Hydrogen redistribution under stress-induced diffusion and corresponding fracture behaviour of a structural steel

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ABSTRACT

Hydrogen redistribution under stress-induced hydrogen diffusion and corresponding fracture behaviour of a 960 MPa grade martensitic steel were studied. Slow strain rate tensile (SSRT) tests after hydrogen pre-charging were performed and the fracture surface was observed and analysed. The strain rate ranged from 10^{-6} to 10^{-4} s⁻¹. In the pre-charged sample with a certain hydrogen content of 0.62 ppm, hydrogen distribution was homogeneous before the SSRT test. After tensile testing, brittle fracture features appeared in the centre of the fracture surface, while ductile features appeared in the surrounding area. Brittle region size increased with the strain rate slowing down in the range from 10^{-4} to 5×10^{-6} s⁻¹, while it stabilised at the strain rate slower than 5×10^{-6} s⁻¹. Relationship between the strain rate and the brittle region size was established and discussed based on the present data of hydrogen content in the material.

ARTICLE HISTORY

Received 11 January 2017 Revised 21 April 2017 Accepted 25 April 2017

KEYWORDS

Slow strain rate test; hydrogen pre-charging; diffusion; structural steel; brittle region size; hydrogen redistribution

Introduction

Hydrogen distribution plays an important role in hydrogen embrittlement (HE) of metals and alloys [1,2]. High-strength steel usually exhibits susceptibility to HE [3–8], causing hydrogen-induced delayed fracture [2]. High-strength steel has been widely used as a structural material in many applications. Previous studies [9,10] showed that stresses below the alloy elastic limit could appreciably modify hydrogen concentration and tensile stress could increase hydrogen solubility [1]. This means that the initial hydrogen distribution (after pre-charging without mechanical loading) would change when stress is applied to the high-strength steel. On the other hand, certain hydrogen content is inevitably retained in steel during its fabrication processes, such as smelting and acidic pickling [2].

There are many research reports on HE of highstrength steel, and majority of them focus on characterising degradation after hydrogen charging. Some researchers have studied hydrogen distribution in different microstructures [11,12], or analysed the crack path of hydrogen-induced cracking [13,14]. Dynamic hydrogen charging, which means hydrogen was introduced into the specimen during mechanical loading, was mainly used in the above studies [15,16]. Hydrogen content was not fixed during deformation process. Since a certain amount of hydrogen is present in the specimen, it is important to know how the hydrogen distribution changes when a tensile stress was applied to the specimen, and how it affects material fracture behaviour. Current literature lacks such reports.

Hydrogen affects material mechanical behaviour based on the time-dependent diffusion and enrichment. The strain rate affects tensile test duration time. Strain rate effects on hydrogen diffusion and enrichment were studied in this paper. Relationships between applied stress and hydrogen redistribution, along with microstructure evolution, are very important for understanding hydrogen-induced delayed fracture.

Experimental methods

Materials

The material examined in this study is a commercial hot-rolled 960 MPa grade high-strength steel, which was tempered at about 600°C. The chemical composition in weight percent is 0.2% C, 1.2% Mn, 0.22% Si, 0.3% Cr, 0.3% Mo, with Fe balance. Specimen for microstructure observations was ground to 5000 grit, and then mechanically polished and etched in 4% natal solution. Light microscope and scanning electron microstructure.

Slow strain rate tests

The specimen of slow strain rate tensile (SSRT) used in this study is shown in Figure 1. The tensile specimens

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Figure 1. Tensile specimen schematics, and the thickness is 1.5 mm.

were 1.5 mm thick, and their surfaces were wet-ground with SiC paper up to 2000 grit, and then ultrasonically cleaned in acetone and ethanol. The strain was recorded from the cross-head displacement. Hydrogen charging was implemented in $0.2 \text{ mol } L^{-1}$ sulphuric acid (H_2SO_4) solution at a current density of 1 mA cm⁻² for 15 min at room temperature. Only the gauge region of the tensile specimen was charged. After pre-charging, the sample was washed with deionised water, dried, and then cadmium was plated on the sample surface for about 45 min. Then the sample was cleaned and dried again followed by installation onto the tensile machine to immediately start the SSRT. The above procedure took about 15 min. Three tensile strain rates of 5×10^{-5} , 10^{-5} and 5×10^{-6} s⁻¹ were mainly studied in this work. The other two strain rate of 10^{-4} and 10^{-6} s⁻¹ were used to verify the result. Each major strain rate was tested three times.

Fracture morphology was observed by Qyanta FEG 450 scanning electron microscope (SEM). In addition, hydrogen concentration was also measured. The specimen size of hydrogen concentration test was cut for the $25 \times 5 \times 1.5$ mm³, which is similar to the gauge region of tensile specimen. Before hydrogen charging, the surfaces were also wet- ground and cleaned as mentioned above. To ensure the data accuracy and repeatability, five specimens were used to measure hydrogen content after pre-charging, using the same conditions as SSRT specimens. Hydrogen concentration was measured by G4 PHOENIX at 800°C for 20 min.

Hydrogen permeation tests

Usually, the specimen used for hydrogen permeation tests was like a coin. However, in this work, to facilitate pre-strain, the sample shape was first like the tensile specimens with the gauge width of 30 mm, length of 72 mm and thickness of 2 mm, then pre-strained to plastic deformation of 1, 2 or 3%. After that, the gauge section was cut down to perform hydrogen permeation test.

Double electrolytic cell for hydrogen permeation test was used in this study; before the permeation test, the specimen was ground with SiC paper up to 5000 grit then ultrasonically cleaned in acetone. The hydrogencharging solution in the charging side was 0.2 mol L⁻¹ NaOH + 0.22 g L⁻¹ thiourea (H₂NCSNH₂) with 1 mA cm⁻² charging current density at room temperature, approximately 25°C, while the solution in the escaping side was 0.2 mol L⁻¹ NaOH.

Results

Microstructure

Figure 2 exhibits the 960 MPa grade high-strength steel microstructure and fracture surface of hydrogen-free sample, and the horizontal direction is the rolling direction in the light micrograph, which was marked by white arrow in Figure 2(a). The microstructure of the steel is full lath martensite and almost isotropy, which is more easily distinguished in the SEM image as Figure 2(b). The fracture surface of hydrogen-free sample has mainly dimples and a few small regions of brittle features, as indicated by yellow arrows in Figure 2(d).

Slow strain rate tests

The nominal stress-nominal strain curves of SSRT after hydrogen pre-charging are shown in Figure 3. The strain is not measured with a local extensometer but from the cross-head displacement. Therefore, the slope of the linear part of the curve does not correspond to the elastic modulus of the steel. Overall, the elongation decreased with the strain rate slowing. The duration time is about 3 min for the stretching of the hydrogen-free sample, 51 min at $5 \times 10^{-5} \text{ s}^{-1}$, 237 min at $1 \times 10^{-5} \text{ s}^{-1}$, and 460 min at $5 \times 10^{-6} \text{ s}^{-1}$ for the pre-charged samples. When the tensile rate is quick enough, the effect of diffusible hydrogen in the lattice could be ignorable, because the duration time of tensile test is short and not enough for hydrogen diffusion and enrichment to cause the change of mechanical property [17]. However, when the strain rate was slow, such as 10^{-5} and $5 \times 10^{-6} \text{ s}^{-1}$, there is long enough time for hydrogen enrichment. Thus, the total elongation was affected. Moreover, an average and the standard deviation of hydrogen concentration in this work was 0.62 ± 0.1 ppm. So, the small elongation loss is mainly because of the low hydrogen concentration of the precharging specimen.

After SSRT, SEM was used to observe the fracture morphology. Figure 4 shows the fracture surface of the pre-charged specimen. Figure 4(a-e) show the overall morphology of the fracture surface at five strain rates, and Figure 4(f-h) show enlarged view of Figure 4(d). It is clear that entirely brittle fracture occurred in the centre of the fracture surface, as shown in Figure 4(f), and the fracture behaviour changed to mixed mode of shallow dimples and brittle crack, as indicated by



Figure 2. Microstructure of the 960 MPa grade high-strength steel: (a) light micrograph; (b) SEM image; (c) and (d) are fracture morphology of hydrogen-free sample.



Figure 3. The nominal stress-nominal strain curves of the hydrogen pre-charged specimens.

yellow arrows in Figure 4(g), then completely ductile fracture at the edge of the fracture surface, as shown in Figure 4(h). As mentioned above, three samples were tested at each mainly studied strain rate, and the average size of the brittle region was calculated. L is defined as the size of the brittle region. It was found that L becomes larger with the strain rate decrease, as indicated by the yellow lines in Figure 4(a–c), which is 1.62 ± 0.12 mm,

 2.55 ± 0.11 mm and 2.97 ± 0.07 mm corresponding to the strain rate of 5×10^{-5} s⁻¹, 1×10^{-6} s⁻¹ and 5×10^{-6} s⁻¹. Thus, the fracture morphology of SSRT for the pre-charged sample here is absolutely different from the situation of dynamic charging during SSRT. For example, Wang's work [18] showed that hydrogeninduced cracking occurred in the surrounding of the specimen under dynamic hydrogen charging of SSRT,



Figure 4. SEM images obtained after pre-charging at different strain rates: (a) 10^{-4} s^{-1} ; (b) $5 \times 10^{-5} \text{ s}^{-1}$; (c) 10^{-5} s^{-1} ; (d) $5 \times 10^{-6} \text{ s}^{-1}$; (e) 10^{-6} s^{-1} ; (f, g and h) enlarge regions labelled as 1, 2 and 3 in (d).

and the centre of specimen exhibited ductile fracture. This indicated that the situation is exactly contrary to the pre-charged specimens. It is easy to understand because the hydrogen entered into the sample from the surface during dynamic charging.

Hydrogen permeation test

Hydrogen permeation test under different plastic strain was studied. Figure 5 shows the hydrogen permeation curves for the samples with different pre-strain of 0 (asreceived), 1, 2 and 3%. After testing, apparent hydrogen



Figure 5. Hydrogen permeation curves for the specimen as-received and after pre-strained of 1, 2 and 3%.

diffusion coefficients were calculated using the following formula [2]:

$$D = W^2 / 6t_{0.63} \tag{1}$$

where *D* is the apparent hydrogen diffusion coefficient, *W* is the thickness of the specimen, $t_{0.63}$ is the delay time, which could be expressed at 0.63 time to reach the steady current.

The details of *D* and $t_{0.63}$ for the four different prestrain are listed in Table 1. When the strain increases, *D* decreases. The value of *D* is the largest at the conditions without any plastic deformation. For example, comparing the *D* values of 0 and 3%, the *D* value decreased from 6.8×10^{-7} to 1.8×10^{-7} cm² s⁻¹.

Kim and Kim [19] showed that D did not change with elastic deformation. However, when the specimen was plastically deformed in tension, apparent hydrogen diffusion coefficient decreased. Thus, D is not a constant in the plastic range, and decreases with plastic strain increasing. This is mainly because the as-received material is hot-rolled and tempered at 600°C, and the dislocation density in the material is low. So the saturation current value of the hydrogen permeation curve is high. After plastic deformation, the dislocation density

Table 1. The details of *D* and $t_{0.63}$ of four different deformation conditions.

Pre-strain, %	t _{0.63} , s	D , 10^{-7} cm ² s ⁻¹
0	2430	6.8
1	4525	3.6
2	7275	2.2
3	8915	1.8

in the material increased. Dislocation is usually considered to be hydrogen traps. Therefore, the saturation current of hydrogen permeation curves became small.

Discussion

Hydrogen homogenisation

The total time from hydrogen charging to beginning of tensile test was about 1.25 h (charging 15 min + cadmium coating 45 min + installing onto the tensile machine 15 min). The formula $x = 2\sqrt{Dt}$ was used to roughly estimate the diffusion distance of hydrogen, where x is the hydrogen diffusion distance and D is apparent hydrogen diffusion coefficient without strain, which is 6.8×10^{-7} cm² s⁻¹. Then x is calculated to be about 1.1 mm. Hydrogen was charged from two sides of the specimen, while the sample thickness is 1.5 mm. Therefore, hydrogen distribution already reached homogeneous before tensile loading. Furthermore, the interval time between hydrogen charging finishing and cadmium plating beginning is only about 2 min, it is very short. So it is assumed that there is no hydrogen leaving the sample.

Relationship between L and $\dot{\epsilon}$

For pure iron, it is calculated that hydrogen can move together with dislocation motion when the strain rate is less than $10^{-7}\rho_{\rm H} \,{\rm s}^{-1}$ [2], where $\rho_{\rm H}$ is the movable dislocation density. For martensitic steel, the density of dislocation is very large, especially after tensile to plastic deformation. The strain rate used in this experiment is slow, so hydrogen is easy to move with dislocation

Table 2. Detailed data of the strain rate \dot{e} and the brittle region size *L* of the fracture surface.

έ, s ^{−1}	<i>L</i> , mm
10 ⁻⁴	0.8
5×10^{-5}	1.62 ± 0.12
10 ⁻⁵	2.55 ± 0.11
5×10^{-6}	2.97 ± 0.07
1 × 10 ⁻⁶	2.91

together. During the stretching process, dislocation and hydrogen move together to the centre of the sample, then caused brittle fracture in the centre of specimen. The specimen section size used in this work is 5 mm width and 1.5 mm thickness. Thus, the plane strain condition does not meet in the thickness direction but in a state of plane stress. Therefore, dislocation mainly moved along the width direction. As a result, the brittle region size L, i.e. hydrogen-induced cracking, would change along the width of the brittle zone on the fracture surface. While in the thickness direction, brittle region was through the sample.

Detailed information of the size of brittle region Land strain rate $\dot{\varepsilon}$ are listed in Table 2. Relationship between L and $\dot{\varepsilon}$ can be fitted as shown in Figure 6, where the size of the brittle region increases with the tensile rate decrease. However, when the strain rate is slow enough, the value of L tends to be stable. This means that the value of L might become a constant. It should be mentioned that the hydrogen content in the present work is a constant, i.e. 0.62 ppm. It is believed that the brittle region size will be related to hydrogen content. For a sample with hydrogen content higher than 0.62 ppm, L would properly increase. According to Abbassi's [20] work, the exponential relationship between the size of nucleation of microvoid and $\dot{\varepsilon}$ was observed. According to Figure 6, here, the relationship between *L* (mm) and $\dot{\varepsilon}$ (s⁻¹) in the present situation was exponentially fitted as follows:

$$L = 3.02e^{-13167\dot{\varepsilon}}$$
(2)

To verify this formula, $\dot{\varepsilon}$ of $6 \times 10^{-3} \text{ s}^{-1}$ was carried out to a same pre-charged sample. According to equation (2), when $\dot{\varepsilon} = 6 \times 10^{-3} \text{ s}^{-1}$, $L \approx 0 \text{ mm}$, which means that there is no brittle area on the fracture surface. The corresponding fracture surface is shown in Figure 7. It exhibited dimples feature either in the centre or in the edge on the fracture surface. There is exactly not brittle area. The tensile test lasted only about 3 min. During the short time, hydrogen accumulation degree is too small to cause the change in mechanical properties [17], including the fracture mode. Thus the hydrogeninduced brittle cracking would not occur.

Hydrogen distribution on the fracture surface

Figure 4(g) shows that brittle region is also accompanied with dimples. Previous work [21–23] indicated that hydrogen promoted cracking propagation by promoting void growth in front of crack tip. Abbassi's [20] research showed that largest strain occurred at the centre of the fracture surface for the plate tensile specimen. Local strain decreases from the centre to the edge of specimen. When the stress gradient and hydrogen concentration gradient existed in the specimen, Fick's first law can be changed as [2]

$$J = J_c + J_\sigma = -D\frac{\partial c}{\partial x} + \frac{Dc}{RT}\overline{V_H}\frac{\partial \sigma_h}{\partial x}$$
(3)



Figure 6. Relationship between the size of brittle region and the strain rate.



Figure 7. Fracture surface obtained at 6×10^{-3} s⁻¹ strain rate: (a) the whole fracture surface; (b) zoomed in boxed area marked in (a).

where σ_h is the hydrostatic stress, $\frac{\partial c}{\partial x}$ is the hydrogen concentration gradient, *D* is the hydrogen diffusion coefficient, $\overline{V_H}$ is the partial molar volume of hydrogen dissolved in the materials. It is assumed that hydrogen diffusion in the material reaches steady, i.e. $\frac{\partial c}{\partial t} = 0$ and J = 0, then the relationship between hydrostatic stress and hydrogen concentration c_σ can be obtained by the formula (3), it can be expressed as [2] follows:

$$c_{\sigma} = c_0 \exp \frac{\overline{V_H} \sigma_h}{RT} \tag{4}$$

After plastic deformation occurs, the maximum strain occurs at the centre of specimen due to the multiplication of dislocations and vacancies [20], thus hydrogen was concentrated and trapped in the centre of specimen. As the strain increases, stress-induced hydrogen diffuses from the edge to the centre, thus the trapped hydrogen increases further. Finally, hydrogen-induced cracking, i.e. brittle fracture occurs at the centre of the sample. And if time is enough, the higher the stress, the higher the hydrogen concentration. Above all, the relationship between the diffusible hydrogen and local strain can be described as hydrogen concentration increases with local strain increase [24]. Thus, for the plate specimen, the highest hydrogen concentration locates in the centre of the fracture surface. Therefore, the strain gradient from the centre to the edge of the fracture surface decreased with the tensile rate decrease. Previous work showed that tensile properties and plastic deformation mechanisms depend on the strain rate, and fracture strain increased with the strain rate increases [25]. Other researcher [26] studied the hydrogen redistribution during tensile test after hydrogen pre-charging using a notched specimen, and found the stress concentration in the root of the notch is near the surface of the specimen.

On the other hand, hydrogen-induced cracking is related to the hydrogen concentration. According to the relationship (4), when the local strain is high enough, hydrogen concentration might reach higher than the critical value and cause HE. The corresponding schematic is shown in Figure 8. Here, ε_c is the critical strain of causing HE. Brittle region formed at the region with strain higher than ε_c . For a certain material and a certain hydrogen content (0.62 ppm, in this work), ε_c is a constant [2]. With the tensile rate decrease, the size of the brittle region increased. It is worth reminding that the combination of the size of the brittle region and hydrogen concentration is not clear. Thus, further research will focus on finding this relationship among the brittle region size, hydrogen content and strain rate.

HE in steels has been discussed mainly in terms of hydrogen-enhanced decohesion (HEDE) [27],



Figure 8. Schematics of the relationship between L and ε for the three tensile test rates.

hydrogen-enhanced localised plasticity (HELP) [28] and hydrogen-enhanced and strain-induced vacancies (HESIV) [22,23]. The HE of fully ferritic steels was ascribed to the HELP effect [29]. In lath martensitic steel, the HE was reported to be associated with HELP and HEDE [30]. In this work, for the sample without hydrogen charging, the dominated dimple feature on the fracture surface (Figure 2(d)) indicates that coalescence of voids and cracks contributed to the final fracture. Since the brittle region size is decades micrometre (larger than the grain size), the brittle fracture regions result from brittle crack propagation through multiple grains. The martensite-related cracking associated with decohesion is considered to be the cause of the partial brittle fracture surface. For the sample with hydrogen prechaging, except the entirely brittle region in the centre of the fracture surface (Figure 4(f)), hydrogen charging increased the brittle size in the mixed region of brittle linkage region indicated by yellow arrows and shallow dimple strip indicated by yellow curve in Figure 4(g). The dimple strip indicates that hydrogenassisted crack propagation was caused by decohesion and ductile cracking. Moreover, the size of the dimple strip is related to the strain rate, while the critical strain ε_c is a constant. This suggested that a HEDErelated mechanism was at work. On the other hand, the presence of hydrogen enhanced the increase in strain-induced trapping sites and reduced ductile crack growth resistance. The strain-induced increase in the hydrogen absorption capacity was correlated with the susceptibility to HE. This suggested that the HELP theory played a role. Based on this, both HEDE and HELP mechanisms assist the damage evolution in the steel in a cooperative manner.

Conclusions

In this paper, hydrogen redistribution and fracture behaviour of the 960 MPa grade martensitic steel was studied using SSRT method after pre-charging. The following conclusions are drawn from this work:

- (1) HE sensibility increases with tensile rate decrease.
- (2) Fracture surface can be divided into three parts. Brittle fracture occurs at the centre of the fracture surface, while ductile fracture appears at the edge of the specimen. There is a mix mode region between the centre and the edge of the fracture surface.
- (3) For a certain amount of hydrogen exited in the steel, the relationship between the size of brittle region (L) and strain rate (\u00ec) can be expressed as

$$L = 3.02e^{-13167\dot{\epsilon}}$$

Thus, the size of the brittle fracture zone increased at lower tensile rate, while the brittle size might reach a maximum when the strain rate is slow enough. Future work will focus on the relationship among the brittle size, strain rate and hydrogen content in this structural steel. It hopes that an effective method will be developed to evaluate the steel lifetime to avoid hydrogen damage.

Disclosure statement

No potential conflict of interest was reported by the authors.

Funding

This project was supported by the National Natural Science Foundation of China under grant Nos. 51371037 and 51571029 and the National Program on Key Basic Research Project (973 Program) under grant No. 2014CB643301 and State Key Laboratory of Development and Application Technology of Automotive Steels (Baosteel Group).

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