Influence of Sheared Edge on Hydrogen Embrittlement Resistance in an Ultra-high Strength Steel Sheet

Masataka YOSHINO,^{1)*} Yuki TOJI,³⁾ Shusaku TAKAGI³⁾ and Kohei HASEGAWA²⁾

1) Steel Research Laboratory, JFE Steel Corporation, 1, Kawasaki-cho, Chuo-ku, Chiba, 260-0835 Japan.

2) Steel Research Laboratory, JFE Steel Corporation, 1, Kokan-cho, Fukuyama, Hiroshima, 721-8501 Japan.

3) Steel Research Laboratory, JFE Steel Corporation, 1-1, Minamiwatarida-cho, Kawasaki-ku, Kawasaki, Kanagawa, Japan.

(Received on November 22, 2013; accepted on February 19, 2014; originally published in *Tetsu-to-Hagané*, Vol. 99, 2013, No. 4, pp. 302–311)

Automotive parts made from steel sheets normally have sheared edges, which have been reported to decrease the hydrogen embrittlement (HE) resistance of ultra-high strength steel (UHSS) sheets. However, the mechanism on the detrimental effect of the sheared edge on HE resistance is not yet clearly understood. In this study, the influence of the edge condition in UHSS sheets on the HE property was investigated using a 1 180 MPa grade steel sheet. The HE resistance of specimens with the edges ground or as-sheared was evaluated by the U-bend method. Two types of as-sheared specimens, which had been bent so that either the burnished surface or the fracture surface was the outer side, were prepared. The specimens with the ground edges did not fracture under any conditions. The fracture stress of the fracture surface specimens was significantly lower than that of the burnished surface specimens. Microcracks were observed at the edge of the specimens except for the ground specimens, and larger microcracks were observed in the fracture surface specimens. Fracture stress drastically decreased as the microcrack length increased. The threshold of the stress intensity factor K decreased with increasing diffusible hydrogen content. When the threshold stress intensity factor at each diffusible hydrogen content was defined as K_H , the fracture condition was described as $K > K_H$. The reason why the fracture stress in the as-sheared specimen decreased was considered to be that K increased due to the microcracks introduced by bending.

KEY WORDS: delayed fracture; diffusible hydrogen; dual-phase steel; shearing; stress intensity factor.

1. Introduction

Ultra-High Strength Steel (UHSS) sheets with tensile strength (TS) over 980 MPa have been applied to automotive parts for weight saving and improvement of the crash-worthiness of car bodies.^{1,2)} However, a risk of hydrogen embrittlement fracture called delayed fracture has been reported in high strength steel bolts with TS over 1 100 MPa.³⁾ Delayed fracture is considered to be caused by diffusible hydrogen which enters the steel from the environment. Therefore, not only high formability suitable for press-forming of automotive parts, but also sufficient hydrogen embrittlement resistance is required of UHSS sheets.

The factors which cause delayed fracture in press-formed parts are applied strain, residual stress and diffusible hydrogen. Several methods of evaluating the influence of these factors on delayed fracture susceptibility have been reported.^{4–11)} Hosoya *et al.* reported the hydrogen embrittlement susceptibility of high strength steel sheets which were evaluated by the 4-point bending and U-bend methods.⁴⁾ Toji *et al.* systematically investigated the influence of strain, stress and diffusible hydrogen content on hydrogen embrittlement resistance, and suggested a 3-dimensional map concept in

which the conditions for the occurrence of delayed fracture are evaluated by strain, stress and diffusible hydrogen content.⁵⁾ Hayashi *et al.* suggested an evaluation technique in which hydrogen is introduced by a cathodic charge technique into a steel sheet tightened with a bolt after U-bending.⁶⁾ In the report, Hayashi *et al.* showed that hydrogen cracking occurred remarkably early in specimens without edge grinding as compared with edge-ground specimens.

UHSS sheets are subject to press-forming without edge grinding after blanking by shearing or punching, and as a result, press parts produced from UHSS sheets have sheared or punched edges.¹²⁾ Therefore, in order to evaluate the hydrogen embrittlement resistance of press-formed automotive parts, it is important to understand the hydrogen embrittlement behavior of UHSS sheets with sheared edges.

In this study, the influence of the edge condition, applied stress, bending strain and diffusible hydrogen content on hydrogen embrittlement resistance in UHSS sheets with sheared edges was investigated in detail. Furthermore, the mechanism of the detrimental effect of the sheared edge on hydrogen embrittlement resistance was discussed.

2. Experimental Procedure

The steel used in this study was a 1 180 MPa grade ferrite-

* Corresponding author: E-mail: ma-yoshino@jfe-steel.co.jp

DOI: <http://dx.doi.org/10.2355/isijinternational.54.1416>

Table 1. Mechanical properties of steel studied.

YS (MPa)	TS (MPa)	El (%)
906	1 214	14.5

Fig. 1. SEM micrograph of steel studied (F: Ferrite, M: Martensite).

Fig. 2. Schematic illustration of as-sheared specimen.

martensite dual-phase steel sheet (thickness: 1.6 mm).^{1,2)} The mechanical properties and SEM microstructure of the steel are shown in **Table 1** and **Fig. 1**. The steel has the complex microstructure which contains approximately 40% of ferrite and 60% of martensite in volume fraction. The size of both grains is approximately 10 μm or less. The steel sheet was sheared to dimensions of 100 mm $\times$ 30 mm, as shown in **Fig. 2**. The rolling direction was parallel to the longitudinal direction of the specimen. Two longitudinal edges were sheared so that the burnished surface and the fracture surface on both edges were at the same side. Specimens with the edges ground after shearing (hereafter referred to as “ground specimen”) were also prepared.

Figure 3 shows a schematic illustration of the preparation procedure of U-bending specimens for the hydrogen embrittlement test. Specimens were bent perpendicular to the longitudinal direction with a bending radius (R) of 5 mm or 10 mm into a U-shape. Two types of as-sheared specimens were prepared by bending so that the burnished surface (**Fig. 4(a)**) or the fracture surface (**Fig. 4(b)**) was the outer side of the specimen. Hereafter, the specimens shown in **Figs. 4(a)** and **4(b)** are referred to as “burnished surface specimen” and “fracture surface specimen”, respectively. After bending, a maximum surface stress of 900 MPa was applied to the top of the specimen in the longitudinal direction by tightening its spring-back with a bolt. The surface stress at the top of the specimen was measured by means of X-ray diffractometer with the iso-inclination method.¹³⁾

The time to fracture of these specimens was investigated by immersing the specimens in a solution of hydrochloric

Fig. 3. Preparation procedure of specimen for hydrogen embrittlement test (R : bending radius).

Fig. 4. Macroscopic photographs of specimens bent to bending radius $R = 5$ mm; (a) burnished surface specimen and (b) fracture surface specimen.

acid (HCl) with pH 1 to 4 up to a maximum time of 96 h. The reason for ending the test at 96 h is that there have been no specimens so far which fractured after 96 h in 200 h immersion tests. The amount of hydrogen that entered the steel when immersed in the HCl solutions was measured by thermal desorption analysis (TDA) using gas chromatography. A 5 mm width specimen for TDA was cut from the top of each bent specimen and it was kept in liquid nitrogen to prevent the emission of diffusible hydrogen. Before inserting the specimen into TDA device, it was polished in a dry condition followed by the ultrasonic cleaning. It took approximately 10 minutes after taking out the specimen from the liquid nitrogen container until TDA analysis of the specimen starts. A GC7000-T produced by J-SCIENCE LAB Co. Ltd. was used for the analysis. The heating rate was 200°C/h, and the sampling interval was 5 min. In this study, the integration of the first evolution peak (below about 200°C) was defined as the diffusible hydrogen content.

3. Results

3.1. Influence of Shearing on Hardness Near Edge

Figure 5 shows SEM micrographs of the as-sheared edge (Fig. 5(a)) and the ground edge after shearing (Fig. 5(b)) at the ND–RD section. The ratio of burnished surface to the thickness was approximately 25%. The burnished surface and the fracture surface generated by shearing were removed by grinding (Fig. 5(b)). **Figure 6** shows the change in hardness on the ND–TD section of the as-sheared steel sheet with distance from the sheared edge. The hardness test was performed with a load of 20 g and a load time of 15 s using a micro Vickers hardness tester. Also, the data of bur-

Fig. 5. SEM micrographs of steels at ND–RD section surface; (a) as-sheared surface and (b) ground surface after shearing.

Fig. 6. Change in hardness of the as-sheared steel with distance from sheared edge.

nished surface was measured at the part approximately 100 μm away from ND surface, and the date of fracture surface was measured at the part approximately 50 μm away from another ND surface. Although a high hardness value of HV480 was measured near the sheared edge, the hardness decreased with the distance from the sheared edge and converged on a steady value of HV360–400 at positions around 200 μm or more from the edge. Therefore, it is considered that the volume affected by shearing was approximately within 200 μm from the sheared edge.

3.2. Influence of Sheared Edge on Fracture Behavior

Figure 7 shows the influence of the edge condition on the relationship between the applied stress and time to fracture of the bent specimens ($R = 10$ mm) when immersed in the pH 1 HCl solution. The ground specimens did not fracture at any applied stress. Thus, their fracture stress was estimated as over 900 MPa. On the other hand, the burnished surface specimen fractured at 900 MPa and the fracture surface specimens fractured at 450 MPa or more, of which fracture occurred from the edge. Therefore, the critical fracture stress decreased significantly in the as-sheared specimen in comparison with the ground specimen. These results were qualitatively in agreement with the results reported by Hayashi *et al.* in an investigation using the cathodic charge technique.⁶⁾ Furthermore, the critical fracture stress in the fracture surface specimen decreased more drastically as compared with that in the burnished surface specimen.

Figure 8 shows the influence of the edge condition on

Fig. 7. Relationship between applied stress and time to fracture of bent steel ($R = 10$ mm) when immersed in pH 1 HCl solution.

Fig. 8. Change in content of diffusible hydrogen entering specimen ($R = 10$ mm) with immersion time in pH 1 HCl solution.

Fig. 9. SEM micrographs of top of bent specimens ($R = 5$ mm); (a) burnished surface specimen, (b) fracture surface specimen, and (c) overview of specimen (arrow indicates observation direction).

the diffusible hydrogen content which entered the bent specimens ($R = 10$ mm) when immersed in the pH 1 HCl solution. The relationship between immersion time and diffusible hydrogen content was almost the same in each specimen. This result indicates the as-sheared specimens fractured at clearly lower stress even though almost the same quantity of diffusible hydrogen entered the as-sheared specimens as compared with that entered the ground specimens. After the diffusible hydrogen content reached its peak at 5 h, it decreased with the immersion time. This is attributed to the fact that the average diffusible hydrogen content of the overall thickness measured by TDA decreased due to dissolution of the surface of the specimen by the long-term immersion, where larger strain was induced.¹⁴⁾ The influence of dissolution of the specimens by immersion in the HCl solution on the evaluated results is discussed in section 4.1.

SEM photographs taken from around the top of the specimen bent with $R = 10$ mm after shearing are shown in **Fig. 9**. Although no cracks were observed in any specimen before bending, cracks were observed at the edge of the as-sheared specimen after bending. No cracks were observed in the ground specimen after bending. The crack observed in the burnished surface specimen (**Fig. 9(a)**) was small, being about $3 \mu\text{m}$. However, in comparison with the burnished surface specimen, the crack observed in the fracture surface specimen (**Fig. 9(b)**) was about $30 \mu\text{m}$, and it is opened widely in the thickness direction. The reason for generation of large cracks in fracture surface specimen was considered that the stress concentration easily occurred when bending; it is due to formation of ripped morphology in the thickness direction at the edge of fracture specimen by shearing. **Figure 10** shows photographs which compare the crack at the top of the fracture surface specimen ($R = 10$ mm) before and after immersion in the pH 1 HCl solution. Many cracks with sizes of several dozen to 100 micrometers were observed before immersion (**Fig. 10(a)**). The crack indicated by the arrow, which was generated by bending, propagated until it penetrated in the thickness direction when the specimen was immersed in the pH 1 HCl solution for 1.5 h (**Fig. 10(b)**). According to these results, the fact that the critical fracture stress in the as-sheared specimens decreased drastically in comparison with the ground specimen is attributed to the crack at the edge which was generated by bending,

Fig. 10. Macroscopic photographs of fracture surface specimen ($R = 10$ mm) before and after immersion in pH 1 HCl solution for 1.5 h; (a) before immersion and (b) after immersion for 1.5 h. Arrows show the same cracks before and after immersion.

and the difference in the critical fracture stress between the burnished surface specimen and the fracture surface specimen is considered to be due to the difference in the length or propagation behavior of the crack.

3.3. Influence of Diffusible Hydrogen Content on Fracture Behavior

Figure 11 shows the relationship between applied stress and time to fracture of burnished surface specimens (**Fig. 11(a)**) and fracture surface specimens (**Fig. 11(b)**) respectively, when specimens were immersed in pH 1, 3 and 4 HCl solutions. Although the time to fracture under pH 3 was slightly longer than that under pH 1, the critical fracture stress under pH 3 was the same as that under pH 1. That is, the critical fracture stress of burnished surface specimen and fracture surface specimen was 650 MPa and 200 MPa, respectively, and it was independent of the pH of the solution. In contrast, when immersed in the pH 4 HCl solution,

Fig. 11. Stress-time to fracture curves of specimen ($R = 10$ mm) when immersed in HCl solutions with various pH levels; (a) burnished surface specimen and (b) fracture surface specimen.

Fig. 12. Change in content of diffusible hydrogen entering the ground specimen ($R = 10$ mm) with immersion time in HCl solutions with various pH levels.

the burnished surface specimen did not fracture at 900 MPa, and the critical fracture stress in the fracture surface specimen increased from 200 MPa to 650 MPa. **Figure 12** shows the change in the content of diffusible hydrogen entering the ground specimen ($R = 10$ mm) with immersion time in pH 1–4 HCl solutions. The lower pH showed higher diffusible hydrogen content than the higher pH in any immersion time. The maximum values of diffusible hydrogen under the pH 1, 3 and 4 solutions were 0.54 ppm, 0.30 ppm and 0.17 ppm, respectively. Therefore, it is considered that the increase of the critical fracture stress in pH 4 HCl solution was due to a decrease in the diffusible hydrogen content entering the steel.

Fig. 13. Comparison of time to fracture curves of burnished surface specimens with bending radii of 5 mm and 10 mm when immersed in HCl solutions with various pH levels; (a) pH 1, (b) pH 3 and (c) pH 4.

3.4. Influence of Bending Radius on Fracture Behavior

The stress–time to fracture curves of the burnished surface specimen and the fracture surface specimen ($R = 5$ mm) when immersed in pH 1–4 HCl solutions are shown in **Figs. 13** and **14**, together with the results of the $R = 10$ mm specimen which were shown in Fig. 7. The critical fracture stress in the burnished surface specimen did not depend on the bending radius, and was unchanged at 650 MPa in the pH 1 and 3 solutions (**Figs. 13(a)** and **13(b)**) and at 900 MPa in the pH 4 solution (**Fig. 13(c)**).

However, the fracture surface specimen ($R = 5$ mm) fractured at 150 MPa in both the pH 1 and 3 solutions (**Figs. 14(a)** and **14(b)**), but did not fracture at $R = 10$ mm. In other words, the critical fracture stress decreased when the bending radius was decreased. Meanwhile, the time to fracture when the specimens with both $R = 5$ mm and $R = 10$ mm fractured was almost the same under all pH levels in both the burnished surface specimen and the fracture surface specimen, indicating that the influence of the bending radius on the time to fracture was small.

Fig. 14. Comparison of time to fracture curves of fracture surface specimens with bending radii of 5 mm and 10 mm when immersed in HCl solutions with various pH levels; (a) pH 1, (b) pH 3 and (c) pH 4.

4. Discussion

In this chapter, in order to clarify the reason why the critical fracture stress was different between the burnished surface specimen and the fracture surface specimen first, the influence of dissolution of the specimen during immersion in the HCl solution on the evaluated result is investigated, and then the deterioration mechanism of the critical fracture stress in as-sheared specimens is discussed from the point of view of local hydrogen accumulation near the sheared edge and material damage near the sheared edge by bending.

4.1. Influence of Dissolution of Specimen on Critical Fracture Stress

Figure 15 shows the weight loss (Fig. 15(a)) and the amount of dissolution from the edge in the width direction of the specimen (Fig. 15(b)) when ground specimens ($R = 10\text{ mm}$) were immersed in pH 1–4 HCl solutions. As the weight loss when specimens were immersed in the pH 3 and 4 HCl solutions was below 2%, dissolution was essentially

Fig. 15. Change in (a) weight reduction and (b) dissolved distance from edge of ground specimens ($R = 10\text{ mm}$) when immersed in HCl solutions with various pH levels.

Fig. 16. Change in crack length of specimens ($R = 10\text{ mm}$) with immersion time in pH 1 HCl solution.

insignificant. However, when immersed in the pH 1 HCl solution, approximately 6% of the specimen was dissolved in 24 h, and approximately 28% was dissolved in 96 h. In addition, the dissolution of the specimen in the width direction from the edge in the pH 1 HCl solution was about $60\text{ }\mu\text{m}$ in 24 h and about $260\text{ }\mu\text{m}$ in 96 h. Almost the same results were obtained with the burnished surface specimen and the fracture surface specimen.

Figure 16 shows the change in crack length with immersion time in the pH 1 HCl solution for the burnished surface specimen and fracture surface specimen with $R = 10\text{ mm}$ when surface stress of 900 MPa was applied. The crack introduced by bending propagated over 0.5 mm in 4 h in the burnished surface specimen and in 1.5 h in the fracture surface specimen. The crack observed in the fracture surface specimen propagated at a constant rate of about 2.4 mm/h. In contrast, although the crack observed in the burnished

surface specimen propagated slowly at approximately 0.8 mm/h until about 1 h after crack initiation, it propagated rapidly at 9.2 mm/h after 1 h.

As shown in Fig. 7, the time to fracture in the burnished surface specimen and the fracture surface specimen was a maximum of 10 h in the pH 1 HCl solution. Based on the fact that the dissolution distance from the edge was about 30 μm after immersion for 10 h (Fig. 15(b)), the hardened layer which was generated by shearing sufficiently remained. Furthermore, the crack growth rate in the burnished surface specimen and the fracture surface specimen was two or three orders larger than the dissolution rate of the specimen, as shown in Fig. 16. That is, it is obvious that the crack at the edge propagated rapidly as a result of immersion in the HCl solution rather than dissolution of the volume affected by shearing. Consequently, it is considered that the influence of dissolution of the specimen by immersion in the HCl solution on the critical fracture stress was negligible in the pH 1 HCl solution, as well as in the pH 3 and 4 solutions.

4.2. Influence of Local Hydrogen Accumulation Near Sheared Edge

Hydrogen is usually understood to be trapped by material defects such as dislocations and point defects,^{15,16)} and diffusible hydrogen entering steel increases with increasing strain and/or material defects.^{14,17,18)} For instance, Toji *et al.*¹⁴⁾ investigated the influence of rolling strain on the diffusible hydrogen content entering 1 180 MPa grade DP steel sheets, which is the same steel as that used in this study, and reported that the diffusible hydrogen content entering the steel sheets increased with increasing rolling strain. Since shearing is a process which involves large plastic deformation, large plastic strain is also introduced at the sheared edge compared with the part which is not affected by shearing deformation. Therefore, the diffusible hydrogen content at the sheared edge was investigated.

Steel sheets without deformation or with cold-rolling with 15% reduction were used in this investigation. This is because it is difficult to extract a sample which only includes the edge part from the top of bent specimens. According to the following equations, the equivalent strain induced by cold-rolling with 15% reduction is almost equal to that at the top of the bent specimen ($R = 5$ mm).

$$\varepsilon_{eq} = \frac{2}{\sqrt{3}} \ln \left(\frac{t_0}{t} \right) \dots \dots \dots (1)$$

$$\varepsilon_{eq} = \frac{t_0}{2R + t_0} \times \frac{2}{\sqrt{3}} \dots \dots \dots (2)$$

where ε_{eq} is the equivalent strain, t_0 is the thickness of the prepared steel sheet, t is the thickness after cold-rolling and R is the bending radius.

From these steel sheets, 60 mm length $\times$ 1 mm width specimen with the longitudinal direction parallel to the rolling direction was cut from the sheared edge at 1 mm intervals by using abrasive cutting. The reason for using abrasive cutting was to avoid the generation of new sheared edges except for the original sheared edge.

Quantitative analysis of diffusible hydrogen entering the specimen was performed after immersion in a 0.01% ammonium thiocyanate (NH_4SCN) solution for a maximum of 96

h. The NH_4SCN solution is commonly used for hydrogen charging in the FIP test.²⁰⁾ Takagi *et al.* reported that the diffusible hydrogen content entering steel can be controlled by changing the amount of NH_4SCN in the solution with hardly any dissolution.^{21,22)} The 0.01% NH_4SCN solution used in this study introduces almost the same diffusible hydrogen content as the pH 3 HCl solution introduced into the bent steel specimens. Additionally, 6 specimens with a 60 mm length $\times$ 1 mm width, which were immersed at the same time, were analyzed at once by TDA to enhance measurement accuracy.

Figure 17 shows the hydrogen evolution curves of the as-sheared specimen when immersed in the 0.01% NH_4SCN solution for 24 h. An obvious hydrogen evolution peak was detected below 200°C in the part from the edge to 1 mm of the specimen. However, no hydrogen evolution peak was detected at any temperatures at the part 1 mm or more from

Fig. 17. Influence of sampling position on hydrogen evolution behavior of steel immersed for 24 h in 0.01% NH_4SCN solution.

Fig. 18. Change in content of diffusible hydrogen entering specimens with immersion time in 0.01% NH_4SCN solution; (a) as-sheared and (b) cold-rolled with 15% reduction.

the edge. These results indicated that the diffusible hydrogen locally accumulated near the sheared edge in the as-sheared steel sheet.

The changes in diffusible hydrogen content with immersion time in the specimen with and without cold-rolling with 15% reduction are shown in Fig. 18. In the specimen without cold rolling (Fig. 18(a)), the diffusible hydrogen at the part from the edge to 1 mm became constant at approximately 0.14 ppm after immersion for 24 h, and was higher than that of the part 1 mm or more away from the edge. On the other hand, in the specimen which was cold-rolled with 15% reduction (Fig. 18(b)), no difference in the diffusible hydrogen content was observed between the specimen at the part from the edge to 1 mm and the part 1 mm or more from the edge, and the diffusible hydrogen content increased with increasing immersion time. Although the hardened layer which was generated by shearing remained up to approximately 72 h immersion as shown in Fig. 15, no additional increment of entered diffusible hydrogen by shearing strain was observed at the part from the edge to 1 mm in the specimen which was cold-rolled with 15% reduction. It is considered that the contribution of the strain introduced by shearing at the edge became relatively smaller by strain of 15% cold-rolling, although the reason for this phenomenon did not explicit enough in the study. On the other hand, the critical fracture stress of burnished surface specimen and fracture surface specimen was clearly different as shown in Figs. 13 and 14, though both the specimens have the same sheared edges. Consequently, even if the local hydrogen accumulation near sheared edge was remarkable or not, it is considered that the critical fracture stress was strongly dom-

inated by microcracks which were introduced by bending as shown in Fig. 9.

4.3. Influence of Crack Length on Fracture Stress

Figure 19 shows the histograms of the crack length on the sheared edges of the burnished surface specimens and the fracture surface specimens bent with bending radii of 5 mm and 10 mm. The cracks in the range of ± 5 mm from the center on the top of the bent specimen were measured, and the crack length was defined as the linear distance between the crack opening area at the edge and the crack tip. The number, average crack length and maximum crack length of the cracks showed little change in the burnished surface specimen when the bending radius was reduced from $R = 10$ mm (Fig. 19(a)) to 5 mm (Fig. 19(c)). On the other hand, in the fracture surface specimen, cracks were observed at 5 places at $R = 10$ mm (Fig. 19(b)), but this number increased eightfold by bending at $R = 5$ mm (Fig. 19(d)), which introduced larger strain than $R = 10$ mm. Furthermore, at $R = 5$ mm, the average crack length and the maximum crack length were $30 \mu\text{m}$ and $78 \mu\text{m}$, respectively. Therefore, in the fracture surface specimens, cracks clearly increased in both number and size as the bending radius decreased. When the burnished surface specimen and fracture surface specimen were compared at $R = 5$ mm, the critical fracture stress in the fracture surface specimen was lower than that in the burnished surface specimen (Fig. 7), even though the number of cracks in the burnished surface specimen was larger than that in the fracture surface specimen. Although the influence of the number of cracks on the critical fracture stress has not been clarified in this study, it is considered

Fig. 19. Histogram of crack length in sheared edges of bent specimens; (a) burnished surface specimen ($R = 10$ mm), (b) fracture surface specimen ($R = 10$ mm), (c) burnished surface specimen ($R = 5$ mm) and (d) fracture surface specimen ($R = 5$ mm).

Fig. 20. Influence of maximum crack length on critical fracture stress of specimens when immersed in HCl solutions with various pH levels.

that the critical fracture stress was strongly influenced by the crack length rather than by the number of cracks.

The HCl immersion test results of the burnished surface specimen and fracture surface specimen were summarized by applied stress and the maximum crack length, as shown in Fig. 20. The lines in the figure indicate the maximum stress at which fracture did not occur; that is, fracture did not occur on the low stress side of this line. The critical fracture stress decreased at the maximum crack length of 8 μm in the pH 1 and 3 HCl solutions, and it significantly decreased to 150 MPa or less when the maximum crack length was more than 23 μm . Although the critical fracture stress in the pH 4 HCl solution did not decrease until the maximum crack length of 12 μm , it decreased to 650 MPa and became constant at 23 μm or more. Therefore, there is a tendency for the critical fracture stress to decrease with increasing the maximum crack length, and it is considered that the decrement of the critical fracture stress and the dependence of the critical fracture stress on the maximum crack length were strongly influenced by the diffusible hydrogen content.

Generally, the relationship between crack length and critical fracture stress can be denoted by the following equation, which uses the stress intensity factor K .

$$K = \sigma \sqrt{\pi a} \dots\dots\dots (3)$$

where K is the stress intensity factor, σ is the applied stress, and a is the crack length. In addition, it has been reported that the threshold stress intensity factor decreased when diffusible hydrogen entered steel.²³⁾ When the threshold stress intensity factor at each diffusible hydrogen content was defined as K_H , the fracture condition was described as $K > K_H$.

The relationship among crack length, applied stress and occurrence of fracture in Fig. 20 was rearranged by the diffusible hydrogen content and stress intensity factor, as shown in Fig. 21. The line in the figure indicates the minimum stress intensity factor at which fracture occurred. The diffusible hydrogen content on the horizontal axis is the maximum diffusible hydrogen content when the specimen is immersed in HCl solutions of each pH level, as shown in

Fig. 21. Dependence of hydrogen cracking on stress intensity factor K and diffusible hydrogen content in steel studied.

Fig. 12. K_H greatly decreased to 2.3 $\text{MPa}\cdot\text{m}^{1/2}$ when the amount of diffusible hydrogen increased to 0.30 ppm (pH 3 HCl solution) from 0.17 ppm (pH 4 HCl solution), and it became constant at diffusible hydrogen levels over 0.30 ppm. Accordingly, the reason why the fracture stress in the pH 1 and 3 HCl solutions significantly decreased in comparison with that in the pH 4 HCl solution was considered to be because $K > K_H$ was satisfied at a relatively lower stress intensity factor due to the large decrement of K_H resulting from the increment of the diffusible hydrogen content.

Wriedt and Oriani reported that the hydrogen content entering a material increased with the increment of tensile elastic stress.²⁴⁾ When hydrostatic pressure affects a stress concentration region such as a notch or crack, it is considered that fracture is accelerated by accumulation of hydrogen at the stress concentration region due to the stress-induced diffusion.²⁵⁻²⁷⁾ Furthermore, in a study of the influence of hydrogen accumulation at the crack tip, Gerberich and Chen assumed that fracture occurs when the local hydrogen concentration at the region where the hydrostatic pressure shows its maximum reaches a threshold limit for fracture, and proposed a model in which the threshold stress intensity factor is proportional to the ratio of local hydrogen concentration the crack tip and that at the part which is not affected by stress.²⁸⁾ Although the present study did not determine whether hydrostatic pressure was present at the edge of the U-bent specimens, it seems reasonable to think that the hydrogen embrittlement threshold stress intensity factor K_H would be influenced by that local accumulated hydrogen. Consequently, in order to understand the dependence of the diffusible hydrogen content on K_H in detail, an interpretation considering the accumulation of diffusible hydrogen at the crack tip is an issue in the future.

5. Conclusions

The influence of the edge condition after shearing, applied stress, bending strain and diffusible hydrogen content on hydrogen embrittlement resistance in an UHSS sheet was investigated. The deterioration mechanism of hydrogen embrittlement resistance in an UHSS sheet with a sheared edge was also discussed. The results obtained in this study are summarized as follows:

- (1) The fracture stress of the as-sheared specimens was

lower than that of the ground specimen. It especially decreased in specimens which were bent so that the fracture surface was on the outer side. Critical fracture stress also decreased with increasing diffusible hydrogen content.

(2) The fractures in the as-sheared specimens occurred by propagation of the microcracks which were introduced at the edge of the specimen by shearing and bending.

(3) The fracture stress in the fracture surface specimens showed a further decrease when the bending radius was reduced, although that of the burnished surface specimens was independent of the bending radius. The reason why the fracture stress decreased in the fracture surface specimens was due to generation of larger cracks at the edge resulting from the reduction of the bending radius.

(4) Although the fracture stress in the as-sheared specimens decreased with increasing crack length, the decrement of fracture stress in a pH 4 HCl solution was relatively smaller than that in pH 1 and 3 HCl solutions.

(5) When the results with or without fracture at each diffusible hydrogen content were arranged by the stress intensity factor K , the threshold stress intensity factor decreased with increasing diffusible hydrogen content.

(6) The reason why the critical fracture stress in a bent UHSS sheet with a sheared edge decreased in comparison with the ground specimen was considered to be because $K > K_H$ was satisfied at a relatively lower stress due to generation of microcracks by bending at the edge which had been damaged by shearing.

REFERENCES

- 1) Y. Toji, K. Hasegawa, H. Shigemoto, H. Kawabe, T. Fujita, Y. Tanaka, H. Nakamura, H. Ishida and H. Sakamoto: *Rev. Automot. Eng.*, **30** (2009), 159.
- 2) Y. Toji, K. Hasegawa, K. Kawamura, H. Kawabe and H. Shigemoto: *Materia Jpn.*, **48** (2009), 129.
- 3) S. Matsuyama: Delayed Fracture, The Nikkan Kogyo Shimibun LTD., Tokyo, (1989).
- 4) Y. Hosoya, S. Tsuyama, Y. Nagataki, S. Kanetoh, T. Izuishi and Y. Tanaka: *NKK Tech. Rep.*, **145** (1994), 33.
- 5) Y. Toji, S. Takagi, M. Yoshino, K. Hasegawa and Y. Tanaka: *Tetsu-to-Hagané*, **95** (2009), 887.
- 6) K. Hayashi, N. Fujita, S. Takebayashi, M. Takahashi and Y. Sakuma: *Materia Jpn.*, **44** (2005), 254.
- 7) K. Hayashi, N. Fujita and M. Takahashi: *CAMP-ISIJ*, **18** (2005), 1559.
- 8) S. Takagi, Y. Toji, K. Hasegawa, Y. Tanaka, N. Rössler, B. Hammer and T. Heller: *Int. J. Automot. Eng.*, **2** (2010), 7.
- 9) S. Takagi, Y. Toji, K. Hasegawa, Y. Tanaka, N. Rössler, B. Hammer and T. Heller: *Mater. Sci. Technol. Conf. Exhib.*, (2010), No. 3, 1769.
- 10) M. Matsuno, H. Suzuki, K. Takai and Y. Hagihara: *CAMP-ISIJ*, **22** (2009), 1383, CD-ROM.
- 11) M. Matsuno, H. Suzuki, K. Takai and Y. Hagihara: *CAMP-ISIJ*, **23** (2009), 1285, CD-ROM.
- 12) K. Mori, Y. Abe and Y. Suzui: *J. Mater. Process. Technol.*, **210** (2010), 653.
- 13) Standard Method for X-Ray Stress Measurement (2002) -steel edition- reprinted ed. by JSMS Committee on X-ray Study of Mechanical Behavior of Materials, The Society of Materials Science, Japan, Kyoto, (2006).
- 14) Y. Toji, S. Takagi, M. Yoshino, K. Hasegawa and Y. Tanaka: *CAMP-ISIJ*, **21** (2008), 1453, CD-ROM.
- 15) R. A. Oriani: *Acta Metall.*, **18** (1970), 147.
- 16) T. Yamaguchi and M. Nagumo: *ISIJ Int.*, **43** (2003), 514.
- 17) M. Nagumo, K. Ohta and H. Saitoh: *Scr. Mater.*, **40** (1999), 313.
- 18) Y. Toji, S. Takagi, K. Hasegawa and K. Seto: *ISIJ Int.*, **52** (2012), 274.
- 19) A. Dalloz, A-F. Gourgues, A. Pineau and J. Besson: *Mater. Sci. Technol. Conf. Exhib.*, **1** (2007), 167.
- 20) Fédération Internationale de la Précontrainte: Report on Prestressing Steel: 5. Stress Corrosion Cracking Resistance Test for Prestressing Tendons, Vol. 5, Cement and Concrete Association, England, Slough, (1980).
- 21) S. Takagi, Y. Toji, M. Yoshino and K. Hasegawa: *ISIJ Int.*, **52** (2012), 316.
- 22) S. Takagi and Y. Toji: *ISIJ Int.*, **52** (2012), 329.
- 23) T. Komamura and T. Shoji: *Trans. Jpn. Soc. Mech. Eng.*, **61** (1995), 283.
- 24) H. A. Wriedt and R. A. Oriani: *Acta Metall.*, **18** (1970), 753.
- 25) H. P. Van Leeuwen: *Corrosion*, **29** (1973), 197.
- 26) H. P. Van Leeuwen: *Eng. Fract. Mech.*, **6** (1974), 141.
- 27) A. Nozue: Advances in Delayed Fracture Solution ed. by Division of Microstructure and Properties of Materials, ISIJ, Tokyo, (1997), 197.
- 28) W. W. Gerberich and Y. T. Chen: *Metall. Trans. A*, **6A** (1975), 271.