Effect of Heat Treatment on Hydrogen Diffusion and Hydrogen Induced Cracking Behavior of Process Pipe Steel in Sour Environment

[1]Do Kyeong Han, [2]Hwan Gyo Jung, [1]Kyoo Young Kim and [1]Sung Jin Kim
[1]Graduate Institute of Ferrous Technology, Pohang University of Science and Technology
San 31, Hyoja-Dong, Nam-gu, Pohang 790-784, Republic of Korea
[2]POSCO Technical Research Laboratories
Geidong-Dong, Nam-gu, Pohang 790-300, Republic of Korea

ABSTRACT

Influence of heat treatment on the HIC susceptibility in the process pipe steel was investigated with respect to the hydrogen diffusion behavior by employing the electrochemical permeation technique. In order to examine the change in morphology of precipitates in acicular ferrite after the heat treatment, extraction replica technique for TEM analysis was utilized. This study clearly shows that, contrary to other microstructures, in acicular ferrite, iron carbide (Fe_3C) particles are newly precipitated mostly along the grain boundaries by the simulated PWHT conducted at 620 ^oC for 1 hour. In addition, decrease in hydrogen diffusivity attributed to the hydrogen trapping effect by the newly generated Fe_3C leads to an increase in apparent hydrogen solubility in the steel. As a result, the heat treated process pipe steel is more susceptible to HIC.

KEY WORDS: Process pipe steel; sour environment; PWHT; acicular ferrite; Fe_3C precipitate; hydrogen induced cracking; hydrogen permeation;

INTRODUCTION

The process pipe steels used in the petrochemical industry suffer frequently from hydrogen induced cracking (HIC) failures when they are used in a sour environment. [Bruno, 1999] With the depletion of high quality oil and gas, the HIC becomes severe since low quality oil and gas contains a large amount of H_2S which is the major source of diffusible hydrogen in the steel. The atomic hydrogen which is reduced from H^+ ion dissociated from H_2S tries to become the hydrogen molecule by the recombination reaction ($H + H \rightarrow H_2$). In the presence of H_2S, however, the recombination reaction is suppressed and the hydrogen atoms are easily diffused into the steel matrix. The diffusible hydrogen atoms are trapped at various metallurgical defects in the steel such as grain boundaries, dislocations and interfaces between the steel matrix and non-metallic inclusions. As a result, the HIC failures can occur. [NACE MR0175-1993]

The HIC failures in the steel have been reduced greatly by applying advanced steel making technology. Especially, proper procedures of Ca-treatment for inclusion shape control have proved to increase the HIC resistance. [Cicutti, 1997] [Domizzi, 2001] In addition, numerous efforts have been made to develop the sour-resistant steel with optimal microstructures by employing various heat treatment processes. A considerable body of literature has reported for the relationship between microstructures obtained by the different heat treatment conditions and HIC resistance in the sour environment. [Koh, 2004] [Park, 2008] [Torres-Islas, 2008] [Beidokhti, 2009] It has been generally accepted that the heat treatment such as tempering and post-weld heat treatment (PWHT) conducted normally at 500-600 ^oC has a beneficial effect on subsequent HIC resistance in a sour environment. [Kim, 2008] [Robertson, 1980] [Serna, 2005] [Carneiro, 2003] Particularly, Luppo et al. and Tau et al. have reported that the tempering treatment conducted to toughen the low-temperature transformation microstructure, such as martensite and bainite, improves HIC resistance due mainly to reduction in dislocation density, hardness and residual stress. [Luppo, 1991] [Tau, 1996] Furthermore, Kim et al. have investigated influence of PWHT conducted at 620 ^oC on hydrogen diffusion and HIC behavior in the pressure vessel steel by means of the electrochemical permeation technique. [Kim, 2011] They indicated that the heat treatment can be effective in reducing the HIC susceptibility of the steel. This is closely related with the agglomeration of fine carbide particles (Fe_3C) to coarse-sized particle during the heat treatment. It leads to decrease in the total interfacial area of ferrite/Fe_3C acting as a reversible trapping site for hydrogen atom, resulting in low apparent hydrogen solubility in the steel. These phenomena can be identified in a variety of microstructures of the steel ranging from ferrite/pearlite to bainite. [Luppo, 1991] [Radhakrishnan, 1967] [Parvathavarthini, 2001] However, recent study conducted by the present authors has observed an opposite phenomenon in the process pipe steel having acicular ferrite. [Kim, 2012a] The study has revealed that the heat treatment conducted at 620 ^oC for 1 hour on the pipe steel leads to an increase in diffusible hydrogen content in the steel that makes it more prone to HIC.

It is frequently required for the process pipe steel to conduct PWHT at 620 ^oC on welded areas to relieve residual stress after welding process. However, it is technically difficult to do PWHT selectively in the welded areas alone due to the limitation of the process in real field. Consequently, the base metal consisting of acicular ferrite can also be affected by the heat treatment. Although there have been numerous studies concerning the effect of the heat treatment on HIC in the weld metal and heat-affected zone (HAZ), there has been rarely reported that the heat treatment increases the HIC susceptibility in the base metal of the process pipe steel having the acicular ferrite. Moreover, effect of the heat treatment on the hydrogen diffusion behavior in the steel was not conducted in our previous work. [Kim, 2012a] It means that the relationship among the heat treatment, hydrogen diffusion behavior and HIC susceptibility in the process pipe steel have not been clearly established yet.

Therefore, in the present study, effect of heat treatment conducted at 620 ^{o}C on the susceptibility to HIC in the process pipe steel having acicular ferrite was investigated with respect to the hydrogen diffusion behavior by utilizing the electrochemical permeation technique.

EXPERIMENTAL PROCEDURE

Specimen Preparation and Microstructure Analysis

The testing material used in this research was equivalent to API (American Petroleum Institute) X70 grade steel plate containing 0.03 wt.% C. A TMCP was performed to produce the 25 mm thick steel plates and form acicular ferrite in the microstructure. The detailed chemical composition of the tested specimen is listed in Table 1. Yield strength of the steel is around 485 MPa which satisfies the strength requirement of API X70 grade steel.

In order to simulate the PWHT conducted in the real field, a heat treatment was conducted at 620 ^{o}C for 1 hour.

For microstructure observation, the two specimens (the heat-treated and non-heat treated specimen) were ground up to 1,200 grit paper and polished with 0.25 μm diamond suspension. After degreasing with ethanol, the samples were etched using 5 vol.% nital etchant. The microstructure was then examined by using field-emission scanning electron microscope (FE-SEM).

In addition, to examine the precipitates in the steels by transmission electron microscopy (TEM), the extraction replica technique was used. The specimen was polished and etched in AA solution composed of 890 ml of methanol, 100 ml of acetylacetone and 10 g of tetramethyl ammonium chloride. The surface was then coated with carbon and it was removed from the surface using the AA solution. The replicas were then placed on copper grids and examined by TEM.

HIC Test

The HIC test in reference to NACE TM0284 was carried out to evaluate the effect of the heat treatment on the subsequent HIC resistance of the steel. [NACE TM0284-2003] After the test, ultrasonic detector was employed to measure size and distribution of the HIC cracks in the tested specimens. The HIC occurrence level was measured in terms of the crack length ratio (CLR). The amount of diffusible hydrogen was also measured after the HIC test in reference to JIS Z3113 method. [JIS Z3113-1975] For the reproducibility of data, five independent HIC tests were performed, followed by the measurement of the diffusible hydrogen content in the steel.

Electrochemical Hydrogen Permeation Test

The hydrogen permeation behavior through the steel membrane was electrochemically measured by using the Devanathan and Stachurski cell. [Devanathan, 1962] The electrochemical hydrogen permeation test was performed in reference to ISO17081 standard. [ISO17081- 2004]

The prepared steel membrane, with 1 mm thickness, was assembled between two electrochemical permeation cells and argon (Ar) gas was continuously applied to both sides of the steel sheet to suppress surface oxidation. Then, the thin palladium (Pd) film was electrochemically deposited to the detection side of the steel membrane. Detailed deposition method is explained in the following subsection.

After the deposition, the hydrogen detection cell was assembled with the steel membrane. The detection side was then filled with 0.1M NaOH solution purged with Ar gas. The Pd-plated surface exposed to the solution was maintained at a potential of 250 mV_{SCE}, at which the dominant reaction was oxidation of hydrogen atoms. Once the background current reached a steady state below 1 $\mu A\cdot cm^{-2}$, NACE TM0284-96A solution (a mixture of 5.0 wt.% NaCl and 0.5 wt.% glacial acetic acid dissolved in distilled water) was poured into the hydrogen charging cell and H_2S gas was purged continuously during the test.

The cathodic current density of 1.5 $mA\cdot cm^{-2}$ was applied to the specimen to prevent from formation of a sulfide film like FeS or any other corrosion products on the steel surface. The hydrogen oxidation current in the detection side was measured continuously as a function of time during the permeation test. Volume of the testing solutions was around 500 ml for each chamber and it was enough to maintain constant concentration during the test. Testing temperature was kept constant at 25 ^{o}C. Simple schematics of the permeation cell and electrical connections are shown in Figure 1.

Electrodeposition of Thin Palladium Film

The Pd film was electrochemically deposited on the steel surface in the hydrogen detection side to increase the efficiency of the hydrogen oxidation reaction ($H \rightarrow H^{+} + e^{-}$) by minimizing the hydrogen recombination. The hydrochloric acid was used as an acid cleaning solution. The prepared coating bath was composed of a mixture of 2.54 g of $PdCl_2$ and 500 ml of 28% aqueous ammonia solution. A galvanostatic current density of 2.83 $mA\cdot cm^{-2}$ was cathodically applied for 2 minutes.

Data Analysis

The hydrogen diffusion parameters of the effective diffusivity, permeability and apparent solubility are calculated after the hydrogen permeation test.

The hydrogen flux (mol $H\cdot m^{-2}\cdot s^{-1}$) through the steel membrane is measured in terms of steady-state current density, I_{ss} ($A\cdot m^{-2}$), and converts into hydrogen flux according to the following equation 1

$$J_{ss} = I_{ss} / nF \qquad (1)$$

The permeability, $J_{ss}L$ ($mol\cdot s^{-1}\cdot m^{-1}$) is calculated by the following equation 2

$$J_{ss}L = I_{ss}L / nF \qquad (2)$$

Where L is the thickness of the sample, n is the number of the electrons that are participated in the electrochemical reactions and F is the Faraday's constant.

The effective diffusivity, D_{eff} ($m^2\cdot s^{-1}$) is calculated by the time-lag method.

$$D_{eff} = L^2 / 6t_{lag} \qquad (3)$$

The t_{lag} is the time to achieve when $J(t)/J_{ss}$ becomes 0.63.

The apparent solubility, C_{app} ($mol\cdot m^{-3}$) is determined by applying Fick's 1st law which is for the time independent diffusion.

$$S_{app} = J_{ss}L / D_{eff} \qquad (4)$$

Table 1 Chemical composition of the tested specimen (wt.%)

Chemical Composition (wt.%)								
C	Mn	Si	P+S	Al	Nb+Ti+V	Cu+Ni	Ca	Cr+Mo
0.03	1.22	0.21	0.006	0.035	0.06	0.14	0.003	0.23

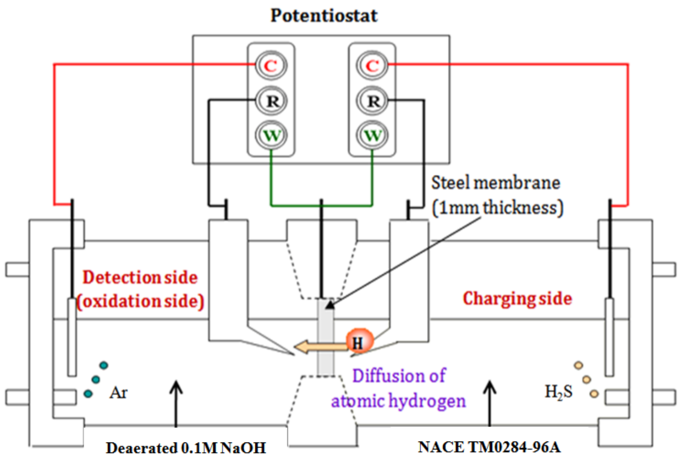
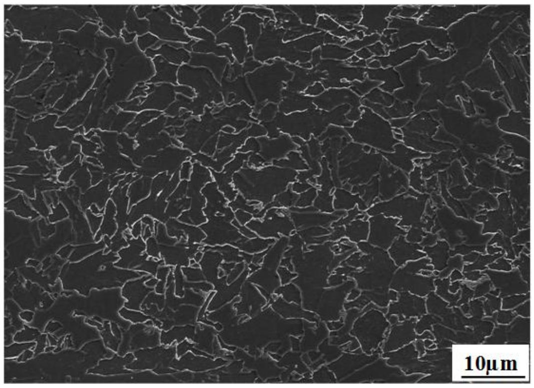


Figure 1 Simple schematic showing the hydrogen permeation test cell and electrical connections.

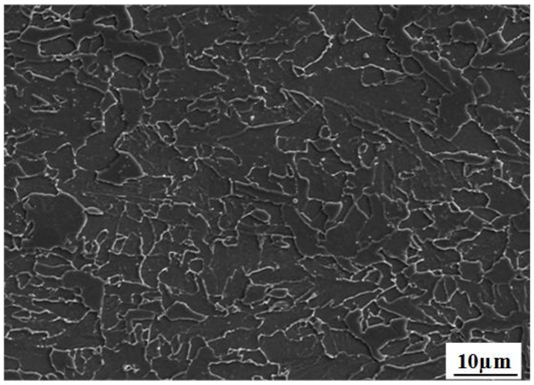
RESULTS AND DISCUSSION

Microstructure

The typical microstructure of the API X70 grade process pipe steel after the 5 vol.% nital etching is shown in Figure 2. The FE-SEM images indicate that there is no significant difference in microstructure of the steel irrespective to the heat treatment. The microstructure is primarily composed of acicular ferrite with an average grain size of 7.5 µm.



(a) Non-heat treated

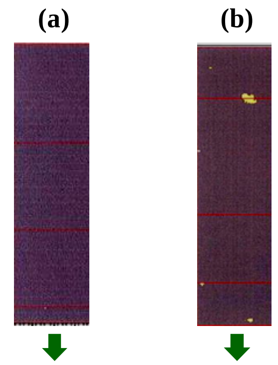


(b) Heat treated

Figure 2 Typical microstructure of (a) non-heat treated and (b) heat treated steel observed by FE-SEM.

HIC Resistance and Diffusible Hydrogen Contents

The results of the HIC test in reference to NACE TM0284 standard testing method are presented in Figure 3. [Kim, 2012a] For the reproducibility and reliability of the test result, five independent HIC tests were conducted. As shown in Figure 3, it is not possible to observe the HIC crack by ultrasonic detector for the non-heat treated specimen, whereas HIC is detected from the heat treated steel. It suggests that the heat treatment increases the subsequent HIC susceptibility in the steel when exposed to the sour environment. The HIC cracks occurred mainly near the surface of the heat treated steel and the following table shows the average crack length ratio (CLR) value. Even though difference in the average value of CLR is not large, it is clearly identified that the additional heat treatment conducted at 620°C for 1 hour increases the susceptibility to HIC.



	(a) Non-heat treated	(b) Heat treated
CLR (%)	0	5.03

Figure 3 Level of HIC occurrences of the (a) non-heat treated and (b) heat treated steel detected by ultrasonic analysis. [Kim, 2012a]

The HIC susceptibility is also evaluated by the amount of diffusible hydrogen in the steel. After the HIC test, the amount of absorbed diffusible hydrogen was measured by the modified JIS Z3113 method and the result is presented in Figure 4. The error bar indicated in the Figure 4 means the average value of five independent test data. According to this data, it is considered that the measured diffusible hydrogen content is larger in the heat treated steel than in the non-heat treated steel. The HIC occurrence level and the diffusible hydrogen contents in the steel before and after the heat treatment can also be identified in our recent investigation. [Kim, 2012a]

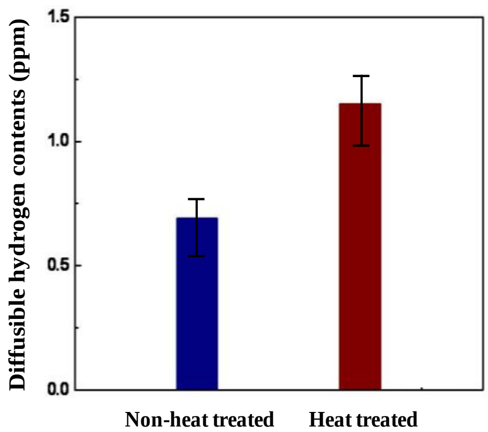
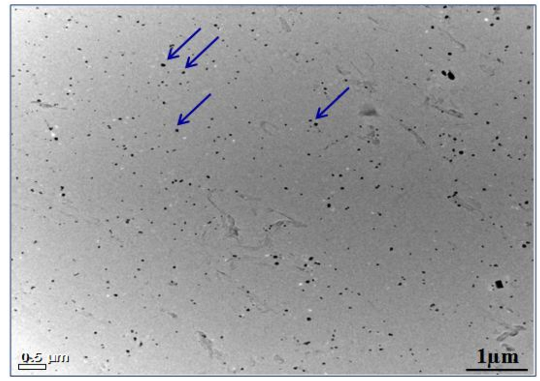


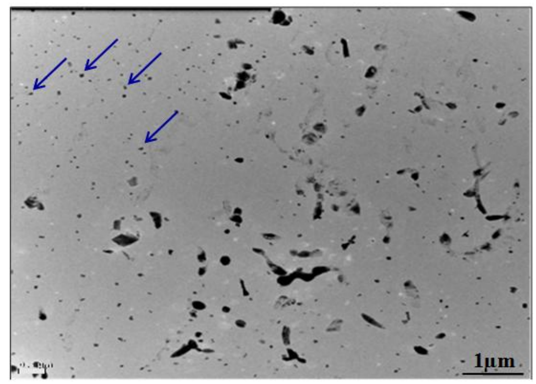
Figure 4 Diffusible hydrogen content in the steel after the HIC test. [Kim, 2012a]

There have been many studies to identify the effect of additional heat treatment such as PWHT or tempering on hydrogen assisted cracking phenomenon. They have found that the heat treatment leads to decrease the diffusible hydrogen content in steels and increase the HIC resistance when it is subsequently exposed to the sour environment. It is generally believed that the heat treatment such as tempering conducted normally at 500-600 ^{o}C decreases the hydrogen trapping effect caused by the reduction in pre-existed dislocations acting as reversible trapping site for hydrogen atoms. A lot of steels consisting of various microstructures ranging from ferrite/pearlite, bainite to martensite have been evaluated and consistent results have been obtained. However, this study demonstrates the totally different behavior in the process pipe steel having acicular ferrite. The diffusible hydrogen content in the heat treated steel is larger than that in the non-heat treated steel. Since it is considered that larger amount of diffusible hydrogen is closely related with higher number of reversible trap density for hydrogen in steel, it is expected that, contrary to other microstructures, in acicular ferrite, hydrogen trapping sites are increased by the heat treatment. This may be caused by the increase in newly-generated hydrogen trapping sites which overtake the decrease in pre-existed trapping sites during the heat treatment. To clearly identify the newly generated hydrogen trapping site in the acicular ferrite during the heat treatment, the extraction replica technique was utilized and fine sized precipitates were observed.

Figure 5 represents the TEM images of precipitations before and after the heat treatment and the difference in morphology of precipitates are clearly observed. This shows that, after the heat treatment, larger sized precipitates over 200 nm were newly generated in the microstructure. In contrast, there is no big change in the morphology of fine sized precipitates of around 40 nm, as marked in the Figure 5 by blue arrows. [Kim, 2012a]



(a) Non-heat treated



(b) Heat treated

Figure 5 TEM images of (a) non-heat treated and (b) heat treated steel attained by the extraction replica technique. [Kim, 2012a]

Those fine and large sized precipitates were characterized by energy dispersive spectroscopy (EDS) analysis and the results are presented in Figure 6. [Kim, 2012a] As shown in Figure 6 (a) and (b), the fine sized precipitates are identified as TiC or TiNb(C,N). In contrast, the large sized precipitations are identified as iron carbide (Fe_3C) which has been reported as reversible trapping site for hydrogen atoms. In general, fine sized precipitates such as Tic or TiNb(C,N) is classified as the irreversible trapping sites for hydrogen atoms and they are not closely related with the hydrogen related problem. [Zhao, 2003] Although coarse sized precipitates are incoherent to the steel matrix and they can act as crack initiation site, nano-sized precipitates are not actively involved in cracking or embrittlement process. Therefore, the increase in diffusible hydrogen content in the steel and HIC susceptibility after the heat treatment is mainly attributed to the newly generated coarse sized iron carbide, acting as reversible trapping site for hydrogen atoms.

The superior resistance to HIC of the steel having acicular ferrite comes from the fine grain size and the absence of precipitates such as Fe_3C in the microstructure. Particularly, the absence of carbide in acicular ferrite is resulted from TMCP involving the accelerated cooling process in the manufacturing process. It is believed that the absence of carbide in the microstructure decreases the hydrogen trapping effect, resulting in high resistance to the cracking and embrittlement phenomena. However, the iron carbide particles are newly precipitated in the acicular ferrite after the heat treatment. It suggests that the carbon atoms supersaturated in acicular ferrite due to the accelerated cooling process are gradually diffused and precipitated mostly along the grain boundaries in the form of Fe_3C. Since the hydrogen atoms are reversibly trapped at the interface between the iron carbide and the steel matrix, [Jeng, 1990][Johnson, 1987][He, 1996][Hong, 1983][Parvathavarthini, 1999] higher number of carbide precipitates is expected to increase the diffusible hydrogen content in steel and HIC susceptibility. The change in hydrogen trapping process before and after the heat treatment is the complete opposite of that in other microstructures such as ferrite/pearlite or bainite structure. In order to form those microstructures, the TMCP is not needed during the manufacturing process and therefore the carbon atoms are not supersaturated in the matrix but they are precipitated in the form of carbide or pearlite depending on the cooling rate. In this case, additional heat treatment leads to the agglomeration of the pre-existing carbide particles to form larger particles or become the spheroidized carbides. Some researchers have found that the agglomeration of the carbide particles leading to decrease in total interfacial area acting as hydrogen trapping site results in increase in hydrogen diffusivity and decrease in apparent hydrogen solubility in steel. [Jeng, 1990] [Johnson, 1987] Although morphology of the carbide particles would be different depending on the heat treatment condition, it is apparent that the heat treatment conducted at 620 ^{o}C for 1 hour leads to the emergence of Fe_3C in the acicular ferrite. Consequently, the heat treated steel can be more prone to HIC problem when exposed to sour environment.

Nevertheless, the influence of the change in hydrogen trapping site in the acicular ferrite on hydrogen diffusion behavior has not been clearly understood. Therefore, in the following subsection, the difference in hydrogen diffusion behavior in the steel before and after the heat treatment is discussed by utilizing the electrochemical hydrogen permeation technique.

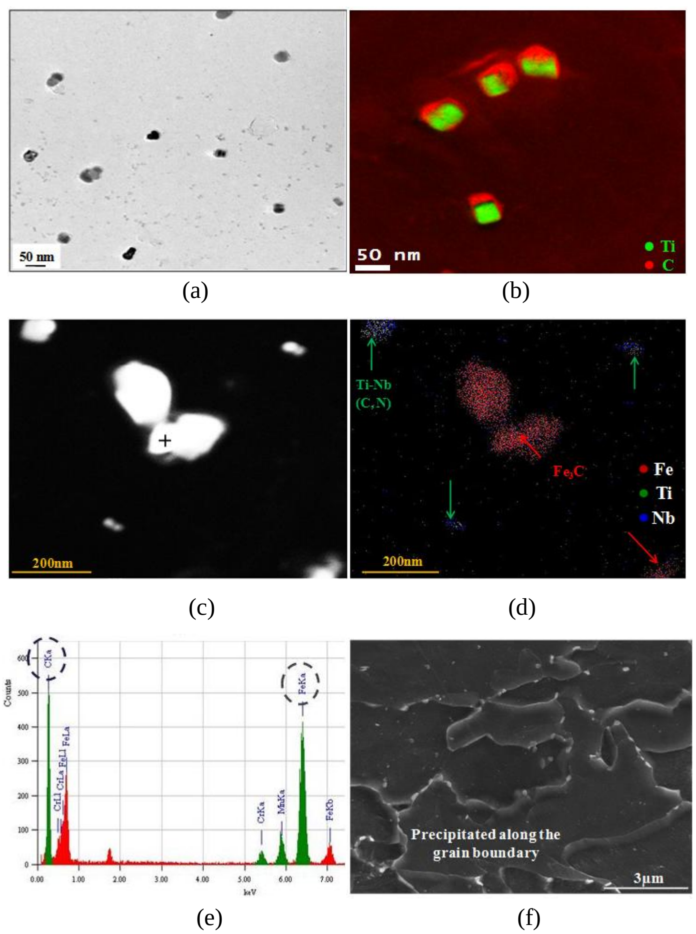


Figure 6 Morphology and EDS analysis of the (a), (b) small sized precipitates and (c)-(f) large sized precipitates in the heat treated steel [Kim, 2012a]

Hydrogen Permeation Behavior

Influence of the heat treatment on the hydrogen diffusion parameters was clearly evaluated by the electrochemical permeation technique. Figure 7 shows typical hydrogen permeation transients for the tested specimens, which represent standard deviation of permeation transient value out of 5 independent experiments. Before analyzing the permeation data, the Pd-plated surface on the detection side of the steel membrane was observed to identify the stability of the Pd film. Since the reliability of the permeation data depends highly on the characteristics of the Pd coating layer, the uniform deposition of the coating is one of the essential factors in the success of the permeation test. [Kim, 2012b] The experimental evidence for this phenomenon can also be identified in our previous work. [Park, 2011] Figure 8 shows morphology of the Pd film. There are no pores and defects in the coating layer and the steel surface is fully covered with small Pd particles. It suggests that the hydrogen oxidation rate is high enough to obtain the reliable permeation data. The hydrogen diffusion parameters determined from the average permeation data are listed in Table 2. Effective hydrogen diffusivity (D_{eff}) was calculated by the break-through method and other diffusion parameters were determined in reference to ISO17081. During the hydrogen permeation test, the cathodic current is continuously applied and the sample surface was kept clean, indicating that any physical diffusion barrier, such as iron sulfide film was not generated.

Table 2 indicates that the heat treatment decreases the effective hydrogen diffusivity (D_{eff}) and increases the apparent hydrogen solubility (S_{app}) in the steel. The effective hydrogen diffusivity has been known as a function of trap density and magnitude of the trap depth in the steel matrix. [Oriani, 1970] Considering this fact, the decrease in effective diffusivity for the heat treated steel suggests that the hydrogen diffusion path is lengthened or the amount of reversible hydrogen trapping site is increased in the steel which is in a good agreement with the TEM observation. The emergence of Fe_3C by the heat treatment provides higher amount of trapping sites in the acicular ferrite and delays the transport of diffusible hydrogen in the steel. However, it is hard to figure out the difference in steady-state current density between two curves which means that there is no significant difference in the permeability ($J_{ss}L$) irrespective to the heat treatment. Gel'd et al. have also proved that the hydrogen permeability depends greatly on the microstructure obtained by the different heat treatment. [Gel'd, 1965] Since there is no big difference in the microstructure between two tested steels, the calculated permeability values are almost the same for both heat treated and non-heat treated steels.

The Fick's law informs that there is inverse relationship between the effective diffusivity and apparent solubility. It means that the lower effective diffusivity yields the higher apparent solubility only when the hydrogen permeability is constant. Since the heat treated steel shows delayed break-through time compared to the non-heat treated steel, the heat treatment decreases the effective hydrogen diffusivity and increases the apparent hydrogen solubility in the steel. This is in a good agreement with the results obtained by the modified JIS Z3113 method.

This study demonstrates that, contrary to other microstructures, in acicular ferrite, the iron carbide (Fe_3C) particles are newly generated and precipitated mostly along the grain boundaries by the heat treatment. In addition, slower diffusion of hydrogen atoms due to the trapping effect by the interface between Fe_3C and steel matrix leads to an increase in apparent hydrogen solubility in the steel. As a result, the steel can be more prone to HIC.

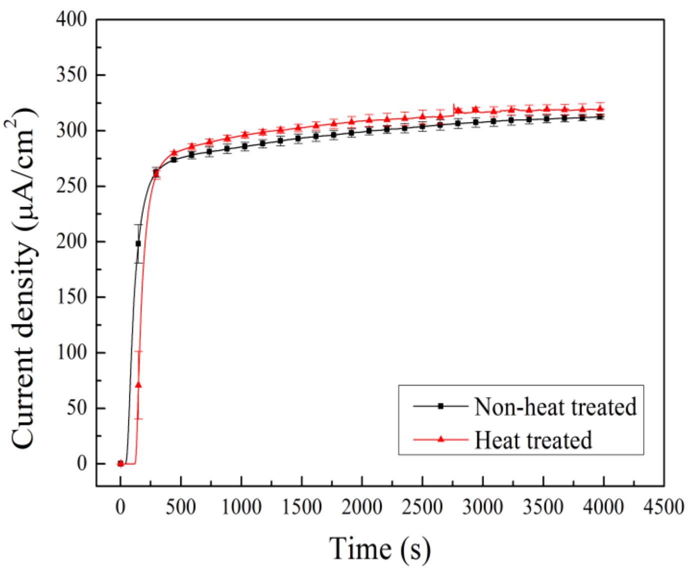
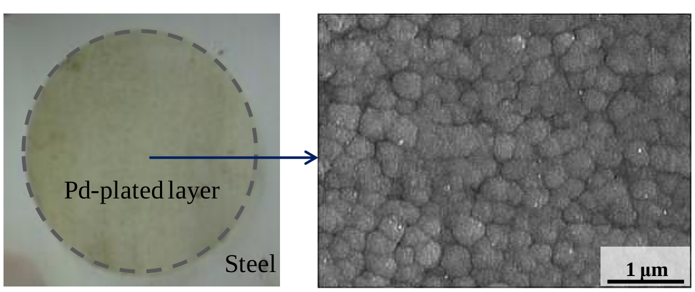


Figure 7 Hydrogen permeation curves of the steels depending on the heat treatment

Table 2 Hydrogen diffusion parameters determined after the hydrogen permeation test
(D_{eff}: effective diffusivity, $J_{ss}L$: permeability, S_{app}: apparent solubility)

Specimen	Non-heat treated	Heat treated
$\mathbf{D_{eff}}$ (x10^{-9} $m^2 \cdot s^{-1}$)	1.46	0.46
$\mathbf{J_{ss}L}$ (x10^{-8} $mol \cdot m^{-1} \cdot s^{-1}$)	2.98	3.05
$\mathbf{S_{app}}$ (x10 $mol \cdot m^{-3}$)	2.05	6.56



(a) Low magnification (b) High magnification

Figure 8 Surface morphology of Pd-plated layer examined by: (a) digital camera and (b) FE-SEM

CONCLUSIONS

Effect of the heat treatment on the hydrogen diffusion behavior of the API X70 grade process pipe steel exposed to aqueous sour environment was investigated in terms of the change in hydrogen trapping sites. The experimental results obtained in this study are summarized as follows:

1) The microstructure is consisted of acicular ferrite with an average grain size of 7.5 μm. There is no observable difference in the microstructure irrespective of the heat treatment.

2) The heat treatment conducted at 620 °C for 1 hour leads to increase in diffusible hydrogen content in the steel and decrease in HIC resistance when the steel is subsequently exposed to the sour environment.

3) The TEM observation and EDS analysis indicate that the precipitates with a size around 200-600 nm is newly generated and precipitated mostly along the grain boundaries after the heat treatment. This means that the supersaturated carbon atoms in the acicular ferrite matrix are gradually diffused and precipitated in the form of Fe_3C by the heat treatment. Since those newly generated carbides act as reversible hydrogen traps, the diffusible hydrogen content increases and the HIC susceptibility decreases when the steel is exposed to the sour environment.

4) The change in hydrogen diffusion and trapping process after the heat treatment was verified by utilizing the electrochemical permeation technique. The hydrogen permeation transient shows that the effective hydrogen diffusivity (D_{eff}) is decreased after the heat treatment. The slower diffusion of hydrogen atoms due to the trapping effect by the interface between Fe_3C and steel matrix leads to an increase in apparent hydrogen solubility (S_{app}) in the steel. This is in a good agreement with the result obtained by the modified JIS Z3113 method. These results suggest that the heat treatment modified the hydrogen trapping process in the acicular ferrite that makes the process pipe steel more susceptible to HIC.

REFERENCES

Beidokhti, B, Dolati, A, and Koukabi, AH (2009) "Effects of alloying elements and microstructure on the susceptibility of the welded HSLA steel to hydrogen-induced cracking and sulfide stress cracking" *Materials Science and Engineering* A507, No 1, pp 167-173.

Bruno, TV, Christensen, C, and Hill, RT (1999). "History and development of TM0284" *NACE Corrosion* 99, paper 99422.

Carneiro, RA, Ratnapuli, RC, and Lins, VFC (2003) "The influence of chemical composition and microstructure of API linepipe steels on hydrogen induced cracking and sulfide stress cracking" *Materials Science and Engineering* A357, Issue 1-2, pp 104-110.

Cicutti, CE, Madias, J, and Gonzalez, JC (1997). "Control of microinclusions in calcium treated aluminium killed steels" *Ironmaking and Steelmaking*, Vol 24, No 2, pp 155-159.

Devanathan, MAV, and Stachurski, Z (1962). "The adsorption and diffusion of electrolytic hydrogen in palladium" *Proceedings of the royal society of London*, series A, Vol 270, No. 1340, pp 90-102.

Domizzi, G, Anteri, G, and Ovejero-Garcia, G (2001). "Influence of sulphur content and inclusion distribution on the hydrogen induced blister cracking in pressure vessel and pipeline steels" *Corrosion Science*, Vol 43, pp 325-339.

Gel'd, PV, Gol'tsov, VA, Kvater, LI, and Sklyuev, PV (1965). "The effect of structure on the hydrogen-permeability of steel" *Metal science and heat treatment*, Vol 7, No 4, pp 218-220.

He, SK, Wang, GS and Wang, SN (1996) "Influence of sulphureous impurity and microshrinkage on HAC behavior of super-strength steel" *Acta Metallurgica*, Vol 9, No 6, pp 619-625.

Hong, GW, and Lee, JY (1983) "The interaction of hydrogen and the cementite-ferrite interface in carbon steel" *Journal of Materials Science*, Vol 18, pp 271-277.

Jeng, HW, Chiu, LW, Johnson, DL, and Wu, JK (1990) "Effect of pearlite morphology on hydrogen permeation, diffusion, and solubility in carbon steels" *Metallurgical and Materials Transactions A*, Vol 21A, pp 3257-3259.

Johnson, DL, Krauss, G, Wu, JK, and Tang, KP (1987) "Correlation of microstructural parameters and hydrogen permeation in carbon steel" *Metallurgical and Materials Transactions A*, Vol 18A, pp 717-721.

Kim, SJ, Jung, HG, and Kim KY (2011) "Effect of post weld heat treatment on the hydrogen trapping behavior of pressure vessel steel" *Proceedings of the NACE International Conference*, Houston, TX, paper 11292.

Kim, SJ, Jung, HG, and Kim, KY (2012a) "Effect of post-weld heat treatment on hydrogen-assisted cracking behavior of high-strength process pipe steel in a sour environment" *Scripta Materialia*, Vol 67, pp 895-898.

Kim, SJ and Kim, KY (2012b) "Electrochemical hydrogen permeation measurement through high-strength steel under uniaxial tensile stress in plastic range" *Scripta Materialia*, Vol 66, pp 1069-1072.

Kim, WK, Jung, HG, Koh, SU, and Kim KY (2008) "The effect of metallurgical factors on SOHIC initiation in HIC free linepipe steels" *Proceedings of the 18th International Offshore and Polar Engineering Conference*, ISOPE, Vancouver, paper TPC-462.

Koh, SU, Yang, BY, and Kim, KY (2004). "Effect of alloying elements on the susceptibility to sulfide stress cracking of line pipe steels" *Corrosion*, Vol 60, No 3, pp 262-274.

Luppo, MI, and Ovejero-Garcia, J (1991) "The influence of microstructure on the trapping and diffusion of hydrogen in a low carbon steel" *Corrosion science*, Vol 32, No 10, pp 1125-1136.

Oriani, RA (1970) "The diffusion and trapping of hydrogen in steel" *Acta metallurgica*, Vol 18, Issue 1, pp 147-157.

Park, GT, Koh, SU, Jung, HG, Kim, KY (2008). "Effect of microstructure on the hydrogen trapping efficiency and hydrogen induced cracking of linepipe steel" *Corrosion Science*, Vol 50, Issue 7, pp 1865-1871.

Park, GT, Jung, HG, Koh, SU, and Kim, KY (2011) "Effect of constant load on hydrogen diffusion and trapping on API X65 grade linepipe steel" *International Journal of Offshore and Polar Engineering*, IJOPE, Vol 21, No 1, pp 70-74.

Parvathavarthini, N, Saroja, S, and Dayal, RK (1999) "Influence of microstructure on the hydrogen permeability of 9%Cr-1%Mo ferritic steel" *Journal of Nuclear Materials*, Vol 264, Issue 1-2, No 1, pp 35-47.

Parvathavarthini, N, Saroja, S, Dayal, RK, and Khatak, HS (2001) "Studies on hydrogen permeability of 2.25% Cr-1% Mo ferritic steel: correlation with microstructure" *Journal of Nuclear Materials*, Vol 288, pp 187-196.

Radhakrishnan, TP, and Shreir, LL (1967) "Hydrogen permeation through iron and steel by electrochemical transfer-II. Influence of metallurgical factors on hydrogen permeation" *Electrochimica Acta*, Vol 12, pp 889-903.

Robertson, WM, and Thompson, AW (1980) "Permeation measurements of hydrogen trapping in 1045 steel" *Metallurgical Transaction A*, Vol 11A, pp 553-557.

Serna, S, Martı´nez, H, Lo´pez, SY, Gonza´lez-Rodrı´guez, JG, Albarra´n, JL (2005) "Electrochemical technique applied to evaluate the hydrogen permeability in microalloyed steels" *International Journal of Hydrogen Energy*, Vol 30, pp 1333-1338.

Tau, L, Chan, SLI, and Shin, CS (1996) "Hydrogen enhanced fatigue crack propagation of bainitic and tempered martensitic steels" *Corrosion Science*, Vol 38, No 11, pp 2049-2060.

Torres-Islas, A, González-Rodríguez, JG, Uruchurtu, J and Serna,S (2008) "Potential effect over the stress corrosion cracking behavior of a X-70 microalloyed pipeline steel in dilute $NaHCO_3$ solutions" *Proceedings of the NACE International Conference*, New Orleans, LA, paper 08482.

Zhao, MC, Tang, B, Shan, YY, and Yang, K (2003) "Role of microstructure on sulfide stress cracking of oil and gas pipline steels" *Metallurgical and Materials Transactions A*, Vol 34A, pp 1089-1096.

ISO 17081 (2004) "Method of measurement of hydrogen permeation and determination of hydrogen uptake and transport in metals by an electrochemical technique"

JIS Z3113 (1975) "Method for Measurement of Hydrogen Evolved from Deposited Metal", Japan

NACE TM0284 (2003) "Evaluation of Pipeline and Pressure Vessel Steels for Resistance to Hydrogen-Induced Cracking" Houston, TX, USA

NACE Standard MR0175, "Sulfide Stress Cracking Resistant Metallic Materials for Oilfield Equipment," Houston, TX, 1993