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「水素輸送におけるパイプライン材の材料特性評価」 完了報告書

事業 ID: 2023021277

事業名: 水素輸送におけるパイプライン材  
の材料特性評価

事業者名: JFE スチール株式会社

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## 1. 事業概要

二酸化炭素排出量を削減するためにパイプラインによる水素ガス輸送が想定されるが、高圧条件下での使用には適切な材料性能を特定することが重要である。このプロジェクトは、開発した材料を用いて、高圧水素下での材料特性を評価する技術を開発すると共に、自社開発したパイプライン用材料の安全性について業界基準を踏まえ評価することを目的とする。

## 2. 背景

パイプラインによる水素輸送において、輸送効率とプロジェクト経済性のためには高圧輸送が必要であるが、高圧条件下で使用可能な材料性能要件を明確にすることが重要である。現在の水素パイプライン規格 ASME B31.12-2019 「Hydrogen Piping and Pipeline」では、高圧条件である設計係数が 40%を超える場合には、水素ガス中の破壊に対する十分な耐性(破壊靱性)を備えたパイプライン材料を使用することが求められている。また、ASME B31.12-2019 規格では、ASTM E1681 規格による KIH 判定に基づく破壊安全性評価を必要としている。しかし、ASTM E1681 規格の破壊靱性試験は平面ひずみ条件が必要となるが、パイプライン材料のような延性材料では、この条件を達成するのは困難であり、材料の破壊靱性を評価する試験方法を確立する必要がある。

## 3. 研究目的

パイプライン材料のような延性材料に対応した水素中での破壊靱性評価法の提案と高圧水素条件下で使用可能な材料性能要件を明らかにし、自社材の耐水素適合性を評価する

## 4. 事業成果

高圧水素パイプラインの必要靱性に関する技術レポート

タイトル: Fracture toughness evaluation of X65 linepipe steel under high pressure hydrogen



## Fracture toughness evaluation of X65 linepipe steel under high pressure hydrogen

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### ABSTRACT

Hydrogen gas will be employed to reduce the carbon footprint and meet climate change goals, and a pipeline system will be necessary to transport this gaseous hydrogen. Since high pressure transmission of large volumes of hydrogen gas is expected to increase from the viewpoint of transportation efficiency, it is important to identify the correct material performance requirements under high pressure conditions. The current hydrogen pipeline code ASME B31.12 “Hydrogen Piping and Pipeline” requires that pipe materials shall be qualified for adequate resistance to fracture in hydrogen gas. According to the standard, a fracture toughness test to measure  $K_{IH}$  under a hydrogen environment needs to be conducted based on ASTM E1681. While the fracture toughness test provided in ASTM E1681 is required for the basic plain strain condition, it is difficult to achieve that condition with high toughness materials such as API X65 linepipe. Hence, it is necessary to define testing methodologies for high toughness materials. This study aims to establish evaluation methods for fracture toughness under high pressure hydrogen conditions and to evaluate the fracture toughness of materials for high pressure hydrogen pipelines. Fracture toughness tests based on ASTM E1820 by the unloading compliance method were conducted under air and high pressure conditions, and the difference in fracture behavior under the air and high pressure hydrogen conditions was investigated from the viewpoint of the early stage of crack initiation. The critical crack size was analyzed using the failure assessment diagram (FAD) concept, which is specified in Article KD-10 of ASME BPVC. The FAD analysis of a longitudinal semi-elliptical surface crack flaw revealed that the X65 HFW linepipe possesses a sufficient safety margin for weld fracture.

**KEY WORDS:** Hydrogen pipeline; HFW; Fracture toughness; FAD calculation

### INTRODUCTION

Hydrogen gas will be employed to reduce the carbon footprint and meet climate change goals, and a pipeline system will be necessary to transport this gaseous hydrogen. Since high pressure transmission of large volumes of hydrogen gas is expected to increase from the viewpoint of transportation efficiency, it is important to identify the correct material performance requirements under high pressure conditions. However, significant deterioration of mechanical properties under gaseous hydrogen environments has been observed in various linepipe steels due to hydrogen embrittlement. It has also been reported that fracture toughness is reduced under high pressure hydrogen (Hoover et al., 1981; Gutierrez-Solana et al., 1982; Cialone et al., 1985; Lam et al., 2009; Stalheim et al., 2010; Ishikawa et al., 2022).

The current hydrogen pipeline code ASME B31.12 “Hydrogen Piping and Pipeline” requires that pipe materials shall be qualified for adequate resistance to fracture in hydrogen gas based on Article KD-10 of ASME BPVC, Sec. VIII, Division 3. In most cases, the above-mentioned code uses the stress intensity  $K_{IH}$ , which is a linear-elastic parameter of fracture mechanics. In linear elastic fracture mechanics, which was developed to quantitatively describe the behavior of brittle materials, a plastic zone in front of a crack leads to blunting of the crack tip, and through this, to de-escalation of the loading situation.

According to the code, fracture toughness testing under hydrogen environments needs to be conducted to evaluate fracture toughness in hydrogen  $K_{IH}$  using the method for determination of  $K_{IH}$  specified in ASTM E1681. While the fracture toughness test provided in ASTM E1681 is required for the basic plain strain condition as a linear-elastic parameter of fracture mechanics, it is difficult to achieve that condition with high toughness materials. Recently, commonly-used grades for pipelines have been developed to secure high toughness behavior, which is beneficial in terms of the safety margin in loading situations such as those at cracks. Linear-elastic fracture mechanics allows plastic zones as long as they are about two orders of magnitude smaller than the dimensions of the ligament. The size of the plastic zone depends on the material grade and increases with lower yield strength. Evaluation of stable crack initiation resistance by the  $J_{IC}$  test based on ASTM E1820, which can evaluate the fracture toughness value more widely (Kalwa, 2022), has been proposed. However, it is not clear that the fracture toughness evaluation method in ASTM E1820 is applicable under high pressure hydrogen gas, because the standard assumes an air condition, as shown in Fig. 1.

Therefore, this study aims to establish evaluation methods for fracture toughness under high pressure hydrogen for high toughness materials and to evaluate their fracture toughness. As the evaluation method, fracture toughness tests based on ASTM E1820 using the unloading compliance method were carried out under a high pressure hydrogen condition, and the difference of fracture behavior under air and high pressure hydrogen conditions was investigated. The toughness requirement based on an assumed surface crack located on the inner surface of the pipe was also calculated by the ECA (Engineering Critical Assessment) method in API 579-1/ASME FFS-1 based on an FAD calculation. The applicability of API X65 HFW pipe to high pressure hydrogen pipelines was discussed based on the ECA results.



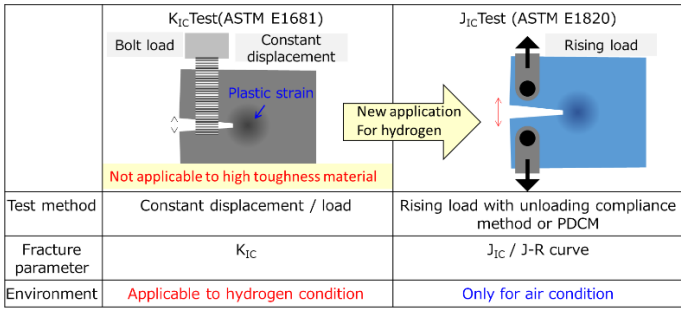
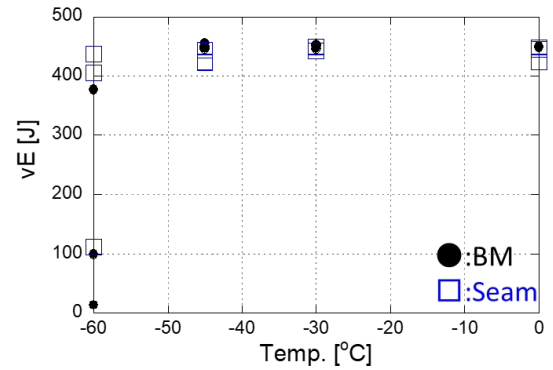
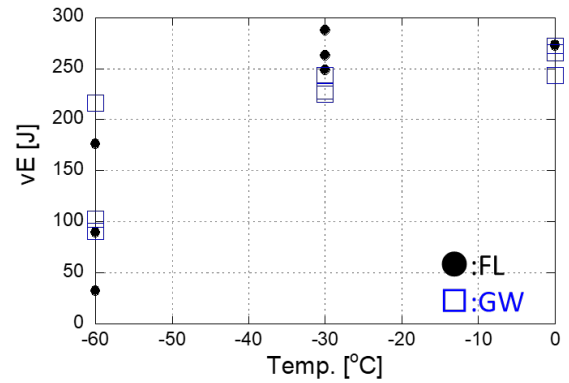


Fig. 1 Difference of fracture toughness test methods



(a) Base metal and seam weld



(b) Girth weld and fusion line of girth weld

Fig. 2 Charpy test results of tested material

EXPERIMENTAL PROCEDURE

Material

The test pipe was a newly-developed HFW linepipe “MightySeam®,” which is an API X65 grade product with a high-quality weld seam (Toyoda, 2012). The dimensions of the test pipe were 16 inches (406.4 mm) in outside diameter (OD) and 16.7 mm in wall thickness (WT). Girth welded joints were prepared by gas metal arc welding (GMAW) under the welding conditions in Table 1. Table 2 shows the tensile properties of the base material of the tested pipes in the transverse direction. Both the yield stress and the tensile strength of the weld metal (WM) were higher than those of the base metal, and the WM showed perfectly overmatched tensile properties. V-notched Charpy tests were conducted. Figures 2 show the results of V-notched Charpy tests with the notch position located in the base metal, the center of the longitudinal weld seam, the center of the girth weld and the fusion line of the girth weld.

Table 1 Example of girth welding conditions

Welding consumable	CRC-70S (AWS A5.18 ER-70S-6)	
Shielding gas	Root	Fill and cap
	75 % Ar / 25 % CO <sub>2</sub>	85 % Ar / 15 % CO <sub>2</sub>
Representative current-voltage-speed	Root ~200 A / ~14 V / ~254 mm/min Fill ~250 A / 23 V / ~406 mm/min Cap ~200 A / 24 V / ~330 mm/min	
Heat input	Root 0.69 kJ/mm Fill 0.82 kJ/mm Cap 0.86 kJ/mm	
Preheating	50 °C	
Macrostructure		

Table 1 Tensile properties of tested materials in T-direction

Grade	Type	YS [MPa]	TS [MPa]
API X65	BM	501	614
API X65	GW	613	679

Residual Stress Measurement

For the ECA calculations, the distribution of the residual stresses in the thickness direction is necessary data as a secondary stress in the ECA analysis. Therefore, the residual stresses of the tested HFW linepipe steel were measured by the modified internal residual stress (MIRS) method, which was improved from the DHD (Deep Hole Drilling) method to improve the accuracy of deep hole machining. A hole was drilled through the pipe from the outer side at the target position, as shown in Fig. 3. After drilling, the diameter of the hole was measured at frequent intervals through the full thickness. The residual stresses were calculated from the differences between the measured diameters before and after stress release based on elasticity theory. In this study, residual stress measurements were performed at the center of the seam weld and the fusion line of the girth weld.

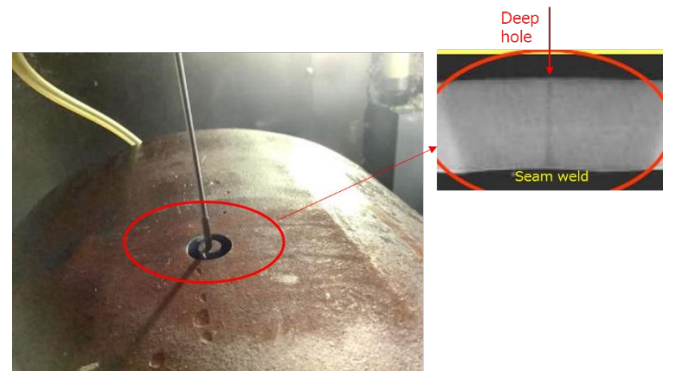


Fig. 3 Drilling position for MIRS method

### Fracture toughness test

A fracture toughness test based on ASTM E1820-2019 was conducted under air and 21 MPa gaseous hydrogen. The crack extension was measured by the unloading compliance method. The configuration of the specimen for the fracture toughness test under high pressure gaseous hydrogen is shown in Fig. 4. A CT specimen with a thickness of one-half inch (1/2 inch-CT) was used. All specimens were machined with a side-groove. Specimens were taken from the pipe wall as well as the seam weld and girth weld. “BM specimens,” in which the notch was located in the base metal, were taken from the inside pipe wall, which is close to the hydrogen contact surface in a hydrogen pipeline. “Seam specimens,” in which the notch was located at the center of the seam weld, were also taken from the inside pipe wall. “GW specimens,” in which the notch was located at the center of the girth weld, were taken from the inside pipe wall, and “FL at GW specimens,” with the notch located at the fusion line of the girth weld, were taken from the inside pipe wall. Three specimens from each notch location were subjected to a fracture toughness test in a pure hydrogen atmosphere with a pressure of 21 MPa, and one specimen from each location was also subjected to a fracture toughness test in air in order to compare the difference in the fracture behaviors under the air and high pressure hydrogen conditions.

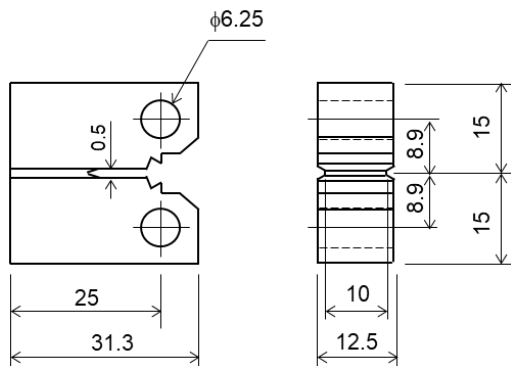


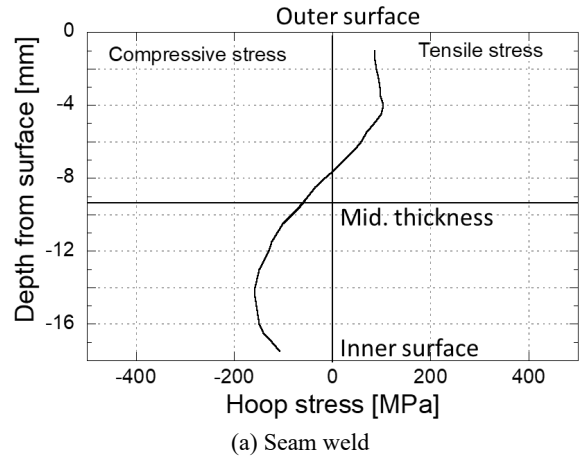
Fig. 4 Fracture toughness test specimen (0.5TCT)

## RESULTS AND DISCUSSION

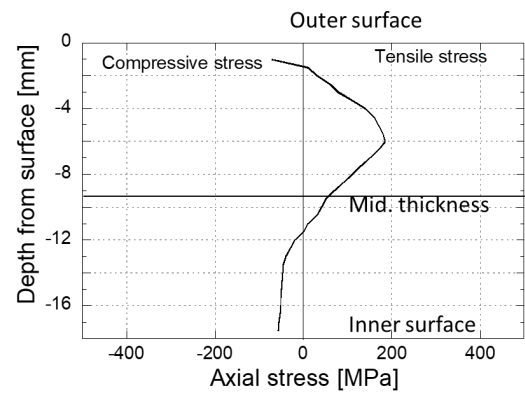
### Residual stress in HFW pipe

Figure 5 shows the relationship between the residual stress and depth from the outer surface. In the seam weld results shown in Fig. 5 (a), residual stress in the hoop direction indicates tension on the outer surface and compression on the inner surface. It is considered that the hoop residual stress distribution occurs as a result of the pipe forming process, that is, expansion of the outer surface and compression of the inner surface. In the results for the fusion line of the girth weld in Fig. 5 (b), residual stress in the axial direction indicates tension on the outer side and compression on the inner side. It is considered that the axial residual stress distribution occurs due to the girth weld, which is influenced by the weld pass.

In the integrity assessment based on the ECA analysis for hydrogen pipelines in this study, the residual stress at the inner surface is most important for considering secondary stress in the FAD calculation because a surface crack is assumed to be located on the inner surface of the pipe wall, which is close to the hydrogen contact surface.



(a) Seam weld



(b) Fusion line of girth weld

Fig. 5 Residual stress distribution

### Fracture toughness test in hydrogen

The fracture toughness test was conducted under air and 21 MPa gaseous hydrogen. The crack extension was measured by the unloading compliance method. Figure 6 shows the relationship between the applied load  $P$  and the crack opening displacement  $V_g$ . The peak load in the  $P$ - $V_g$  curve for the high pressure hydrogen condition was smaller than that of the air condition in both the base metal and seam weld specimens. Figure 7 shows the  $J$ - $\Delta a$  curves obtained from the load and the difference of the unloading compliance based on ASTM E1820. The  $J$ - $\Delta a$  curves for the high pressure hydrogen condition are located at much lower positions than those for the air condition. This means that a stable crack due to hydrogen tends to initiate and propagate easily.

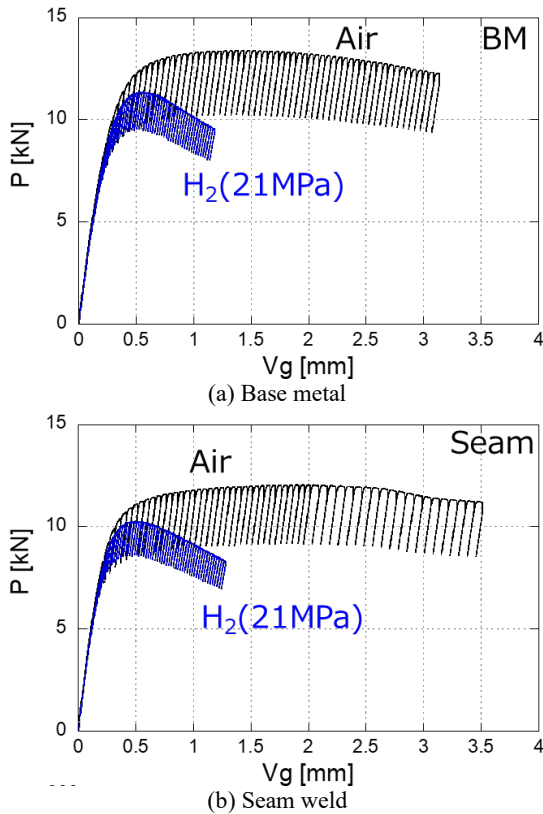
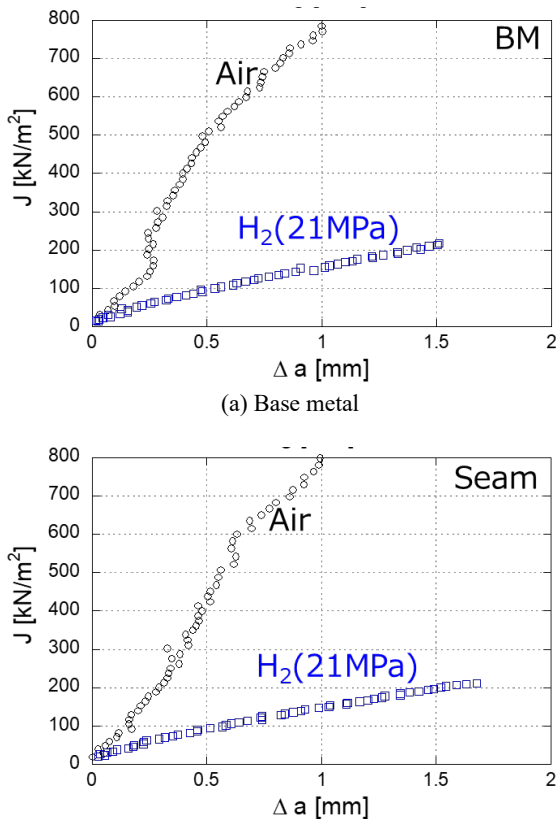


Fig. 6 Relationship between applied load  $P$  and crack opening displacement  $V_g$  in fracture toughness test



(b) Seam weld

Fig. 7 J- $\Delta a$  curves of X65 HFW linepipe

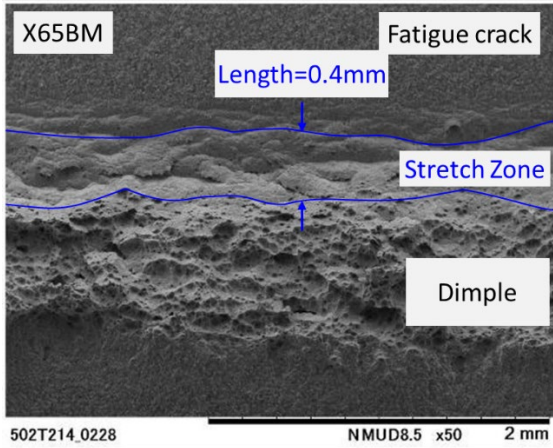
After the fracture test, detailed observation of the fracture surface by SEM was conducted to investigate the early stage of crack initiation. Figure 8 shows the fracture surfaces of the base metal specimens. Under the air condition, as shown in Fig. 8 (a), a stretch zone with a length of approximately 0.4 mm formed at the initial fatigue crack tip, after which dimples were observed as a typical feature of ductile crack initiation. Under the high pressure hydrogen condition in Fig. 8 (b), the stretch zone was not clearly observed at the initial fatigue crack tip, and it appeared to be a quasi-cleavage fracture surface, which is a typical feature of hydrogen-related fracture immediately after an initial fatigue crack. The detailed observation was conducted by tilting the observation field in order to find the stretch zone at the initial fatigue crack tip. Figure 9 shows the result of tilted observation for the hydrogen condition using the tilting angle of 80°. Under the high pressure hydrogen condition, a short length of stretch zone was observed, and its size was only 0.02 mm when converted for the effect of tilting, as shown in Fig. 10. The stretch zone length represented pre-deformation of crack initiation in the hydrogen condition, and was much smaller than that in the air condition. This tendency was the same in the seam weld specimen, GW specimen and FL at GW specimen.

Fracture toughness should be determined as a crack initiation point. Generally speaking, a plastic zone in front of an initial crack leads to blunting due to the localization of deformation, and a stable crack initiates as the result of critical local deformation at the initial crack tip, which is strongly related to the size of blunting. The critical blunting is observed as the result of the stretch zone length on the fracture surface in the fracture toughness test specimen, as shown in Fig. 11. According to ASTM E1820, fracture toughness  $J_{IC}$  is defined as the intersection point between the J- $\Delta a$  curve obtained from the experimental data and the 0.2 mm offset line from the blunting line as an engineering determination method. This definition is widely used for the air condition. The intersection point is conservative in comparison with the actual stretch zone length in the result for the stretch zone length in Fig. 7(a). On the other hand, the stretch zone length under the high pressure hydrogen condition is much smaller than 0.2 mm, which is the value of the offset. This means that fracture initiation under the high pressure hydrogen condition should be determined at an earlier stage of the J-R curve compared to the original determination for the air condition. Therefore, the intersection point between the blunting line calculated by the function of yield stress and the J-R curve, as shown in Fig. 12, is proposed as the fracture determination for the high pressure hydrogen condition. As also shown in Fig. 12, the intersection point is very close to the length of the stretch zone in the high pressure hydrogen condition.

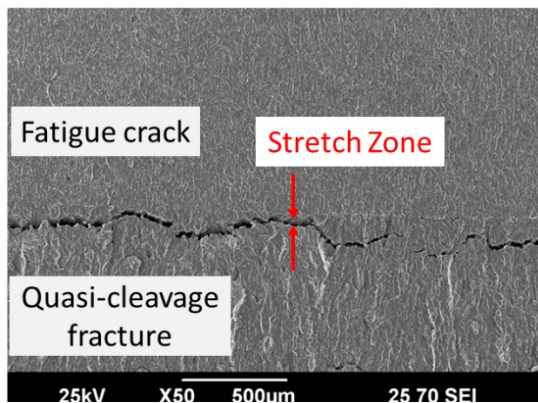
Table 3 shows the results of the evaluation using the critical J-value for crack initiation proposed above as  $J_{IC}$  and the original determination using the 0.2 mm offset line as  $J_{C0.2}$ . Each J value representing the critical fracture toughness with stable crack growth can be recalculated as  $K(J)$  by Eq. (1). The MOTE (Minimum of three equivalent) values of  $K(J_c)$  are used for the integrity assessment based on the FAD analysis in the following section.

$$K_J = \sqrt{\frac{JE}{(1-\nu^2)}} \quad (1)$$

where  $K_J$  is the stress intensity factor determined from a value of  $J$ , and  $E$  and  $\nu$  are the elastic modulus and the Poisson's ratio of the material, respectively.



(a) Air condition



(b) 21 MPa high pressure hydrogen condition

Fig. 8 Fracture surface of base metal specimen

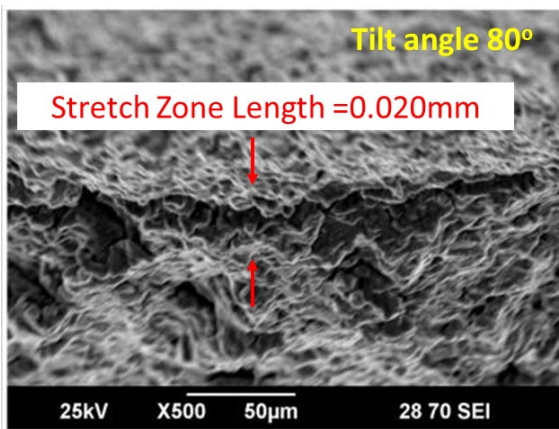


Fig. 9 Stretch zone under high pressure hydrogen condition

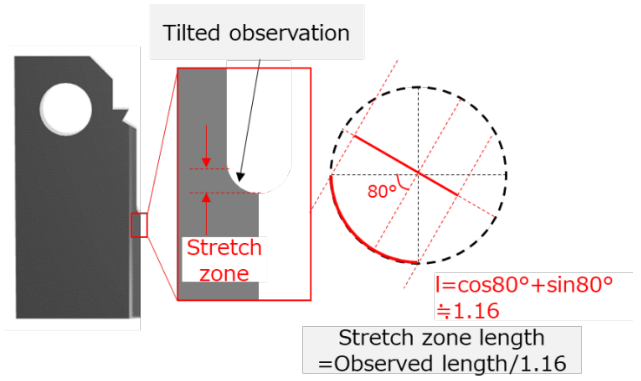


Fig. 10 Calculation method of stretch zone under high pressure hydrogen condition

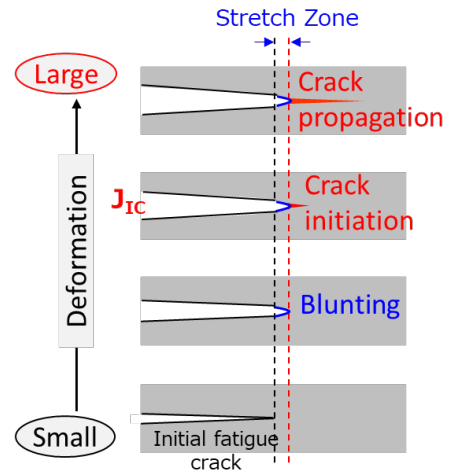


Fig. 11 Schematic illustration of definition of crack initiation

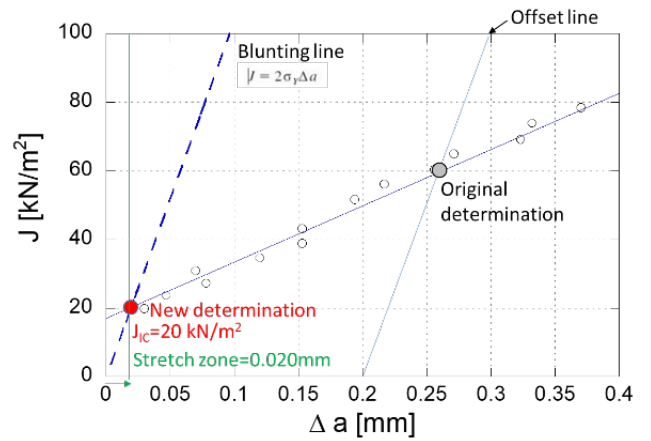


Fig. 12 New determination of crack initiation in J-Δa curve under high pressure hydrogen condition

Table 3 Critical fracture toughness in hydrogen condition

	BM				Seam			
	J <sub>IC</sub>	K(J <sub>IC</sub> )	J <sub>CO,2</sub>	K(J <sub>CO,2</sub> )	J <sub>IC</sub>	K(J <sub>IC</sub> )	J <sub>CO,2</sub>	K(J <sub>CO,2</sub> )
1	20	67	60	117	23	72	60	117
2	21	69	64	120	23	72	57	114
3	21	69	68	124	26	77	58	115
MOTE	21	68	64	120	24	74	58	115

	GW				F.L. at GW			
	J <sub>IC</sub>	K(J <sub>IC</sub> )	J <sub>CO,2</sub>	K(J <sub>CO,2</sub> )	J <sub>IC</sub>	K(J <sub>IC</sub> )	J <sub>CO,2</sub>	K(J <sub>CO,2</sub> )
1	20	67	55	112	16	60	55	112
2	16	60	52	108	16	60	55	112
3	21	69	56	113	17	62	57	114
MOTE	19	65	54	111	16	61	56	112

MOTE: Minimum of three equivalent value

$$K_r = \sqrt{\frac{EJ_f}{(1 - \nu^2)}}$$

**FRACTURE ASSESSMENT BASED ON API579/ASME FFS**

The toughness requirement based on an assumed surface crack located at the inner surface of the pipe was calculated by using the ECA in API 579-1/ASME FFS-1 based on an FAD calculation. In the integrity assessment in this study, a surface crack is assumed to be located on the inner surface of the pipe wall because the inside pipe wall is close to the hydrogen contact surface.

FAD is a method for assessing the safety and integrity of cracked and damaged metallic structures. The FAD method adopts an assessment curve which uses the ratio of the stress intensity factor  $K_I$  to the fracture toughness  $K_{IC}$ , defined as  $K_r$ , as the vertical (fracture) axis, and the ratio of the applied load  $P$  to the plastic collapse load  $P_c$ , defined as  $L_r$ , as the horizontal (plasticity) axis. If the service (assessment) point falls inside the assessment curve, the structure is considered safe, and otherwise, the structure is deemed unsafe, as shown in Fig. 13.

In this study, the toughness requirement was calculated based on Grade X65 linepipes with the size of 16" (406.4 mm) OD and 16.7 mm WT geometry, with a surface flow parallel to the seam weld and a surface flow parallel to the girth weld. The flaw has a semi-elliptical shape with a surface length of 25 mm and a flaw depth of 3 mm. The design factor was set from in the range from 0.4 to 0.72. The material fracture toughness  $K_{IC}$  under the high pressure hydrogen condition was obtained by the unloading compliance method in ASTM E1820, as shown in Table 3, as the value of MOTE  $K(J_c)$ .

It has been reported that the seam weld residual stress of HFW pipes remains as a result of the pipe forming press but is tensile on the outer surface and compressive on the inner surface (Igi et al., 2014). The seam weld residual stress of the tested HFW also displayed the same tendency. Therefore, the secondary stress derived from the seam weld residual stress in the hoop direction, which is the flaw opening direction, is considered to be zero because compressive residual stress remained on the inner surface. The residual stress in the axial direction showed tension on the outer side and compression on the inner side, but because the distribution of axial residual stress depends on the weld pass, the ECA calculation in this study was conducted for a secondary stress (residual stress) condition of zero and the maximum value of 200 MPa in the girth weld assessment.

The corresponding values of  $K_r$  and  $L_r$  for different values of residual stress and material toughness were computed, and the crack driving force for fracture was obtained as the toughness requirement for a high pressure hydrogen pipeline. Figure 14 shows the relationship between the crack driving force with each residual stress condition and the design factor (fd) for Grade X65 linepipes, together with the obtained material toughness of the tested material in the base metal, seam weld, girth weld and fusion line at the girth weld. As the design factor increases, the crack driving force for fracture increases, which means the toughness

requirement also increases. The obtained material toughness based on the new determination of the tested material exceeds both the minimum requirement fracture toughness in ASME B31.12 and the calculated toughness requirement based on the FAD calculation shown in Fig. 14. Based on these results, it is concluded that the developed API X65 HFW pipe "Mighty Seam" is applicable to high pressure hydrogen pipelines.

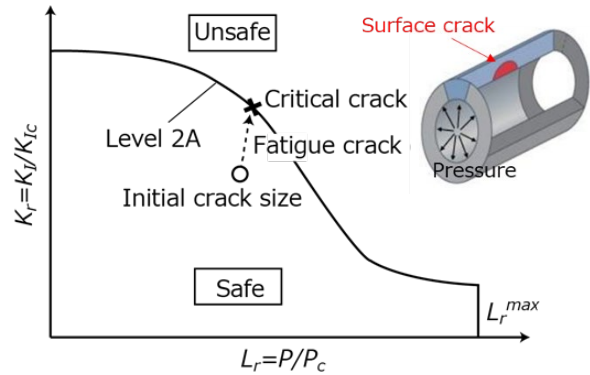
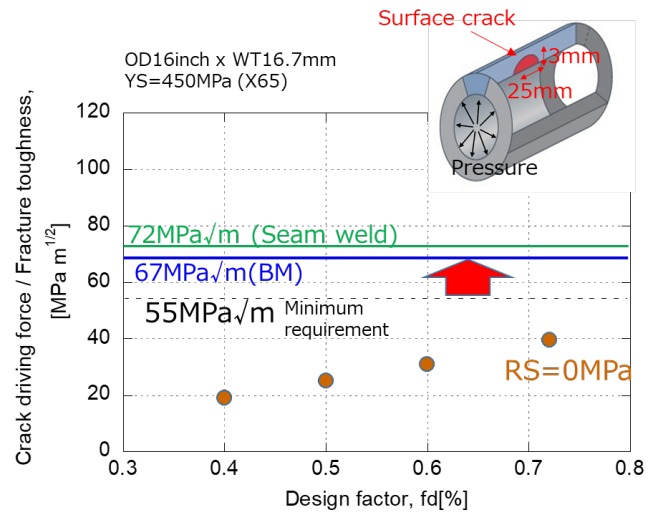
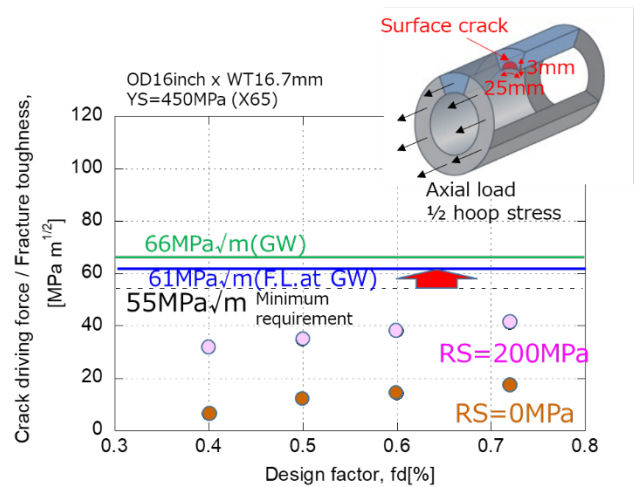


Fig. 13 Failure assessment diagram (FAD)



(a) Assessment of axial crack in base metal (BM) and seam weld



## (b) Assessment of circumferential crack in girth weld

Figure 14 Fracture assessment of pressurized pipe with assumed surface crack

## CONCLUSIONS

In this study, fracture toughness tests based on ASTM E1820 were conducted under air and high pressure hydrogen conditions, and the difference of the fracture behaviors under the two conditions was investigated using an API Grade X65 HFW pipe. The toughness requirement based on an assumed surface crack located in the inner surface of the pipe was also calculated by the ECA method in API 579-1/ASME FFS-1 based on an FAD calculation. The applicability of the API X65 HFW pipe to high pressure hydrogen pipelines was discussed based on the ECA results. The following conclusions were obtained.

- (1) In a seam-welded HFW pipe, residual stress in the hoop direction indicates tension on the outer surface and compression on the inner surface. Residual stress in the axial direction indicates tension on the outer side and compression on the inner side.
- (2) In comparison with the air condition, a stable crack due to hydrogen initiates and propagates easily from an initial crack in the  $J_{IC}$  test, and the size of the stretch zone under the high pressure hydrogen condition is much smaller than that under the air condition. Determination of crack initiation is proposed using the intersection between the linear J-R approximate line and the blunting line as  $J_{IC}$ .
- (3) The obtained  $J_{IC}$  of the tested material, including the seam weld and girth weld, exceeded both the minimum requirement for fracture toughness in ASME B31.12 and the calculated crack driving force based on the ECA. Therefore, it was concluded that the developed API X65 HFW pipe "Mighty Seam<sup>®</sup>" is applicable to high pressure hydrogen pipelines.

## ACKNOWLEDGEMENTS

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