



Full Length Research Paper

# Crystallographic study of hydrogen-induced twin boundary separation in type 304 stainless steel under cyclic loading



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## ARTICLE INFO

## Keywords:

- A. Stainless steel
- B. SEM
- B. TEM
- C. Hydrogen embrittlement
- C. Interfaces
- C. Corrosion fatigue

## ABSTRACT

This study focused on the martensitic transformation during fatigue crack growth in twinned crystals using small compact-tension specimens to elucidate the hydrogen-induced twin boundary separation in type 304 stainless steel. In the uncharged specimen, martensite variants were formed with their habit planes parallel to the most highly shear-stressed slip plane. The crack extended in martensite that was earlier formed ahead of the crack tip and deflected from the twin boundary. Hydrogen-induced twin boundary separation occurred predominantly at the interface between martensite and austenite in a medium stress intensity range. As the stress intensity range increased, martensite variants symmetrically arranged with respect to the twin plane dominated, suggesting that a slip-off crack growth mechanism was facilitated by hydrogen.

## 1. Introduction

Stainless steels used in the chemical, marine, and energy industries often suffer from stress corrosion cracking (SCC) and corrosion fatigue (CF) in harsh environmental conditions. Many mechanisms have been proposed for SCC and CF; these can be classified into two categories, *i.e.*, dissolution-based mechanisms under anodic conditions and mechanisms based on the adsorption of environmental species generated from cathodic reactions [1,2]. In particular, the hydrogen-assisted cracking (HAC) mechanism [3–5] (included in the latter category) is widely accepted for austenitic stainless steels. Metastable austenitic stainless steels, where the  $\alpha'$  martensitic transformation occurs during deformation, exhibit severe hydrogen embrittlement [6–9] and significantly increased crack growth rates due to interstitially dissolved hydrogen [10–12]. Quasi-cleavages and flat facets are most prevalent in SCC and CF of metastable austenitic steels [12–14]. A micro-tensile test study of hydrogen-charged type 304 austenitic steel revealed that the excess hydrogen generated by the martensitic transformation diffuses to the surrounding austenite, leading to quasi-cleavage fracture along the habit plane between martensite and austenite [15]. Further, a finite element analysis study of hydrogen diffusion around a crack tip demonstrated that martensitic transformation drastically increases the local hydrogen concentration in the retained austenite, consequently causing cracking therein [16]. On the other hand, the occurrence of flat facets can be related to twin boundaries [17–19]. Tensile tests of Fe–(10–20)Ni–17Cr–2Mo alloys (in mass%) in 1 MPa hydrogen gas revealed [18] that the flat facets related to twin boundaries were

mainly observed in the 10Ni alloy and they were decreased with increasing Ni content. San Marchi et al. [19] performed tensile tests of metastable austenitic steels in 40 MPa hydrogen gas, and observed that the occurrence of flat facets tends to increase in 300-series stainless steels that have lower equivalent nickel contents. The tendency toward the deformation-induced martensitic transformation also increases with decreasing nickel content. Meanwhile, Alyousif and Nishimura studied SCC of 304, 316 and 310 austenitic stainless steels in HCl solutions and boiling saturated magnesium chloride solutions [20–22] and proposed the mechanism for intergranular hydrogen embrittlement associated with the formation of  $\alpha'$  martensite [22]. Further, in sensitised austenitic stainless steels, it was reported that intergranular hydrogen embrittlement [23] and hydrogen-enhanced crack growth [24] were not attributed to the precipitation of carbides, but to the deformation-induced martensite formation along the grain boundaries. Therefore, it is also hypothesised that the martensitic transformation during deformation would be a crucial factor in the twin-boundary-mediated HAC of metastable austenitic stainless steels. The present study focussed on the martensitic transformation at a crack tip to examine the hydrogen-induced twin boundary separation in metastable austenitic steel. To readily observe stable crack growth at the twin boundary, fatigue tests were performed using a set of small twinned specimens of type 304 steel with and without hydrogen pre-charging in combination with post-fatigue-test metallographic characterisation.

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**Table 1**  
Chemical compositions of steels used in this study.

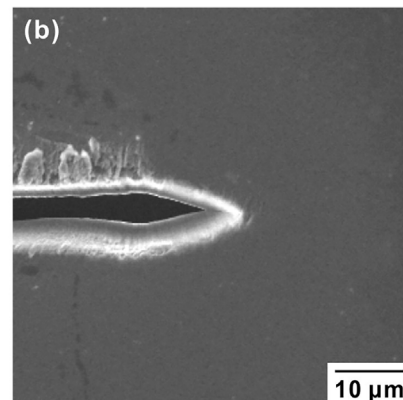
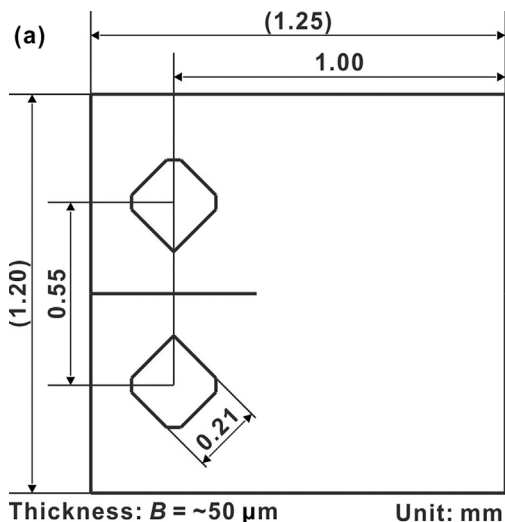
	(mass%)						
	C	Si	Mn	P	S	Ni	Cr
JIS-SUS304	0.05	0.58	1.24	0.025	0.003	8.09	18.54
Fe–0.24 C	0.24	< 0.01	< 0.01	< 0.003	0.0004	–	–

## 2. Materials and methods

The material used in this study was a commercial type of 304 (JIS-SUS304) austenitic stainless steel. For comparison, an Fe–0.24 mass% C martensitic steel was also used. Table 1 shows the chemical compositions of the steels. Disc-shaped samples were prepared with a diameter of 19 mm and an approximate thickness of 0.5 mm. Coarse-grained samples with an average grain size of  $\sim 80 \mu\text{m}$  were obtained through heat treatment at 1403 K for 4 h followed by air cooling. These samples were thinned to  $\sim 50 \mu\text{m}$  by grinding with emery paper. The sample surface was electrochemically polished at a voltage of 40 V in an electrolyte of  $\text{HClO}_4\text{:C}_6\text{H}_{14}\text{O}_2\text{:C}_5\text{H}_5\text{OH:H}_2\text{O}$  (1:1:7:1 in volume fraction) at room temperature for electron backscatter diffraction (EBSD) analysis. The crystal orientation was determined using a scanning electron microscope (SEM) equipped with an EBSD analyser and the TexSEM Laboratories software OIM v.7.1.0. Small compact-tension (CT) specimens 50  $\mu\text{m}$  thick and 1 mm wide were fabricated using a laser beam, as shown in Fig. 1. A through-thickness notch was introduced on a twinned crystal. The tip of the notch was trimmed using a focused ion beam (FIB) (Fig. 1b). The notch plane and direction were arranged roughly parallel to (111) and  $[1\bar{1}\bar{2}]$ , respectively, based on EBSD analysis.

Fatigue crack growth tests were performed at room temperature in laboratory air using a piezoelectric actuator with a position accuracy of 30 nm. Constant fatigue cycling was imposed at a load ratio of 0.1 and a cyclic frequency of 1 Hz with load feedback control. The tests were interrupted after predetermined numbers of loading cycles so that the crack growth increment for each duration is several micrometres, and the crack length was monitored using a SIGMA KOKI microscope with an autofocus white-light interferometer. The stress intensity factor range,  $\Delta K$ , for the ASTM standard CT specimen is given by the following equations:

$$\Delta K = \frac{\Delta P}{B\sqrt{W}} F\left(\frac{a}{W}\right), \quad (1)$$



**Fig. 1.** (a) Shape and dimensions of the small CT specimen and (b) SEM micrograph of crack tip fabricated by FIB.

$$F\left(\frac{a}{W}\right) = \frac{2 + \frac{a}{W}}{\left(1 - \frac{a}{W}\right)^{2/3}} \left[ 0.886 + 4.64\left(\frac{a}{W}\right) - 13.32\left(\frac{a}{W}\right)^2 + 14.72\left(\frac{a}{W}\right)^3 - 5.6\left(\frac{a}{W}\right)^4 \right], \quad (2)$$

where  $\Delta P$  is the applied load range;  $B$  is the thickness;  $W$  is the width;  $a$  is the crack length. In this study, the specimen sizes do not satisfy the ASTM standard. Therefore, the stress intensity factor range values are denoted as  $\Delta K_Q$ . For the uncharged specimen, the fatigue test was performed at a constant load range of 1.0 N. To reduce the hydrogen release during the fatigue test in the hydrogen-charged specimen, the load range was increased by 10% after each duration until the nominal crack growth rate exceeded  $\sim 2.5 \times 10^{-6} \text{ mm cycle}^{-1}$ , i.e., the fatigue test was performed at a load range of 1.6 N for  $\Delta K_Q > 5.8 \text{ MPa}\sqrt{\text{m}}$ . To measure the surface relief due to the martensitic transformation, scanning white-light interferometry was employed with a vertical resolution of 0.1 nm and a horizontal resolution of 0.148  $\mu\text{m}$ . After pre-cracking, one specimen was cathodically charged with hydrogen in an aqueous solution of sulfuric acid ( $\text{pH} = 3.5$ ) held at a temperature of 353 K and a current density of  $27 \text{ A m}^{-2}$ . The sulfuric acid aqueous solution was prepared using analytical grade 95%  $\text{H}_2\text{SO}_4$  ( $\text{Cl} \leq 1 \text{ ppm}$ ,  $\text{Pb} \leq 5 \text{ ppm}$ ,  $\text{As} \leq 0.1 \text{ ppm}$ ,  $\text{Se} \leq 1 \text{ ppm}$  and  $\text{Fe} \leq 3 \text{ ppm}$ ) and ion-exchanged water. Based on an estimate of the diffusion velocity [25], the charging time was chosen so that the 50- $\mu\text{m}$ -thick specimen was saturated with hydrogen. The saturated hydrogen content under corresponding hydrogen charging conditions was measured to be  $\sim 100 \text{ mass ppm}$  by thermal desorption spectrometry [15]. The fatigue crack growth test for the hydrogen-charged specimen was started in 0.5 h after hydrogen charging and was completed in 8 h. Based on an estimate of the hydrogen concentration distribution on the assumption that the hydrogen-saturated specimen with 50  $\mu\text{m}$  in thickness is left for 8 h, it can be found that hydrogen releases from the region within  $\sim 6 \mu\text{m}$  beneath the surface. Therefore, the effect of hydrogen release during the fatigue test on the mechanical response would not be significant.

Post-fatigue-test examinations were conducted using EBSD and transmission electron microscopy (TEM). Samples for TEM were milled perpendicular to the fatigue surface using FIB. The sample surface was chosen considering the Kurdjumov–Sachs (K–S) orientation relationship between austenite and martensite. The microstructure developed by the fatigue process in the uncharged specimen was examined using an FEI Tecnai F20 system operated at an accelerating voltage of 200 kV. The flat fracture surfaces formed in the hydrogen-charged specimen were examined by automatic beam scanning with a step size of 0.08  $\mu\text{m}$  at an

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