13], and there were many cracks on the specimen surface (Fig. 2). Thus, SCC susceptibility increased. However, for X-70 pipeline steels, the evolution

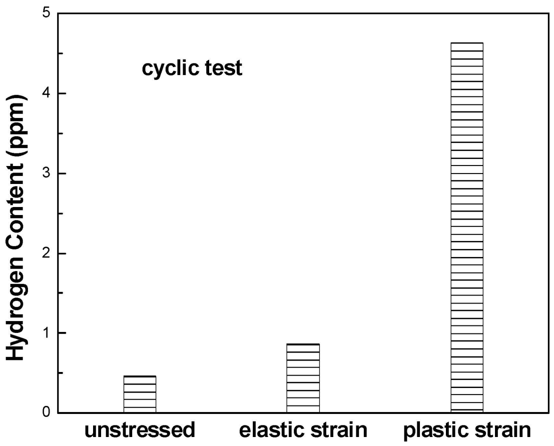

Figure 10 Hydrogen content for X-70A L specimens without loading, stressed at elastic and plastic strains in a NS4 solution purged with 5% CO_2 + 95% N_2 at OCP.

of hydrogen was very weak around OCP. Moreover, specific conditions must be fulfilled for hydrogen uptake under this weak circumstance [14]. Therefore, there should be the presence of poisons or the occurrence of slow deformation, both enabling hydrogen entry into the material [15]. In this study, there were no poisons used, so it was most likely to be the strain induced hydrogen entry into pipeline steel. Generally, strain could be divided into elastic strain and plastic strain. Therefore, it was necessary to clarify the role of elastic strain or plastic strain on strain induced hydrogen entry in near-neutral pH SCC around OCP. Thus cyclic tests were carried out, as shown in Fig. 7. When the specimens were cyclically loaded, it was considered that the strain during the cyclically load in solutions was elastic strain since the stress and strain changed along DE and CD in Fig. 7 although plastic deformation might take place at some locations to some extent. For the X-70A L specimen strained in the soil solution purged with 5% CO_2 + 95% N_2 at OCP on SSRT machine, 143.9 h later, the specimen did not fail and then it was broken by continually straining. There were some quasi-cleavage features on the facture surface (Fig. 8). All of these indicated that plastic strain was necessary for SCC in near-neutral pH solutions, which could induce hydrogen accumulation at the tri-axial stress field ahead of crack tip. When the cyclic tests were performed using fatigue machine under the conditions of load controlled and 2.319×10^{-4} Hz, after 400 cycles, the specimen did not fail yet and there was not any crack appeared on the specimen surface. When the load was raised a little, it took 59 cycles for this sample to fracture. The fractograph in Fig. 9b and hydrogen content measured in Fig. 10 presented that there was large difference for the specimens tested at elastic strain and plastic strain. There was much higher hydrogen content for the specimen imposed by plastic strain. Therefore it could be suggested that hydrogen in near-neutral pH SCC was related to plastic deformation.

In fact, the solubility of hydrogen in the stressed X-70 pipeline steel lattice was determined by its chemical potential. In an inhomogeneously stressed steel, care must be taken in defining the chemical potential, since there might not be a unique value [16]. For a stressed steel subjected to a generalized state of stress, a unique chemical potential could be given by [16]

$$\mu_{\mathrm{H}} = \mu_{\mathrm{H}}^0 + \bar{w}_{\mathrm{H}} - w_{\mathrm{H}} \tag{1}$$

where μ_{H}^0 was chemical potential of hydrogen at zero stress, $\bar{w}_{\mathrm{H}} = \frac{\partial w}{\partial n_{\mathrm{H}}}$ was the partial molal strain energy of component H (the change in the strain energy of the steel because of the addition of n_{H} moles of hydrogen), and w_H was work done by per mole addition of hydrogen. For a dilute solution of hydrogen in iron, assuming uniform applied stress within a small region of the body and assuming $\overline{V}_{\mathrm{H}}$ was isotropic under uniaxial tensile stress, the solubility was [16]:

$$C_{\mathrm{H}}^{\sigma} = C_{\mathrm{H}}^{0} \exp\left(\frac{\sigma_{11}\bar{V}_{\mathrm{H}}}{3RT}\right) \tag{2}$$

where C_{H}^{σ} was the hydrogen solubility in stressed steel, C_{H}^{0} was the solubility in the unstressed steel. According to Liang's conversion [17], the total hydrogen concentration as a function of both the hydrostatic stress and the equivalent plastic strain was:

$$C_{\mathrm{H}}^{\sigma,\varepsilon} = C(\sigma_{kk}, \varepsilon^{p}) \tag{3}$$

where $C_{\mathrm{H}}^{\sigma,\varepsilon}$ was the hydrogen concentration in stressed and plastic straining solid, σ_{kk} was the hydrostatic stress and the ε^{p} was the effective plastic strain. At elastic condition,

$$\sigma_{kk} = \frac{2(1+\nu)}{(3\pi\nu)^{1/2}} K_I \cos\frac{\theta}{2} \tag{4}$$

where K_{I} was the mode I stress intensity factor, ν was possion ratio, r and θ were polar co-ordinates. If plasticity was included at the crack tip

$$\sigma_{kk} = \frac{2}{3}(1+\nu)\sigma_{y} \tag{5}$$

where σ_{y} was the yield strength. So it could be seen that when the steel was in the plastic zone, the hydrostatic stress and the plastic strain were large, and the hydrogen was more than that at elastic strain according to the Equation 3. The high content of hydrogen could influence the mechanical properties and the SCC of pipeline steels since the entropy and internal energy of pipeline steels could be altered by hydrogen. The presence of hydrogen and its interaction with stress could greatly increase the cracking propagation rate. Thus interaction of hydrogen and plastic strain facilitated SCC in near-neutral pH environments.

However, anodic dissolution could not be ruled out in the mechanism of SCC in near-neutral pH environments

[8, 18, 19]. It should be noted that the cracking caused by anodic dissolution and hydrogen involvement was not necessarily separate and distinct phenomena. Both processes could occur simultaneously, and might operate in parallel or sequentially to promote cracking.

5. Conclusion

SCC in pipeline steels in contact with near-neutral pH environments could become severe as the potential was reduced to very large cathodic protection potential. Precharging with high-pressure hydrogen could promote SCC due to the increase in hydrogen content. In pipeline field, plastic strain rather than elastic strain was necessary for SCC to occur in the environments used. The mechanism for near-neutral pH SCC might be plastic strain induced hydrogen entry that facilitated SCC. However, dissolution could not be ruled out of this mechanism.

Acknowledgements

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