

In Situ Observation of Hydrogen-Induced Cracking Propagation Behavior

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ABSTRACT

Hydrogen-induced cracking (HIC) is one of the major issues of line pipe exposed to sour environments. There are some guidelines on materials requirements for carbon and low alloy steels for H₂S-containing environments in oil and gas production. Generally, HIC susceptibility is evaluated after the test duration, typically 96 h, in accordance with NACE Standard TM0284-2016. However, HIC propagation behavior during HIC test has not been fully understood. In this study, a new in situ HIC measurement method has been developed in order to make the connection between HIC propagation behavior and microstructure. This technique is based on the combination of an automatic ultrasonic wave inspection system and a scanning electron microscopy (SEM) observation. HIC propagation rate and HIC propagation behavior of carbon steels with different textures were investigated, using this in situ technique. Texture components of tested steels were changed by controlled rolling process in the alpha-gamma dual phase region. The {100} intensity parallel to the rolling plane was developed with increasing controlled rolling reduction in the alpha-gamma dual phase region. HIC propagation rate increased and crack length of HIC grew in a staircase pattern with time when the {100} texture was highly developed. In addition, HIC propagation behavior could be overlapped with fracture surface, just like a projection mapping. The overlapping could make the connection between HIC propagation behavior and HIC fracture surface. Microstructure and texture just under the HIC fracture surface was also characterized by SEM and an electron backscatter

diffraction pattern method. The results obtained in this study showed that HIC propagation behavior was affected by a texture. In addition, the new in situ HIC observation technique, which can make the direct connection between HIC propagation behavior and microstructure, revealed a detailed HIC propagation behavior and an effect of microstructure.

KEY WORDS: carbon steel, hydrogen-induced cracking, hydrogen sulfide, microstructure, oil and gas, pipelines

INTRODUCTION

In oil and gas industry, pipeline for natural gas transportation is exposed to wet H₂S environments (sour environments) in some cases. The presence of H₂S in the sour environment enhances hydrogen entry into the steel as a result of catalytic action of H₂S. Absorbed hydrogen atoms in the steel diffuse and accumulate in a local area and finally cause several types of environmental cracking such as hydrogen-induced cracking (HIC), sulfide stress cracking (SSC), and stress oriented hydrogen-induced cracking. HIC is one of the major issues of line pipe exposed to sour environments. There are some guidelines on materials requirements for carbon and low alloy steels for H₂S-containing environments in oil and gas production. In general, HIC susceptibility, such as crack sensitivity ratio (CSR), crack length ratio (CLR), and crack area ratio (CAR), is evaluated after the test duration, typically 96 h, in accordance with NACE Standard TM0284-2016.¹

It is well known that HIC susceptibility is affected by inclusions, precipitates, microstructure, and

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hardness.²⁻⁶ Elongated nonmetallic inclusions (especially MnS) and insoluble coarse particles (Nb(C,N), TiN) induce the initiation of HIC. As HIC susceptibility is increased with maximum hardness of the steel, banded microstructure and center segregated zone also decrease HIC resistance. Therefore, many researchers and manufacturers agree that there are three important factors to achieve resistance to HIC: clean steel, uniform microstructure, and low hardness. Researchers have also been examining the effects of texture on HIC resistance⁷⁻¹² because crystallographic texture is altered through thermomechanical control process (TMCP) variations and influence alloy toughness. Assuming that HIC occurs parallel to the rolling plane, it has been postulated that reducing the number of {001} planes parallel to the rolling plane results in enhanced HIC resistance.

It is recognized that HIC is observed in sour environments without external stresses. The internal hydrogen pressure theory is typically used to explain HIC.^{2-3,11-12} It is postulated that hydrogen atoms diffuse through the lattice and recombine as H₂ gas which accumulates at discontinuities, principally inclusion/metal interfaces. When the gas pressure becomes sufficiently high, voids and cracks develop and propagate. The gas pressure depends upon the hydrogen content according to Sievert's law. A proposed concept of HIC initiation has been discussed by Ikeda, et al.²⁻⁵ When the absorbed hydrogen concentration (C₀) from a corrosion reaction is larger than a threshold hydrogen concentration of material (C_{th}), HIC initiates; when the C₀ value is smaller than the C_{th} value, HIC does not initiate. Here, the C_{th} is defined as a value which is the minimum hydrogen concentration to produce HIC in the material. The relationship between the critical hydrogen concentration for HIC initiation (C_{th}) and inclusion size at HIC initiation can be derived as follows. The critical fugacity of hydrogen gas (P_{crit}) for starting unstable crack growth at the crack tip of the precrack length (a_{crit}) is expressed as follows:¹³

$$P_{\text{crit}} = C_1 (a_{\text{crit}})^{-1/2} \quad C_1: \text{constant} \quad (1)$$

where P_{crit} is the critical fugacity of hydrogen gas in precrack $2 \times (a_{\text{crit}})$. Here, a_{crit} is taken to be the critical length of a nonmetallic inclusion such as an elongated MnS for HIC initiation. Sievert's law indicates the relationship between P_{crit} and C_{th}:

$$C_{\text{th}} = C_2 (P_{\text{crit}})^{1/2} \quad C_2: \text{constant} \quad (2)$$

Hara, et al.,¹⁴ showed that C_{th} as a critical hydrogen permeability (Per_{crit}) was expressed as a function of the critical length of inclusions (a_{crit}) and the maximum hardness of the center-segregation zone (Hv_{max}). In addition, the prediction model that indicates how the sour-resistant line pipe steels should be produced was also proposed.¹⁴ Once HIC initiates,

hydrogen gas pressure drives crack propagation and could be assisted by other hydrogen embrittlement mechanisms including hydrogen-enhanced localized plasticity and hydrogen-enhanced decohesion.¹⁵⁻¹⁶ Furthermore, the hydrogen concentration at the crack tip is likely to increase as a result of the hydrostatic stresses at the crack tip and increased dislocation density in the plastic zone of the crack. Hydrogen accumulation within hydrogen-induced cracks has recently been measured by three-dimensional neutron tomography.¹⁷

In recent years, considerable studies on HIC propagation using in situ HIC observation techniques were suggested and a few in situ HIC observation methods were proposed by Kushida and Kudo¹⁸ and Sawamura, et al.¹⁹ In situ HIC measurement technique developed by Kushida was based on the combination of the electrochemical hydrogen permeation method and the automatic ultrasonic wave inspection system. Therefore, the in situ technique can make the connection between HIC propagation behavior and hydrogen permeability. On the other hand, using in situ HIC observation system developed by Sawamura, et al., HIC testing could be conducted in accordance with NACE Standard TM0284-2016. This in situ system made it possible to make the connection between HIC propagation behavior and HIC susceptibility, such as CAR and CLR. However, the effect of microstructure on HIC propagation behavior during HIC test has not been fully understood. Up to now, there has not been an in situ observation method that can make the direct connection between HIC propagation behavior and microstructure.

In this study, a new in situ HIC measurement method has been developed and HIC propagation behavior was investigated, using various steels with different texture. HIC test was conducted in accordance with NACE Standard TM0284-2016 and HIC propagation behavior was measured using an in situ HIC observation instrument at the same time. A C-scan movie which shows HIC propagation behavior was made and overlapped with HIC fracture surface, just like a projection mapping. In addition, the cross-sectional microstructures just under the HIC fracture surface were observed using scanning electron microscopy (SEM) and electron backscatter diffraction pattern (EBSP), in order to make the connection between HIC propagation behavior and microstructure.

EXPERIMENTAL PROCEDURES

Materials

The low alloy carbon steel with a chemical composition given in Table 1 was used as test material in this study. Figure 1 shows a schematic drawing of TMCP condition. The low alloy carbon steels, after being austenitized, were rolled into 20-mm-thick plates and accelerate-cooled from 700°C to ambient temperature in

TABLE 1

Chemical Composition of Test Material (wt%)

C	Si	Mn	Others	Bal.
0.04	0.3	1.55	Ni, Cu, Nb, Ti, Al	Fe

order to deteriorate the HIC resistance. At the controlled rolling stage, three controlled rolling conditions at the non-recrystallization temperature were selected and the microstructures of R0, R5, and R10 steels were controlled. R0 steel was completely controlled rolled in gamma region. On the other hand, R5 steel was 50% controlled rolled in gamma region and 50% controlled rolled in alpha-gamma dual phase region. R10 steel was completely controlled rolled in alpha-gamma dual phase region. These reduction ratios in alpha-gamma dual phase region were 0, 5, and 10, respectively. In this study, the reduction ratio is defined as the ratio of the plate thickness after rough rolling to the plate thickness after controlled rolling in alpha-gamma dual phase region. In this case, ferrite-martensite banded structures were formed in all of the tested steels. In addition, integration of {100} plane parallel to the rolling direction was enhanced in R5 and R10 steels because of the controlled rolling in alpha-gamma dual phase region.²⁰

Hydrogen Permeation Method

Hydrogen permeability and corrosion potential were measured using an electrochemical hydrogen permeation method with nickel-plated specimens on the anode-side surface.²¹ The test specimens were taken from the surface of the test materials. 5.0% sodium chloride, 0.28 wt% sodium acetate, and 5.38 wt% acetic acid (5.0 wt% NaCl + 0.93 N (CH₃COOH + CH₃COONa)) (high-strength line pipe [HLP] solution pH 3.0) was used as the test solution, in order to maintain solution pH value. This test solution was originally adopted in the Iron and Steel Institute of Japan (ISIJ) HLP Research Committee as an optional HIC test

solution²²⁻²⁵ and is currently proposed as HLP solution for NACE Standard TM0284-2016. Test solution was deaerated with nitrogen and purged with H₂S before the hydrogen permeation test. The dimension of the specimen was 20 mm width × 40 mm length × 1 mm thickness. Figure 2 shows the schematic illustration of a hydrogen permeation system. The hydrogen-entry side chamber was filled with a test solution (HLP solution pH 3.0) and hydrogen-withdraw side (anode side) chamber was filled with a 0.1 N sodium hydroxide (NaOH) solution. The specimen was set at 0.148 V vs. silver-silver chloride electrode (Ag/AgCl). The measured specimen area was approximately 2 cm² and the specimen thickness (L) was 1.0 mm. One side surface of each specimen was polished mechanically, electropolished, and then nickel-plated in a Watt bath.¹³ Based on the observed hydrogen permeation flux, the observed permeation current density (J) during the test start was measured. Hydrogen permeation (Per) is described as the hydrogen permeation through the steel multiplied by the specimen thickness.²⁶⁻²⁷

$$\text{Per} = J \times L \quad (3)$$

where J is the permeation current density and L is the specimen thickness through which hydrogen permeates.

In Situ Hydrogen-Induced Cracking Observation

HIC test was performed in accordance with the NACE Standard TM0284-2016 and HIC propagation behavior was measured using an in situ HIC observation instrument at the same time. The test specimens were taken from the test material and emery-polished down to #320. Configuration of the specimen was 20 mm width × 100 mm length × 20 mm thickness. HLP solution pH 3.0 was used as a test solution, in the same way as the hydrogen permeation test. Figure 3 shows the schematic drawing of an in situ HIC observation instrument. The instrument used in this study

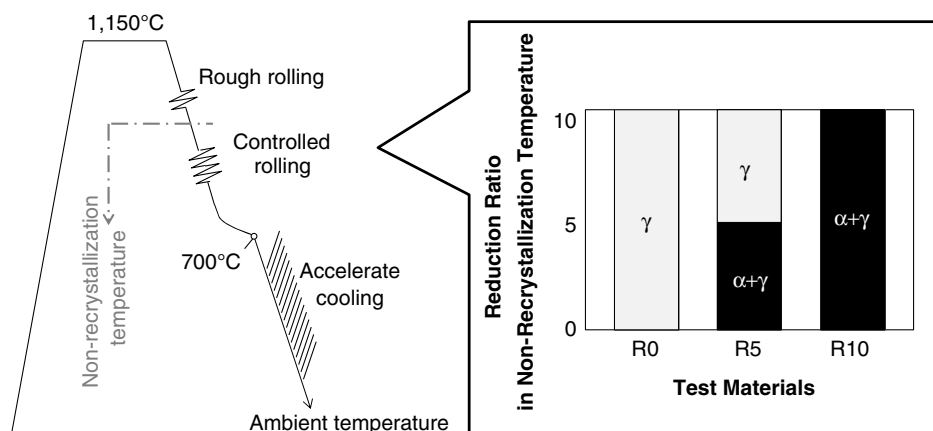


FIGURE 1. Schematic drawing of TMCP condition.

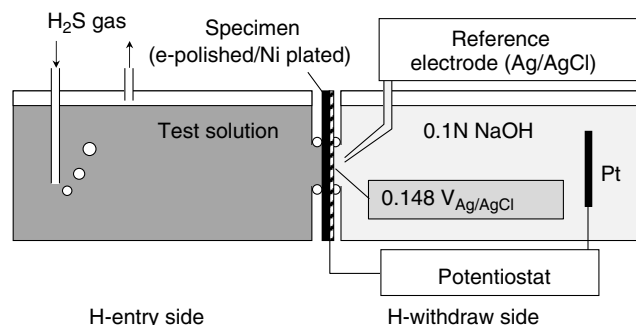


FIGURE 2. Schematic illustration of hydrogen permeation system.

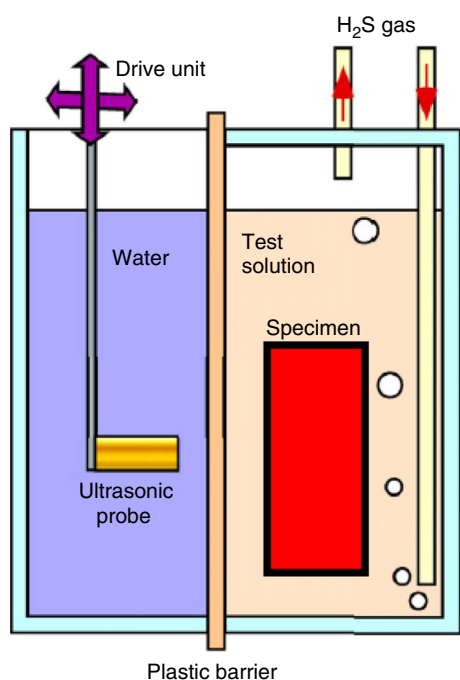


FIGURE 3. Schematic drawing of in situ HIC observation instrument.

was originally developed by Sawamura, et al.¹⁹ The HIC detection system was composed of a 10 MHz ultrasonic probe in the detection side, and the detection side chamber was separated from the HIC testing side by a plastic barrier. The HIC testing-side chamber was filled with a test solution (HLP solution pH 3.0) and the detection side was filled with water. C-scan depth was set in the range from the surface to the mid-thickness of tested plate. C-scan mapping was automatically performed during HIC test. The auto-scan was undertaken at 20 min to 30 min intervals to observe HIC propagation behavior. Test temperature of the solution was kept at 25°C by a water bath with constant temperature. Using C-scan maps as the frames of a movie, a C-scan movie that shows HIC propagation behavior was made. Figure 4 shows in situ HIC observation system. After the testing, specimens that were machined from a close part of HIC, which occurred around quarter-thickness of tested plates, were

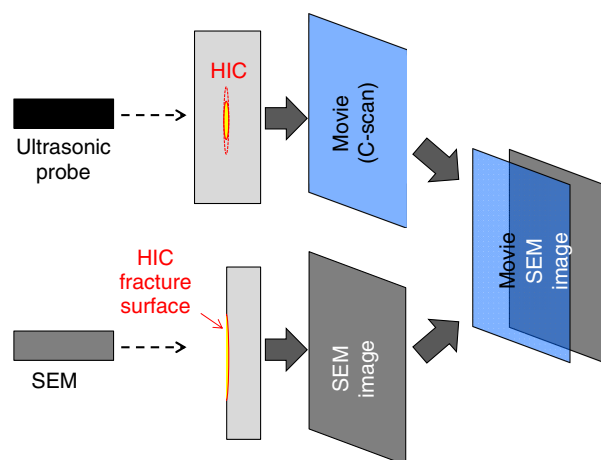


FIGURE 4. Schematic drawing of in situ HIC observation system.

notched, put into liquid nitrogen, and cut, in order to show the HIC fracture surface. HIC fracture surfaces were observed by SEM. Finally, HIC propagation behavior obtained from C-scan movie was overlapped with fracture surface, just like a projection mapping as shown in Figure 4.

Cross-Sectional Microstructure of Hydrogen-Induced Cracking Fracture Surface

Specimens were cut and cross-sectional microstructures just under the HIC fracture surface were observed, in order to make the connection between HIC propagation behavior and microstructure. The cross-section microstructures were observed by SEM and EBSD.

RESULTS AND DISCUSSION

Materials

Figure 5 shows optical micrographs at the surface of the tested plates with 20 mm thickness. This location corresponds to the location that hydrogen atoms enter through. All plates consisted of ferrite-martensite banded microstructures. In R0 steel, the shape of ferrite grain was polygonal because R0 steel was completely controlled rolled in gamma region and the reduction ratio in alpha-gamma region of R0 steel was 0. On the other hand, in R5 and R10 steels, the shape of ferrites were elongated, as ferrites in these steels were deformed by controlled rolling in alpha-gamma region. The reduction ratio in alpha-gamma region of R5 and R10 steels were 5 and 10, respectively.

Figure 6 shows SEM images at the quarter-thickness of the tested plates with 20 mm thickness. This location corresponds to the location where HIC occurred as described later. At this location, all plates also consisted of ferrite-martensite banded microstructures. The area fraction of martensite was from

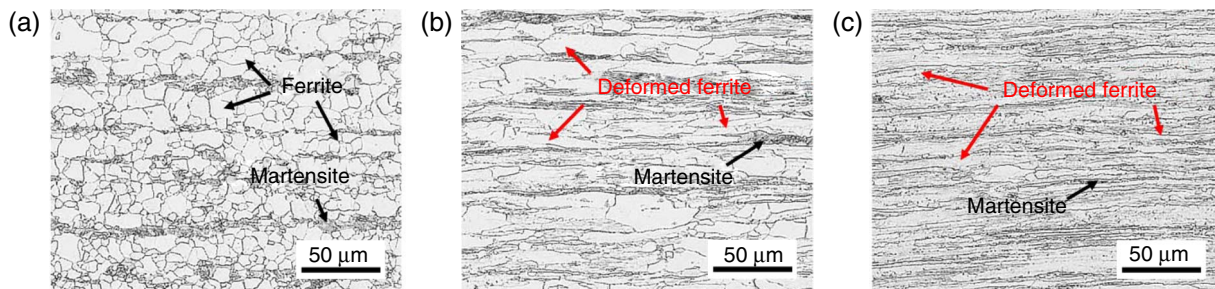


FIGURE 5. Optical micrographs at the surface of tested plates. (a) R0 steel, (b) R5 steel, and (c) R10 steel.

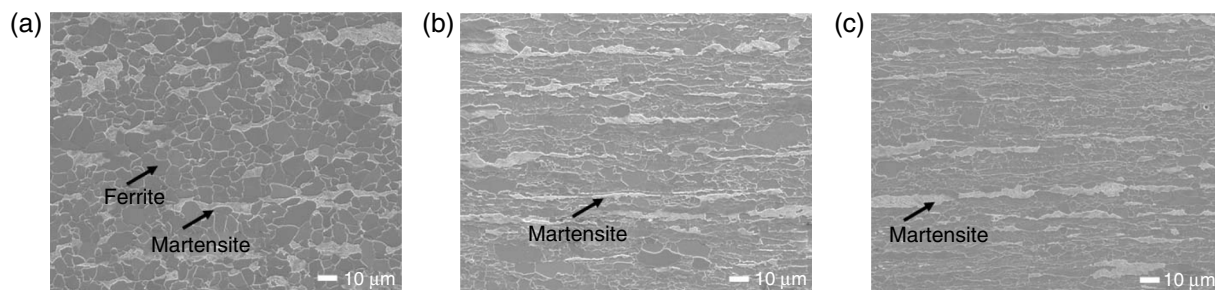


FIGURE 6. SEM images at the quarter-thickness of tested plates. (a) R0 steel, (b) R5 steel, and (c) R10 steel.

12% to 13% in all of the test material, regardless of the reduction ratio in alpha-gamma region. It could be understood that the same area fraction was obtained as a result of the same accelerate-cooling start temperature which was below austenite-to-ferrite transformation temperature. In this study, the measured austenite-to-ferrite transformation temperature of the test material was 770°C, and 700°C was selected as an accelerate-cooling-start temperature.

Figure 7 shows the intensity plots at the quarter-thickness of the tested plates with 20 mm thickness. Maximum {100} intensity was increased with increasing reduction ratio in alpha-gamma region, as controlled rolling in alpha-gamma dual phase region enhanced the integration of {100} plane parallel to the rolling direction.²⁰

Hydrogen Permeation Method

Figure 8 shows the time dependence of hydrogen permeability and corrosion potential of R0 and R10 steels. Corrosion potential of R0 steel was the same as that of R10 steel and there was no big difference between hydrogen permeability of R0 steel and that of R10 steel, regardless of the reduction ratio in alpha-gamma region.

In Situ Hydrogen-Induced Cracking Observation

Figure 9 shows the C-scan maps of tested steels at 24 h duration. Blue area in Figure 9 corresponds to the area where HIC did not occur. Areas in red, green, and light blue in Figure 9 correspond to the areas where HIC occurred. Those differences in colors between

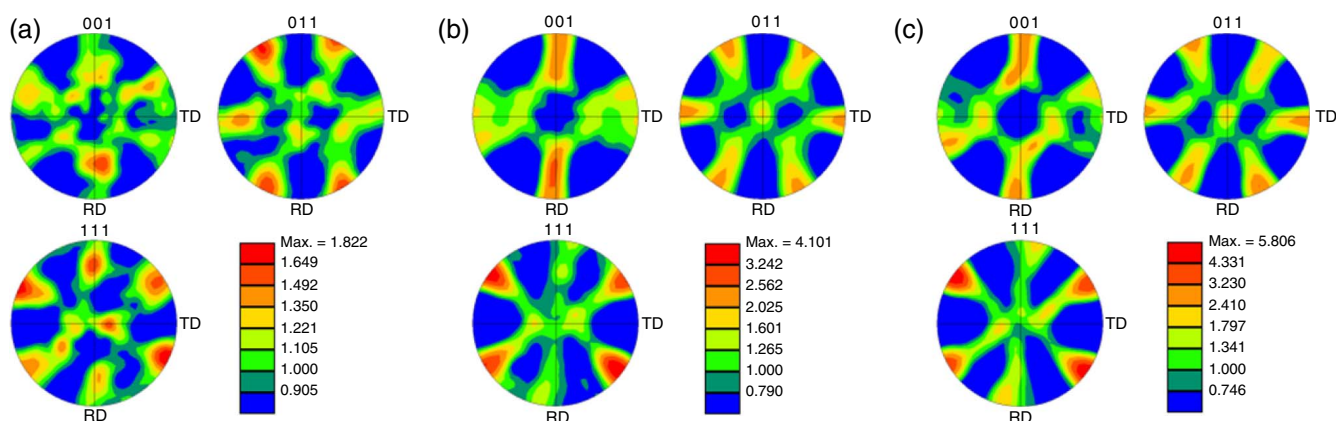


FIGURE 7. Intensity plots at the quarter-thickness of tested plates. (a) R0 steel, (b) R5 steel, and (c) R10 steel. RD: rolling direction. TD: transverse direction.

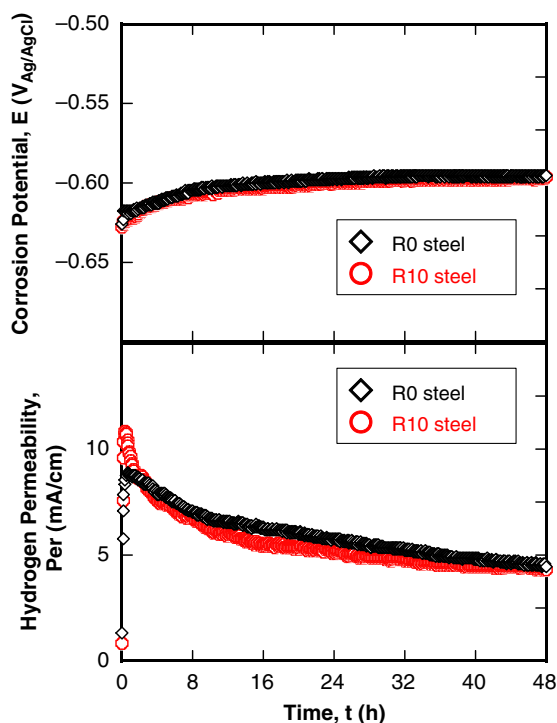


FIGURE 8. Time dependence of hydrogen permeability and corrosion potential.

red, green, and light blue around HIC depend on echo intensity. HIC occurred in all tested steels. The area where HIC occurred increased significantly with increasing the reduction ratio in alpha-gamma dual phase region. In addition, the shape of HIC which occurred in R5 and R10 steels were elongated in rolling

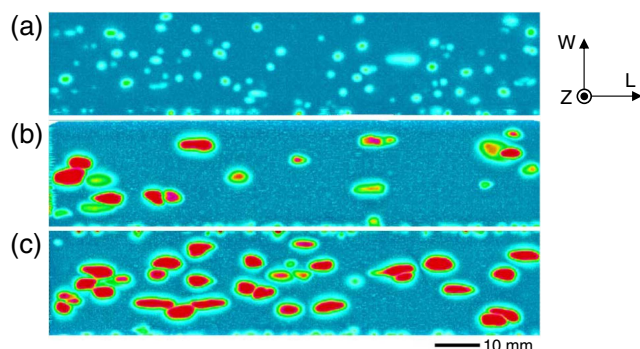


FIGURE 9. C-scan maps of tested steels at 24 h duration. (a) R0 steel, (b) R5 steel, and (c) R10 steel.

direction as shown in Figure 9. The factors that affect HIC propagation behavior are thought to be a ferrite-martensite banded structure, the shape of ferrite grain, the texture of ferrite, and the strain (dislocation density) in the elongated ferrite, as the area fraction of martensite was the same for all tested materials. Figure 10 shows an example of C-scan map with the depth profile of HIC. HICs occurred in various depth positions. The fracture surface of HIC occurring at only the quarter-thickness portion, indicated in the blue area in Figure 10, was shown in order to equalize the measurement position of HICs and to analyze HICs under the same condition.

The C-scan movie which shows HIC propagation behavior was made, using C-scan maps as the frames of a movie. In addition, as a result of overlapping the movie on a HIC fracture surface, the HIC propagation behavior which is shown in the C-scan movie was related to the HIC fracture surface which was observed by SEM as shown in Figure 11. By analyzing these overlapped movies, HIC initiation sites and propagation behaviors were determined. The crack lengths of HICs were measured using a part of the overlapped movie, and the time dependence of the crack length from the HIC initiation site was obtained, as shown in Figure 12. The crack length of HIC occurring in R5 steel quickly increased, whereas the crack length of HIC occurring in R0 steel gradually grew, as shown in Figure 12. This result suggests that HIC propagation rate increases with increasing reduction ratio in alpha-gamma dual phase region. In other words, development of the {100} texture brings an increase in HIC susceptibility. In addition, Figure 12 revealed that crack length of HIC grew in a staircase pattern with time when the {100} texture was highly developed. Especially, it is noteworthy that HIC propagation was arrested once in R5 steel, as indicated by an arrow in Figure 12.

HIC fracture surface was observed carefully in order to find a microstructural reason for the arrest. Figure 13 shows SEM images of HIC fracture surface near the location where HIC was arrested once. As indicated by white arrows in Figure 13(a), steps were found near the arrested location. The location indicated by "b" in Figure 13(a) was observed with high-magnification, as shown in Figure 13(b). The specimen was cut, and cross-sectional microstructures just under the HIC fracture surface were observed as shown in Figure 13(b), in order to make the connection between HIC propagation behavior and microstructure.

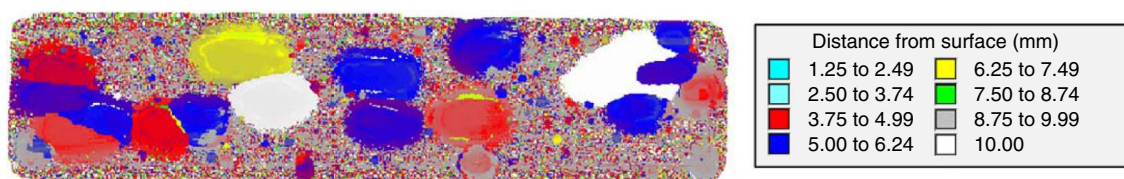


FIGURE 10. C-scan map with the depth profile of HIC occurring in R5 steel after the HIC testing.

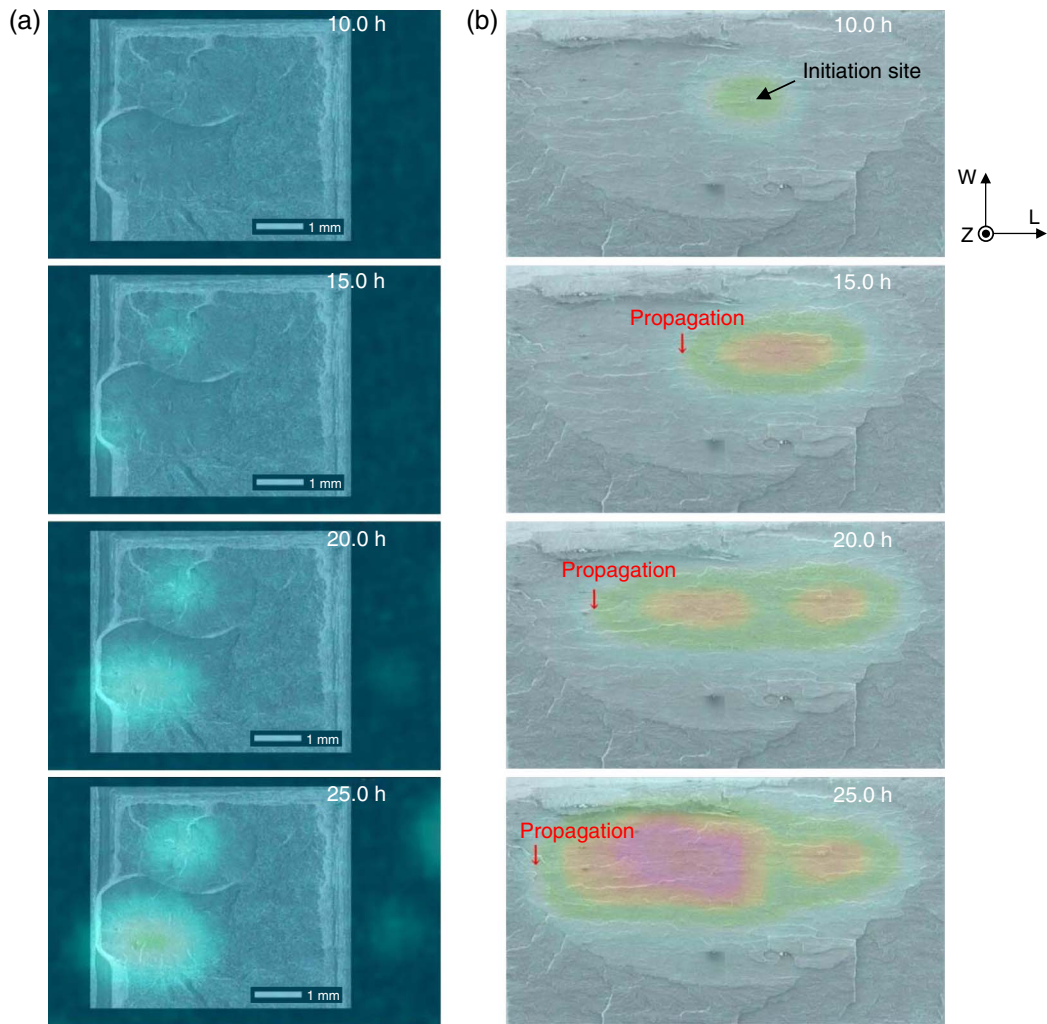


FIGURE 11. In situ observation of HIC propagation behavior. (a) R0 steel, and (b) R5 steel.

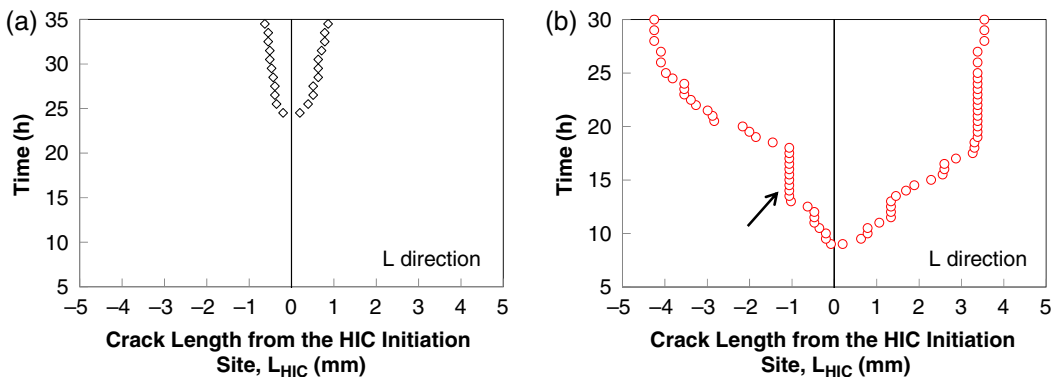


FIGURE 12. Time dependence of the crack length from the HIC initiation site. (a) R0 steel, and (b) R5 steel.

Cross-Sectional Microstructure of Hydrogen-Induced Cracking Fracture Surface

The cross-sectional microstructure just under the HIC fracture surface was observed by SEM and EBSP. Figure 14 shows cross-sectional microstructure: SEM image, inverse pole figure (IPF) map, and

Kernel average misorientation (KAM) map near the location where HIC was arrested once.

Non-deformed ferrite was found just under the step where HIC was arrested once. As indicated by a white arrow in Figure 14(a), ferrite was found just under the step that was observed near the arrested

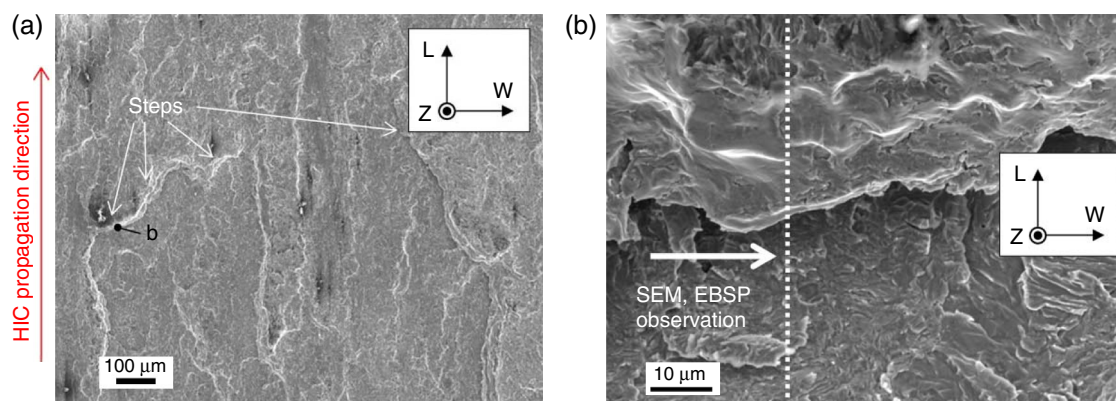


FIGURE 13. SEM images of HIC fracture surface in R5 steel. (a) Low magnification, and (b) high magnification.

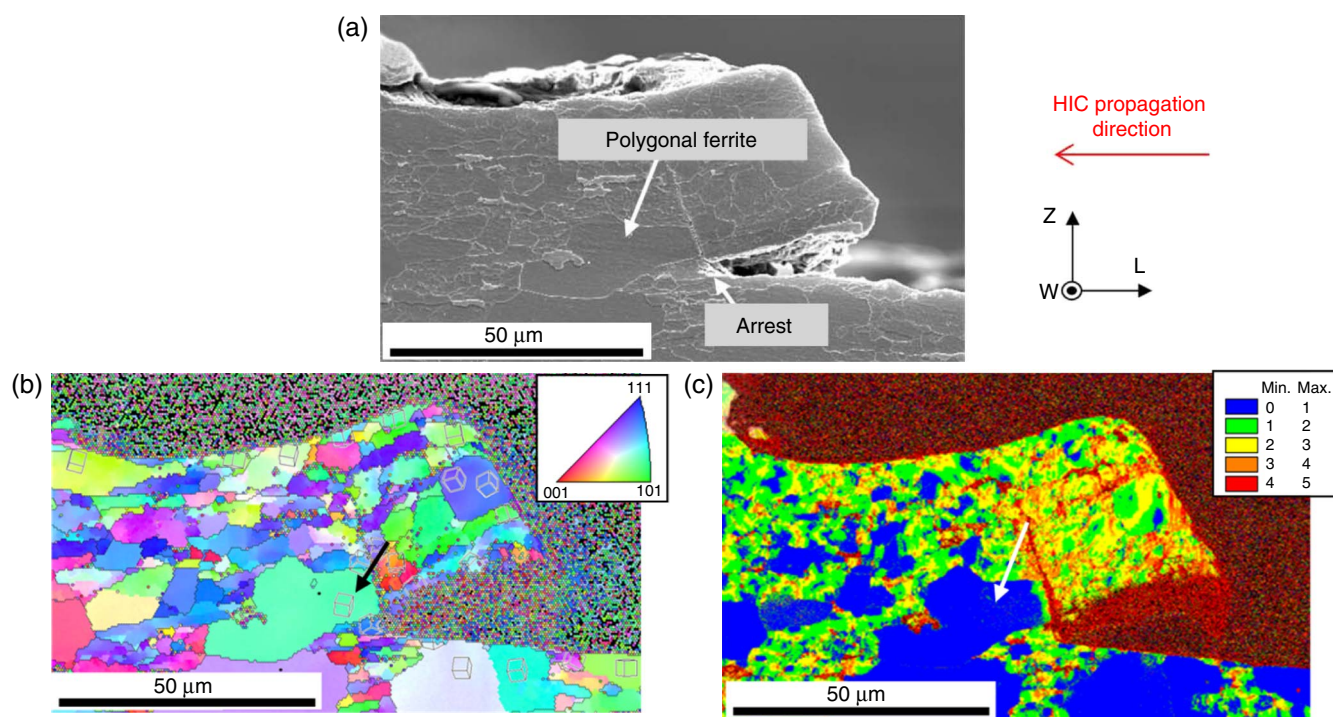


FIGURE 14. Cross-sectional microstructure near the location where HIC was arrested once. (a) SEM image, (b) IPF map, and (c) KAM map.

location. The shape of this ferrite was not so elongated, and it looked like a polygonal ferrite. In addition, the {100} plane of this polygonal ferrite was not parallel to the rolling direction, as indicated by the cube in Figure 14(b). Moreover, blue areas in Figure 14(c) correspond to the area where misorientation between neighboring measured points was below 1° . Therefore, there was no strain in the ferrite grain located just under the step. These results suggest that the ferrite located just under the step is non-deformed ferrite which was not deformed at controlled rolling stage. Thus, the factors that affect HIC propagation behavior are thought to be the orientation of this ferrite and the strain in this ferrite.

CONCLUSIONS

A new in situ HIC observation method has been developed in this study. This technique is based on the combination of an automatic ultrasonic wave inspection system and a scanning electron microscopy observation. The advantage of this method compared with previous in situ observation method is that it can make the direct connection between HIC propagation behavior and microstructure. The effect of microstructure on HIC propagation behavior was investigated, using the various steels with different textures and the new in situ HIC observation technique. Texture components of tested steels were changed and {100}

intensity parallel to the rolling plane was developed with increasing controlled rolling reduction in the alpha-gamma dual phase region. Main conclusions are as follows:

❖ HIC propagation rate increased and crack length of HIC grew in a staircase pattern with time when {100} texture was highly developed.

❖ HIC propagation behavior could be overlapped with fracture surface, just like a projection mapping. As a result, steps were observed near the location where HIC was arrested once. In addition, non-deformed ferrite was found just under the step where HIC was arrested once.

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