INTRODUCTION

When atomic hydrogen diffuses into steel, it causes cracking of the steel by either hydrogen-induced cracking (HIC) or sulfide stress corrosion cracking (SSCC) [1, 2]. Since these failures often lead to serious engineering and economic consequences without warning, much effort has been made towards research on this subject. SSCC is a specific type of corrosion that is especially observed in steels used in the gas and oil industry [3].

There are two forms of cracking, intergranular cracking and transgranular cracking, which are influenced by several environmental and mechanical factors, such as the electrochemical potential, load, deformation rate, and microstructure. Since the SSCC often occurs at high cathodic potential and low strain rate, the SSCC resistance depends not only on the strength level but also the surface conditions, because the total SSCC failure time includes crack initiation and propagation [4, 5]. The crack initiation generally occurs at micro-cracks from pits at grain boundaries, pearlitic colonies, and banded phases in steels [6]. The pit initiation and propagation depend on the crystallographic texture [7, 8]. The intergranular cracking of steels under SSCC conditions is usually observed at low pH and high pH ranges over a restricted range of electrochemical potentials, and can mainly be explained by a film rupture and dissolution model [9]. The passive film on a steel surface involves an intergranular SSCC mechanism, and can be broken by plastic deformation at a crack tip during loading, and halide ions in a corrosive environment [10]. For transgranular cracking in SSCC, much controversy in data has been reported, especially for conditions of near-neutral and moderately low pH. For example, the transgranular SSCC of steels was observed when a great amount of hydrogen diffuses into steels in diluted near-neutral solution, at potentials around open circuit potential values. However, hydrogen-induced cracking is not exclusively involved [11, 12].

There are several models to explain the SSCC behaviors, such as the anodic dissolution model, film rupture model, and electro-mechano model [13–15]. One of the common views in the SSCC models is that the models try to explain the SSCC behaviors by met-

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The Effects of Crystallographic Texture and Hydrogen on Sulfide Stress Corrosion Cracking Behavior of a Steel Using Slow Strain Rate Test Method1

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Received October 7, 2013; in final form, April 8, 2014

Abstract—The effects of pre-charged hydrogen inside steel and the hydrogen ions on its surface on the sulfide stress corrosion cracking (SSCC) behavior was studied by slow strain rate tests. The specimen had an ASTM grain size number of about 11. Most of precipitates were 30–50 nm in size, and their distribution density was about 10^8 mm^-2. The crystallographic texture consisted of major α-fiber (〈110〉//RD) components with a maximum peak at 〈115〉(110) relatively close to 〈001〉(110), and minor γ-fiber (〈111〉//ND) components with a peak slightly shifted from 〈111〉(112) to 〈332〉(113). Hydrogen was pre-charged inside the steel by a high-temperature cathodic hydrogen charging (HTCHC) method. SSCC and corrosion tests were carried out in an electrolytic solution (NaCl : CH₃COOH : H₂O : FeCl₂ = 50 : 5 : 944 : 1, pH = 2.7). The corrosion potentials and the corrosion rates of the specimen without hydrogen charging for 24 hours were –490 mV_SHE and 1.2 × 10^-4 A cm^-2, and those with charging were –520 mV_SHE and 2.8 × 10^-4 A cm^-2, respectively. The corrosion resistance in the solution with 1000 ppm iron chloride added was decreased significantly, such that the corrosion potential and corrosion rate were –575 mV_SHE and 3.5 × 10^-4 A cm^-2, respectively. Lower SSCC resistance of the pin-hole pre-notched specimen was observed at the open circuit potential than at the 100 mV cathodically polarized condition. Pre-charged hydrogen inside of the specimen had a greater influence on the SSCC behavior than hydrogen ions on the surface of the specimen during the slow strain rate test.

Keywords: sulfide stress corrosion cracking (SSCC), slow strain rate test, crystallographic texture

DOI: 10.1134/S0031918X14130031

1 The article is published in the original.
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allurgical variables, like dislocation and precipitates, chemical variables like ions and hydrogen, and mechanical variables like stress distribution and strain rates. Since SSCC is a complicated phenomenon that includes metallurgical, chemical, and mechanical variables, various test methods to evaluate SSCC behavior have been developed for scientific and engineering purposes, such as potentiodynamic tests, constant load tests, and slow strain rate tests [16]. Accordingly, one plausible method to verify the transgranular SSCC mechanism is to perform SSCC tests in moderate acid solution with halide ions at a free corrosion potential, using a slow strain rate test method. Hence, the objectives of this study are to systematically analyze the effect of crystallographic texture and hydrogen on the SSCC behavior of steels for verification of the SSCC mechanism, and to obtain applicable industrial information about pre-notched specimens.

EXPERIMENTAL METHOD

1. Sample Preparation

The specimens used in this study were steels supplied by POSCO, Korea. Table 1 shows the composition of the specimens. Two types of specimens were prepared in this study: smooth and pin-hole pre-notched specimens. Pin-hole-shape notches were prepared around a cylindrical specimen to study the effect of hydrogen concentration on the SSCC tip by controlling the output beam diameter of an Nd:YAG pulse laser to about 0.01 mm (JSC Plasma, UK).

Figure 1 shows a schematic of the specimen, which was machined in its longitudinal direction to be perpendicular to the rolling direction of the as-received thick steel plate. The machined specimens were heat treated at 1233 K for 900 sec and then tempered at 873 K for 1800 sec followed by oil quenching to room temperature. Figure 2 shows the thermal history of the specimen. Hydrogen was introduced by high-temperature cathodic hydrogen charging method (HTCHC), the amount of which was determined using a wet method [17].

2. Characterization

The microstructure was observed by optical microscopy (Nikon 200, USA) after polishing and etching with nital solution (HNO₃ : C₂H₅OH = 1 : 9). A failed surface was observed by scanning electron microscopy (Jeol JSM 6400, Japan). Precipitates of the specimen were observed by transmission electron microscopy (Jeol JTM 2010-F, Japan). The TEM sample was prepared by the twin-jet polishing method (Struers, TenuPol-5, USA) at the condition of 5% HClO₄ + 95% CH₃OH, 233 K and 25 V. Chemical analysis was carried out by energy dispersive spectroscopy (EDS, Oxford, UK).

The crystallographic texture was determined by X-ray diffractometry (Bruker D8 Discover, Germany) and EBSD (electron back-scattered diffraction, TSL, USA), respectively. The specimen used for texture analysis was prepared into longitudinally sectioned pieces 15 mm in length and 10 mm in width. The pieces were cold-mounted and polished using 0.3 μm diamond paste. The specimen for EBSD was finally subjected to vibratory polishing (vibratory polisher, Buehler, Vibromet-2, USA) to remove the residual

Table 1. Chemical composition of a steel [wt %]

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.066</td>
<td>0.1</td>
<td>1.31</td>
<td>0.016</td>
<td>0.0026</td>
<td>0.049</td>
</tr>
<tr>
<td>Nb</td>
<td>V</td>
<td>Ca</td>
<td>Na</td>
<td>N</td>
<td>Fe</td>
</tr>
<tr>
<td>0.041</td>
<td>0.043</td>
<td>0.0078</td>
<td>0.0077</td>
<td>0.05</td>
<td>bal.</td>
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