Reliability Assessment of New Factor Proposed for Characterizing Hydrogen Embrittlement Sensitivity of Austenitic Steels using in-situ SP Test

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1. Introduction

In this study, a simple screening technique based on the hydrogen embrittlement (HE) sensitivity of the hydrogen energy materials such as austenitic stainless steels was developed using an in-situ small-punch (SP) test. In order to investigate the susceptibility of HE on hydrogen energy materials, the in-situ SP tests were carried out in the range from room temperature to low temperatures under high pressure hydrogen gas environment. The HE behaviors of austenitic stainless steels were evaluated qualitatively and quantitatively. As an influencing factor of the HE behaviors suitable for the in-situ SP test, a relative ratio of reduction of thickness (RRT) was proposed. The RRT represents the relative reduction ratio of the specimen thickness based on ductility, similar to the RRA obtained by the SSRT test. Regarding on RRT, the measurement reliability of thickness at fracture part is important. At this time, the influence of the punch velocity on the HE sensitivity during in-situ SP testing was also examined. Especially, the HE behavior of the high-Mn steels which will be considered as candidate materials for storage and transportation of the hydrogen gas was evaluated. Consequently, a screening technique for determining the use environmental conditions (pressure, temperature, stress, etc.) of the steels will be established by confirming the effectiveness of the influencing factor RRT obtained by the in-situ SP test method based on the reliability assessment.

2. Experimental Procedures

To perform the in-situ SP test under a high-pressure hydrogen gas atmosphere, three types of steel were used: two austenitic stainless steels of STS304L and SUS316L, and 24.5% Mn steel, all have an FCC (face-centered cubic) structure. Tables 1 delineate the mechanical properties, respectively. Table 2 describes the SP test conditions applied. Variations in HE sensitivity with changes in test temperature and punch velocity were investigated.

Figure 1 shows the schematics of the in-situ SP test apparatus used under a pressurized hydrogen gas environment and at low temperatures. A small-size test specimen (10 × 10 × t0.5 mm) was placed between the upper and lower dies, then a 3mm diameter steel ball (HRC: 60 or greater) was placed on its center. A compressive load was applied to the ball through a punch. To conduct in-situ SP tests at low temperatures, the apparatus used at room temperature [1, 2] was modified to allow vaporizing liquid nitrogen (LN_2) to create the low-temperature test environment. The specimen was fixed between the upper die and lower dies. A pressure gauge was used to monitor gas pressure during charging and testing. A ball valve allows gas to enter into the lower die, while a one-touch connector used to disconnect the SP test kit from the gas charging system when the gas was charged to a specified pressure level. To set the specimen into the SP test kit, an O-ring was inserted into a groove formed into the lower die to prevent gas leakage and maintain gas tightness in the die.

Table 1 - Mechanical properties of each steel .

Properties / Materials	Yield Strength [MPa]	Tensile Strength [MPa]	Elongation [%]
STS304L	304	614	65.0
SUS316L	205	515	60.0
24.5% Mn	393	875	72.1

Table 2 - Test conditions for in-situ SP tests to evaluate hydrogen embrittlement behaviors in austenitic steels.

Specimen dimension [mm]	10 × 10 × t0.5 (±0.005)
Ball diameter [mm]	3.0
Gas environment	N_2 gas , H_2 gas (99.999%)
Gas pressure [MPa]	10, 15
Punch velocity [mm/min]	1.0, 0.1, 0.01
Test temperature [ºC]	Room temperature, 40, -60, -80, -100 and -120

3. Experimental Results

Figure 2 plots typical load-displacement curves obtained by SP tests under 10 MPa N_2 and H_2 gas environment at room temperature, respectively.

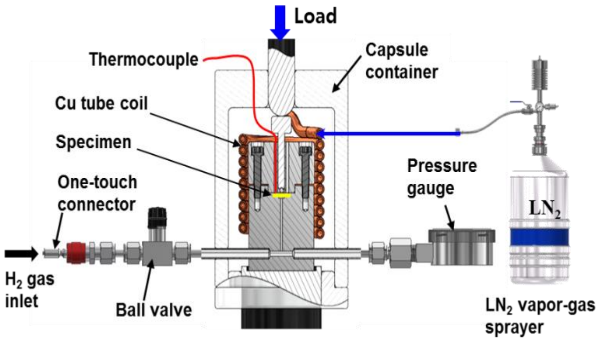

Fig. 1 Schematics of an in-situ SP test apparatus with capsule type insulation container under a high-pressure H_2 gas environment and at low temperatures.

From the load-displacement curve obtained under N_2 gas environment (without showing any embrittlement behavior), certain test parameters, such as yield load, maximum load, and SP absorbed energy, could be obtained [3]. The curve can generally be divided into four or five regions [4], namely: elastic bending (Region I); plastic bending across the entire test specimen (Region II); the transition from bending to membrane behavior (Region III); membrane stretching (Region IV); and fracture (Region V), which includes necking after the peak load until fracture of the specimen occurs. On the other hand, if the HE phenomenon occurred under H_2 gas environment, a decrease in fracture load and corresponding displacement was observed deviating from the curve representing a non-HE behavior under N_2 gas environment. Also, it can be found that the HE typically occurred in the membrane stretching regions of III and IV on the load-displacement curve of Fig. 2.

As shown in Fig. 3, fractures in the inert gas environment remained a significant thinning

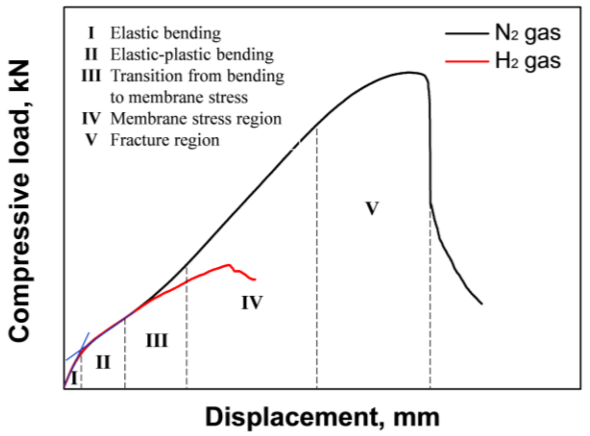

Fig. 2 Typical load-displacement curves obtained from steel specimens under high-pressure N_2 gas and H_2 gas environment at room temperature.

during plastic deformation, whereas in the hydrogen gas environment, brittle fracture occurred without thinning. Therefore, a comparison between the RTs measured at both gas environments can be obtained as a ratio, RRT, which can satisfactorily represent ductility, a characteristic related to HE sensitivity. Therefore, in this study, RT and RRT were adopted as influencing factors representing the HE behaviors of materials when using the in-situ SP test. Once the final thickness was obtained, Eqs. (1) and (2) can be used to calculate RT and RRT, respectively, as follows:

$$RT = (1 - t_f / t_0) \times 100\% \quad (1)$$

$$RRT = RT_{H_2} / RT_{N_2} \quad (2)$$

where $\boldsymbol{t_0}$ is the initial thickness of the specimen before the SP test and $\boldsymbol{t_f}$ is the average thickness measured in the fractured part of the specimen after the SP test.

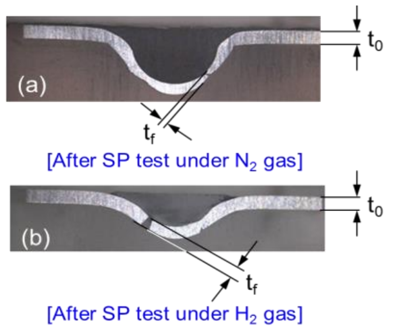

Fig. 3 Cross-sectional views after SP test (a) under N_2 gas and (b) under H_2 gas environments.

4. Summary

This study established a unique hydrogen embrittlement evaluation factor and criteria called RRT suitable for the simple in-situ SP testing. Based on this, it can be provided the basis for establishing safety design standards for hydrogen energy equipment, pressure vessels, and piping materials. International standardization activities are also required.

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[1] CEN Workshop Agreement CWA 15627, (2007), Small Punch Test Method for Metallic Materials - Part B: A Code of Practice for Small Punch Testing for Tensile and Fracture Behaviour, Comite Europeen de Normalization (CEN; European Committee for Standardization)..