# Loading-Frequency Effects on Fatigue Crack Growth Behavior of a Low Carbon Steel in Hydrogen Gas Environment

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# 1. Introduction

It is necessary to clarify the load frequency and duration time effects on the fatigue crack growth behavior to estimate the fatigue life and strength [1,2]. It has been reported that the fatigue crack growth rate of austenitic stainless steel and aluminum alloy in hydrogen gas environment accelerate at a low test frequency and it saturates below a certain frequency. This tendency is based on the slip-off mechanism of fatigue crack propagation. However, the fatigue crack growth rate of carbon steel in hydrogen gas exhibits quasi-cleavage fracture with brittle striations [3]. It may have another mechanism for the fatigue crack growth [4,5]. Therefore, there is a possibility that the loading frequency effect differs from austenitic stainless steel and aluminum alloy.

In this study, in order to understand the basic and qualitative loading frequency dependency on fatigue crack growth behavior, two kinds of fatigue test are carried out. One is a low strain rate test, and the other is a trapezoidal wave test. The fracture surfaces are observed in detail with an Scanning Electron Microscope (SEM).

# 2. Experimental procedure

2.1 In-situ Observation System

The testing apparatus used in this study consists of three parts: a plate bending fatigue test machine, an environmental chamber and a Microscope for observation. Fatigue crack growth behavior in a gas environment can be monitored with the Microscope.

# 2.2 Material and Specimen

The materials used in this study is a low carbon steel (JIS S10C) rolled cylindrical bar with a diameter of 22-mm. Specimens were machined after annealing at 1173K for one hour. Tables 1 and 2 show the chemical composition and mechanical properties of the annealed material. Fig. 1 shows the specimen configuration. The specimen was mechanically polished with #2000 emery paper and buffed with 0.05  $\mu$ m alumina suspension. Then a small blind hole was introduced at the center of the specimen. After that, the specimen was reannealed

Table 1 Chemical composition (wt %)									Table 2 Mechanical properties			
С	Si	Mn	Р	S	Cu	Al	Ni+Cr		$\sigma_{\rm s1}$ [MPa]	$\sigma_{\rm B}$ [MPa]	$\sigma_{\rm T}$ [MPa]	ψ[%]
0.13	0.22	0.39	0.01	0.02	0.09	0.01	0.01		203	372	771	67.7
Small artificial hole									d=0.1 h=0.05 θ=120°			
(a)									(b) Enlargement of a hole			

Fig. 1 Specimen configuration

in vacuum at 873K for one hour to remove the residual stress and the internal hydrogen.

## 2.3 Testing Method

Fatigue crack growth tests were carried out under displacement controlled fully reversed bending in 0.18 MPa hydrogen gas or in nitrogen gas. The temperature of the environment was kept constant at 313 K during each test. In order to determine the loading condition, a strain gauge was placed on the back surface of the specimen. The fatigue process was observed through the glass window.

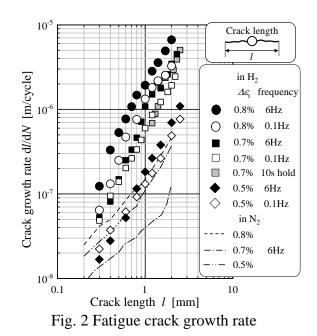
- 3. Results and Discussion
- 3.1 Crack growth behavior in hydrogen gas

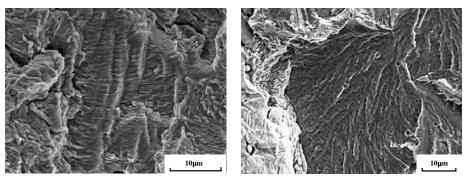
In order to clarify the hydrogen effects on the fatigue crack growth behavior, fatigue tests were carried out in a hydrogen gas and in a nitrogen gas. After the fatigue tests, fracture surface morphologies were observed with an SEM.

Fig. 2 shows the fatigue crack growth rate in nitrogen and in hydrogen gas environment. The fatigue crack growth rate in hydrogen gas was higher than that in nitrogen. The fatigue crack growth rate in hydrogen was more than 10 times higher than that of in nitrogen gas.

Fig. 3 shows typical fatigue fracture surfaces of (a) in nitrogen gas and (b) in hydrogen gas tested at  $\Delta \varepsilon_i=0.80\%$ . Fig. 3 (a) shows ductile fatigue striations. Fig. 3 (b) shows quasi-cleavage facets with brittle striations.

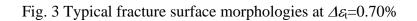
Fig. 4 shows the brittle striation spacing on the fracture surface tested in hydrogen gas. Symbols  $\blacksquare$  and  $\bullet$  represent the data of  $\Delta \varepsilon_t = 0.70\%$ , and  $\Delta \varepsilon_t = 0.80\%$ , which show that the crack growth rate become higher, brittle striation spacing become wider. Brittle striation spacing was related to crack growth rate.





(a) In nitrogen

(b) In hydrogen



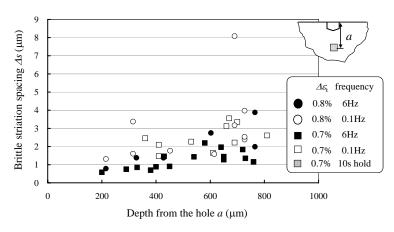


Fig. 4 Brittle striation spacing in hydrogen

3.2 General characteristics of loading frequency effect on fatigue crack growth behavior in hydrogen gas

### 3.2.1 Fatigue crack growth rate

In the case of many other metallic materials, the fatigue crack growth rate usually increases as the loading frequency decreases [1,2]. In order to examine whether the fatigue crack growth rate of the low carbon steel reveals the same tendency, two types of fatigue tests were added. One is a triangular wave with a lower strain rate, and the other with duration time at the every maximum and minimum load, as shown in Fig. 5. Fig. 2 shows that the fatigue crack growth rates reveal rather complex behavior related to the loading frequency. There are both cases; acceleration and deceleration in spite of the low frequencies. The crack growth data having different load frequencies at  $\Delta \varepsilon_t = 0.50\%$  in hydrogen gas ( $\blacklozenge$  and  $\diamondsuit$ ) cross each other. The fatigue crack growth rate at low strain rate accelerates in the low growth rate region, while decelerates in the high growth rate region. In the case of  $\Delta \varepsilon_t = 0.80\%$ , the data at 0.1 Hz ( $\Box$ ) is lower than that at 6 Hz ( $\blacksquare$ ). In the that of 6 Hz ( $\blacksquare$ ).

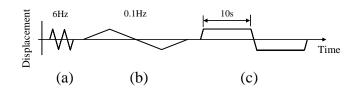


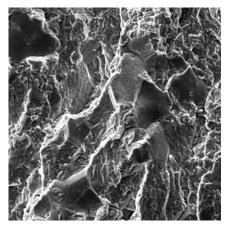
Fig. 5 Wave forms

3.2.2 Brittle striation spacing

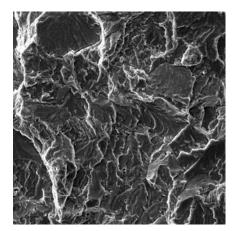
The fatigue crack growth rate usually increases as the brittle striation spacing expands, as described in section 3.1. Here, the load frequency effect on the brittle striation spacing is investigated. Fig. 4 shows that the brittle striation spacing of the low strain rate test and holding time test are slightly wider than those of high strain rate tests ( $\bullet$  and  $\bigcirc$ ,  $\blacksquare$  and  $\square/\square$ ). On the other hand, the fatigue crack growth rate at the low strain rate and of load holding test are lower than that at the high strain rate. This relationship between the brittle striation spacing and the fatigue crack growth rate conflicts with common notion.

### 3.2.3 Fracture surface morphologies

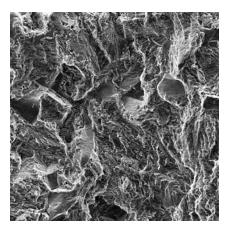
Deceleration tendency of the fatigue crack growth rate at low load frequency did not reflect on brittle striation spacing. In this section, changes in fracture surface morphologies were observed.



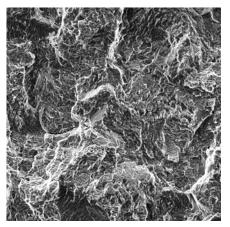
(a-1)  $\Delta \varepsilon_t = 0.50\%$ , d=250µm



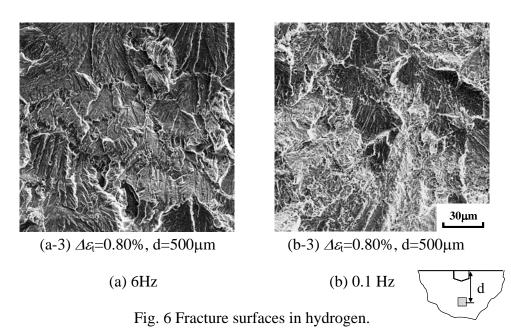
(a-2)  $\Delta \epsilon_t = 0.50\%$ , d=500 $\mu$ m



(b-1)  $\Delta \epsilon_t = 0.50\%$ , d=250µm



(b-2)  $\Delta \epsilon_t = 0.50\%$ , d=500 $\mu$ m



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Fig. 6 shows the comparison between the fracture surfaces tested at high strain rate and low strain rate under  $\Delta \varepsilon_t = 0.5\%$ ; (a) 6 Hz, (b) 0.1 Hz. The mechanical condition having the same depth d is almost the same. Fig (a-1) and (b-1) are the fracture surfaces at the low growth rate region, (a-2) and (b-2) at a high growth rate region. Grain boundaries facets are seen in (a-1) and quasi-cleavage with brittle striations are seen in (a-2). However, (b-1) is more ductile than (a-1) and (b-2).In the case of higher strain range of  $\Delta \varepsilon_t$ =0.80%, the situation are the same; the quasi-brittle surface are predominant in (a-3), while small portion in (b-3). The fracture surface of the test with duration time reveals similar tendency to that at a low load frequency.

The decrease of fatigue crack growth rate at low strain rate could be explained by decrease of the area ratio of quasi-cleavage. It is considered that fatigue crack growth rate accelerate both in ductile area and in quasi-cleavage area by hydrogen, but quasi-cleavage is faster propagation mode.

3.3 Detailed Observation of loading frequency effect on Brittle-like fracture

Loading frequency effect on the brittle fracture area does not clarified. In this section, brittle-like fracture is divided into two processes. One is the formation of starter for brittle-like fracture. The other is propagation of brittle striation. In order to clarify the loading frequency effects on the fatigue crack growth rate of the brittle fracture area, loading frequency effects on both processes are discussed.

## 3.3.1 Formation of starter for brittle-like fracture

In this part, loading frequency effects on the formation of starter for brittle-like fracture were observed. Fig. 7 shows the relationship between the area ratio of quasi-cleavage fracture and the depth from the hole. Arrows indicate the depth at which quasi-cleavage fracture first appeared. At 0.1 Hz, the depth of first quasi-cleavage fracture was deeper than that of 6Hz. It is considered that a lower load frequency may make harder to form the starter for brittle-like fracture

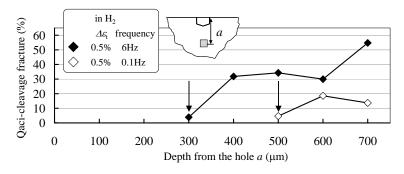
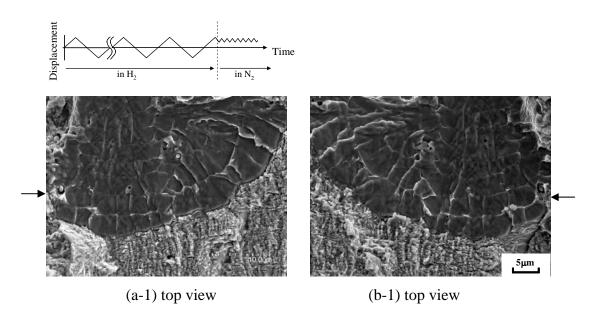


Fig. 7 Relationship between the area ratio of the quasi-cleavage surface and the depth from a hole

### 3.3.2 Propagation of brittle striation area

One of the possibilities of brittle striation formation process is as follows. First, a crack tip opens by slip deformation and blunted at the loading part. After that, a crack tip starts propagation. Brittle striation is formed when propagation starting. In this part, brittle striation formation process and loading frequency effects on the crack propagation of brittle striation area were investigated.

In order to know whether a crack tip has blunting process, matching of brittle striations were conducted. To leave the clear step formed during the loading part of the last cycle, the crack was lengthened in a load sequence as shown in Fig. 8. Figs 8 (a-1) and (b-1) are the top view of mating fracture surfaces and (a-2) and (b-2) are tilted images. The striation indicated by arrows shows the last striation; the step is higher than that of others and there is no striation in front of the step. It is considered that crack propagated after blunting process.



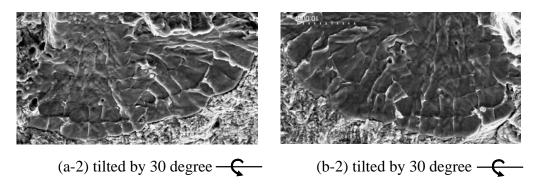
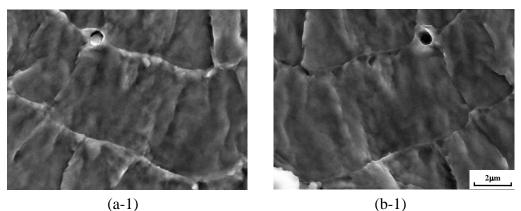
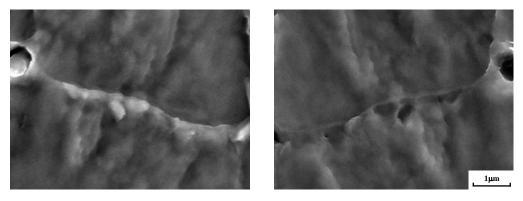
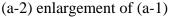


Fig. 8 Blunting at the brittle striation on the mating fracture surfaces



(b-1)





(b-2) enlargement of (b-1)

Fig. 9 Brittle striation formation on the mating fracture surfaces

In order to know the crack propagation process of a brittle striation, matching of the brittle striations on the mating fracture surfaces were conducted Fig. 9 shows matching images of high magnification. The inclusion is seen on the brittle striation. Dimples at the inclusion and along the striations look ductile ones. Between striations, fracture surfaces are not flat but wavy. It seems slip deformation exists in propagation process. It is considered that, propagation process in this case is similar to ductile tear fracture.

To clarify the loading frequency and hydrogen effects on the crack growth rate in the brittle striation region, two kinds of crack growth tests were carried out in load sequences as in Figs 10 (a-1) and (b-1). Changes in crack growth behavior are directly observed on the same grain. Fig. 10 (a) and (b) do not show apparent change in brittle striation spacing. However, (b) shows some effect of load duration time on brittle striation propagating area. Last three striation steps are more distinct than those of preceding cycles and deformation between the striations. Hydrogen diffused around the crack tip may also assist the deformation.

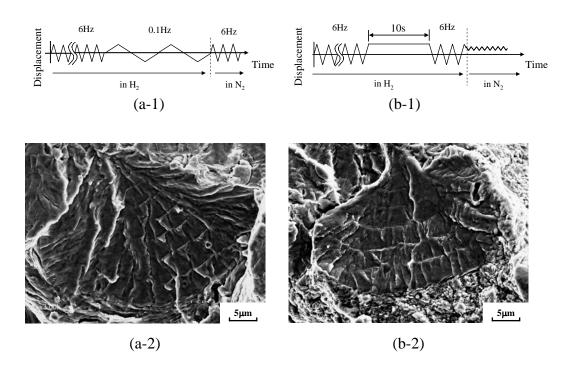


Fig. 10 Effects of the load frequency and the duration time on the brittle striation formation ( $\Delta \varepsilon_t = 0.80\%$ )

## 4. Summary

In this study, loading frequency effects of a low carbon steel (JIS S10C) on the fatigue crack growth rate were investigated. In the case of many other metallic materials, the fatigue crack growth rate usually increases as loading frequency decreases. However, in this case fatigue crack growth rate shows rather complex behavior. There are both cases; acceleration and deceleration in spite of the low frequency. In this case, the decrease of fatigue crack growth rate at low strain rate could be explained by decrease of the area ratio of quasi-cleavage.

To estimate the loading frequency effect of this material, it is necessary to consider the effects on the ductile fracture and on the brittle-like fracture.

At ductile fracture area, crack is accelerated by low strain rate similar to many other materials.

At the brittle-like area, it is considered that loading frequency affect forming the starter of brittle-like fracture and propagation of brittle striation. One of the possible effects of loading frequency is as follows.

Based on the experimental results, brittle striation forming process starts opening crack tip by slip deformation and blunting. After that the crack tip starts propagating. Propagation process is similar to ductile tear fracture. Low strain rate makes slip deformation easier and helps blunting. Hydrogen also can help blunting. Low strain rate and load holding makes brittle striation becomes harder to start propagation. Therefore, quasi-cleavage fracture decreases.

At brittle striation propagating process, slip deformation seem to occur. Therefore, it is possible that propagation process has load frequency effect. However, in this case, these did not appear clearly.

The crack growth rate at low strain rate is determined by the mutual relationship between the brittle like fracture and ductile fracture.

However, the study in the wide range of the condition should be conducted in the future.

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