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# Effect of environmental and metallurgical factors on hydrogen induced cracking of HSLA steels

Wan Keun Kim<sup>a</sup>, Seong Ung Koh<sup>b</sup>, Boo Young Yang<sup>b</sup>, Kyoo Young Kim<sup>a,\*</sup>

<sup>a</sup> Department of Material Science and Engineering, Pohang University of Science and Technology, San 31, Hyoja-Dong, Nam-gu, Pohang 790-784, Republic of Korea <sup>b</sup> POSCO Technical Research Center, Geidong-Dong, Nam-gu, Pohang 790-704, Republic of Korea

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# ABSTRACT

Hydrogen induced cracking (HIC) resistance of two high strength low alloy (HSLA) steel plates equivalent to API X70 grade was evaluated in various test solutions with different H<sub>2</sub>S partial pressures and pH values. Results showed that H<sub>2</sub>S partial pressure is the key parameter affecting HIC resistance. Hydrogen permeation rate was affected by both H<sub>2</sub>S partial pressure and pH of test solutions, whereas the apparent hydrogen diffusivity was determined mainly by pH value in case of H<sub>2</sub>S partial pressure less than 0.1 atm. HIC in the steels primarily nucleated at inclusions and/or clusters containing the Al and Ca oxides. HIC resistance was determined by diffusible hydrogen amount with different microstructures.

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### 1. Introduction

Hydrogen induced cracking (HIC) is a kind of the hydrogen embrittlement which occurs in the form of surface blisters and/ or internal cracks in the absence of applied stress [1]. HIC is developed by a sulfide corrosion process on the steel surface in the presence of hydrogen sulfide in the solution. The most accepted corrosion reactions of steel exposed to a sour gas are:

Anodic reaction : 
$$Fe \rightarrow Fe^{2+} + 2e^{-}$$
 (1)

Dissociation reactions :  $H_2S \rightarrow H^+ + HS^-$ 

$$\mathrm{HS}^{-} \to \mathrm{H}^{+} + \mathrm{S}^{2-} \tag{3}$$

$$\begin{array}{ll} \mbox{Cathodic reaction}: & 2H^+ + 2e^- \to 2H \ (\mbox{atomic hydrogen}) \\ & \to H_2 \uparrow_{(gas)} \end{array} \tag{4}$$

The H<sup>+</sup> ions present in acidic solution or produced by dissociation reactions in neutral and alkali solution combine at the cathode with electrons, released by the steel, to form atomic hydrogen on the steel surface. The hydrogen atoms combine to form molecular gaseous hydrogen; however, the presence of hydrogen sulfide gas (H<sub>2</sub>S) in acidic solution or hydrogen sulfide ions (HS<sup>-</sup>) in neutral and alkali solution reduces the rate of hydrogen gas formation on the steel surface [2-4]. Thus, some amount of atomic hydrogen diffuses into the steel. The diffused hydrogen encounters trapping sites and is collected there, forming hydrogen gas at very high pressures. The hydrogen trapping sites are metallurgical defects, such as non-metallic inclusions, large precipitates and bands of hard microstructures. Generally, these sites are planar and oriented in the direction parallel to the rolling direction of pipeline steels. As hydrogen is collected at the trapping sites, the high pressure developed causes a stress concentration at the edge of the site. Consequently, a crack is formed and it propagates in the direction parallel to the rolling direction of pipeline steels [2].

Susceptibility of steels to HIC is closely related with metallurgical parameters, especially distribution of defects such as nonmetallic inclusions and secondary phases. Another important parameter affecting HIC is the environment to which steels are exposed. The test environment of NACE TM0284 standard HIC test method is unnecessarily severe compared to the actual sour service conditions. In order to use pipeline steels properly according to actual service environments, it is necessary to evaluate HIC resistance of steels in the practical range of environmental severity [5]. The environmental factors critically affecting HIC are considered to be pH and H<sub>2</sub>S partial pressure, P<sub>H<sub>2</sub>S</sub>, of in-service environments because both the pH and  $P_{H_2S}$  affect the diffusion of hydrogen atoms in steels [6,7]. However, the effect of such factors on the HIC of HSLA steels is not clearly understood.

In this study, the effect of pH and  $P_{H_2S}$  of test solution on HIC resistance of two HSLA steel plates was studied in terms of hydrogen diffusion. HIC nucleation and propagation were investigated by metallurgical factors such as microstructure, inclusion and secondary phase.





<sup>\*</sup> Corresponding author. Tel.: +82 54 279 2134; fax: +82 54 279 4499. E-mail address: kykim@postech.ac.kr (K.Y. Kim).

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а

b

# 2. Experimental procedures

## 2.1. Specimens and HIC test

Two HSLA steel plates which belong to X70 grade according to API 5L specification were used as specimens of Steel A and B, composed of 0.03 wt.%C-0.25 wt.%Cr and 0.05 wt.%C-0.25 wt.%Cr, respectively. These two steel specimens were taken from the commercial products of API X70 grade linepipe steel. They were continuously casted in the commercial steel mill and reheated to the temperature range of 1150–1200 °C. In the rolling mill, a thermomechanically controlled process (TMCP), combination of the controlled rolling and on-line accelerated cooling process, was applied to produce the steel plates. The accelerated cooling process was performed by water spray on the steel in the temperature range of 800–500 °C.

HIC tests were performed for 96 h in nine different test solutions using varied combinations of  $P_{H_2S}$  and pH value as listed in Table 1. The solution with pH 2.7 is a NACE TM0284. A solution containing 5 wt.% NaCl and 0.5 wt.% CH<sub>3</sub>COOH dissolved in distilled water, and the solutions with pH 4.0 and 5.0 are buffer solution which compensated by adding sodium acetate (CH<sub>3</sub>COONa) in the NACE TM0284 A [8].  $P_{H_2S}$  means the percent concentration of H<sub>2</sub>S gas which is included in N<sub>2</sub> gas. After HIC testing, cracking sensitivity was measured in terms of crack area ratio (CAR) by using ultrasonic detector.

The microstructural details of tested steels such as size and distribution of inclusions were investigated. Fracture surfaces were analyzed using optical microscope (OM) and scanning electron microscope (SEM) in order to examine the crack initiation and propagation. To determine the chemical composition of inclusions which acted as HIC nucleation sites, energy dispersive spectroscopy (EDS) was used.

## 2.2. Hydrogen measurement

Table 1

### 2.2.1. Electrochemical hydrogen permeation test

To investigate the hydrogen diffusion behavior in steel, hydrogen permeation tests were performed with nine different solutions similar to HIC tests. The tests were conducted in a dual cell called a "modified Devanathan-Stachurski cell" as shown in Fig. 1a. This cell is composed of two compartments, a hydrogen generating cell (input side) and a hydrogen oxidizing cell (detection side). In the hydrogen input side, hydrogen atoms enter the steel by diffusion while the amount of hydrogen diffused through the steel plate is measured in term of oxidizing current density in the detection side [9]. The input side is filled with test solutions, and potential is not applied. On the other hand, the detection side is filled with 0.1 N NaOH solution, and an overvoltage of 250 mV<sub>SCE</sub> is applied in order to oxidize the diffused hydrogen. The thin Pd layer was electroplated on the detection side of the specimens in PdCl<sub>2</sub>  $5.08 \text{ g} + \text{NH}_4\text{OH}$  1 L solution [10]. The thickness of specimens is 1 mm. Hydrogen permeation rate (HPR) was measured by the hydrogen oxidation current density.

The apparent hydrogen diffusivity in steels can be determined by breakthrough time method or relaxation time method [11]. In this study, the breakthrough time method was used to determine the hydrogen diffusivity for the tested steels and the relationship

Test solution chemistry.								
Solution no.	1	2	3	4	5	6	7	8
P <sub>H2S</sub> (atm) pH	1 2.7	1 4.0	1 5.0	0.1 2.7	0.1 4.0	0.1 5.0	0.01 2.7	0.01 4.0

9

0.01

5.0



Potentiostat

0.1N NaOH

H-detection side

 $V_1$ 

Steel membrane

Rolling direction

NACE

TM0284-96 solution

H-input side

Cock

Fig. 1. Schematics for hydrogen measurements; (a) modified Davanathan–Stachurski cell and (b) diffusible hydrogen measurement apparatus.

Hot bath

between the apparent hydrogen diffusivity ( $D_{app}$ ), thickness of specimen (L) and breakthrough time ( $t_b$ ) is as follows;

$$D_{\rm app} = 0.5^{L^2} / \pi^2 t_{\rm b} \tag{5}$$

Hydrogen permeation tests were performed only for the steel which exhibited HIC according to severity of testing environments since one of the objectives in this study was to investigate the effect of hydrogen diffusion on HIC level (in terms of CAR) of tested steels according to severity of testing environments.

# 2.2.2. Diffusible hydrogen amount measurement

The diffusible hydrogen amount included in two steels was measured by the modified JIS Z3113 method as presented in Fig. 1b [12]. To determine the critical hydrogen amount value which initiates HIC, hydrogen charging methods were applied differently according to HIC sensitivity of the steels. Since HIC did not occur in steel A in sour service tests, a forced hydrogen charging method was applied. Cathodic hydrogen charging was performed for 48 h by applying current to the specimen, steel A. For steel B, hydrogen charging was conducted by controlling pH and  $P_{H_{2S}}$  like HIC tests. After the hydrogen charging, specimens were inserted into glycerin immediately. During the process, ice water was used as a medium to prevent hydrogen escape from the specimens. The processing time was kept to less than 2 min. After confirming discharging of all diffusible hydrogen from the specimen for two days,

-H<sub>2</sub>S

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