

Hydrogen Exposure Effect on Tensile Strength of High Strength steel Sharp Notched Specimen

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1. Introduction

Recently, hydrogen energy is expected a next energy of fossil fuels and several products which use hydrogen energy are developed. For example, the development of fuel cell vehicles and hydrogen automobiles are advanced, and many parts of these products and infrastructure installations of hydrogen supply; pipe lying and storage tank are manufactured from a variety of steels. Steels cause hydrogen embrittlement, especially, high strength steels exhibit high hydrogen embrittlement susceptibility [1-2]. Although many researches have been studied to understand hydrogen embrittlement [3-5], it is not yet satisfactorily understood. In order to use high strength steel in hydrogen environment, the effect of hydrogen to high strength steel were discussed.

Almost failures of industrial products are caused by stress concentration zone such as crack and defect. Additionally, hydrogen concentrates at high stress field. Therefore, the tensile property considered stress concentration was measured. In our previous study, we studied the tensile tests of 1300MPa tensile strength of SCM435 with a sharp notched specimen in 0.6MPa hydrogen gas environment [6]. As a result, the notch tensile strengths of hydrogen gas 48h exposed specimens were higher than that of hydrogen gas non-exposed specimens. As increase of hydrogen content causes decrease of tensile strength, it is thought that increase of tensile strength for hydrogen gas exposed specimens are caused by other factors.

In this study, in order to examine the hydrogen concentration distribution which is caused by hydrogen exposed time, the tensile tests were conducted using the notched specimens with different hydrogen exposed time. To investigate the effects of hydrogen concentration distribution on tensile strength is also useful for considering a use condition of manufactured products. Furthermore, the effect of hydrogen exposure on the tensile strength at several gas pressure was investigated.

2. Specimen and experimental procedure

2.1. Specimens

Quenched and tempered SCM435 was used in the present study with the chemical composition listed in Table 1. The circumferentially notched round bar specimens with a notch root radius of $\rho=0.03\text{mm}$, minimum cross-section diameter of $d=6\text{mm}$ and depth of notch of $t = 1.0\text{mm}$ are shown in Fig.1. The notch root was polished by alumina powder (powder size: $0.05\mu\text{m}$) to avoid the effects of machined flaw or surface roughness.

2.2. Experimental procedure

The tensile tests were carried out at a cross head speed was $5.0 \times 10^{-4}\text{mm/s}$ in hydrogen gas environment with gas pressure 0.6MPa at room temperature. Hydrogen gas exposed condition was gas pressure 0.6MPa at room temperature and exposed time were 0 (non-exposed), 3, 6, 7.5, 9, 12, 19, 24 and 48h. How to decide the exposed time are discussed below. The tensile test of hydrogen gas non-exposed specimen was started right after filling a gas. Tensile tests of hydrogen gas exposed specimens were exposed to hydrogen gas for 3h-48h before starting the tests. The tensile tests of other pressure conditions were carried out at 0.3, 0.6, 5.0 and 9.5MPa the same procedure of testing at 0.6MPa pressure. The tensile tests at 0.3 and 0.6MPa were carried out with hydrogen gas 48h exposed specimens, and tensile tests at 5.0 and 9.0MPa were carried out with hydrogen gas non-exposed specimen and hydrogen gas 48h exposed specimen.

3. Experimental results and discussions

3.1. Hydrogen contents

In order to decide the saturation exposed time to hydrogen gas, calculation of theoretical hydrogen contents and a thermal desorption spectrometry (TDS) analysis was conducted.

Table1 Chemical composition (wt%)

C	Si	Mn	P	S	Ni	Cr	Mo	Cu
0.38	0.22	0.84	0.024	0.021	0.08	1.11	0.15	0.12

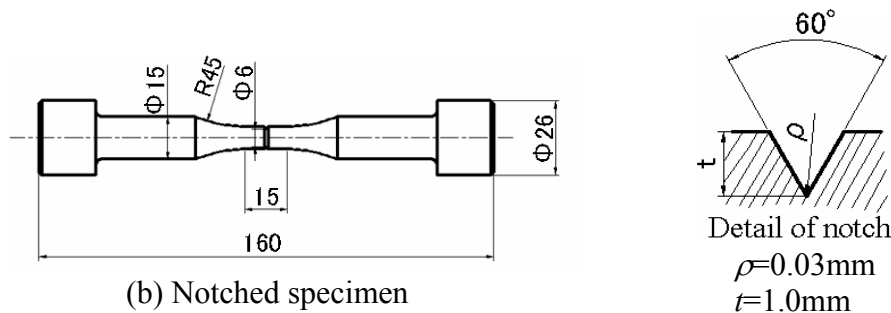


Fig.1 Specimen configuration.

3.1.1. Theoretical curve of Hydrogen contents

Consider an infinite cylinder of radius r_0 . The rate of C/C_0 ; C is the concentration at the point r at time t and C_0 is the concentration at the point of cylinder surface ($r= r_0$) of the cylindrical ordinate, is derived from Fick's equation at constant temperature [7].

$$C / C_0 = 1 - 2 \sum_{n=1}^{\infty} \frac{\exp\left(-\frac{D\beta_n^2 t}{r_0^2}\right) J_0\left(\frac{\beta_n r}{r_0}\right)}{\beta_n J_1(\beta_n)} \quad (1)$$

Where D is the diffusion coefficient under experimental conditions ($D=1.0 \times 10^{-10}$ m²/s for SCM435 [8]), J_0 , J_1 are Bessel coefficients of zero and first order, β_n is a root of $J_0(\beta)=0$. From the Eq. 1, the rate of C_{ave}/C_0 ; C_{ave} is the average hydrogen concentration in an infinite cylinder at time t , is follows,

$$C_{ave} / C_0 = 1 - 4 \sum_{n=1}^{\infty} \frac{\exp\left(-\frac{D\beta_n^2 t}{r_0^2}\right)}{\beta_n^2} \quad (2)$$

The theoretical curve of hydrogen concentration at center of infinite cylinder with diameter $d'=6$ mm derived from Eq. 1 is shown in Fig.2(a). This cylinder's size was decided from the size of minimum cross-section diameter (6mm) for the tensile test specimen. Hydrogen diffusion time is adequate for 20-30h at the center of cylinder according to Fig.2(a). The theoretical curve of average hydrogen concentration of infinite cylinder with diameter $d'=6$ mm derived from Eq. 2 is shown in Fig.2(b). The data obtained from TDS analysis are average hydrogen content in measurement sample, therefore the results of TDS analysis are effective by compared with Fig.2(b).

3.1.2. Results of TDS analysis

Hydrogen gas exposure and a TDS analysis were conducted with referring the theoretical exposed time obtained in the previous chapter. The exposed condition was with the gas pressure 0.6MPa at room temperature and exposed specimens had a diameter of 6mm and a length of 30mm. The exposed times were 0, 6, 24, 48, and 96h. After exposed, the measurement samples with thickness 2.0mm were cut off from the middle of specimens and measured by the TDS, where the measurement of the sample's thickness was sufficiently shorter than the exposure tests specimen's length, hydrogen diffusion condition of the measurement samples were the same as that of an infinite cylinder.

The results of TDS analysis are shown in Fig.3. As a result, the exposed time for 48h was adopted as saturation time by comparing the results of TDS analysis and theoretical curve. The exposed times of the tensile tests were decided voluntarily within 48h.

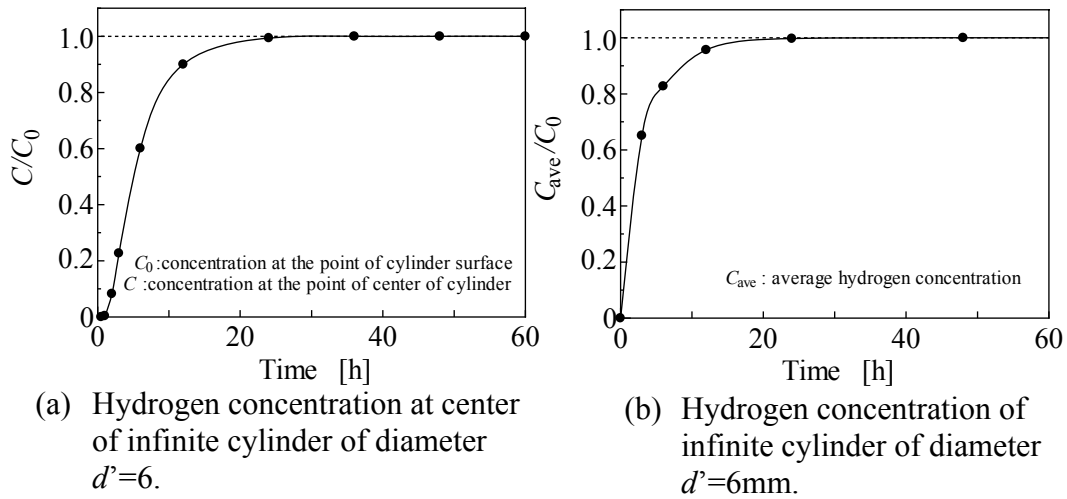


Fig.2 Theoretical curves of hydrogen concentration.

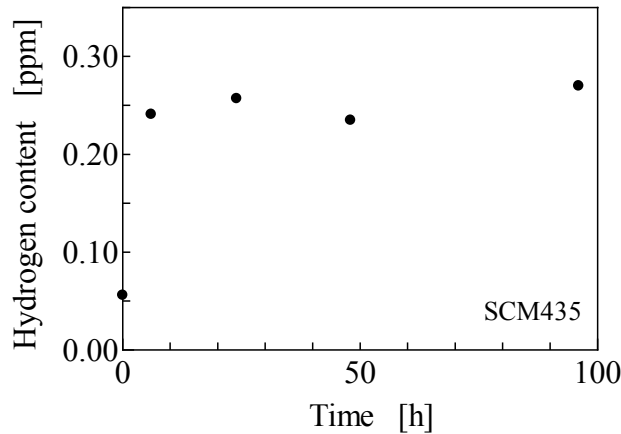


Fig.3 Relationship between hydrogen content and exposed time.

3.2. Effect of hydrogen gas exposed time on tensile strength

The stress-elongation curves of hydrogen gas 48h exposed specimen and non-exposed specimen are shown in Fig.4, and the relationship between tensile strength and hydrogen gas exposure time is shown in Fig.5. The tensile strengths at hydrogen gas exposed time between 0-6h are low levels, between 6-19h increases with increasing exposed time and between 19-48h are high levels.

The fracture surfaces were examined in consideration of the effect of hydrogen gas exposed time on tensile strength. The fracture surfaces of the 48h exposed specimen and the non-exposed specimen were observed on the scanning electron microscope (SEM) and images are shown in Fig.6. Both the 48h exposed specimen and the non-exposed specimen are exhibited an area which is seemed to be ductile fracture in the very vicinity of notch root (Fig.6(a)), an intergranular

fracture in the vicinity of the notch root (Fig.6(b)) and a quasi-cleavage fracture inside of an intergranular fracture appearance area (Fig.6(c)). As both fracture surfaces are similar, it makes no difference about the fracture mechanism to exposed time. It is considered that the increase of the tensile strength is caused by the change of hydrogen concentration distribution with increase of hydrogen gas exposed time. Therefore, the hydrogen concentration distribution at each exposed time is calculated from Eq.2 and is shown in Fig.7. The exposed time of non-exposed specimen is calculated as testing time 15min.

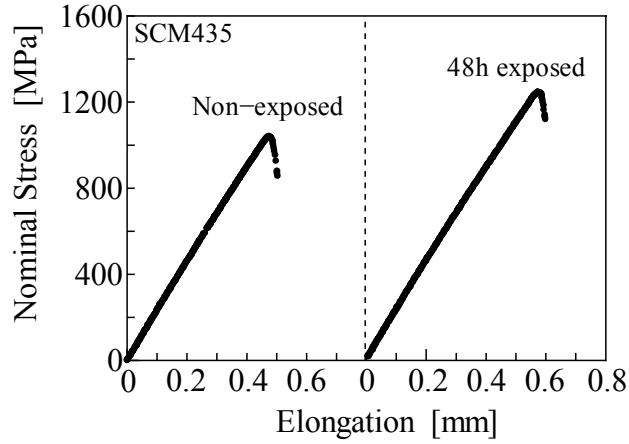


Fig.4 Relationship between nominal stress and elongation.

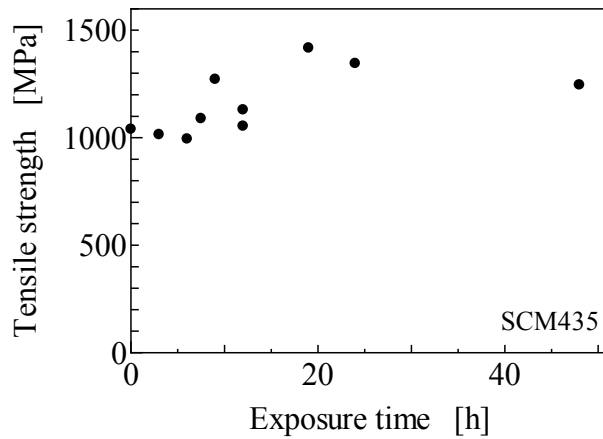
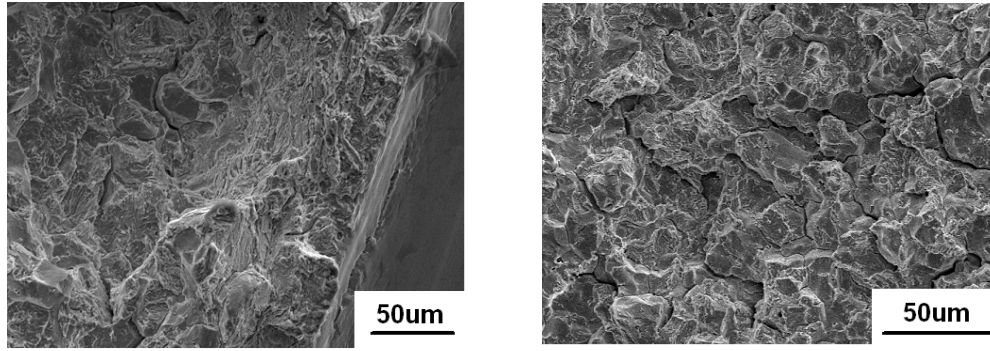
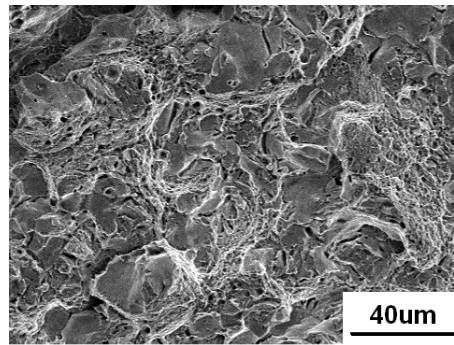


Fig.5 Relationship between hydrogen concentration and depth from specimen surface.



(a) Ductile fracture.

(b) Intergranular.



(c) Quasi-cleavage.

Fig.6 SEM images of fracture surfaces.

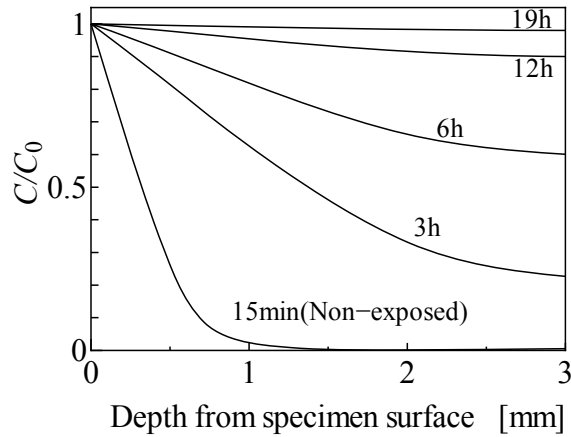


Fig.7 Relationship between hydrogen concentration and depth from specimen surface.

There is the difference of the hydrogen concentration between specimen surface and center until hydrogen diffuses sufficiently. It is thought that the difference of the hydrogen concentration distribution causes the difference of the tensile strength. As the hydrogen exposed time is short and the difference of hydrogen concentration distribution is not uniformed, the tensile strength is low. On the

other hand, as the hydrogen exposed time is long and the difference of hydrogen concentration distribution is uniformed, the tensile strength is high. This is the reason that about 20h diffusion time that hydrogen diffuses homogeneously in specimen is coincident with 19h exposed time that the tensile strength is the highest value. However, as these hydrogen concentration distributions are default values which are not considered stress loading, it is different from actual hydrogen concentration distributions at fracture. An effect of the hydrogen concentration distribution given by the stress condition to tensile strength is investigated in the next assignment.

3.3. Effect of hydrogen gas pressure on tensile strength

The relationship between tensile strength and hydrogen gas pressure is shown in Fig.8. The tensile strength decreases with increasing the hydrogen gas pressure. The difference of tensile strength between hydrogen gas non-exposed specimen and hydrogen gas 48h exposed specimen testing at 5.0 and 9.5MPa are smaller than that of testing at 0.6MPa. In testing at 9.5MPa, the tensile strength of the hydrogen gas 48h exposed specimen was lower than that of the hydrogen gas non-exposed specimens. Due to increase of the hydrogen concentration at notch root that is caused by increase of gas pressure, the tensile strength is low. Therefore, as the effect of hydrogen concentration at notch root to the tensile strength is more effective than the effect of the hydrogen concentration distribution to the tensile strength, the hydrogen concentration distribution effect was not appeared in testing at 5.0 and 9.5MPa.

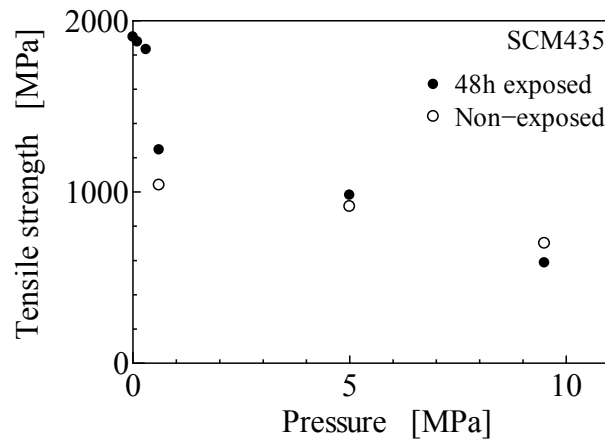


Fig.8 Relationship between tensile strength and hydrogen gas pressure.

4. Conclusions

The effects of hydrogen gas exposed time and hydrogen gas pressure on 1300MPa tensile strength of SCM435 steel with a sharp notched specimen have been investigated. The results of this study can be summarized as follows.

- (1) The notch tensile strength increases with increasing hydrogen gas exposed time until hydrogen diffuses homogeneously at 0.6MPa gas pressure.
- (2) The notch tensile strength decreases with increasing hydrogen gas pressure.

Acknowledgement

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