An Evaluation Method for Hydrogen Embrittlement of High Strength Steel Sheets Using U-bend Specimens

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(Received on May 1, 2020; accepted on June 5, 2020; originally published in Tetsu-to-Hagané, Vol. 105, 2019, No. 9, pp. 927–934; J-STAGE Advance published date: July 18, 2020)

An evaluation method for hydrogen embrittlement property of high strength steel sheet has been proposed in this study. To take into consideration of the effect of plastic strain in addition to the effects of applied/residual stress and diffusible hydrogen, U-bend specimens have been adopted because steel sheets for automobiles are usually used after press forming into various parts. After U-shape bending, the specimen was loaded using a bolt. The proposed evaluation method is based on the measurement of critical hydrogen content or critical hydrogen charging condition for hydrogen embrittlement fracture at given stress and strain conditions. The hydrogen charging current density was increased in step-wise manner until cracking was observed, and cracking was detected by optical observation and by monitoring voltage between the sample and a counter electrode. The critical hydrogen contents for specimens with varied applied stress were obtained by means of thermal desorption spectroscopy. For the critical hydrogen content, both the hydrogen contents in strained portion of the specimen and no-strained portion were measured. The former is affected by introduced dislocations caused by straining and the latter is thought to be proportional to the hydrogen fugacity. Both critical hydrogen contents tended to be decreased slightly when the applied stress was relatively high.

KEY WORDS: hydrogen embrittlement; high-strength steel; U-bend; cathodic charging; sheet steel.

1. Introduction

High strength steel sheets have been applied to automobiles to achieve weight reduction and collision safety. In particular, the high strength steel sheets with the tensile strength of 980 and 1 180 MPa have been applied for the automotive structural parts. However, there is a risk of hydrogen embrittlement which significantly reduces toughness with increasing the steel strength. Hydrogen embrittlement is defined as a brittle fracture phenomenon due to diffusible hydrogen in steels. In the 1960s, there were some cases of failure of high strength bolts with 1 300 MPa tensile strength grade used for bridges and constructions which led to strenuous investigations of evaluation of hydrogen embrittlement susceptibility. There have been many reports of the evaluation for hydrogen embrittlement properties using circumferentially notched round bar specimens simulating stress concentration at the notch root of threads of bolts.

There are some proposed methods to evaluate hydrogen embrittlement susceptibility of the high strength bolt steels, which are constant load test (CLT), slow strain rate technique (SSRT), test and conventional strain rate technique (CSRT) test with hydrogen pre-charging or simultaneously hydrogen charging.

On the other hand, demands for evaluation of hydrogen embrittlement of the high strength steel sheets have been increasing in recent years because of the strengthening of the structural parts, although no incident of hydrogen embrittlement of steel sheet for automobiles has been reported. As for an evaluation method of hydrogen embrittlement properties of high strength steel sheets, 4-point bending tests have been proposed as well as SSRT and CSRT of steel sheets. In these methods the relationship between critical hydrogen content causing a fracture and applied stress is evaluated. Since most of the automobile parts made of steel sheets are processed by punching, shearing and press forming, it is necessary to take account of the effect of plastic strain as a controlling factor of hydrogen embrittlement in addition to the factors of material property (strength and microstructure), applied stress and diffusible hydrogen content which are generally considered as the controlling factors for hydrogen assisted fracture of high strength bolts.

CLT using U-bend specimens is one of the proposed evaluation methods for hydrogen embrittlement considering...
the effect of plastic strain. In the method, the plastic strain is introduced by U-shape bending and stress was applied by tightening the U-bend specimen with a bolt and nut and then, hydrogen is introduced. This is a true-false type test method to evaluate the hydrogen embrittlement property by the presence or absence of fracture. Using this method, the effects of plastic strain, applied stress and absorbed hydrogen content on hydrogen embrittlement of the steel sheets have been investigated, and the hydrogen cracking conditions of the steel sheet have been mapped in the three-dimensional space with the axes of applied strain, applied stress, and hydrogen content. If one specimen is used for one testing condition, a large number of specimens and a large amount of testing time are consumed to evaluate the hydrogen embrittlement property. Accordingly, novel testing methods are required to evaluate hydrogen embrittlement property efficiently.

This study proposes a new hydrogen embrittlement tests using a U-bend specimen of the high strength steel sheets. The hydrogen charging condition for the U-bend specimen with given applied plastic strain and stress is changed to obtain higher hydrogen content in a step-wise manner and the critical hydrogen charging condition, in other words, the critical hydrogen content for fracture is evaluated. This method is expected to not only reduce the number of required specimens but also efficiently evaluate the accurate critical hydrogen content for hydrogen assisted cracking. Cathodic hydrogen charging is adopted because it can easily control hydrogen content. First, hydrogen content obtained under various hydrogen charging conditions is examined to obtain conditions to effectively control the hydrogen content.

Currently, automotive steel sheets are being developed to achieve both high strength and high ductility. An example is TRIP (Transformation Induced Plasticity) -aided bainitic ferrite steel containing retained austenite in bainitic ferrite matrix, which is expected to be used as the next-generation high strength steel sheets for automobile structural parts because it possesses high strength and high ductility due to strain-induced martensitic transformation of the retained austenite. Martensitic transformation in the presence of hydrogen enhances hydrogen embrittlement when the low alloy TRIP-aided steels are pre-charged with hydrogen and then, plastic strain deformation, however, the transformation does not occur actually in a service environment. Therefore, to make the evaluation method proper and applicable to steels including TRIP-aided steels, in the evaluation method we propose in this paper, a specimen is plastically deformed and loaded, and then it is charged with hydrogen to simulate the hydrogen uptake in the service environment after the press forming process. In this study, hydrogen embrittlement properties of tempered martensitic steels, which does not show strain-induced phase transformation, were evaluated using the proposed method to verify the adequacy of the method and to establish an appropriate evaluation method.

2. Experiment

2.1. Concept of the Evaluation Method for Hydrogen Embrittlement Properties Using U-bend Specimens Charged with Hydrogen

Figure 1 shows a schematic diagram of the testing method to evaluate a critical hydrogen content for hydrogen embrittlement using a step-wise increase in hydrogen charging efficiency. Electrochemical hydrogen charging was used in this method because the electrochemical hydrogen charging allows us to control hydrogen content in steel easily by controlling current density and hydrogen charging solution. Cathodic hydrogen charging is better than hydrogen charging by immersion in an acidic media because it can avoid corrosion which disturbs the visual detection of cracking during the test.

In this method, the presence of crack initiation was examined under a given hydrogen charging condition, and if no crack was found for certain testing time, the current density was increased. The process was repeated until cracking was observed and the critical hydrogen charging condition causing cracking due to hydrogen embrittlement was obtained. A similar step-wise test was subsequently carried out and the increase in current density was terminated just below the critical hydrogen charging current density. The hydrogen contents in the cracked specimen and that in the specimen charged with hydrogen at the highest current density which did not cause cracking was measured using thermal desorption spectrometry to determine the critical hydrogen content. The bending radius of the U-bend specimen and the stress applied by tightening the feet of the specimen using a bolt and a nut were varied and the critical hydrogen charging condition was obtained for each bending radius and applied stress condition. The initiation of cracks was monitored through in-situ interval photographing.

2.2. Materials

Two kinds of tempered martensitic steels with a tensile strength of 1 500 MPa grade produced from JIS-SCM435 steel of 1.6 mm thickness were used in this study. One was produced from ingot (SCM435M) prepared with the process as will be described below and the other was a commercially available product (SCM435C). Chemical compositions of steels used are listed in Table 1. For the SCM435M, vacuum melted ingot was hot forged and hot-rolled to 5 mm thickness, and quenched from annealing at 900°C for 900 s, snap-tempered at 150°C for 1 800 s and tempered at 400°C for 1 800 s followed by grinding to reduce the thickness to 1.6 mm. For the SCM435C, the commercial SCM435 steel sheet of 6 mm thickness was normalized at 800°C for 1 800 s, quenched from annealing at 900°C for 900 s and tempered at 400°C for 1 800 s. Then, the thickness of the steel was reduced to 1.6 mm by grinding.

Figure 2 shows SEM micrographs of the SCM435M and the SCM435C. The samples were etched in 5% Nital etchant.
The microstructure of both the SCM435M and SCM435C steels consisted of martensite and the diameter of prior austenite grain of the SCM435C was smaller than that of the SCM435M. Tensile strength ($\sigma_t$), yield strength ($\sigma_y$) and total elongation (TEl) measured for the JIS13B half-size tensile specimen of the SCM435M were $1514 \text{ MPa}$, $1297 \text{ MPa}$, and $10.4\%$, respectively. On the other hand, the $\sigma_t$ of the SCM435C measured by using the JIS5 tensile specimen was $1549 \text{ MPa}$, the $\sigma_y$ was $1416 \text{ MPa}$, and the TEl was $7.0\%$.

2.3. Preparation of U-bend Specimens

Figure 3 schematically shows an example of the preparation procedure of U-bend specimens. A rectangular specimen of $100 \text{ mm} \times 30 \text{ mm} \times 1.6 \text{ mm}$ whose rolling direction was parallel to the longitudinal direction was bent in U shape. The surface of the specimen was as-ground. The edge of the specimen was polished by the waterproof SiC paper of a grid size of #600 to remove the residual stress caused by machining. U-bending was carried out at a crosshead speed of 2 mm/min using a jig equipped with a punching bar of a diameter of 10 mm. After U-bending, stress was applied by tightening the bolt as shown in Fig. 3. The strain of the top of the U-bend specimen was measured by using a strain gauge, and the stress was calculated by using the following Eq. (1).

$$\sigma = E\varepsilon$$ ........................................... (1)

where $\sigma$, $E$, and $\varepsilon$ are stress, Young’s modulus, and strain, respectively. The stress on the outer side of the top of the U-bend specimen was defined as 0 MPa when U-bending was conducted. However, the actual stress is the sum of residual stress due to the spring back after U-bending and the stress applied by tightening the bolt.

2.4. Evaluation Method of Hydrogen Embrittlement Properties of U-bend Specimens

U-bend specimens were charged with hydrogen by using galvanostatic hydrogen charging in 3 wt% NaCl aqueous solutions with the addition of 0–3 g L$^{-1}$ of NH$_4$SCN as schematically shown in Fig. 4. A Pt wire as a counter electrode was set in the cell. The hydrogen charging solution in the acrylic plastic cell was kept at 30°C by circulating temperature-controlled water in the jacket for better reproducibility. A digital camera was set for in-situ observation of hydrogen embrittlement cracking, and interval photographing was carried out. Moreover, the voltage between the sample and the counter electrode was recorded by a data logger to detect the crack initiation.

As described above (Fig. 1), if cracking did not occur within a certain time, hydrogen content in the steel was increased by increasing the current density. This process was repeated step-by-step until cracking was detected to determine the critical condition for cracking. Another U-bend specimen was used to obtain the highest hydrogen content just before fracture. Hydrogen contents in a sample cut out from the specimen suffered from cracking and a sample cut out from a specimen charged with hydrogen at the highest current density just before fracture. TDS analysis was carried out for the hydrogen measurements. A critical hydrogen content was defined as the maximum diffusible content just before cracking.

Table 1. Chemical composition of steels (mass%).

|       | C   | Si  | Mn  | P   | S   | Cr  | Mo  | Ni  | Al  | O   |
|-------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| SCM435M | 0.35 | 0.21 | 0.74 | 0.006 | tr  | 1.00 | 0.22 | tr  | 0.031 | 0.0024 |
| SCM435C | 0.35 | 0.29 | 0.69 | 0.008 | 0.004 | 0.97 | 0.18 | 0.01 | –   | –   |

Fig. 2. SEM micrograph of (a) SCM435M and (b) SCM435C.

Fig. 3. Preparation procedure of a U-bend specimen ($R$: bending radius, 10 mm).

Fig. 4. Schematic drawing of a setup for hydrogen embrittlement test.
hydrogen content in a specimen which showed no hydrogen embrittlement cracks.

2.5. Hydrogen Thermal Desorption Analysis

Hydrogen content in the specimen was measured using thermal desorption spectrometry (TDS). Typical hydrogen desorption curves of the steel are shown in Fig. 5. The specimens were charged with hydrogen in a 3 wt% NaCl aqueous solution and a 3 wt% NaCl aqueous solution containing 0.03 g L\(^{-1}\) NH\(_4\)SCN at a current density of 2 A m\(^{-2}\) for 48 h. The heating rate was 200\(^\circ\)C/h for this measurement but some other measurements were carried out at 100\(^\circ\)C/h. A standard leak gas system was used for calibration of the hydrogen desorption rate. The measured hydrogen partial pressure was converted to the hydrogen desorption rate. The hydrogen desorption rate up to a certain temperature was integrated, and that was divided by the weight of the specimen to obtain the hydrogen content. The content of diffusible hydrogen desorbed from room temperature to 300\(^\circ\)C was regarded as the diffusible hydrogen content. Since the diffusible hydrogen which diffuses at ambient temperature plays a key role in hydrogen embrittlement, diffusible hydrogen content was evaluated as a determining parameter in this study. The specimens after hydrogen embrittlement testing were kept in liquid nitrogen to avoid hydrogen desorption before TDS.

3. Results and Discussion

3.1. Hydrogen Absorption Property

Figure 6 shows hydrogen desorption curves measured at the heating rate of 100\(^\circ\)C/h of the SCM435M steel charged with hydrogen in an aqueous solution of 3 wt% NaCl with 3 g L\(^{-1}\) NH\(_4\)SCN at a current density of 3 A m\(^{-2}\) for 48 h. The heating rate was 200\(^\circ\)C/h for this measurement but some other measurements were carried out at 100\(^\circ\)C/h. A standard leak gas system was used for calibration of the hydrogen desorption rate. The measured hydrogen partial pressure was converted to the hydrogen desorption rate. The hydrogen desorption rate up to a certain temperature was integrated, and that was divided by the weight of the specimen to obtain the hydrogen content. The content of diffusible hydrogen desorbed from room temperature to 300\(^\circ\)C was regarded as the diffusible hydrogen content. Since the diffusible hydrogen which diffuses at ambient temperature plays a key role in hydrogen embrittlement, diffusible hydrogen content was evaluated as a determining parameter in this study. The specimens after hydrogen embrittlement testing were kept in liquid nitrogen to avoid hydrogen desorption before TDS.

In addition, the effects of a current density and an NH\(_4\)SCN concentration on the content of hydrogen introduced in specimens were investigated in advance to the hydrogen embrittlement test. Specimen with dimensions of 10 mm \(\times\) 10 mm \(\times\) 1.6 mm was cut from the SCM435C steel sheet and was charged with hydrogen for 48 h with varied current densities and concentrations of NH\(_4\)SCN which is known to accelerate hydrogen entry into metals probably by acting as poison for hydrogen evolution reaction. Hydrogen content introduced in the specimens with the varied conditions was measured using TDS. Figure 8 shows the effects of the current density and NH\(_4\)SCN concentration on the resulted diffusible hydrogen content at a range of current density the measurements shown in Fig. 5. This difference in peak position is because hydrogen desorption from the hydrogen trapping site in the steel is limited to the thermal dissociation. Figure 7 shows the relationship between hydrogen content and hydrogen charging time. Diffusible hydrogen content tended to increase until 6 h. The diffusible hydrogen content became almost constant at about 2 wppm (weight parts per million) indicating that the in-depth hydrogen distribution became homogeneous. According to this result, the time of each step of the hydrogen embrittlement test was determined to be more than 6 h in which U-bend specimens with the same thickness of 1.6 mm was used.

3.2. Hydrogen Desorption Property

Figure 8 shows hydrogen desorption curves measured at the heating rate of 100\(^\circ\)C/h of the SCM435M steel charged with hydrogen in an aqueous solution of 3 wt% NaCl with 3 g L\(^{-1}\) NH\(_4\)SCN at a current density of 3 A m\(^{-2}\) for 48 h. The heating rate was 200\(^\circ\)C/h for this measurement but some other measurements were carried out at 100\(^\circ\)C/h. A standard leak gas system was used for calibration of the hydrogen desorption rate. The measured hydrogen partial pressure was converted to the hydrogen desorption rate. The hydrogen desorption rate up to a certain temperature was integrated, and that was divided by the weight of the specimen to obtain the hydrogen content. The content of diffusible hydrogen desorbed from room temperature to 300\(^\circ\)C was regarded as the diffusible hydrogen content. Since the diffusible hydrogen which diffuses at ambient temperature plays a key role in hydrogen embrittlement, diffusible hydrogen content was evaluated as a determining parameter in this study. The specimens after hydrogen embrittlement testing were kept in liquid nitrogen to avoid hydrogen desorption before TDS.

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from 0.3 to 300 A m\(^{-2}\) and varied NH\(_4\)SCN concentrations in a 3 wt% NaCl aqueous solution. The hydrogen content increased with increasing the current density between 0.3 and 300 A m\(^{-2}\) in the 3 wt% NaCl aqueous solution without NH\(_4\)SCN. In contrast, when hydrogen was introduced in the solutions with additive NH\(_4\)SCN, hydrogen content increased significantly. However, the hydrogen content did not increase at the current density of more than 1 A m\(^{-2}\) in the NH\(_4\)SCN-containing solutions although hydrogen content increased in the low current density region. Thus, if NH\(_4\)SCN addition is necessary to achieve critical hydrogen content for cracking in the hydrogen embrittlement test, the low current density region was desirable to control hydrogen content by changing current density.

Figure 9 shows the relationship between hydrogen content at a low current density between 0.1 and 1.5 A m\(^{-2}\) in a 3 wt% NaCl aqueous solution containing 0.3 g L\(^{-1}\) NH\(_4\)SCN. It was confirmed that the hydrogen content linearly increased with logarithmic increasing of current density in the low current density region. However, the specimen surface was corroded when hydrogen charging was carried out at a current density of 0.1 A m\(^{-2}\). This was presumably because the potential of the specimen was in the active region, although the specimen was cathodically polarized. Taking account of the consumption of cathodic current density for reduction reaction of dissolved oxygen given in Eq. (2), which is roughly 0.3 A m\(^{-2}\), the minimum current density for hydrogen charging was set at 0.3 A m\(^{-2}\) in the hydrogen embrittlement evaluation method in the present study.

\[
\text{O}_2 + 2\text{H}_2\text{O} + 4\text{e}^- \rightarrow 4\text{OH}^- \quad \text{................................ (2)}
\]

To investigate the effects of plastic deformation and applied stress on hydrogen content, the hydrogen contents in a couple of portions in the U-bend specimen of the SCM435M with a bending radius of 10 mm were compared. The applied stress on the outer side of the top was 0 or 1 000 MPa. Hydrogen charging was carried out at a current density of 10 A m\(^{-2}\) at 30°C. Figure 10, one portion was taken from the top and the other was from a foot corresponding to the deformed region (Strain) and undeformed region (No strain), respectively, and the hydrogen content in each sample was measured using TDS. Figure 11 shows hydrogen desorption curves of deformed and undeformed parts with and without applied stress during hydrogen charging. The hydrogen content of the deformed part was remarkably higher than that of the undeformed part. This result suggests that dislocations and vacancy clusters introduced by plastic deformation acted as
hydrogen trapping sites.\textsuperscript{22})

The diffusible hydrogen content was almost constant regardless of applied stress. It is known that absorbed hydrogen content is changed by applied hydrostatic stress and hydrogen is accumulated in the tensile stress field.\textsuperscript{12,23})

The reason that hydrogen content at the deformed part was not affected by the applied stress was that the stress was symmetrically distributed in the thickness direction of the U-bend specimen. That is, in the top of the U-bend part, the stress at the center in the thickness direction was 0 and tensile and compressive stresses are in the outer and inner sides, respectively. Therefore, it is considered that hydrogen concentration was increased in the outer side whereas that was decreased in the inner side so that the change in hydrogen concentration was offset in the thickness direction because the hydrogen content was the average value throughout the thickness direction. It was, therefore, considered that the hydrogen content in the bending position does not change even if fracture takes place and stress is released.

3.2. Crack Detection by Observation of the Voltage of Galvanostatic Changing

Figure 12 shows an example of a change in the voltage between the specimen and the Pt counter electrode measured during the hydrogen embrittlement test of a U-bend specimen of the SCM435M steel with a bending radius of 10 mm and the applied stress of 1 000 MPa. The solution of hydrogen charging was a 3 wt% NaCl aqueous solution with 0.03 g L\textsuperscript{−1} NH\textsubscript{4}SCN. 8 h after starting the test, the current density was instantly increased, which led to a sharp decrease in voltage. At the time of about 12 h, an obvious increase and following gradual decay of voltage were observed. Comparing with photos taken every 1 min during the test, it was confirmed that the sudden increase in voltage corresponded with the crack initiation. The change of the voltage was probably caused by the appearance of a fresh metal surface due to the crack initiation resulting in an increase in the potential of the specimen at the galvanostatic condition. This result suggests that monitoring voltage is useful to detect crack initiation.

3.3. In-situ Observation of Crack Initiations and Propagations

Figure 13 shows typical crack initiation and propagation behaviors of hydrogen embrittlement on a U-bend specimen. Hydrogen was introduced to the U-bend specimen of the SCM435M at the current densities of (a) 30 A m\textsuperscript{−2} and (b) 300 A m\textsuperscript{−2} in a 3 wt% NaCl aqueous solution. In both hydrogen charging conditions, the crack was initiated at the center in the width direction and was propagated outward in the width direction with time. The change in the crack length with time obtained from the images is shown in Figure 14. When the crack was branched, the maximum crack length was regarded as the crack length and the other branches were ignored. The crack growth rate at high current density was obviously higher than that at low current density. The preferential occurrence of initial crack at the center in the width direction might be because the stress was applied by bolt tightening and the stress around the center in the width direction was the highest.

3.4. Effects of Applied Stress on the Critical Hydrogen Content

U-bend specimens with a bending radius of 10 mm and with applied stress from 500 to 1 500 MPa were charged with hydrogen and the critical hydrogen contents for hydrogen embrittlement for the conditions were obtained. Figure 15 shows the relationship between the critical hydrogen content and the applied stress. Hydrogen contents of deformed and undeformed parts cut out from the U-bend specimen.
were measured. In the figure, “Strain Crack” and “No Strain crack” represent diffusible hydrogen contents in the deformed and undeformed portions (Fig. 10), respectively, in a specimen which showed hydrogen assisted cracking. Furthermore, “Strain” and “No Strain” indicate the maximum diffusible hydrogen contents of the deformed and undeformed portions, respectively, in a specimen that did not show cracking. A gap of hydrogen contents between “Crack” and “No Crack” was small except for that of a specimen with the applied stress of 500 MPa. The small gap indicated that the quantitativeness of the analysis of the critical hydrogen content obtained by the proposed evaluation method was high. Thus, it was confirmed that the critical condition of crack initiation can be efficiently obtained. The critical hydrogen content of both deformed (“Strain”) and undeformed (No Strain) portions of a specimen with the applied stress of 1 500 MPa was slightly lower than that with the applied stress of 1 000 MPa whereas the difference between the critical hydrogen contents for 500 and 1 000 MPa was not clear.

The hydrogen content was affected by the plastic strain and the hydrogen content of the deformed portion (“Strain”) was obviously higher than that of the undeformed portion (“No Strain”). The increased hydrogen content in the deformed portion was presumably attributed to multiplicated dislocations and vacancy clusters caused by the U-bending, which acted as hydrogen trapping sites. When hydrogen embrittlement properties of the high strength steels are evaluated based on the hydrogen content in the deformed portion, the diffusible hydrogen content is affected by the increased hydrogen trap site density due to plastic strain. Therefore, the hydrogen content affected by plastic deformation is not an appropriate parameter for hydrogen embrittlement property, but the parameter reflecting the hydrogen fugacity of the environment should be considered. In this sense, the critical hydrogen content of the undeformed portion is more plausible. In this study, therefore, the critical hydrogen content of the undeformed part was examined, and that was in the range of 1–2 wppm. Since it is known that hydrogen uptake under atmospheric corrosion condition of the similar steels is no more than 0.15 wppm, it is predicted that hydrogen embrittlement will not occur on practical parts in the actual service condition as far as the degrees of plastic strain and the applied stress are similar or lower to that of the U-bend specimens used in this study. In this study, the degree of hydrogen uptake in the electrochemical hydrogen charging conditions used for the hydrogen embrittlement tests is high compared to the actual hydrogen uptake of steels in the service environment.

The fracture surfaces of the tested U-bend specimens with a bending radius of 10 mm and applied stress of 1 500 MPa are shown in Fig. 16. The hydrogen charged specimens exhibited intergranular fracture surface near the center in the thickness direction whereas shear lip region was also observed near the outer side surface of the U-bend portion. Since it is known that the crack initiation in tempered martensite steel takes place typically at the prior austenite grain boundary, the fractography suggests that the crack initiation site in the U-bend specimen was located around the center in the thickness direction. It is considered that plastic

Fig. 15. Relationship between applied stress and hydrogen content in strained and non-strained portions of SCM435C U-bend specimens.

Fig. 16. Scanning electron micrographs of fracture surface of a U-bend specimen (R = 10 mm, 1 500 MPa) of SCM435C.
strain and the corresponding increase in hydrogen concentration in this central region are low compared to that in the vicinity of the surface.\textsuperscript{25,20} Accordingly, it is suggested that the tensile stress is expected to be highest near the center in the thickness direction and that the maximum tensile stress is the predominant factor to determine the position of the crack initiation and the following fracture. The dependence of the critical diffusible hydrogen content for cracking based on the non-deformed portion on the stress applied to the outer side surface of the top of the U-bend specimen was not apparent in Fig. 15. This result suggests that the stress applied on the outer side surface did not cause any obvious change in the maximum tensile stress near the center, though the applied stress was varied from 500 to 1 500 MPa.

For further understanding, clarification of the stress distribution in the thickness direction and its change according to the change in the applied stress is required. Hence, investigation of stress and plastic strain distributions inside of the U-bend specimen are now ongoing by using the finite element method (FEM). Another future subject is to clarify the effect of plastic strain on hydrogen embrittlement by changing the bending radius because hydrogen embrittlement properties were evaluated for only one plastic strain condition in the present study.

4. Conclusion

We have proposed an evaluation method to determine the critical hydrogen concentration for the crack initiation employing U-bend specimens charged with hydrogen using galvanostatic polarization, in which the cathodic current density is increased in a step-wise manner. The validity of the method was verified by applying the method to a quenched and tempered martensitic steel sheet of 1 500 MPa grade. We have successfully confirmed that the critical hydrogen content can be obtained efficiently using the method for both in the deformed and undeformed portions of the U-bend specimens. The critical hydrogen content for cracking in the deformed portion includes the content of hydrogen trapped to defects introduced by plastic deformation which act as hydrogen trapping sites. Whereas, the critical hydrogen content in the undeformed portion reflects the fugacity of hydrogen in the environment.

By employing in-situ interval photographing, the crack initiation and propagation behaviors could be recorded. The observation indicated that the crack growth rate under the hydrogen charging at higher current density was higher than that at lower current density, which denoted that higher hydrogen fugacity promoted faster growth of cracking. Since a sudden change in the voltage between the specimen and the counter electrode was correspondent to the crack initiation recorded by the interval photographing, it can be said that the monitoring of the voltage is useful to detect the crack initiation.

The critical hydrogen content for cracking was measured in various conditions of the applied stress on the outer surface of the top of the U-bending specimen. When the applied stress was 1 500 MPa which was equivalent to the tensile strength of the tested steel, the critical hydrogen content for cracking slightly decreased. However, the change of the critical hydrogen content was not clear in the low applied stress range. According to the observation of the fracture surface, the crack initiation site was around the center in the thickness direction, suggesting that the tensile stress was the highest around the center. To establish the hydrogen embrittlement evaluation method, it is necessary to grasp the maximum tensile stress in the specimen. To improve the hydrogen embrittlement evaluation method for high strength steels considering the relationship between the maximum tensile stress and hydrogen fugacity, estimation of the stress distribution in the thickness direction employing FEM, for example, is in the future subject.

Acknowledgment

This article is based on results obtained from a project commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

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