Comparison of Constant Load, SSRT and CSRT Methods for Hydrogen Embrittlement Evaluation Using Round Bar Specimens of High Strength Steels

Tetsushi CHIDA,1)* Yukito HAGIHARA,2) Eiji AKIYAMA,3) Kengo IWANAGA,4) Shusaku TAKAGI,5) Masao HAYAKAWA,6) Hiroyuki OHISHI,7) Daisuke HIRAKAMI8) and Toshimi TARUI9)

1) Steel Research Laboratories, Nippon Steel & Sumitomo Metal Corporation, 1-8, Fuso-cho, Amagasaki, Japan.
2) Formerly Sophia University. 3) Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai, 980-8577 Japan. 4) Neturen Co., Ltd., 1-586-1, Higashi-une, Ako, Hyogo, 678-1185 Japan.
5) Steel Research Laboratory, JFE Steel Corporation, 1-1, Minamiwatarida-cho, Kawasaki-ku, Kawasaki, 210-0855 Japan. 6) National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki, 305-0047 Japan.
9) Nippon Steel & Sumikin Technology Co., Ltd., 12 Nakamachi, Muroran, 050-0087 Japan.

(Received on October 8, 2015; accepted on April 7, 2016; originally published in Tetsu-to-Hagané, Vol. 100, 2014, No. 10, pp. 1298–1305)

Resistance to hydrogen embrittlement of low alloy steels was evaluated based on their critical hydrogen content and critical stress. Constant load test (CLT), Slow Strain Rate Test (SSRT) and Conventional Strain Rate Test (CSRT) were carried out using JIS-SCM435 and V-added steels in six laboratories. It was confirmed that the same test results were obtained in different laboratories under the same test conditions. Furthermore, the relationships between diffusible hydrogen content and nominal fracture stress obtained by means of CLT and SSRT were similar to each other. In CSRT, the nominal fracture stress was higher than that in CLT and SSRT under the same absorbed hydrogen content in the specimens. In SSRT and CSRT, fracture surfaces showed Quasi-cleavage mode under small hydrogen content, while they showed Inter-granular fracture under large hydrogen content. In order to compare the three methods considering the concentration of hydrogen in stress field, locally accumulated hydrogen content under the same fracture stress is calculated. The locally accumulated hydrogen under the same applied stress, in other words, critical hydrogen content to hydrogen embrittlement, is the following order; SSRT < CLT < CSRT in JIS-SCM435, and CSRT < CLT ≈ SSRT in V-added steels.

KEY WORDS: hydrogen; hydrogen embrittlement; delayed fracture; constant load test; SSRT; CSRT; high strength steel.

1. Introduction

Demand for high-strength steel bolts with tensile strengths of more than 1 000 MPa has been increasing in order to reduce the weights and sizes of construction structures, bridges and automobile parts. However, it is well known that high strength steels suffer from delayed fracture, which is one type of mechanical degradation, caused by a small amount of hydrogen absorbing from atmospheric environment.1) For the safe use of the high-strength steels, it is necessary to forecast whether delayed fracture occurs or not in actual service environments.

In several laboratories test methods for evaluating delayed fracture susceptibility of high strength steels are proposed from the viewpoint of the degree of degradation of mechanical property by cathodic hydrogen charging. Yamasaki et al. proposed a test method2,3) based on critical diffusible hydrogen content Hc. Hc is defined as the maximum diffusible hydrogen content up to which a steel specimen does not fail under a constant load. This concept was originally suggested by Troiano et al.4) In this test method, it is assumed that no delayed fracture will occur when Hc is higher than absorbed diffusible hydrogen content in the actual environment. Yamasaki et al. showed a good relationship between the laboratory evaluation result based on Hc and probability of failure obtained by means of exposure tests using a large number of high-strength steel bolts in atmospheric environments. Kushida et al. proposed a similar test method. They evaluated fracture stress of notched tensile specimens under cathodic hydrogen charging conditions which simulate acidified environment in the area such as pit bottoms or crevices.5) Yamasaki and Kushida used constant load tests (CLT) for the evaluation of susceptibility to delayed fracture.

Recently, susceptibility to delayed fracture is evaluated by Slow Strain Rate Test (SSRT)6–12) and Conventional

* Corresponding author: E-mail: chida.m27.tetsushi@jp.nssmc.com
DOI: http://dx.doi.org/10.2355/isijinternational.ISIJINT-2015-565

© 2016 ISIJ
Strain Rate Test (CSRT)\cite{13-16} which can complete the delayed fracture test in a shorter time than CLT. The relationship between $H_C$ and fracture stress can be obtained by SSRT and CSRT. However, comparison among CLT, SSRT and CSRT has not been investigated systematically.

In this study, relationship between diffusible hydrogen content and fracture stress obtained by means of CLT, SSRT and CSRT were compared. Some of the experiments with the same procedure were conducted in some laboratories simultaneously using the same specimens in order to confirm the reproducibility and universality of the test methods.

2. Experimental Method

2.1. Materials and Specimen Shapes

Low alloy steels, JIS (Japanese Industrial Standards) SCM435 and V-added steel, were used in this study. SCM435 is generally used as the material for high-strength steel bolts. The V-added steel is one of recent developed steels with excellent resistance to delayed fracture. The steel contains nano-sized carbides which act as hydrogen trapping sites.\cite{2,5} The chemical compositions of the steels are shown in Table 1.

For SCM435, the wire rod was drawn into wires with a diameter of 10 mm. The wires were quenched and tempered by a high frequency induction heating (Steel S11) as shown in Fig. 1. Table 2 shows the mechanical properties evaluated by tensile tests using round bar specimens with parallel part diameter of 10 mm and gauge length of 100 mm.

For V-added steel, the ingot made in a laboratory vacuum melting furnace was forged into bars with a diameter of 16 mm. The bars were normalized for 60 minutes at 920°C followed by air-cooling, and heat treated in the condition shown in Fig. 2 (Steel V14). The bars were tempered at 150°C for 30 minutes immediately after quenching to reduce the risk of quenching crack between quenching and tempering. Table 2 shows the mechanical properties evaluated by tensile tests using round bar specimens with parallel part diameter of 8 mm and gauge length of 56 mm.

The specimen shape for hydrogen embrittlement testing is shown in Fig. 3. To simulate stress concentration in threaded parts of actual bolts, the round bar specimen was with a circumferentially notch with a depth of 1 mm and its notch root radius was 0.12 mm. The stress concentration factor of the specimen was approximately 5.

2.2. Evaluation Method of Hydrogen Embrittlement Susceptibility

Three methods - CLT, SSRT, and CSRT were used to evaluate the hydrogen embrittlement susceptibility. Prior to the hydrogen embrittlement tests, hydrogen absorption test was conducted to investigate appropriate hydrogen charging conditions.

2.2.1. Hydrogen Absorption Test

Hydrogen absorption test was conducted by the following two methods using un-loaded specimens.

Cathodic charging tests were conducted at Sophia University (SPU). The electrolyte was a 0.1 mol/L–NaOH aqueous solution. The current density was varied between 10–80 A/m².

<table>
<thead>
<tr>
<th>Steels</th>
<th>YS/MPa</th>
<th>TS/MPa</th>
<th>El. (%)</th>
<th>R. A. (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SCM435 (S11)</td>
<td>1 003</td>
<td>1 103</td>
<td>12</td>
<td>33</td>
</tr>
<tr>
<td>V steel (V14)</td>
<td>1 339</td>
<td>1 443</td>
<td>15</td>
<td>60</td>
</tr>
</tbody>
</table>

Table 1. Chemical composition of steels used.

<table>
<thead>
<tr>
<th>Steels</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Al</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>SCM435</td>
<td>0.35</td>
<td>0.24</td>
<td>0.79</td>
<td>0.023</td>
<td>0.016</td>
<td>0.036</td>
<td>1.09</td>
<td>0.15</td>
<td>–</td>
<td>0.005</td>
</tr>
<tr>
<td>V steel</td>
<td>0.41</td>
<td>0.20</td>
<td>0.70</td>
<td>0.005</td>
<td>0.005</td>
<td>0.035</td>
<td>1.19</td>
<td>0.65</td>
<td>0.30</td>
<td>0.004</td>
</tr>
</tbody>
</table>

Table 2. Mechanical properties of steels used.

Fig. 1. Schematic diagram of heat treatment condition of SCM435.

Fig. 2. Schematic diagram of heat treatment condition of V steel.

Fig. 3. Specimen dimension.
The hydrogen charging time was 72 h for Steel S11 and 120 h for Steel V14. Thereafter, hydrogen measurement was carried out by means of Thermal Desorption Analysis (TDA) using gas chromatography.

Immersion tests were conducted at Neturen Co. Ltd. (NC) using the aqueous solution of NH₄SCN specified in the FIP (Fédération Internationale de la Précontrainte).17,18) Hydrogen content was varied by adjusting the concentration of NH₄SCN between 0.25–20 mass%. The relationship between the immersion time and diffusible hydrogen content was examined for Steel S11 and Steel V14 immersed in 1 mass% and 20 mass% NH₄SCN solution. For the immersed specimens, diffusible hydrogen contents in the specimens were measured by means of TDA after removing the surface corrosion products with #1000 emery paper.

2.2.2. Constant Load Test (CLT)

CLT is a method that estimates the maximum diffusible hydrogen content at which a steel does not fail due to hydrogen embrittlement under a constant load. In this study, two types of hydrogen charging methods were applied as follows.

Tests under high hydrogen concentration conditions were carried out by Nippon Steel & Sumitomo Metal Corporation (NSSMC). Cathodic hydrogen charging method was used for hydrogen absorption. Hydrogen content was varied by changing current density and ammonium thiocyanate (NH₄SCN) concentration in an aqueous solution of 3 mass% NaCl+(0.1–20 g/L) NH₄SCN. The duration of hydrogen charging was 18 h. Zn plating was applied to the specimens after hydrogen charging to prevent hydrogen degassing. Both Steel S11 and Steel V14 were kept at room temperature for 3 h and 96 h after Zn plating, respectively, to homogenize hydrogen distribution in the specimens. The Zn plating was removed subsequent to CLT and hydrogen content in the specimens was measured by means of TDA.

Low hydrogen concentration condition tests were carried out by Neturen Co. Ltd. (NC). The tests were performed by the immersion method using the aqueous solution of NH₄SCN specified in the FIP.17,18) Hydrogen content was varied by adjusting the concentration of NH₄SCN between 0.25–20 mass%. CLTs were performed in NH₄SCN solution heated at 50°C. NH₄SCN solution was poured into the test cell with a jacket in which water of 50°C was circulated to maintain the test temperature as shown in Fig. 4. The immersion CLT results were analyzed as a function of absorbed hydrogen content in un-loaded specimens immersed for 100 h.

The loading stress was set with respect to the maximum tensile stress of the circumferential notched round bar specimens without hydrogen, that is 1 663 MPa. The applied stresses for Steel S11 were 1 497 MPa, 1 330 MPa and 993 MPa and the ratios of the applied stress to the maximum notch tensile stress were 0.9, 0.8 and 0.6, respectively. The stresses applied to Steel V14 were 1 618 MPa, and 1 299 MPa, and their ratios to the maximum notch tensile stress, 2 023 MPa were 0.8 and 0.6, respectively. The maximum tensile stress σ [N/mm² = MPa] of the circumferential notched round bar specimens was calculated from Eq. (1):

$$\sigma = \frac{P}{28.3}$$

where

$$P = \text{the maximum tensile load} \ [\text{N}]$$

* The area of the notched portion was 28.3 mm² for Ø 6.0 mm.

The CLT was finished in the case that no fracture occurred within 100 h after loading. Diffusible hydrogen contents of un-loaded specimens after immersion for 100 h in the same solutions used for the CLT were regarded as the diffusible hydrogen contents of the specimens used for the CLT.

2.2.3. Slow Strain Rate Test (SSRT)

Fracture stresses of specimens charged with hydrogen under various conditions were evaluated by means of SSRT. The tests were conducted at a crosshead displacement rate of 0.005 mm/min. Low displacement rates promote stress-induced diffusion of hydrogen resulting in the hydrogen accumulation in the notch root according to the stress concentration in the specimens. The relationship between hydrogen content and fracture stress evaluated by SSRT can be expected to coincide with that obtained by means of CLT.

SSRT were carried out at the laboratories of Nippon Steel & Sumitomo Metal Corporation (NSSMC) and National Institute for Material Science (NIMS). Hydrogen charging was conducted by cathodic charging method prior to SSRT. Hydrogen charging at NSSMC was conducted for 18 h at a current density of 0.3–2.0 A/m² in a 3 mass% NaCl+(0–3 g/L) NH₄SCN solution and that at NIMS was conducted for 72 h at a current density of 0.4–6.25 A/m² in a 3 mass% NaCl+3 g/L NH₄SCN solution or 0.1 mol/L –NaOH solution. Hydrogen content was controlled by changing test solutions and current densities. To prevent hydrogen degassing during SSRT, the specimens were Zn plated at NSSMC laboratory and Cd plated at NIMS laboratory. After plating, at NSSMC laboratory, Steel S11 and Steel V14 specimens were kept at room temperature for 3 h and 96 h respectively, to homogenize the hydrogen distribution in the specimens. At NIMS laboratory, the homogenization treatment was not conducted. Instead, hydrogen charging time was 72 h which is long enough to homogenize hydrogen concentration.

Hydrogen concentration near fracture surface was measured by means of TDA with gas chromatography at NSSMC laboratory and quadrupole mass spectrometry at NIMS laboratory. The fracture surfaces were observed by
using an Electron Scanning Microscope (SEM) to investigate the fracture mode.

2.2.4. Conventional Strain Rate Test (CSRT)

It is known that SSRT is conducted under an extremely slow strain rate so that hydrogen distribution can reach an equilibrium state through stress-induced diffusion. In contrast, CSRT\(^{13-16}\) is performed under a high strain rate similar to ordinary tensile testing rate which makes the stress-induced diffusion negligible. Therefore, hydrogen content at the crack initiation site of hydrogen embrittlement in the vicinity of the notch root is equivalent to the average hydrogen content in the specimen. It is required to the specimens to be charged with hydrogen uniformly prior to CSRT. The hydrogen charging time to achieve homogeneous hydrogen distribution is determined in advance.

CSRT were carried out at several laboratories in institutions and companies. Sophia University (SPU), Nippon Steel & Sumitomo Metal Corporation (NSSMC), and Mitsubishi Steel MFG. Co. LTD. (MSM) carried out CSRT to evaluate hydrogen embrittlement property of Steel S11. SPU, NSSMC, and National Institute for Material and Science (NIMS) conducted CSRT of Steel V14. The threaded part of the specimen was coated with a silicone plastic before hydrogen charging to avoid fracture there. The shank part including notched area of the specimen, 20–40 mm from the center, was charged with hydrogen by cathodic hydrogen charging method. Electrolyte was a 3 mass\% NaCl+0–3 g/L NH\(_4\)SCN aqueous solution or a 0.1 mol/L–NaOH aqueous solution. The current density was varied in the range of 0.25–80 A/m\(^2\). Hydrogen charging time until the hydrogen distribution became homogeneous throughout the circumferential notched round bar specimen was about 72 h for Steel S11 and about 120 h for Steel V14. The hydrogen charging time for Steel S11 was adopted 72–144 h at SPU, 144 h at NSSMC, and 72 h at MSM, and that for Steel V14 was 120 h at SPU, 144 h at NSSMC, and 336 h in NIMS.

Immediately after hydrogen charging, CSRT was conducted at a crosshead displacement rate of 1 mm/min. Subsequently, Hydrogen concentration near fracture surface was analyzed by means of TDA using gas chromatography. In addition, the fracture surfaces were observed by means of SEM to investigate the fracture mode.

2.3. Hydrogen Analysis Method

Diffusible hydrogen content in the specimen was measured by means of TDA using gas chromatography or quadrupole mass spectrometry. The heating rate was 100\(^\circ\)C/h. Diffusible hydrogen content was defined as the summation of the first peak evolved below 300\(^\circ\)C.

3. Results

3.1. Hydrogen Absorbing Behavior

The TDA results of Steel S11 with cathodic electrolysis hydrogen charging showed that the peak temperature of the hydrogen desorption curve was about 100\(^\circ\)C, which was equivalent to that in previous studies\(^{13,14}\) on similar SCM steels with tensile strengths of 1 300 MPa and 1 500 MPa. As shown in Fig. 5, the peak temperature of the hydrogen desorption curve for Steel V14 was approximately 150\(^\circ\)C in the case of small absorbed hydrogen content. On the other hand, the peak temperature was approximately 100\(^\circ\)C in the case of large absorbed hydrogen content. Steel V14 has hydrogen trapping sites with high activation energy, such as fine VC precipitation. The reason that the difference in the peak temperature depend on the absorbed hydrogen content can be that hydrogen initially occupies high energy hydrogen trap sites.\(^{21}\)

The relationship between immersion time in 1 mass\% and 20 mass\% NH\(_4\)SCN aqueous solutions and diffusible hydrogen content was investigated without loading. The results are shown in Fig. 6. For Steel S11, at both NH\(_4\)SCN concentrations, diffusible hydrogen content increased during the initial 20 h, and then became constant. Therefore, it is assumed that diffusible hydrogen concentration becomes satisfactorily homogeneous in the specimen in 20 h. In contrast, for Steel V14, diffusible hydrogen content continued to increase within 100 h. The result means hydrogen charging requires more than 100 h for Steel V14 to obtain homogeneous hydrogen distribution.

![Fig. 5. Hydrogen curves of various hydrogen content for Steel V14 charged in aqueous solutions of 0.1 M NaOH at current densities (CD) in the range from 10 to 80 A/m\(^2\).](image1)

![Fig. 6. Relationship between immersion time in NH\(_4\)SCN solution and diffusible hydrogen content for Steel S11 and Steel V14 (without load).](image2)
and diffusible hydrogen content for Steel S11 and Steel V14 is shown in Fig. 7. In both steels, the time to fracture became longer as diffusible hydrogen content decreased. Similar tendencies were observed at other stress levels. The maximum hydrogen content at which specimens does not fracture for 100 h is defined as the critical hydrogen content, $H_C$. $H_C$ depends on the stress conditions and the kind of materials.

The relationships between applied stress and diffusible hydrogen content for Steel S11 and Steel V14 are shown in Fig. 8. The open marks in the figure denote minimum hydrogen content among the fractured specimens. The solid marks in the figure denote the maximum value among the non-fractured specimens (except for specimens with hydrogen content equivalent to or higher than that of the open mark). In both steels, fracture stress decreased as diffusible hydrogen content increased. Under the same hydrogen content, the fracture stress of Steel V14 is higher than that of Steel S11.

### 3.3. SSRT

The relationships between the fracture stress obtained by means of SSRT and hydrogen content for Steel S11 and Steel V14 are shown in Figs. 9 and 10, respectively. The fracture stress decreased as hydrogen content increased in both steels. In the case of Steel S11, the relationship between the fracture stress and hydrogen content was similar in the two laboratories. The fracture modes observed in the areas around the crack initiation site were quasi-cleavage (QC) within a range of hydrogen content less than 0.5 mass ppm, and inter-granular (IG) crack mixed with QC in a range of hydrogen content more than 0.5 mass ppm. The slope of the decrease in nominal fracture stress with hydrogen content was gradual within a range of hydrogen content less than 0.5 mass ppm. On the contrary, the slope was relatively steeper in a range of hydrogen content from 0.5 to 1 mass ppm. The same tendency was reported on a previous research for boron added tempered martensite steel with a tensile
strength equivalent to that of Steel S11. The result implies that IG crack remarkably decreases fracture stress.

Absorbed hydrogen content in Steel V14 was significantly higher than that in Steel S11 under the same hydrogen charging conditions since fine VC precipitations acted as hydrogen trapping sites. Generally, steels with high tensile strength exhibit a large decrease in nominal fracture stress by hydrogen charging compared to steels with low tensile strength. However, nominal fracture stress of Steel V14 was apparently higher than that of Steel S11 under the same hydrogen content as shown in Figs. 9 and 10.

3.4. CSRT

The relationship between hydrogen content and fracture stress evaluated by means of CSRT for Steel S11 is shown in Fig. 11. In CSRT, it is assumed that average hydrogen content in the test specimen $H_{CSRT}$, is equivalent to the local hydrogen content $H^*$ at the fracture initiation site. Similar relationships between nominal fracture stress and hydrogen content were obtained in three laboratories. The fracture stress was less sensitive to hydrogen content in the low hydrogen content region, whereas the fracture stress decreased sharply when the hydrogen content range was higher than 2.5–3.0 mass ppm. The dependence of the fracture stress on hydrogen content was consistent with a change in the fracture mode. In the region of the low hydrogen content, QC is dominant in the area positioned several tens of μm inside from the tip of the notch at which the fracture initiates. On the contrary, in the region of high hydrogen content, IG comes to mix with QC. IG strongly affected to the fracture stress and indicated high susceptibility to hydrogen embrittlement compared with QC.

The relationship between hydrogen content and fracture stress evaluated by CSRT for Steel V14 is shown in Fig. 12. Similar tendencies were obtained in three laboratories for Steel V14 as well as Steel S11. Thus, CSRT was presumed to be an appropriate testing method that exhibits little deviation in the results. Absorbed hydrogen content in Steel V14 was remarkably higher than that in Steel S11 under the same hydrogen charging conditions. In addition, under the same hydrogen content, Steel V14 fractured at a higher stress than Steel S11 similarly to SSRT. In the region of low hydrogen content less than 5 mass ppm, the fracture stress gradually decreases as hydrogen content increases. However, in the region of high hydrogen content, the fracture stress remarkably decreases as hydrogen content increases. The sharp decrease in the fracture stress was consistent with a change in the fracture surface. In the region of low hydrogen content, QC was dominant at the fracture initiation sites, whereas in the region of high hydrogen content, IG came to be mixed with QC.

4. Discussion

Similarity and difference between CLT, SSRT and CSRT are discussed as follows.

Fracture stresses as a function of diffusible hydrogen contents evaluated by CLT, SSRT and CSRT are compared in Figs. 13 and 14 for Steel S11 and Steel V14, respectively. The marks in CLT denote the results on minimum hydrogen content among the fractured specimens, and the minimum values in the error bars correspond to maximum hydrogen content among the non-fractured specimens. At the same fracture stress level, CSRT shows higher hydrogen content than the other methods, because hydrogen accumulation in the vicinity of notch root through stress-induced diffusion is hard to occur in CSRT. On the other hand, CLT and SSRT provide approximately the same relationship between frac-
ture stress and hydrogen content. The reason is presumably that both test methods provide a similar process to fracture which affords adequate time for stress-induced diffusion of hydrogen.

The relationship between diffusible hydrogen content and fracture stress obtained by means of SSRT and CSRT depends upon the fracture mode. QC is dominant in the region of low hydrogen content, where the fracture stress gradually decreases with diffusible hydrogen content. On the other hand, when hydrogen content exceeds a certain amount, IG appears accompanying QC, and the fracture stress sharply decreases.

It has been reported by Hagihara et al.\cite{13,14} that SSRT and CSRT presented almost the same relationship between the local stress and the local diffusible hydrogen concentration at the initiation site for 1 300–1 500 MPa grade steels. From this logic, the local diffusible hydrogen concentration $H^*$ accumulated by stress-induced diffusion in this study was calculated using Eq. (2),\cite{6} and results obtained from CLT, SSRT and CSRT were compared.

$$H^* = H \times \exp \left\{ \frac{-(\sigma_h - \sigma_{h\min}) \times \Delta V}{RT} \right\} \quad \ldots \ldots \quad (2)$$

Where, $H$ is average diffusible hydrogen content in specimens measured by TDA, and $\sigma_h, \sigma_{h\min}, \Delta V, R, \text{and } T$ denote hydrostatic stress, hydrostatic stress at a position away from the notch bottom of specimens, change in volume produced in the lattice by hydrogen ($2 \times 10^{-6} \text{m}^3/\text{mol}$), gas constant and test temperature at 300 K, respectively. $\sigma_h$ was obtained through Finite Element Analysis (FEA). As the analysis conditions, Marc was used employing a two-dimensional model with the symmetrical axis. Stress-strain curves below TS of the steels obtained by using smooth round bar specimens were used for the analyses. In the region beyond tensile strength, the relationship between stress and strain was postulated according to power law hardening.

Local diffusible hydrogen concentration at the fracture initiation site in CLT and SSRT was obtained by Eq. (2). The relationship between fracture stress and local diffusible hydrogen concentration for Steel S11 is shown in Fig. 15. The Y-axis shows the nominal fracture stress of the specimens at the notched part, and does not denote the local stress at the fracture initiation site. However, in this study, as the shape of every specimen used in all tests is identical, the nominal fracture stress and the maximum local stress have one-to-one correspondence. The local hydrogen concentrations at a given nominal fracture stress of 1 100-MPa-grade Steel S11 obtained by using the respective methods are in the order of SSRT < CLT < CSRT.

The above outcome is different from the previous results\cite{13} reported by Hagihara et al. One possible reason is the difference in the strength level. Since the steels with 1 300 MPa and 1 500 MPa grade tensile strength were used by Hagihara et al., IG was dominantly appeared. On the contrary, QC was dominantly appeared in the present study. It is considered that Steel S11 shows greater plastic deformation under stress than 1 300 MPa grade steel. Takai et al.\cite{20} reported that the interaction between hydrogen and dislocation facilitates occurrence of hydrogen embrittlement. It is supposed that SSRT provides a large interaction between hydrogen and dislocation because hydrogen can diffuse with dislocation moving under a slow strain rate. By contrast, it is supposed that CSRT provides a small interaction between hydrogen and dislocation because hydrogen cannot follow the fast dislocation motion during CSRT. Therefore, it is possible that CSRT provides low susceptibility to hydrogen embrittlement than SSRT even when locally accumulated hydrogen content is the same. In the case of CLT, it is likely to provide a small interaction between hydrogen and dislocation as the dislocation moves suddenly when the specimen is loaded. Nevertheless, the susceptibility of Steel S11 to
hydrogen embrittlement using CLT lies in between SSRT and CSRT. This is probably attributed to that dislocation moves to some extent due to relaxation during CLT.

The relationship between fracture stress and local diffusible hydrogen content for Steel V14 of 1 400 MPa grade is shown in Fig. 16. The local hydrogen concentrations at a given nominal fracture stress of 1 400-MPa-grade Steel V14 obtained by using the respective methods are in the order of CSRT < CLT ≒ SSRT. The result showed different tendency to that in Steel S11. The possible reason is the effect of VC precipitation as hydrogen trapping sites. The hydrogen trapping sites can affect several parameters in Eq. (2). Thus, the validity of applying Eq. (2) should be examined to estimate the locally accumulated hydrogen content strictly.

5. Conclusion

Susceptibilities to hydrogen embrittlement of two kinds of low alloy steels - SCM435 (Steel S11) and V-added steel (Steel V14) - were evaluated by Constant Load Test (CLT), Slow Strain Rate Test (SSRT) and Conventional Strain Rate Test (CSRT) in several laboratories. Relationship between diffusible hydrogen content and fracture stress was compared among the three test methods. The results are as follows.

(1) In CLT, critical diffusible hydrogen content decreased with increase in applied stress. CLT under immersion in NH₄SCN solution and CLT after cathodic hydrogen precharging in two laboratories showed similar tendency.

(2) In SSRT, fracture stress decreased with increase in diffusible hydrogen content. QC (Quasi-cleavage) fracture was observed in low hydrogen content range, whereas IG (Inter-granular) fracture was observed in high hydrogen content range. Similar relationship between diffusible hydrogen content and nominal fracture stress were obtained in two laboratories.

(3) In CSRT, fracture stress decreased with increase in diffusible hydrogen content. QC fracture was observed within a range of small hydrogen content, whereas IG fracture was observed in a range of large hydrogen content. Three laboratories obtained a similar relationship between diffusible hydrogen content and nominal fracture stress.

(4) Steel V14 absorbed higher content of diffusible hydrogen than Steel S11 under the same hydrogen charging conditions.

Moreover, Steel V14 showed a higher nominal fracture stress than Steel S11 at the same diffusible hydrogen content.

(5) By means of CLT and SSRT, a similar relationship between diffusible hydrogen content and nominal fracture stress was obtained both in the case of Steel S11 and Steel V14. The reason is that both of the methods allow hydrogen to diffuse sufficiently during the test.

(6) The order of local critical diffusible hydrogen concentration for Steel S11 was SSRT < CLT < CSRT under the same nominal stress. For Steel S11, CSRT showed lower susceptibility to hydrogen embrittlement than SSRT even under identical local hydrogen concentration. The cause is considered to be the difference in interaction between dislocation and hydrogen during tests. During SSRT the interaction is strong due to slow dislocation motion at a slow strain rate, while during CSRT the interaction is weak because of fast dislocation motion which does not allow hydrogen to follow dislocations.

(7) The order of local critical diffusible hydrogen concentration for Steel V14 was CSRT < CLT ≒ SSRT under the same nominal stress. The result showed different tendency to that in Steel S11. The possible reason is the effect of VC precipitation on several parameters in conventional equation on local hydrogen accumulation.

REFERENCES