



## Hydrogen-assisted quasi-cleavage fracture in a single crystalline type 316 austenitic stainless steel

Motomichi Koyama<sup>a,\*</sup>, Eiji Akiyama<sup>a</sup>, Takahiro Sawaguchi<sup>a</sup>, Kazuyuki Ogawa<sup>a</sup>, Irina V. Kireeva<sup>b</sup>, Yury I. Chumlyakov<sup>b</sup>, Kaneaki Tsuzaki<sup>a,c</sup>

<sup>a</sup> National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan

<sup>b</sup> Siberian Physicotechnical Institute, Tomsk State University, Tomsk 634050, Russia

<sup>c</sup> Department of Mechanical Engineering, Kyushu University, 744 Motoooka, Nishi-ku, Fukuoka 819-0395, Japan

### ARTICLE INFO

#### Article history:

Received 21 March 2013

Accepted 12 June 2013

Available online 22 June 2013

#### Keywords:

A. Stainless steel

B. Galvanostatic

B. SEM

B. TEM

C. Hydrogen embrittlement

### ABSTRACT

Hydrogen embrittlement properties were examined in a single crystalline type 316 austenitic stainless steel. Tensile tests were conducted along the  $\langle 111 \rangle$  and  $\langle 001 \rangle$  directions under hydrogen charging. Hydrogen-assisted  $\{111\}$  quasi-cleavage fracture was observed in both tensile orientations. The degradation of fracture stress and elongation, hydrogen uptake, and hydrogen-induced fracture surface were dependent on the tensile orientation. The tensile orientation dependence of the hydrogen embrittlement properties was shown to result from the deformation twinning behavior. In addition, Ag decoration technique clarified that hydrogen localizes on regions where hydrogen-assisted quasi-cleavage fracture appeared.

© 2013 Elsevier Ltd. All rights reserved.

### 1. Introduction

Hydrogen is introduced by a high hydrogen gas pressure and cathodic electrochemical reactions including corrosion, causing embrittlement in tempered martensitic steels, austenitic stainless steels, etc. [1,2]. For example, austenitic stainless steels are used as reactor materials with boiling water [3,4]. The corrosive atmosphere promotes a delayed fracture that is known as a practical disadvantage of the austenitic stainless steels [3,4]. Therefore, the hydrogen embrittlement and the corrosion-related cracking of austenitic stainless steels have been noted to improve the delayed fracture properties [5–7]. Type 316 austenitic stainless steels that is widely used under corrosive atmospheres also have been known to show hydrogen-assisted stress corrosion cracking [8] and hydrogen embrittlement caused by cathodic hydrogen charging [9,10]. Understanding factors affecting the hydrogen embrittlement is undoubtedly-beneficial to develop advanced austenitic steels.

In the case of hydrogen embrittlement of austenitic steels, deformation twinning has been reported to be an important cause of the hydrogen-induced quasi-cleavage fracture observed [11–14]. The deformation twins can cause stress concentration at the tips of the twin plates [15], and the twin boundaries can act as a crack propagation path [11,12–18]. Based on the combination of

these effects, the twin boundary cracking is promoted at the interception of deformation twinning at the obstacle twin boundaries [15,18]. Since the twinning plane of face-centered cubic structure is  $\{111\}$ , the twin boundary cracking results in  $\{111\}$  quasi-cleavage fracture. However, the contribution of deformation twinning to hydrogen embrittlement is still unclear in austenitic steels. For instance, influences of deformation twin morphology and crystallographic orientation relationship between twinning and tensile directions on hydrogen embrittlement properties have not been clarified yet.

The type 316 stainless steel is a typical austenitic steel that shows deformation twinning and hydrogen embrittlement [9,10]. Assuming that deformation twinning is an important factor for the hydrogen embrittlement, the embrittlement property would depend on the crystallographic orientation due to the twinning behavior since deformation twinning occurs frequently in the  $\langle 111 \rangle$  but not in the  $\langle 001 \rangle$  tensile orientations [19]. From the viewpoint of crystallographic orientation dependence, tensile tests using single crystalline specimens with and without hydrogen charging are helpful for analyzing the correlation between the deformation twinning behavior and the embrittlement property. There have not been any reports on the correlation among stress–strain response, twinning behavior, and hydrogen embrittlement, although hydrogen embrittlement in single crystalline austenitic stainless steels has been discussed in terms of the hydrogen effects on strain localization [20], surface cracking [21], cleavage fracture stress [22], and the change in hardness [23].

\* Corresponding author. Tel.: +49 211 6792 885; fax: +49 211 6792 333.

E-mail address: [m.koyama@mpie.de](mailto:m.koyama@mpie.de) (M. Koyama).

This work aims at correlating the crystallographic orientation dependence of the twinning behavior with the hydrogen embrittlement properties based on stress–strain response, hydrogen uptake, and fracture mode using single crystals. We focus on the crack propagation path, not the crack initiation site, since the typical crack initiation site of the type 316 austenitic stainless steels is known to be the  $\alpha'$ -martensite at the intersections of deformation twins [24] and place an emphasis on the following.

1. Usage of single crystals instead of polycrystals. The disappearance of grain boundary provides a clearer quasi-cleavage fracture and a more homogeneous deformation microstructure. These observations are useful for correlating the morphology of deformation twins with the crack propagation path. Note that the dominant fracture mode of hydrogen-assisted failure and hydrogen effects on stress–strain responses are different from those in polycrystals. Since the dominant fracture mode of a hydrogen-affected type 316 stainless steel is intergranular [9], we will also discuss the correlations between the present results of the single crystals and conventional results about various austenitic steels including polycrystals.

2. Multi-scale microstructure characterizations to analyze the deformation twins. We conducted secondary electron (SE) imaging for fractography, electron channeling contrast (ECC) imaging for observing the morphology of deformation twins in a wide view field, electron backscatter diffraction (EBSD) analysis for verifying the existence of deformation twins with multi-twinning direction, and transmission electron microscopy (TEM) for observing the dislocation substructures and the fine structures of deformation twins.

## 2. Experimental

### 2.1. Tensile test under hydrogen charging

A single crystalline type 316 stainless steel (Fe–16.8Cr–10.4Ni–2.1Mo–1.4Mn–0.10C–( $N < 0.05$ ) in wt.%) was prepared by the Bridgman method. The single crystal growth control was performed at 1823 K under an inert gas atmosphere. The Cr, Ni, Mo, and Mn concentrations were measured by X-ray fluorescence analyses. The amounts of carbon and nitrogen were determined by infrared absorption method after combustion and inert gas fusion-thermal conductivity method, respectively. Note that the carbon concentration is higher than that of standard type 316 stainless steels. Tensile specimens with a gauge dimension of  $4.0 \text{ mm}^w \times 0.3 \text{ mm}^t \times 5.0 \text{ mm}^l$  and grip sections on both ends were cut by spark erosion. Then, their thickness was reduced further to 0.3 mm by mechanical grinding to remove the layers affected by the spark erosion. The tensile tests were conducted at ambient temperature around 294 K with an initial strain rate of  $1.7 \times 10^{-5} \text{ s}^{-1}$  along the  $\langle 111 \rangle$  and  $\langle 001 \rangle$  directions using an Instron type machine. The strains were determined by dividing the displacements by the initial gauge length. The low strain rate was chosen to increase a hydrogen-affected zone through an increase in hydrogen charging time as well as hydrogen-decorated dislocation motion during the tensile test. The initial strain rate of  $1.7 \times 10^{-5} \text{ s}^{-1}$  is lower than that in conventional works which showed clear hydrogen embrittlement in a polycrystalline type 316 austenitic steel [9].

Hydrogen was introduced into the specimens during the tensile tests by electrochemical charging in a 3% NaCl aqueous solution containing 3 g/L of  $\text{NH}_4\text{SCN}$  at a cathodic current density of  $10 \text{ A/m}^2$ . The specimen was contained in a plastic container with the solution. A platinum wire with a diameter of 1 mm was used as the counter electrode. The solution was added continuously to cover the part of the gauge during the tensile tests. The experimental

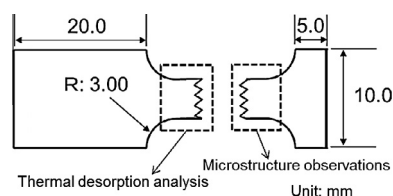
setup was shown schematically in our previous work [25]. The length and the surface area of the Pt wire in the solution were approximately 200 mm and  $600 \text{ mm}^2$ , respectively (namely the anodic current density was about  $0.8 \text{ A/m}^2$ ). Since the specimen and the Pt wire were immersed into the solution in the cell, Pt dissolution and subsequent Pt re-deposition on the specimen might occur during the hydrogen charging. Although the Pt deposition can affect hydrogen charging behavior, the effect was not considered in the present study. Tensile testing and hydrogen charging were started simultaneously, and the hydrogen charging was stopped when the specimen was fractured. Namely, hydrogen charging time is determined by fracture strain divided by the strain rate. Hydrogen charging during tensile testing introduced hydrogen effectively into the samples via diffusion as well as through the motion of hydrogen-decorated dislocations [26–28]. The fractured specimens were used for hydrogen detections and microstructure observations. A half of the fractured specimen was used for the measurement of the hydrogen content, and the other part of the specimen was used for the microstructure observations, schematically described in Fig. 1.

### 2.2. Measurement of hydrogen content

The hydrogen uptake was measured by thermal desorption analysis (TDA) from room temperature to 800 K using mass spectrometry. The TDA was started within 20 min. after the tensile test. The heating rate was 200 K/h. A standard leak gas system of hydrogen using crimper capillary whose hydrogen gas leak rate is was  $5.4 \times 10^{-11} \text{ mol/s}$  was used for calibration. The calibration was done before each hydrogen desorption measurement. The diffusible hydrogen content, which is defined as the amount of hydrogen that diffuses at room temperature, was determined by measuring the cumulative desorbed hydrogen content from room temperature to 450 K, since the peak existing around 350 K in an austenitic steel disappears after exposure under air for 10 days at room temperature [28]. The activation energy for desorption of hydrogen of the peak around 350 K in an austenitic steel was reported to be about 35 kJ/mol. This fact also indicates that the cumulative desorbed hydrogen content around 350 K corresponds to diffusible hydrogen content. The diffusible hydrogen is reported to play a key role in hydrogen embrittlement [29]. We also obtained the desorbed hydrogen content from 450 to 650 K. This was reported to be the hydrogen trapped at the twin boundaries in the austenitic steel [30].

### 2.3. Microstructure observations

Fractographs were obtained by SE imaging at 20 kV. EBSD analysis and ECC imaging were also used for observing the deformation twins with a scanning electron microscope. A beam step of 50 nm was used for the EBSD analysis. The ECCI was operated to reveal the deformation twins of austenitic steels in a wide view field [11,31–33]. The accelerating voltages for the EBSD analysis and ECC imaging were 20 and 15 kV, respectively. The specimens for



**Fig. 1.** A schematic of the fractured specimens used for the tests. The width, thickness, and length of the gauge were 4.0, 0.3, and 5.0 mm, respectively, before the deformation.

Download English Version:

<https://daneshyari.com/en/article/7896428>

Download Persian Version:

<https://daneshyari.com/article/7896428>

[Daneshyari.com](https://daneshyari.com)