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Comparison of hydrogen embrittlement susceptibility of three cathodic protected subsea pipeline steels from a point of view of hydrogen permeation

Timing Zhang^{a,b,**}, Weimin Zhao^{a,*}, Tingting Li^a, Yujiao Zhao^a, Qiushi Deng^a, Yong Wang^a, Wenchun Jiang^{b,**}

^a College of Mechanical and Electronic Engineering, China University of Petroleum (East China), Qingdao 266580, China

^b College of Chemical Engineering, China University of Petroleum (East China), Qingdao 266580, China

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ABSTRACT

Revealing the hydrogen permeation behavior in steel is the foundation to analyze hydrogen induced fracture. Herein, slow strain rate tension tests were used to investigate the susceptibility of X70, X80 and X100 steel to hydrogen embrittlement induced by cathodic polarization, and the subsurface hydrogen concentration (C_0) of three steels was accurately determined by two kinds of hydrogen permeation tests. Results showed that the HE index range of ductile-to-brittle transition should be determined by fracture analysis. The C_0 increased in turn from X70, X80 to X100 at any given applied potential, that's why X100 presented the highest hydrogen embrittlement susceptibility.

1. Introduction

The increasing energy requirement has driven the development of oil and gas transport pipeline around the world, many of which are located at offshore area [1]. However, the offshore environment is highly corrosive due to the presence of sea water. Corrosion is usually addressed through coatings and/or cathodic protection [2]. Generally, various coatings applied on pipeline surfaces can give a first line of defence against corrosion, but given the severity of the offshore environment, such as sea waves, tides, ocean currents [3], fatigue and microbial corrosion [4], these coatings would eventually detach from the pipeline steel with time. Considering the fact that offshore pipelines are often built to serve for several decades, the coating disbondment is not unexpected [2]. In contrast, the cathodic protection is an effective method for long-term protection, so cathodic protection or along with coating has been widely used to prevent subsea pipelines from corrosion. However, hydrogen induced brittle failure, i.e., hydrogen embrittlement (HE), poses a serious threat to the safety operation of pipelines under cathodic protection. One of the reasons for HE is too negative potential applied on steel substrate [5], which means that when the cathodic protection is improperly implemented, especially over protection, hydrogen reduction potential is more positive than the applied potential [6]. This could increase a risk of hydrogen

degradation during service, causing a brittle and unpredictable fracture which might have severe consequences [7]. Therefore, it is really necessary to study the susceptibility of pipeline steels to HE under cathodic protection.

Generally, slow strain rate tension (SSRT) test is widely used in a row of tests to evaluate the susceptibility of materials to HE. The degree of HE is usually assessed by the loss of reduction of cross-sectional area in test condition compared with that in a hydrogen-free atmosphere [8]. A general perception is that the ductility decreases with increasing strength while this inherent brittleness further enhances the susceptibility to HE [9]. But Cabrini [10] deemed that the resistance of steel to HE increases with its ultimate tensile strength until about 700 MPa, and then decreases. However, numerous studies suggested that microstructure, rather than the strength of the steels, plays the predominant role in the HE failing process [2]. Up to now, no agreement has been reached about the effect of different microstructures on HE susceptibility. It has been reported by some researchers that the microstructures containing higher hydrogen trap sites are more effective in reducing the susceptibility to HE because the diffusible hydrogen is the main factor influencing the susceptibility of material to HE [11,12]. For example, Hardie [11] found that the ductility loss was recoverable when a charged steel was left for several days at ambient temperature, because the diffusible hydrogen atoms had diffused out of the

* Corresponding author. Permanent address: College of Mechanical and Electronic Engineering, China University of Petroleum, 66 Changjiang West Road, Qingdao 266580, China.

** Corresponding authors.

E-mail addresses: zhangtm@upc.edu.cn (T. Zhang), zhaowm@upc.edu.cn (W. Zhao), jiangwenchun@upc.edu.cn (W. Jiang).

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specimens. However, other researchers hold the opinion that the higher the hydrogen traps in material, the lower the hydrogen diffusivity and the greater susceptibility of the material to HE [13,14]. The offshore pipelines are usually manufactured by high-strength low-alloy (HSLA) steels, which exhibit fine grain microstructures of ferrite and/or bainite [15–17]. However, the combined effect of these microstructures and hydrogen on the susceptibility of pipeline steels to HE is still a complicated problem [2].

Hydrogen permeation in steel is the precondition for hydrogen induced failure [8]. According to its small size, hydrogen atom may easily penetrate into steel, which leads to an immature failure of steel structure [18]. Therefore, hydrogen permeation tests are mostly used to reveal the hydrogen permeation behavior through steel substrates. The test devices reported in literatures are usually modified basing on Devanathan-Stachurski electrochemical cells [19,20], which are composed of a cathodic cell and an anodic cell, and a flat specimen is sealed between of them during test. Up to now, hydrogen permeation test method has been taken by a great number of researchers. Zheng [21,22] studied the effect of H₂S dissolved in aqueous solution on the hydrogen permeation behavior through pipeline steel in free corrosion condition. Besides, the hydrogen atoms can be introduced into material by cathodic polarization at a constant applied potential [23] or at a constant charging current density [24] in aqueous solution. In general, hydrogen diffusion through a steel is typically characterized by two different phenomena [1]: purely diffusion driven by concentration gradient, which is described by Fick's laws; reactions with specific microstructure defects, i.e., hydrogen traps, which have binding energies with hydrogen higher than lattice sites. Hydrogen diffusivity and sub-surface hydrogen concentration are two important parameters to reveal the permeation properties of hydrogen atoms through steel substrate [8]. Generally, the determination of hydrogen diffusivity in steel substrate is the precondition for the calculation of sub-surface hydrogen concentration [18,25], and the hydrogen diffusivity is commonly calculated using time-to-breakthrough and time-lag method according to reported investigations [1,13,26,27]. Because the hydrogen traps, such as grain boundaries [28], dislocations [29], inclusions and precipitates [8], are inevitably included in steel substrate, the hydrogen diffusivity or hydrogen concentration in steel obtained by hydrogen permeation test is essentially an apparent value, which takes into consideration the combined effects of hydrogen concentration gradient and hydrogen traps.

A review of the hydrogen diffusivities obtained by different authors on API pipeline steels, including grades ranging from X52 to X100, could be found in Fallahmohammadi's work [1]. However, the reported hydrogen diffusivities vary by a factor larger than 10. This may be attributed to the difference of electrochemical composition and the manufacture process of the steels. Besides, the following factors that influencing the variation of hydrogen diffusivity could not be negligible as well. One of the main causes of this variation is applying different calculation method by different researchers. The time-to-breakthrough and time-lag method reported in literatures are deduced from a given transient state of the hydrogen diffusion process. Generally, the hydrogen diffusivity obtained by time-to-breakthrough method is larger than that calculated by time-lag method. To the author's knowledge, the Fourier method and Laplace method [30] are more ideal for the calculation of hydrogen diffusivity, both of which take the whole transient permeation process into consideration, i.e., the hydrogen

diffusivity calculated using this two methods is an averaged value. Another unneglectable reason is the effect of surface condition on hydrogen diffusion behavior. It is worth noting that the electrochemical charging during hydrogen permeation test may lead to the formation of corrosion films at charging side of the specimen [21,22,31], so the diffusion path is actually a duplex system, which includes the surface corrosion film and the steel substrate. Thus, the hydrogen diffusivity obtained by electrochemical hydrogen permeation test cannot reflect the diffusion rate of hydrogen atoms through steel substrate, let alone the sub-surface hydrogen concentration [18,25]. In comparison, the surface condition of the steel specimen under hydrogen gas charging condition is relatively stable, and the hydrogen diffusivity obtained by this method could exactly reveal the diffusion rate of hydrogen atoms through steel, but the relevant studies were almost performed at high temperature [32,33]. For this reason, we have developed a new high-pressure gaseous hydrogen permeation system, which could be used at ambient temperature [8].

To date, the mostly used subsea pipeline is X65 grade, and the application of higher-strength steel is favorable for reducing pipe wall thickness, increasing operating pressure, and improving transportation efficiency. So the study of HE susceptibility and hydrogen permeation behavior in higher-strength steel is helpful to the selection of cathodic protection parameters. In this work, X70, X80 and X100 steels with typical ferrite and/or bainite microstructures were selected as test materials. SSRT tests at different applied potentials were performed to investigate the susceptibility of the three pipeline steels to HE. Subsequently, hydrogen permeation tests under high-pressure gaseous charging and electrochemical charging conditions were used to obtain the hydrogen diffusivity and sub-surface hydrogen concentration, respectively. Finally, the susceptibility of the three pipeline steels to HE was discussed from a point of view of hydrogen permeation properties.

2. Experiment details

2.1. Materials

The materials employed in this study were cut from the base metal area of API X70, X80 and X100 welded pipelines, and the main chemical composition analyzed by spectra is provided in Table 1. It can be seen that the content of all the elements meets the requirement of API 5L Specification. The carbon concentration for the three steels was relatively low, and the increased strength was mainly attributed to the addition of Mn and microalloying elements such as Nb, Ti, and V [34]. The microstructures of the three steels were examined with a LEICA DM2500M optical microscope (OM), as shown in Fig. 1. The X70 steel exhibited a massive ferrite microstructure with the grain size ranging from 5 μm to 20 μm, as shown in Fig. 1a. Compared with X70 steel, the Mo concentration in X80 and X100 steel was relatively higher (Table 1), which could suppress the formation of proeutectoid ferrite, and thus more bainite structures were obtained. For example, the microstructures of X80 were characterized by bainite and a little of massive ferrite, the grain size of which was same to that of X70 steel, as shown in Fig. 1b. For X100 steel, the microstructure was completely composed of bainite (Fig. 1c) [2,35,36]. Moreover, the concentration of Nb, Ti, and V in X100 steel was higher than that in X70 and X80 steel, these elements formed complex carbides, which could pin grain boundaries and prevent recrystallization and grain growth during steel

Table 1
Chemical composition (wt.%) of X70, X80 and X100 steel.

Steel	C	Si	Mn	S	P	Cr	Mo	Ni	Al	Cu	Nb	Ti	V
X70	0.05	0.21	1.58	0.001	0.009	0.26	0.06	0.018	0.041	0.016	0.08	0.011	0.001
X80	0.05	0.21	1.82	0.001	0.007	0.012	0.18	0.033	0.035	0.012	0.07	0.012	0.007
X100	0.06	0.16	1.89	0.001	0.005	0.28	0.24	0.27	0.041	0.02	0.08	0.016	0.016

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