Microscopic Study on the Effect of Hydrogen on Deformation Process near Stage II Fatigue Crack Tip

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1. Introduction

The deleterious effects of hydrogen on the mechanical properties of metallic materials (e.g. decrease of break elongation or fracture toughness, increase of sub-critical crack growth rate) are generally termed hydrogen embrittlement (HE). Among these effects, a particular attention should be paid to the enhancement of fatigue crack growth rate when a structural body containing a crack is operated under a cyclic load. The fatigue crack growth property is known to be strongly dependent on the cyclic slip behavior near the crack tip region [1]. Therefore, it is important to elucidate the effect of hydrogen on cyclic slip deformation at the near-tip region.

A number of studies have adopted polycrystalline metals to investigate the effect of hydrogen on the fatigue crack growth property. The crack tip slip behavior in a polycrystalline metal is, however, constrained by the grain boundary or sensitive to the crystallographic orientation of the grain [2]. In addition, the grain boundary could become the primary crack path due to the damage caused by hydrogen [3]. Hence a single-crystalline metal is suited for the purpose of evaluating the hydrogen effect on the crack tip slip behavior. There have been reports on the hydrogen effects on the sub-critical crack growth using single-crystalline metals with body-centered cubic (BCC) structure [2,4-6]. These studies focus on the crack growth under sustained load condition. On the other hand, there have been few attempts to use single-crystalline metal for studying the hydrogen effects on the crack tip slip under cyclic loading.

In this study, a fatigue crack growth test using a single-crystalline metal is implemented both in hydrogen and non-hydrogen (helium) atmosphere. The effect of hydrogen on the crack tip slip behavior is investigated in detail not only by the fractographic approach but also by the cross-sectional observation of the crack tip region by transmission electron microscopy (TEM).

2. Experiment method

The material used for this study is a single-crystalline Fe-3.2mass%Si alloy. Fig. 1 schematically shows the crystallographic orientation and the geometry of the specimen. The specimen is uniaxially loaded in $[1\bar{1}0]$ direction, and the macroscopic fatigue crack surface is introduced parallel to (110). The choice of this orientation intends to avoid the cracking along {100} face which is the

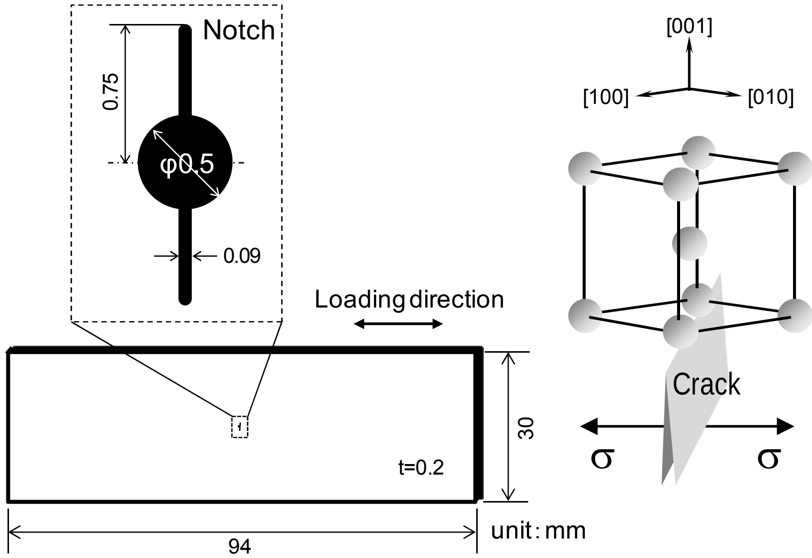

Fig. 1 Geometry and crystallographic orientation of specimen.

normal cleavage plane of this material. Thus, the effect of hydrogen on crack tip slip is expected to be more pronounced. The specimen is cut into rectangular shape, and a through-notch is machined at the center position.

The fatigue crack propagation tests are conducted using a servo-hydraulic testing machine equipped with a gas chamber. In order to clarify the effect of gaseous hydrogen on the crack growth property, a reference test is also performed in inert helium (He) atmosphere. The cyclic loading is conducted by nominal stress control (σ_{max} = 280 MPa, R = 0.05, 1 Hz sinusoidal), and the crack length is optically monitored by a microscope through a window of the chamber. The gauge pressure of each gas atmosphere is kept constant at 0.58 MPa.

The tests are conducted as follows. First, the relationship between applied stress intensity factor range (ΔK) and crack growth rate (da/dN) is preliminarily evaluated both in helium and hydrogen atmosphere. ΔK value is evaluated by the following equation.

$$\Delta K = \Delta\sigma\sqrt{\pi a \sec\frac{\pi a}{2W}} \quad (1)$$

Here, $\Delta\sigma$ is the applied stress range, a is the crack half-length projected to [001] axis, W is the specimen half-width. On the basis of the preliminarily evaluation, the crack growth is next interrupted at a certain ΔK value (20 $\mathrm{MPam}^{1/2}$ in this study) that yields different da/dN in helium and hydrogen atmosphere. This allows a direct comparison of the different crack tip slip behavior at the same mechanical condition.

After the test, the crack surface and the crack tip region are observed in detail by scanning electron microscopy (SEM) and TEM respectively. For TEM observation, the interrupted crack tip region is carefully cut from the specimen and thinned by a special method that combines ion milling technique and focused ion beam (FIB) technique [7]. The TEM observation is conducted by JEM-1300NEF facility at HVEM Laboratory in Kyushu University, Japan. An ultra-high voltage acceleration of 1250 kV, which enables stable observation of magnetic materials, is employed.

3. Results and discussion

Fig. 2 shows the da/dN-ΔK relationships (stage IIb region) in helium and hydrogen atmosphere. The crack growth is significantly facilitated by hydrogen atmosphere (Specimen B). The growth rate is approximately four times larger for Specimen B than for Specimen A at ΔK = 20 (MPam$^{1/2}$), and the ratio of growth rate becomes larger at elevated ΔK region.

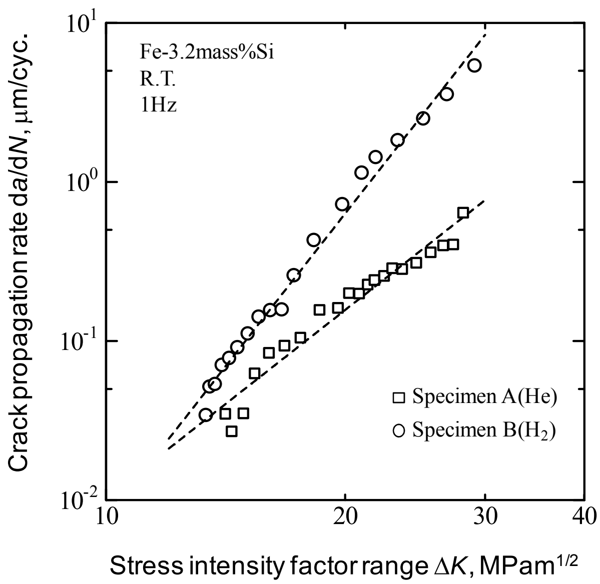

Fig. 2 Relationship between stress intensity factor range and crack propagation rate.

Fig. 3 shows the fatigue crack surface of Specimen A observed by SEM. The image is taken at the position where the crack is driven by ΔK value of 20 MPam$^{1/2}$. Striation pattern almost parallel to [110] direction is clearly seen. The average period of striation is 0.25 μm, which is close to the measured da/dN value in Fig. 2. A cross-sectional TEM image of the crack tip interrupted at the same ΔK value (≈ 20 MPam$^{1/2}$) is shown in Fig. 4. Dense slip bands are formed along $(1\bar{1}2)[\bar{1}11]$ system and reach as far as about 30 μm from the crack. The distinct boundary between slipped/unslipped areas denoted by the dotted line indicates that the slip bands are emitted from the crack tip. The magnified view of the crack periphery reveals that the crack wake is wavy in shape, which corresponds to the

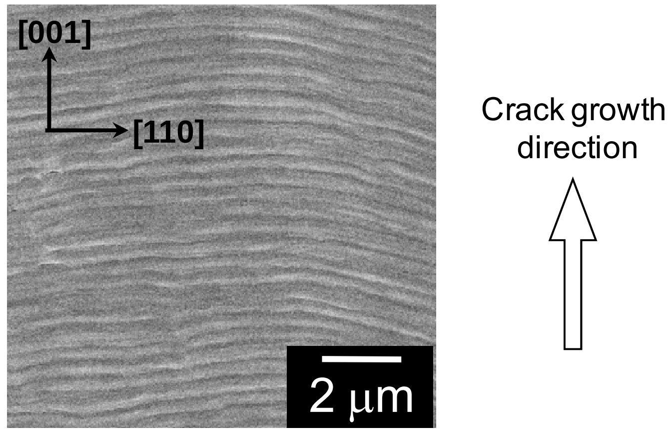

Fig. 3 SEM image of crack surface (Specimen A, ΔK = 20 MPam$^{1/2}$, in He)

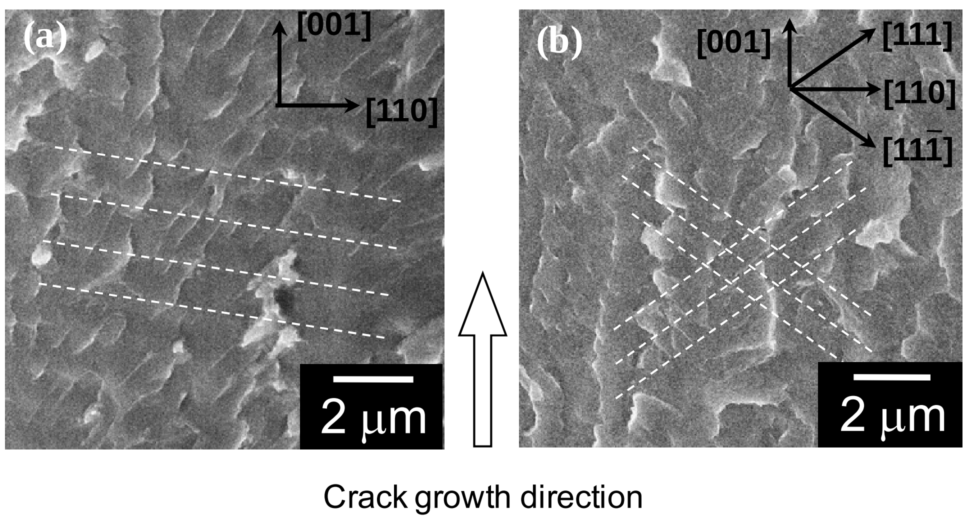

Fig. 5 SEM image of crack surface (Specimen B, ΔK = 20 MPam$^{1/2}$, in H_2)

striation pattern on the crack surface shown in Fig. 3. These observations suggests that the crack tip slip in helium atmosphere is mainly characterized by the activation of two symmetrical slip systems about the crack face (i.e. $(1\bar{1}\bar{2})[1\bar{1}1]/(\bar{1}12)[\bar{1}11]$ pair, which is denoted as 'type A' hereafter). In other words, the crack tip advances as the result of cyclic blunting and re-sharpening known as the 'slip-off' mechanism [1] in helium atmosphere.

In hydrogen atmosphere, on the other hand, the slip behavior is quite different from that in helium atmosphere. Fig. 5 shows the SEM fractography of Specimen B (ΔK = 20 MPam$^{1/2}$). Two types of striation patterns outlined by tear-ridges are found; the one parallel to [110] direction (Fig. 5(a)) and the one parallel to

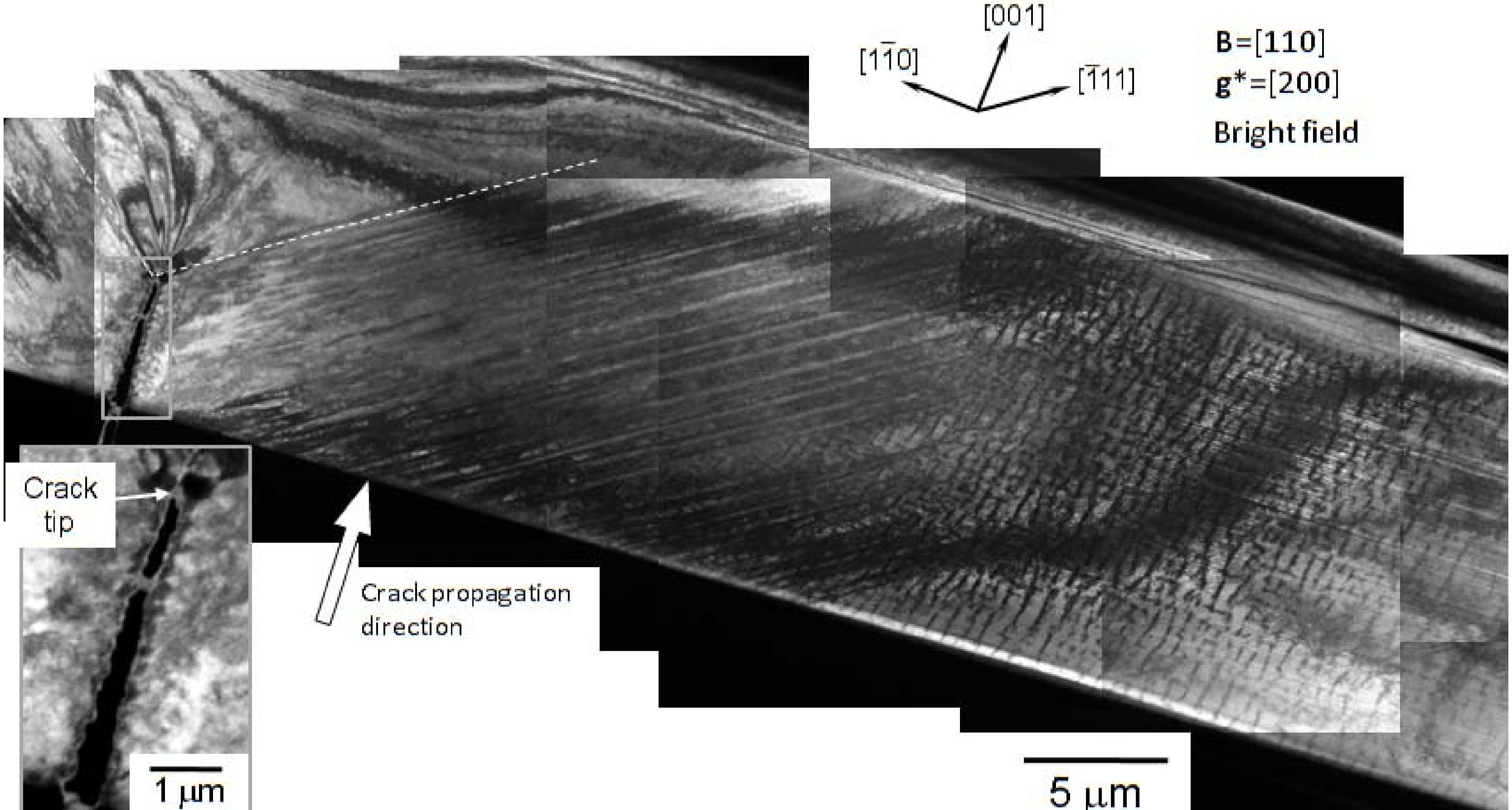

Fig. 4 Cross-sectional TEM image of near-tip region ($\Delta K \approx 20$ MPam$^{1/2}$, in He).

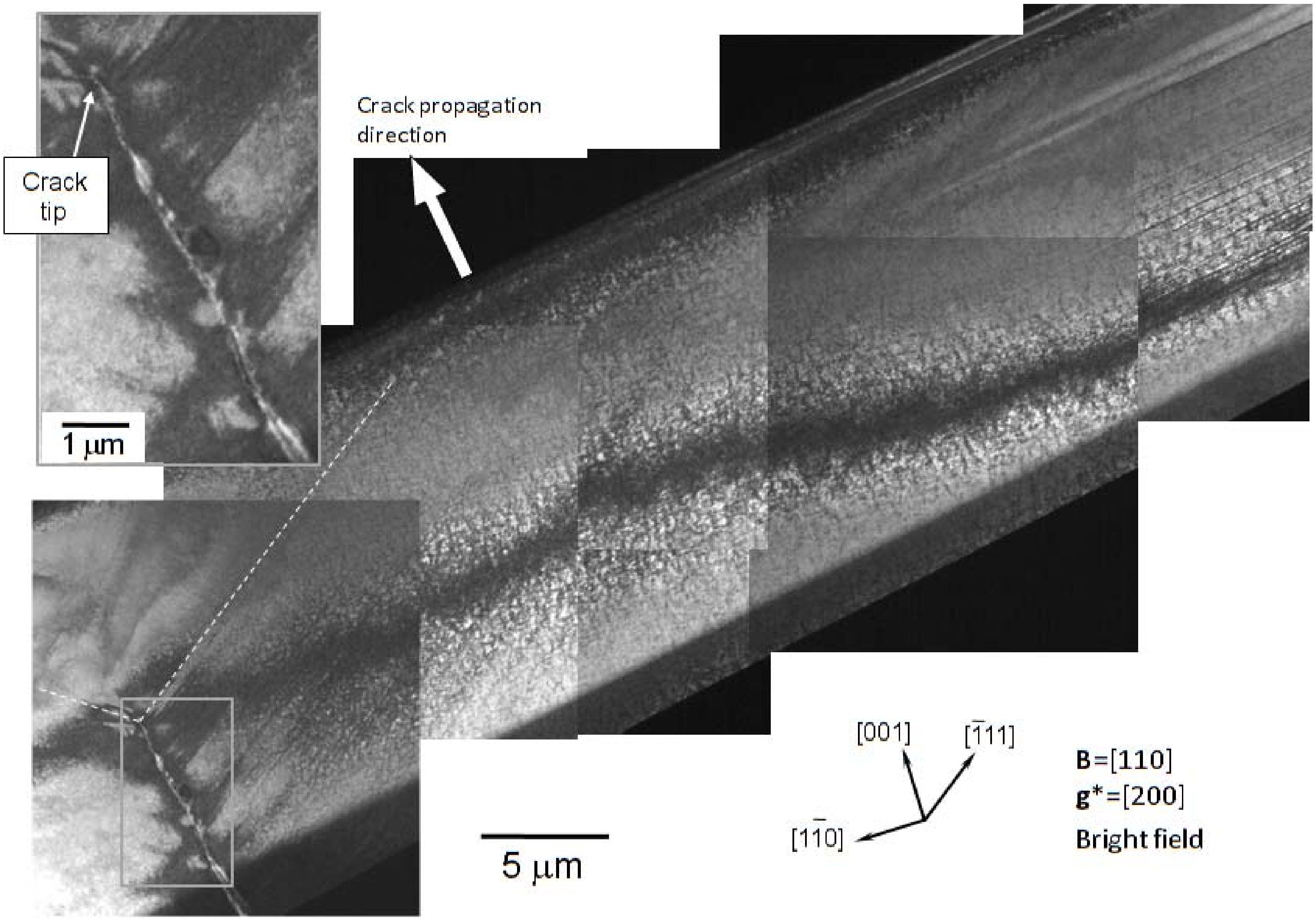

Fig. 6 Cross-sectional TEM image of near-tip region ($\Delta K \approx 20$ MPam$^{1/2}$, in H_2).

$[111]/[11\bar{1}]$ directions (Fig. 5(b)). Although both types are observed on the crack surface, the later one is more pronounced at the center thickness of the specimen. The period of each pattern is close to the measured da/dN value in Fig. 2. The cross-sectional TEM image of the interrupted crack tip is shown in Fig. 6. Here, the slip trace along $[\bar{1}11]$ direction can also be seen although it is not as intense as that observed in Fig. 4. It can be postulated from these observations that, in hydrogen atmosphere, other types of slip systems ($(011)[1\bar{1}1]/(101)[\bar{1}11]$, $(\bar{1}01)[1\bar{1}1]/(0\bar{1}1)[\bar{1}11]$ which are denoted as 'type B' hereafter) are also activated in addition to type A slip. The magnified view of the crack shows that the heavily dislocated region is concentrated to the close vicinity of the crack, and the crack opening displacement is significantly small. The overall appearance of the crack growth in hydrogen is quite brittle compared to that in helium atmosphere.

To interpret the results of microscopic observation from a mechanical point of view, the local stress state near the crack tip is considered. Here, the material is assumed to be a homogeneous isotropic body and an elastic approximation is employed as the first step. Then the resolved shear stress operating on a slip plane close to a mode I crack tip, $\tau_{rss}|_{tip}$, can be described in terms of the singular stress intensity as follows.

$$\begin{aligned}\tau_{rss|tip} &= \frac{K_I}{\sqrt{2\pi r}}\cos\frac{\theta}{2}\left\{ll'\left(1-\sin\frac{\theta}{2}\sin\frac{3\theta}{2}\right)+mm'\left(1+\sin\frac{\theta}{2}\sin\frac{3\theta}{2}\right)+2nn'\nu\right.\\&\qquad\left.+\left(ml'+lm'\right)\sin\frac{\theta}{2}\cos\frac{3\theta}{2}\right\}\\&=\frac{K_I}{\sqrt{2\pi r}}F\left(lmn\,l'm'n'\theta\right)\end{aligned} \tag{2}$$

In Eq. (2), K_I is the mode I stress intensity factor, (r, θ) is the polar coordinate originating from the crack tip, ν is the Poisson's ratio, (lmn) and ($l'm'n'$) are the slip plane normal vector and slip direction vector respectively. The function 'F' can be regarded as the magnitude of stress intensity that depends on the slip system. Calculation of F values on possible slip systems are carried out and the values are compared. The resultant value for type A slip is found to be the largest, and that for type B is 87 % of type A. Since the simultaneous activation of these slip systems is characteristic to the specimen in hydrogen atmosphere, hydrogen may alter the critical shear stress of each slip system and hence causes the different slip behavior. The apparent diversity of operative slip system induced by hydrogen is also found in FCC metal [8]. It is noted, however, that the applicability of the elementary analysis shown here should be validated through a systematical study.

The present investigation successfully captures the clear effect of hydrogen on the slip behavior around a fatigue crack tip i.e. the localization of slip and the simultaneous activation of different pairs of slip systems. These results suggest that the observed crack growth behavior in hydrogen is not simply explained by the two-dimensional slip-off model in which a single pair of symmetrical slip about the crack is considered. A more detailed observation of the dislocation structure along with an appropriate mesoscopic modeling that takes crack tip slip into account is necessary for revealing the mechanism that dominates the brittle crack growth behavior which is now in progress.

4. Summary

The effect of hydrogen on the crack tip cyclic slip deformation is investigated using a single-crystalline Fe-3.2mass%Si alloy. The fracture surface and the near-tip cross-section are observed in detail by SEM and TEM respectively. The cyclic slip-off process by a pair of symmetrical slip system about the crack ($(1\bar{1}\bar{2})[1\bar{1}1]/(1\bar{1}2)[\bar{1}11]$ pair) is operative in helium atmosphere. On the other hand, other types of slip system ($(011)[1\bar{1}1]/(101)[\bar{1}11]/(\bar{1}01)[1\bar{1}1]/(0\bar{1}1)[\bar{1}11]$ pair) are also found to be operative and the slip is localized around the close vicinity of the crack in hydrogen atmosphere.

Acknowledgments

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