Influence of Specimen Surface Roughness on Hydrogen Embrittlement Induced in Austenitic Steels during In-Situ Small Punch Testing in High-Pressure Hydrogen Environments

Hyung-Seop Shin 1,*, Juho Yeo 1 and Un-Bong Baek 2

Abstract: An in-situ small punch (SP) test method has recently been developed as a simple screening technique for evaluating the properties of metallic materials used in high-pressure hydrogen environments. With this method, the test conditions including temperature and gas pressure can easily be adjusted to those used in practice. In this study, specimens of STS316L steel and 18 wt% Mn steel were prepared at two different surface roughness, fabricated using wire-cutting and mechanical polishing. Their effects on hydrogen embrittlement (HE) were evaluated using in-situ SP testing at both room temperature and a lower temperature where HE was shown to occur under 10 MPa hydrogen. Both steels were evaluated using two variables obtained from in-situ SP testing, the SP energy, and the relative reduction of thickness (RRT), to quantitatively determine the effect of specimen surface roughness on HE susceptibility. Their fracture characteristics due to HE under 10 MPa hydrogen showed little difference with surface finish. Surface roughness had a negligible influence on these quantitative factors describing HE, indicating that it is not a dominant factor to be considered in in-situ SP testing when it is used to screen for HE compatibility in steels used in high-pressure hydrogen environments.

Keywords: in-situ small punch test; hydrogen embrittlement; surface roughness; austenitic steels; relative reduction of thickness

1. Introduction

Hydrogen energy is drawing attention in application fields such as mobilities wherein global warming and microparticle emissions are being addressed. Many countries have recently announced road maps for their hydrogen economies in the near future and have been pursuing their respective action plans. Research and development are actively underway to increase the supply of hydrogen fuel cell electric vehicles, expand hydrogen stations, and inexpensively produce green hydrogen.

From a hydrogen economy viewpoint, hydrogen energy expansion should include the use of low-cost metals in the parts and components of devices used in hydrogen production, transportation, and storage. Metals with austenitic structures such as austenitic stainless steels are generally used in hydrogen energy facilities because they can provide sufficient hydrogen embrittlement (HE) resistance, even at low temperatures [1–3]. When austenite stainless steels have low Ni-equivalent content, however, hydrogen can penetrate the metal, causing HE to some extent due to phase transformations resulting from the reduced austenitic phase stability [4]. Therefore, when using steels in hydrogen energy devices, their properties under practical hydrogen use environments must be identified, and reliability should be ensured by accumulating HE characteristic data. As a result, HE behaviors of materials under high-pressure conditions have primarily been evaluated in...
either external hydrogen conditions using slow strain rate tensile testing (SSRT), where a specimen is typically placed in a large autoclave with explosion-proof capabilities, or in internal hydrogen conditions using hydrogen-precharged specimens in ambient air [4–6]. When SSRT tests take place in high-pressure hydrogen environments in particular, extensive equipment and facilities for creating and maintaining external hydrogen test conditions are required [3,7,8] as well as large amounts of cost and manpower for installing, operating, and maintaining autoclave equipment. In practice, it is not feasible to perform SSRTs under high-pressure hydrogen to obtain varieties of HE compatibility data for materials used in devices exposed to actual hydrogen environments, given the breadth of chemical composition and microstructures and thus the number of tests required. A simple test method that easily provides data for screening HE susceptibility without the autoclave equipment would prove useful. Toward that end, a tensile test has been performed in the atmosphere while filling a hollow-type specimen with high-pressure hydrogen [9–11]. This type of specimen has also been used in high-pressure cryogenic environments and tested using SSRT.

Another simple test method for evaluating the HE behaviors in steels, the small punch (SP) test, has been performed, typically for examination under internal hydrogen conditions, using H\textsubscript{2}-precharged specimens in ambient air [12,13]. The SP test has some advantages in that it is a direct mechanical test and does not require a large amount of material, and the small sampling system is practical in examining the nonuniform features, such as weld [14–16]. However, when using H\textsubscript{2}-precharged specimens, the correlation between the H\textsubscript{2} content charged and the H\textsubscript{2} pressure around the material in practice is not clear. Therefore, the mechanical properties of steels in hydrogen must be evaluated in hydrogen use environments that mimic those used in practice during in-situ testing.

We recently developed a simple in-situ SP test method for evaluating the HE behaviors of metallic materials under high-pressure external hydrogen conditions [17,18]. It is an external hydrogen test procedure that investigates embrittlement behaviors of structural steels under high-pressure hydrogen environments without relying on large-scale high-pressure vessels, unlike ASTM G142 and ISO 11114–4 [6,19]. It directly evaluates the embrittlement effects of hydrogen penetrating through the surface of the specimen and diffusing into the material during in-situ SP testing. It has been used to screen for HE susceptibility in various steels used in hydrogen energy applications [20,21].

The influence of high-pressure hydrogen on high-strength materials can vary with the surface roughness of the specimen. Therefore, when performing a tensile test under high-pressure hydrogen, the smooth part of the specimen is typically polished. Specimen surface conditions depend on the fabrication method, such as wirecutting, forming, grinding, or machining. Each creates a different surface roughness and thus, potentially, different HE behaviors. Surface conditions are known to affect corrosion resistance in metals [22], but their effects on HE behaviors have been investigated in machined austenitic stainless steels only on hydrogen-precharged specimens [23]. Results can differ from those found when investigating the direct influence of surface conditions in in-situ testing under external hydrogen conditions.

Test specimens used in the SP test for evaluating HE sensitivity are small in size [14]. They are commonly fabricated by wirecutting or machining specimens from supplied plates or pipes. In the wirecutting process in particular, the surface can be hardened by localized melting during electric discharge machining, causing a denser structure at the surface than general machining. It can affect the HE behaviors occurring on the surface of the specimen when used in in-situ SP testing, which should be performed under high-pressure external hydrogen conditions [17–20]. This process is unlike other cutting processes, wherein the specimen surface is not likely to undergo microstructural changes such as the martensitic transformations seen in austenitic stainless steels [24]. Therefore, investigating the HE effect of surface roughness in specimens directly exposed to high-pressure hydrogen gas is meaningful in establishing the in-situ SP test method and expanding its applicability.
Ogata et al. studied the influences of specimen surface conditions on HE assessments of austenite stainless steels using SSRTs on hollow-type specimens filled with high-pressure hydrogen, finding minimal influence from various surface machining methods [25]. Richardson and others used an SP test to evaluate the effects of surface roughness on deformation and fracture behaviors of 9% Cr steel in air [26], also finding that surface roughness did not change the maximum load or fracture displacement. On the other hand, during the in-situ SP testing, various stress states and gradients are formed in the specimen as a result of flexural deformation of specimen, a phenomenon that is different than what is seen in uniaxial tensile tests of SSRTs. The role of the surface roughness in HE behaviors has yet to be clarified when a stress gradient from bending deformation exists in high-pressure hydrogen environments.

Therefore, in this study, we aimed to determine whether surface roughness influences HE behaviors in test specimens using the in-situ SP test method under high-pressure hydrogen environments. Specimens of STS316L steel and 18 wt% Mn steel with austenite phases were prepared with different surface conditions using wirecutting and mechanical polishing. The in-situ SP test was conducted under a high-pressure gas environment at room temperature (RT) and a lower temperature where severe HE has been shown in these steels [20,21]. The influence of specimen surface roughness on HE behaviors was qualitatively and quantitatively examined based on the applied test temperature and punch velocity. Using this process, the in-situ SP test method can be established as a simple method for screening HE susceptibilities of steels to be used in the hydrogen energy industry.

2. Experimental Procedures

2.1. Specimens

The chemical compositions and mechanical properties of supplied samples of STS316L steel and 18 wt% Mn steel are shown in Tables 1 and 2, respectively. Notably, both steels show large elongations at RT.

| Material            | C  | Si | Mn  | P  | S  | Cr | Ni | Mo | Fe         |
|---------------------|----|----|-----|----|----|----|----|----|------------|
| 18 wt% Mn steel     | 0.62 | -  | 17.98 | 0.02 | 0.02 | -  | -  | -  | Bal.       |
| STS316L steel       | 0.013 | 0.47 | 0.66 | 0.03 | 0.004 | 16.65 | 10.11 | 2.06 | Bal.       |

| Material          | Yield Strength (MPa) | Tensile Strength (MPa) | Elongation (%) |
|-------------------|----------------------|------------------------|----------------|
| 18 wt% Mn steel   | 498                  | 1070                   | 50.2           |
| STS316L steel     | 229                  | 568                    | 63.5           |

A wirecutting process was used to machine the specimens for in-situ SP testing, measuring 10 mm × 10 mm × 0.5 mm, from supplied sheets of steels. The surfaces of the wirecutted samples were either left untouched, designated as “as-received”, or were polished using 600-grit sandpaper, designated as “polished”. Figure 1 shows the appearance of each surface finish, as viewed using scanning electron microscopy (SEM, TECSAN, Czech Republic). Surface roughness was measured using a surface roughness tester (SV-502; Mitutoyo, Japan), and Table 3 compares those measures, expressed as mean and standard deviation. Polished specimens were much smoother ($R_a$ ranged from 0.11 to 0.14 μm) than those not polished ($R_a$ ranged from 2.4 to 2.9 μm). This shows the effects of electric discharge machining. In general, STS316L steel was slightly rougher than 18 wt% Mn steel.
Figure 1. SEM images of specimen surfaces, either as-received or polished (using 600-grit sandpaper).

Table 3. Surface roughness of specimens.

| Condition               | STS316L Steel | 18 wt% Mn Steel |
|-------------------------|---------------|-----------------|
| Wirecutted (as-received)| 2.88 ± 0.195  | 17.68 ± 1.011   |
| Polished (#600 grit)   | 0.14 ± 0.025  | 3.13 ± 0.299    |

Note: $R_a$ = the arithmetical mean of the roughness value; $R_{\text{max}}$ = the maximum roughness depth, respectively. Adapted from ref. [27]. Each value shown is the average across three measures.

2.2. Experimental Procedure

In-situ SP tests were performed on specimens of each surface finish at RT and at temperatures where HE was significant under 10 MPa hydrogen gas ($H_2$), which were $-60 \, ^\circ C$ for STS316L steel and $-40 \, ^\circ C$ for 18 wt% Mn steel, respectively [20,21].

Figure 2 shows the schematic diagram of the in-situ SP test fixture used to apply a high-pressure hydrogen environment and a low temperature. Details of the test procedure, including hydrogen charging and maintaining a low temperature, are described elsewhere [17,20]. Briefly, the specimen was mounted between the lower and upper dies of the SP test fixture at RT. An O-ring was inserted into the groove formed in the lower die to prevent the gas from leaking during the SP test. To maintain an even pressure on the O-ring, the upper and lower dies were tightened to a uniform torque (1.5 N-m) using the torque wrench. The space between the specimen and the lower die was vacuumed and purged three times with high-purity nitrogen gas ($N_2$) to remove impurities, and then $H_2$ was charged into the space to a specified pressure so that one side of the specimen was exposed. The SP test was conducted, placing a steel ball 3 mm in diameter on the specimen, and then applying a compressive load via a punch. The lower die had a 4 mm diameter hole. To conduct the in-situ SP test at low temperatures, once the SP test fixture was mounted to the material testing machine (AG-IS, 5 kN load cell, Shimadzu, Japan), a cooling device was installed. During cooling, a capsule-type container made of Styrofoam was installed around the SP test fixture containing the specimen, allowing the pressure gauge, ball valve, and one-touch connector to remain in the ambient environment. To cool the specimen and the interior of the container to the specified test temperature, liquid nitrogen (LN$_2$) was pumped into the Cu-tube coil and allowed to vaporize in the insulated container. Two T-type thermocouples were used to monitor the specimen temperature.
Table 4 shows the test conditions adopted. Punch velocity was 0.1 mm/min under high-pressure N\textsubscript{2}, and under high-pressure H\textsubscript{2}, three punch velocities (1.0, 0.1, and 0.01 mm/min) were used. At each punch velocity, a compressive load was applied to the specimen via the punch and steel ball. Both the applied load and the displacement of the specimen were measured. Punch displacement obtained from the testing machine was used to infer specimen displacement at the midpoint of its lower surface. The in-situ SP test was performed twice at each test condition.

Table 4. Test conditions applied during the in-situ small punch tests used to examine the effects of surface roughness on hydrogen embrittlement behaviors.

| Specimen Dimension (mm) | 10 × 10 × t0.5 |
|-------------------------|----------------|
| Ball diameter (mm)      | 3.0            |
| Gas purity              | N\textsubscript{2} (99.999%), H\textsubscript{2} (99.999%) |
| Gas pressure (MPa)      | 10             |
| Punch velocity (mm/min) | 1.0, 0.1, 0.01 |
| Test temperature        | Room temperature and low temperatures (−60 °C for STS316L steel, −40 °C for 18 wt% Mn steel) |

Load-displacement curves were obtained for each set of test conditions. When either a fracture or a gas leak developed, the test was terminated. The energy absorbed during the in-situ SP test until fracture could be calculated using the obtained load-displacement curve, and it was defined as the SP energy. If the final fracture did not occur abruptly, the fracture point was defined as the displacement attained when the load dropped 20% from its maximum.

Each recovered specimen was examined using SEM on the side exposed to the high-pressure gas. Fractographic morphologies such as the cracks produced as a result of HE and the surface roughness of the specimen were observed under SEM together with the load-displacement curves.

3. Experimental Results and Discussion
3.1. Qualitative Investigation of Surface Roughness Effect on HE Behaviors

Figure 3a–d show the load-displacement curves and fracture morphologies of STS316L steel specimens, both as-received and polished, after in-situ SP testing under various conditions. Figure 4a–d show the same for 18 wt% Mn steel.
Figure 3. Cont.
Figure 3. Load-displacement curves and fracture morphologies obtained for STS316L steel specimens of two surface finishes, tested under various conditions. (a) Wirecut at RT; (b) Polished at RT; (c) Wirecut at $-60^\circ$C; (d) Polished at $-60^\circ$C.
Figure 4. Cont.
The load-displacement curves obtained for both steels under 10 MPa N$_2$ showed ductile failure regardless of test temperature. The curves are usually composed of five regions: elastic bending, plastic bending, the transition to stretching, plastic membrane stretching, and a final fracture region that includes necking after the applied load reached its maximum [14,28]. Yield load, maximum load, and absorbed SP energy can be determined from the curves [14]. Maximum load was greater during the low temperature test compared to the RT test (due to low-temperature hardening) only for the STS316L steel. In both steels, however, fracture morphologies occurring under 10 MPa N$_2$ showed predominantly large circumferential cracks on the fractured surface, a morphological observation backed by the respective load-displacement curves. They also reached final fracture after remarkable plastic deformation, and the maximum loads and fracture displacements were approximately the same no matter the surface roughness. The effects of surface roughness on the plastic deformations and fractures induced during SP testing under high-pressure
inert gas were minimal. This is consistent with the results reported for 9% Cr steel, wherein surface roughness had a minimal effect on deformation behaviors and fractures [26].

On the other hand, under 10 MPa H₂, punch velocity affected HE differently at each test temperature, but surface roughness remained a minimal influence. The STS316L steel, when tested at RT and 10 MPa H₂, showed HE and a resultant decrease in maximum load and fracture displacement; the load-displacement curve deviated from that obtained under 10 MPa N₂. Fractures occurred with a rapid decrease in load during plastic membrane stretching, indicating a moderate HE effect. However, embrittlement was most noticeable at −60 °C, as seen by others [20]. At this low temperature, punch velocity had a noticeable effect: a drop in maximum load and pronounced fracture displacement occurred regardless of surface roughness. At lower punch velocities, the load began to decrease earlier during plastic bending, and the load-displacement curves slowly deviated from those found with 10 MPa N₂. In these cases, the fracture resulting from HE is defined as the point at which the load drops 20% from its maximum. Eventually, under 10 MPa H₂, the effect of surface roughness was not as pronounced as the effects of other variables. In particular, at −60 °C, lower punch velocities (0.1 mm/min and 0.01 mm/min) created small circular cracks that were partially formed because surface cracks appeared earlier in the latter part of plastic bending.

The 18 wt% Mn steel, tested at RT under 10 MPa H₂, is shown in Figure 4a,b. Fracture displacement was reduced because of HE, and punch velocity was influenced significantly. When the punch velocity was low, the multiple circular cracks that developed during plastic bending region were small, resulting in partial fracture due to HE. At −40 °C, as shown in Figure 4c,d, the lowest punch velocity highlighted brittle fractures early during plastic deformation, indicating significant HE.

Scheme 10 MPa H₂ did not show significant variations related to surface roughness, no matter the test temperature, punch velocity, or type of steel. Whether the surface was as-received or polished, the HE behaviors were very similar, even at low temperatures. This is a great advantage in preparing specimens for the in-situ SP test, and this test remains a simple test method for screening the HE behaviors of structural materials used in hydrogen energy devices. Although friction and surface defects can influence test results, the effect of the specimen surface, by itself, is negligible in the evaluation of HE behaviors of austenitic steels under high-pressure H₂, meaning that surface roughness is not a dominant factor in in-situ SP testing for austenitic steels examined in this study.

3.2. Surface Roughness Effect on Quantitative Evaluation of HE Susceptibility

Our quantitative evaluation of the effect of surface roughness on the HE susceptibility of austenitic steels utilized two ductility-based SP parameters. One was SP energy, the energy absorbed during the deformation and fracture process, derived from the load-displacement curves. Another was a ductility-based factor, the reduction of thickness (ROT), used to assess the HE sensitivities of materials through in-situ SP testing. The initial thickness \( t_0 \) of the specimen and the final thickness \( t_f \) perpendicular to the fractured part of the specimen were measured using a point-micrometer, and ROT was calculated using Equation (1). The relative reduction of thickness (RRT) was then calculated using Equation (2), which compares ROT under high-pressure H₂ to that found under high-pressure inert gas (N₂). It is used as a quantitative measure of HE sensitivity for materials suitable for the in-situ SP test [17,20].

\[
\text{ROT} = (1 - t_f/t_0) \times 100\% \tag{1}
\]

\[
\text{RRT} = \frac{\text{ROT}_{H2}}{\text{ROT}_{N2}} \tag{2}
\]

Both SP energy and RRT, identified from in-situ SP testing, are characterizing factors for quantitatively evaluating the effect of surface roughness on HE susceptibility.

Figure 5 shows the SP energies absorbed to the point of fracture or gas leakage in the in-situ SP tests under various test conditions. The results of each of two tests under a given
set of test conditions are displayed separately so that the effect of surface roughness can be seen. This data display helps to convey the extent of scattering that occurred during in-situ SP testing. Figure 5a,b show that under 10 MPa N₂, STS316L steel represented greater SP energy (6 J) at −60 °C than at RT (3.4~4.0 J), but the difference due to surface roughness was small. Under 10 MPa H₂, the average SP energy at RT was about 1.5 J regardless of punch velocity; this was significantly reduced at −60 °C (to 0.3~1.2 J depending on punch velocity). The values based on surface finish were similar to each other, indicating that SP energy was not affected by surface roughness.

On the other hand, surface roughness played a slight role in 18 wt% Mn steel, as shown in Figure 5c,d. Under RT and 10 MPa N₂, the polished surface showed slightly less SP energy (2.8~3.2 J) compared to the as-received surface (3.5~3.7 J). However, at −40 °C, the values were similar across the two (3.5~4.0 J). Overall, surface roughness rarely affected plastic deformation and fracture behaviors during SP testing under these conditions. Under 10 MPa H₂, however, SP energy at RT ranged from 1.0 to 3.0 J depending on punch velocity,
where lower punch velocities resulted in less SP energy. At \(-40^\circ C\), SP energy was lower than at RT, and the effect of punch velocity seemed more significant. In particular, SP energy was 0.4 J at punch velocities of 0.1 mm/min and 0.01 mm/min. Variations of two tests in 18% Mn steel can be seen when compared to the same two tests of STS316L steel, but overall, surface roughness has insignificant effect on SP energy. Because SP energy represents the deformation energy used until the point of fracture as well as the influence of HE, the effect of surface roughness remains minimal.

Another quantitative characterizing factor, RRT, is compared in Figure 6. The RRTs in STS316L steel shown in Figure 6a, at RT and 10 MPa \(H_2\), ranged from 0.55 to 0.70, no matter the surface roughness or punch velocity. At \(-60^\circ C\) shown in Figure 6b, they were further reduced to 0.15 and to 0.40 depending on punch velocity; however, no distinct difference appeared to result from the surface finish characteristics.

|               | STS316L @ RT | STS316L @ -60°C | 18%Mn @ RT | 18%Mn @ -40°C |
|---------------|--------------|----------------|------------|---------------|
|              | (Wirecut)    | (Polished)     | (Wirecut)  | (Polished)    |
| Punch Velocity|              |                |            |               |
| 0.1 mm/min    | (1)          | (1)            | (1)        | (1)           |
| 0.01 mm/min   | (2)          | (2)            | (2)        | (2)           |
| 0.001 mm/min  |              |                |            |               |

**Figure 6.** Comparisons of RRTs obtained at RT and lower test temperatures at each punch velocity, separated by surface finish for (a,b) STS316L steel and (c,d) 18 wt% Mn steel.

The RRTs in 18 wt% Mn steel at RT and 10 MPa \(H_2\) shown in Figure 6c, showed an effect of punch velocity in that they ranged from 0.45 to 0.95. At \(-40^\circ C\) shown in Figure 6d, they ranged from 0.2 to 0.9, the lowest at a punch velocity of 0.01 mm/min.
The variance in RRT between the two tests was small for STS316L steel and a little larger, in general, for 18 wt% Mn steel, but the variance due to surface roughness was minor. Because RRT describes a loss of ductility due to HE, the effect of surface roughness on HE susceptibility is evidently minimal for these two steels, considering that failure occurred after some amount of plastic deformation due to HE during the in-situ SP tests. Both qualitative and quantitative HE effects induced by surface roughness, when evaluated using in-situ SP testing under 10 MPa H\textsubscript{2}, showed little influence on either STS316L steel or 18 wt% Mn steel, even at temperatures shown to render these steels susceptible to HE. These results are consistent with those of others who applied the SP test and showed that the deformation and fracture behaviors of 9% Cr steel were not affected by specimen surface roughness [26]. This was also confirmed by other results showing that HE behaviors in austenitic stainless steels under high-pressure H\textsubscript{2} were not affected by surface machining [24,25]. Therefore, the effect of surface roughness can be considered negligible compared to other factors of HE revealed by in-situ SP testing, such as punch velocity, gas pressure, and test temperature.

This makes it possible to apply various machining processes in preparing specimens of austenitic steels for in-situ SP testing under high-pressure environments. It was already shown that results obtained using in-situ SP testing are consistent with those found using the usual methods and high-pressure vessels [20]. Variations in HE behaviors with test temperature and punch velocity improve the usefulness of this simple test method. To verify the results of the effect of surface roughness on HE behaviors obtained from austenitic steels for any kind of metal, it is also necessary to examine ferritic and ferritic-pearlitic steels.

4. Conclusions

In this study, the effects of specimen surface roughness on HE behaviors were investigated for the austenitic STS316L and 18 wt% Mn steels by applying an in-situ SP test method under 10 MPa N\textsubscript{2} and H\textsubscript{2}. Results showed that for these two steels, specimen surface roughness rarely affected, or affected to a comparatively negligible degree, HE behaviors when evaluated either qualitatively or quantitatively. With SP energy, the deformation energy remained unchanged until the point of fracture for both wirecutted and polished samples. Similarly, variation in RRT between samples with different surface roughness was very small considering that failure occurred after some amount of plastic deformation due to HE during in-situ SP test. Punch velocity and test temperature were much greater factors affecting HE behaviors. These results improve the usefulness of this in-situ SP test in austenitic steels, and without the need for elaborate specimen preparations, it remains a simple test method for screening HE susceptibility in austenitic steels under high-pressure hydrogen environments.

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