Effect of hydrogen on void initiation in tensile test of carbon steel JIS-S25C

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Abstract. In order to investigate the effect of hydrogen on tensile fracture mechanism of a carbon steel, tensile tests were conducted. Pre-strain specimens (0%, 5% and 10%) were used to study the effect of hydrogen content, since saturated hydrogen content in specimens increases in increasing dislocation density. The tensile strength and the yield stress of hydrogen specimens were almost the same as uncharged. In contrast, the reduction of area of hydrogen charged specimens was smaller than that of uncharged. To reveal the reasons of decrease of the reduction of area, the fracture surface and longitudinal cross section near the fracture surface were observed. On the fracture surface of uncharged specimens, only dimples were observed. On the other hand, dimples and flat fracture surface were observed on the fracture surface of hydrogen charged. On the longitudinal cross section of hydrogen charged specimens, many voids were observed compared to uncharged. From these observations, it is showed that hydrogen gives a rise to the increase of voids and the hydrogen charged specimens break without sufficient necking, thus hydrogen makes the reduction of area smaller.

1. Introduction

In recent years, many researchers [1-7] have been studied about the effect of hydrogen on tensile strength properties. It has been reported that tensile strength and yield stress of hydrogen charged specimens were almost the same as those of uncharged. In contrast, the reduction of area of hydrogen charged specimens was smaller than that of uncharged. During tensile tests, voids are initiated in the specimens and lead to failure. However, there are no studies about the effect of hydrogen on tensile fracture mechanism in terms of void initiation and distribution.

In this study, the effect of hydrogen on void initiation and distribution of a carbon steel was studied to reveal the tensile fracture mechanism. Fracture surface and longitudinal cross section near fracture surface in broken specimens were observed. Longitudinal cross section around necking of unbroken specimens was also observed. And voids distribution in specimens before break was compared with those after break.

2. Materials and experimental procedure

2.1. Materials

Tensile tests were conducted using a normalized 0.24% carbon steel, JIS (Japanese Industrial Standards) S25C [8]. Table 1 shows the chemical compositions of the carbon steel JIS-S25C. Figure 1 shows the shape and dimensions of the tensile specimens. The specimen surface was polished with emery paper #800. Pre-strain specimens (pre-strain: ε_{pre} = 0%, 5% and 10%) were used to study the effect of
hydrogen content, since saturated hydrogen content in specimens increases in increasing dislocation density. Pre-strain was introduced in specimens at cross-head speed of 1 mm/min.

Table 1. Chemical compositions of JIS-S25C. (mass%)

|       | C   | Si  | Mn  | P   | S   | Cu  | Ni  | Cr  | NC  |
|-------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| S25C  | 0.24| 0.22| 0.51| 0.015| 0.017| 0.08| 0.05| 0.16| 0.21|

Figure 1. Shape and dimensions of tensile specimens (in mm).

2.2. Experimental procedure
The specimens were charged with hydrogen by immersing in 20 mass% NH₄SCN solution at 313 K for 48 hours. According to the results of the experiment by Kaneko et al. [9], the hydrogen content of uncharged specimens of JIS-S25C was 0.0 ppm. The hydrogen content of 0% pre-strained hydrogen charged specimens was 0.24 ppm, 5% pre-strained hydrogen charged specimens was 0.67 ppm, 10% pre-strained hydrogen charged specimens was 1.20 ppm. Hydrogen content of hydrogen charged specimens increased in increasing pre-strain. Tensile test was carried out with crosshead speed of 1 mm / min at room temperature in air.

3. Results

3.1. Tensile properties
Table 2 shows tensile properties. Figure 2 shows the stress-strain diagram. The tensile strength σ_B and yield stress σ_UY, σ_UL of hydrogen charged specimens were almost the same as those of uncharged. In contrast, elongation at fracture of hydrogen charged specimens was smaller than that of uncharged. Figure 3 shows the reduction of area. The reduction of area of hydrogen charged specimens was obviously smaller than uncharged and decrease in increasing pre-strain. Thus, the reduction of area tends to decrease in increasing hydrogen content of specimens.

Table 2. Tensile properties.

|       | Uncharged | Hydrogen charged |
|-------|-----------|-----------------|
|       | Upper yield stress σ_UY [MPa] | Lower yield stress σ_LY [MPa] | Tensile strength σ_B [MPa] | Upper yield stress σ_UY [MPa] | Lower yield stress σ_LY [MPa] | Tensile strength σ_B [MPa] |
| S25C  | Pre-strain 0% | 386 | 312 | 499 | 399 | 325 | 494 |
|       | Pre-strain 5% | 382 | 313 | 498 | 375 | 316 | 497 |
|       | Pre-strain 10% | 379 | 311 | 499 | 378 | 309 | 498 |

3.2 Fracture surface
Figure 4 shows fracture surface of uncharged and hydrogen charged specimens. On fracture surface of uncharged specimen (Figure 4 (a), (b) and (c)), only typical dimples were observed. On the other hand, on fracture surface of hydrogen charged specimen (Figure 4 (d), (e) and (f)), typical dimples and flat fracture surface were observed. Most of the flat fracture surfaces were formed circularly around inclusions. Figure 5 shows the relationship between pre-strain and area of the flat fracture surface of hydrogen charged specimens. As the pre-strain increased, the area of the flat fracture surface increased. This result shows that the area of flat fracture surface increase in increasing hydrogen content.
Figure 2. Stress-Strain diagram.

Figure 3. Reduction of area.

(a) Uncharged, $\varepsilon_{\text{pre}} = 0\%$
(b) Uncharged, $\varepsilon_{\text{pre}} = 5\%$
(c) Uncharged, $\varepsilon_{\text{pre}} = 10\%$

(d) Hydrogen charged, $\varepsilon_{\text{pre}} = 0\%$
(e) Hydrogen charged, $\varepsilon_{\text{pre}} = 5\%$
(f) Hydrogen charged, $\varepsilon_{\text{pre}} = 10\%$

Figure 4. Fracture surface.

Figure 5. Relationship between pre-strain and area of flat fracture surface.
3.3. **Longitudinal section through fracture surface**

In order to reveal the tensile fracture mechanism of uncharged and hydrogen charged specimens, longitudinal section of broken and unbroken specimens was observed. Broken specimens were carried out tensile tests to failure. And unbroken specimens were carried out tensile tests up to initiation of necking, just after tensile strength. The longitudinal section of broken specimens was observed near fracture surface and that of unbroken was observed around necking. To study the relationship between voids and metal structure, the cross section was polished by buff and etched by natal (solution of 97% alcohol and 3% nitric acid).

Figure 6 shows the longitudinal section of fracture surface. Almost the voids of the uncharged specimens were formed in longitudinal direction. On the other hand, almost the voids of the hydrogen charged specimens were formed in perpendicular to the longitudinal direction. Almost all voids of hydrogen charged and uncharged specimens were initiated from pearlite structure and inclined to loading direction since voids might occur by slip deformation.

![Loading direction](image)

(a) Uncharged, $\varepsilon_{pre} = 0\%$
(b) Uncharged, $\varepsilon_{pre} = 5\%$
(c) Uncharged, $\varepsilon_{pre} = 10\%$

(d) Hydrogen charged, $\varepsilon_{pre} = 0\%$
(e) Hydrogen charged, $\varepsilon_{pre} = 5\%$
(f) Hydrogen charged, $\varepsilon_{pre} = 10\%$

**Figure 6.** Longitudinal section through fracture surface.

3.4. **Voids distribution**

To examine distribution of voids, voids were counted on each observation area ($500 \, \mu m \times 500 \, \mu m$). The observation area 0 shows the center of specimen. The number of observation area is denoted as shown in Figure 7.

Figure 8 shows the voids distribution. In the unbroken specimens (Fig 8. (a), (c) and (e)), there was no obvious difference the void distribution of hydrogen charged and uncharged specimens. Many voids on longitudinal cross section of uncharged broken specimen (Fig 8. (b), (d) and (f)) were observed near the center of specimens and a few voids near specimen surface. On the other hand, many voids on longitudinal cross section of hydrogen charged broken specimen (Fig 8. (b), (d) and (f)) were observed over the cross section.
**Figure 7.** Observation area of longitudinal section.

**Figure 8.** Voids distribution near fracture surface.
4. Discussion
From the above results and observations, the mechanism of tensile fracture can be explained as follows. Figure 9 shows the mechanism of tensile fracture. In hydrogen charged specimens, many voids initiate over the cross section. And flat fracture surface and voids grow and merge to others, thus hydrogen charged specimens break before necking progress substantially. On the other hand, in uncharged specimen, fewer voids initiate compared with hydrogen charged. Necking progress and the stress distribution at the center of specimens is triaxial stress state. Because stress at the center of specimens is higher than that near surface, more voids initiate in the center of specimens. And the uncharged specimen breaks after the reduction of area becomes larger sufficiently. Hydrogen charged specimens fractured faster than uncharged specimens, because the number of voids is more for hydrogen charged specimens.

Figure 9. Mechanism of tensile fracture.

5. Conclusions
Tensile tests were conducted using hydrogen charged and uncharged specimens of a 0.24% carbon steel JIS-S25C to reveal the effects of hydrogen on void initiation in tensile tests of a carbon steel. The conclusions can be summarized as follows.

(1) The tensile strength and the yield stress of hydrogen specimens were almost the same as those of uncharged. In contrast, the reduction of area of hydrogen charged specimens was smaller than that of uncharged.

(2) On fracture surface of uncharged specimen, only typical dimples were observed. On the other hand, on fracture surface of hydrogen charged specimen, typical dimples and flat fracture surfaces were observed. As pre-strain increased, the area of the flat fracture surface on hydrogen charged specimens also increased because of hydrogen content.
(3) Many voids on longitudinal cross section of uncharged specimen were observed near the center of specimens and a few voids near specimen surface. On the other hand, many voids on longitudinal cross section of hydrogen charged specimen were observed over the cross section.

(4) A flat fracture surface in the hydrogen charged specimens might initiate before the voids initiation.

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