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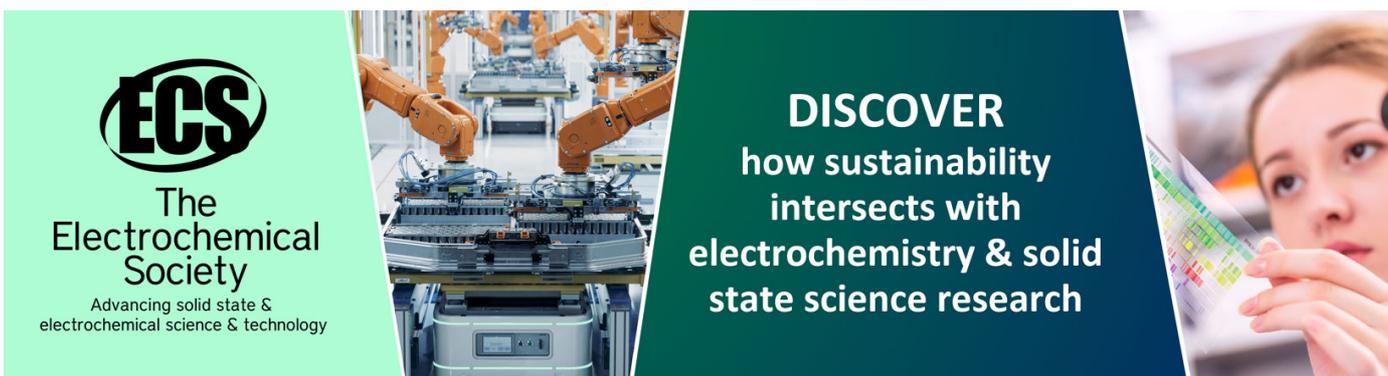
An investigation into the frequency dependence upon the fatigue crack growth rate conducted by a novel fatigue testing method with in-situ hydrogen-charging

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An investigation into the frequency dependence upon the fatigue crack growth rate conducted by a novel fatigue testing method with in-situ hydrogen-charging

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Abstract. The most widely used testing methods for evaluating the hydrogen embrittlement resistance are classified into the following two types: (1) testing in high-pressure hydrogen gas and (2) testing in air using hydrogen pre-charged specimens. Testing in hydrogen gas is technically difficult and expensive, because high-pressure gas equipments composed of such as a high-pressure vessel and piping system has to be introduced. On the other hand, in the case of pre-charging method, the outgassing of hydrogen from the specimen occurs during the test. Therefore, hydrogen embrittlement could not be evaluated accurately by the pre-charging method in long-term tests such as a fatigue test. In a previous study, to evaluate hydrogen embrittlement effectively, a novel experimental method was developed, in which four-point bending fatigue test system was performed with continuous circulation of a hydrogen-charging solution into a pipe specimen. This new testing system using hydrogen-charging solution enables an easy yet reasonable evaluation of hydrogen embrittlement for a long-term fatigue test. In this study, the frequency effect on the crack growth rate due to hydrogen was investigated by this new testing method. Fatigue crack growth tests at a test frequency ranging from 1 to 0.0002 Hz demonstrated that the fatigue crack growth rate was faster in the presence of hydrogen than in the absence of hydrogen at all test frequencies. Further, the increase in crack growth rate became more pronounced with decrease in test frequency. This frequency dependence upon crack growth rate was discussed by considering the difference between the effects of external hydrogen and internal hydrogen.

1. Introduction

Development of practical use of hydrogen as an environment-friendly energy is highly required to solve global warming and resource problem, and consequently the establishment of hydrogen energy society is being proceeded in USA, Europe, Japan, etc. However, in order to ensure the integrity of hydrogen equipments (e.g. hydrogen fuel cell vehicle and hydrogen station), problems of hydrogen embrittlement (i.e., degradation of strength properties of metallic material due to hydrogen penetration into the material) need to be well understood and properly solved.

The strain rate dependence is a well known feature of hydrogen embrittlement [1-7]; i.e., slow strain rate for monotonic loading or low frequency for fatigue loading intensifies the hydrogen effect. Regarding the hydrogen equipments mentioned above, many kinds of components are used and some



of them (e.g., pressure vessel) are subjected to a cyclic load with extremely low frequency. Therefore, the frequency dependence of fatigue properties in the presence of hydrogen is a very important issue.

Recently, Matsuo et al. [8] reported a peculiar frequency dependence of fatigue crack growth in hydrogen gas, which is summarized as follows. The crack growth rate in hydrogen gas was increased with decrease in test frequency and it reached a peak at 0.1 Hz. This can be explained by the hydrogen enhanced successive fatigue crack growth (HESFCG) [9, 10] mechanism in which the increase in crack growth rate is caused by stress-induced hydrogen diffusion and accumulation in the limited area in the vicinity of the crack tip. Surprisingly, the further decrease in frequency inversely reduced the crack growth rate to the level of that in air. The HESFCG mechanism cannot describe this phenomenon. Matsuo et al. [8] pointed out that hydrogen penetrating into the material through the crack surface cannot remain within the small area in the vicinity of crack tip (i.e. hydrogen will diffuse to comparatively large area around crack tip) owing to extremely long loading time in a cycle. Somerday et al. [11] also reported a similar frequency dependence of crack growth in hydrogen gas. They interpreted that the inhibitor effect against hydrogen penetration due to impurity such as oxygen contained in hydrogen gas became pronounced with decrease of test frequency. Both groups conducted the fatigue crack growth tests in hydrogen gas and considered the effect of the hydrogen which came from the outside of a specimen during the tests (so-called *external hydrogen*). In the case of the external hydrogen, both hydrogen penetration and subsequent diffusion need to be considered. This may be a cause that complicates the interpretation of the mechanism of peculiar frequency dependence. In the case of hydrogen that exists inside a specimen prior to the test (so-called *internal hydrogen*), it is possible to discuss about the frequency dependence by focussing only on the effect of hydrogen diffusion and accumulation at crack tip. However, in conventional fatigue test for investigating the effect of internal hydrogen (i.e. the test using pre-charged specimen), the crack growth property at low frequency cannot be evaluated successfully because of considerable hydrogen outgassing from a specimen during the early stage of long-term testing.

In this study, we investigated the frequency dependence of crack growth rate in the presence of internal hydrogen using the method newly developed in the previous study [12], which is a four-point bending test system with internal circulation of hydrogen-charging solution in a pipe specimen.

2. Testing method and specimen

2.1 Testing method

Figure 1 shows the concept of the testing method developed in the previous study [12]. The fatigue crack growth test was conducted by the four-point bending fatigue test system with internal circulation of hydrogen-charging solution in a pipe specimen. The hydrogen-charging solution (20 mass% NH_4SCN) was pumped into the pipe specimen through a flexible tube. Two screw holes were introduced on both ends of the specimen, which were connected to a flexible tube by a screw type fitting. This specimen with internal circulation of hydrogen-charging solution is hereafter called the *H-charging specimen*, while the specimen tested without hydrogen-charging is called the *non-charging specimen*.

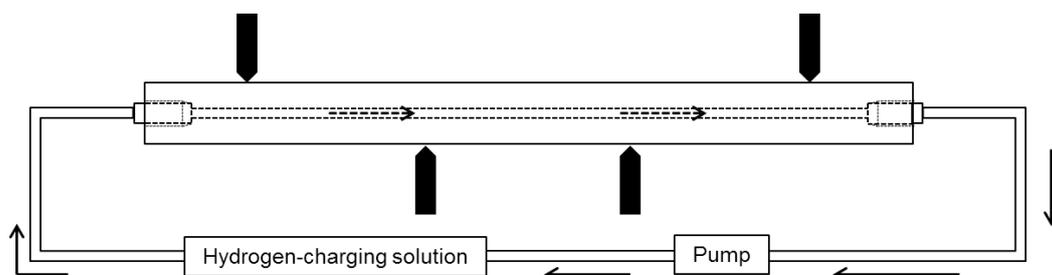


Figure 1. Concept of the testing method.
(Four-point bending fatigue test with internal circulation of hydrogen-charging solution).

2.2 Specimen

An annealed carbon steel (JIS-S35C) was used in this study. The Vickers hardness of this material is approximately 156. The microstructure is composed of ferrite-pearlite with an elongated texture in the rolling direction, as shown in Fig. 2.

Figure 3 shows the geometry of pipe specimen. The specimen surface was polished with a #2000 emery paper and subsequently buff-polished using alumina paste with a particle diameter of 1 μm . After polishing, three drilled holes shown in Fig. 4 were introduced on the specimen surface as a crack starter. Then, stress relief annealing in vacuum at 600 $^{\circ}\text{C}$ for 1h was conducted.

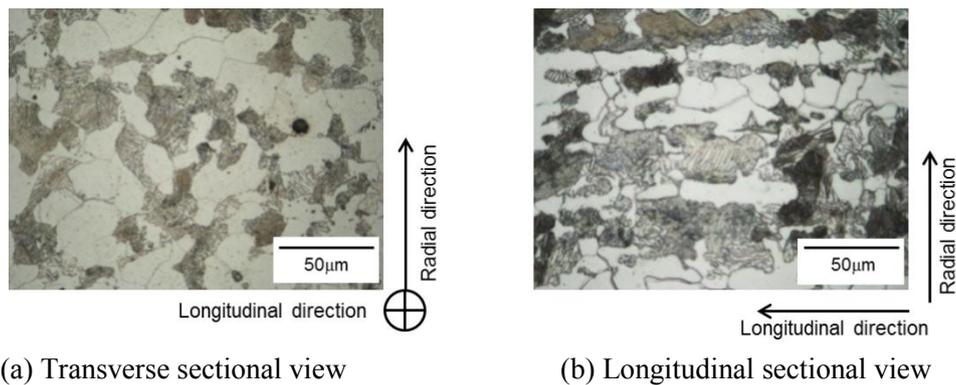


Figure 2. Microstructure of S35C.

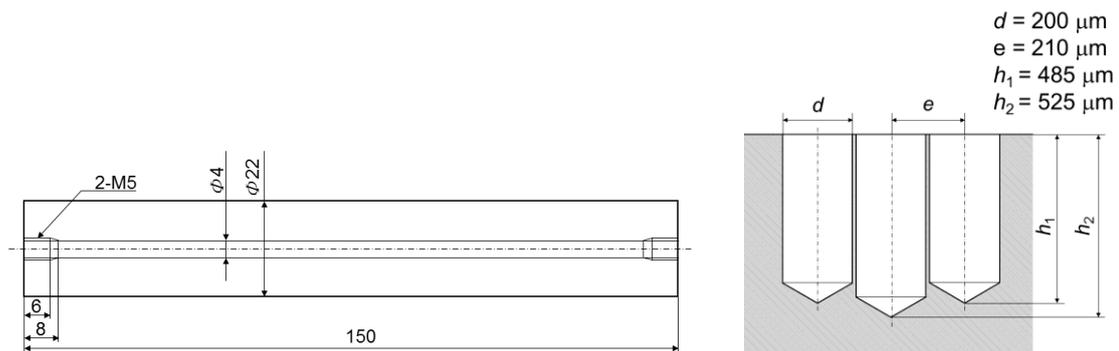


Figure 3. Geometry of pipe specimen (in mm).

Figure 4. Shape and dimensions of drilled holes.

2.3 Fatigue crack growth test

Fatigue crack growth behavior was investigated by the four-point bending fatigue of a H-charging specimen and a non-charging specimen in air at room temperature. The stress amplitude was constant, and the stress intensity factor (SIF) range, ΔK , increased from 5.5 $\text{MPa}\cdot\text{m}^{1/2}$ to 15 $\text{MPa}\cdot\text{m}^{1/2}$ with crack growth (ΔK -increasing test). The stress ratio, R , was 0.1 and the test frequency, f , was 1 Hz. Then, the frequency dependence of fatigue crack growth rate was investigated at test frequencies of 0.0002 Hz, 0.002 Hz, 0.05 Hz, 0.2 Hz and 1 Hz. The stress amplitude was decreased every time the frequency was changed so that the SIF range could be kept around 15 $\text{MPa}\cdot\text{m}^{1/2}$ (ΔK -constant test). The crack growth behavior was observed by an optical microscope at the appointed numbers of cycles to measure the crack length. The fracture surface was observed by a scanning electron microscope. Hydrogen-charging solution was changed every three days in order to maintain the enough amount of hydrogen to supply from the charging solution to the specimen.

3. Results and discussion

3.1 Fatigue crack growth rate obtained by ΔK -increasing test

ΔK -increasing tests were conducted at a stress ratio of $R = 0.1$ and at a test frequency of $f = 1$ Hz under the conditions of continuous hydrogen-charging as well as no hydrogen-charging. The SIF range was initially about $5.5 \text{ MPa}\cdot\text{m}^{1/2}$ and gradually increased up to about $15 \text{ MPa}\cdot\text{m}^{1/2}$ with crack growth. Figure 5 shows the relationship between the crack growth rate, da/dN and the SIF range, ΔK . As reported in our previous study [12], the crack growth rate was enhanced in the H-charging specimen in the relatively high ΔK regime ($\Delta K > 7 \text{ MPa}\cdot\text{m}^{1/2}$). For the present H-charging specimen, after the SIF range reached $15 \text{ MPa}\cdot\text{m}^{1/2}$ in the process of ΔK -increasing test, the test frequency was decreased in a stepwise fashion from 1 Hz to 0.0002 Hz, maintaining a constant SIF range of around $15 \text{ MPa}\cdot\text{m}^{1/2}$ by properly decreasing the stress amplitude. As shown in Fig. 5, the enhanced crack growth rate with frequency effect was observed at $\Delta K = 15 \text{ MPa}\cdot\text{m}^{1/2}$. A further discussion on frequency effect is provided in Section 3.2. Many flat fracture facets of the order of a few grains in size were observed on the fracture surface of the H-charged specimen.

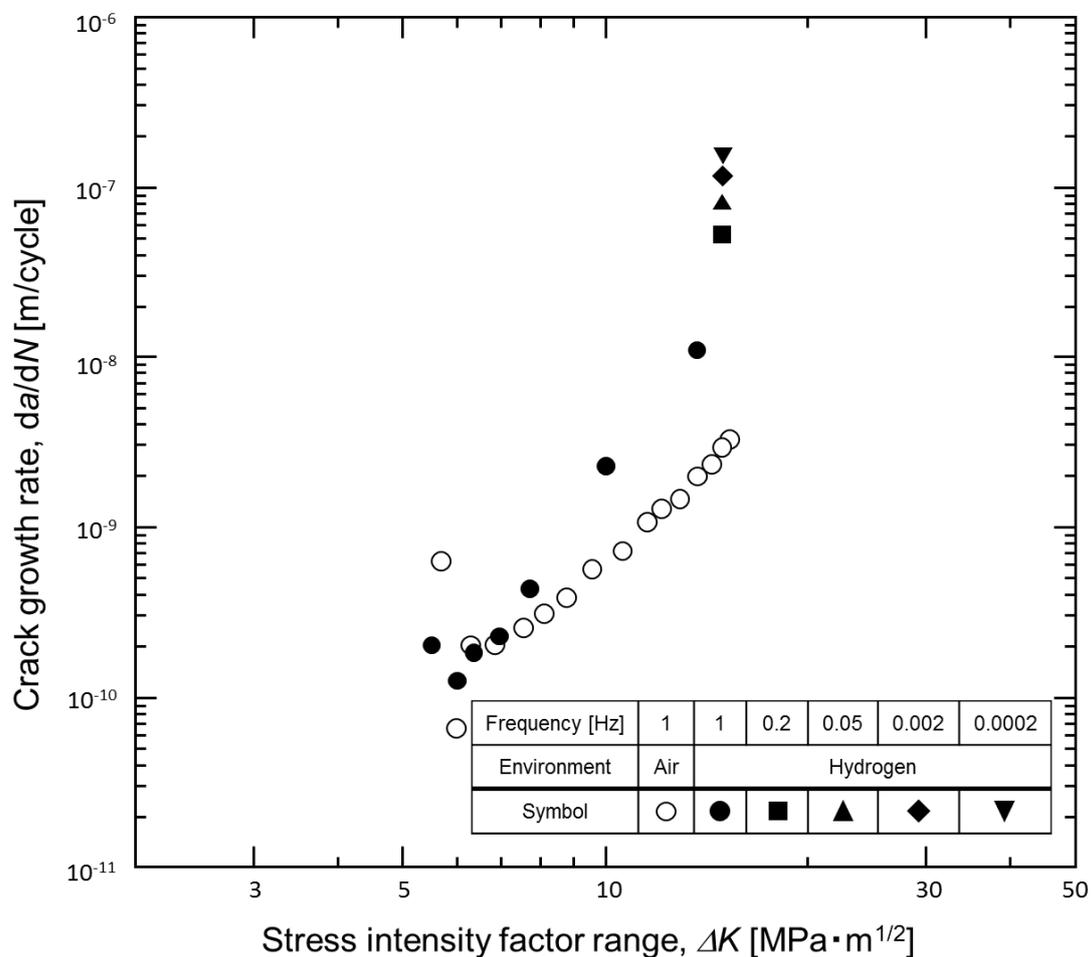


Figure 5. Relationship between crack growth rate and ΔK .

3.2 Frequency dependence of fatigue crack growth rate under continuous hydrogen-charging

The ratio of the crack growth rate of H-charging specimen (da/dN)_H to that of non-charging specimen (da/dN)_{H-free}, is shown as a function of test frequency, f , in Fig. 6. In this study at all test frequency, the crack growth rate is higher for the H-charging specimen than for the non-charging specimen. The rate of (da/dN)_H/ (da/dN) _{H-free} increased as the test frequency decreased. The increase in crack growth rate

was noticeable when the frequency decreased from 1 Hz to 0.05 Hz, whereas the ratio moderately increased with decreasing from $f = 0.05$ Hz to 0.0002 Hz. The ratio reached 52 at 0.0002 Hz (the lowest frequency in this study). This frequency dependence of crack growth rate due to hydrogen is well known as a feature of hydrogen-related fracture [1-8], which has been explained by considering the increase in hydrogen accumulation at the crack tip with decrease in test frequency. As mentioned in the *Introduction*, a peculiar frequency dependence of hydrogen-induced crack growth rate in hydrogen gas was reported by Matsuo et al. [8]; that is, although the increase in crack growth rate became pronounced with decrease to $f = 0.1$ Hz, the crack growth rate did not increase at a frequency of 0.01 Hz anymore and it decreased with further decrease in f (cf. Fig 6). In this study, however, such the peculiar frequency dependence was not observed even at 0.0002 Hz, showing a monotonic increase with decrease in f . In the next section, this different characteristics for frequency dependence will be discussed by focusing on the possible different actions of internal hydrogen and external hydrogen.

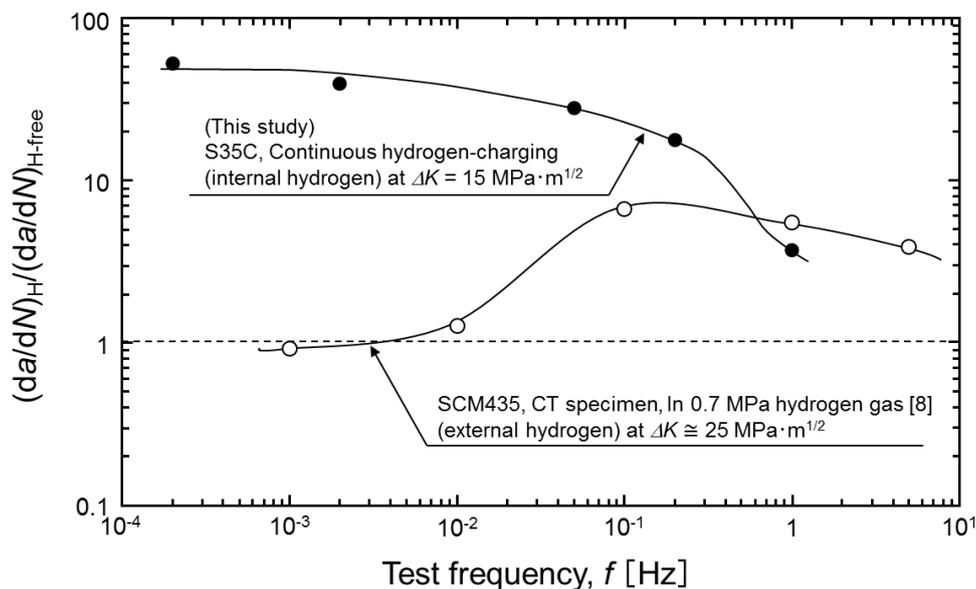


Figure 6. Frequency dependence of $(da/dN)_H / (da/dN)_{H-free}$ under ΔK -constant condition.

3.3 Effect of internal and external hydrogens on the frequency dependence

As shown in Fig. 6, the increase in crack growth rate due to internal hydrogen tends to be saturated at an extremely low frequency, approaching to a value of about 60 in the ordinate. In the case of external hydrogen, the increase rate reached a peak of about 7 at $f = 0.1$ Hz. This different actions of internal and external hydrogens can be explained from the viewpoint of the mechanism of hydrogen accumulation at the crack tip. Figure 7 shows the schematic illustrations of hydrogen accumulation at the crack tip. External hydrogen penetrates easily into the material directly through the newly formed crack surface and thereby more hydrogen accumulates at the crack tip. On the other hand, internal hydrogen needs to diffuse inside the material to accumulate at the crack tip. For this case, internal hydrogen requires much longer time for accumulation than external hydrogen. As a result, the frequency corresponding to the maximum influence on the crack growth is supposed to become lower for internal hydrogen than for external hydrogen. The peak of increase rate for internal hydrogen may exist at a further low frequency less than 0.0001 Hz. Unfortunately, this could not be verified in this study because of time-consuming fatigue test. However, the following two points are worthy to note: (i) the different accumulation mechanisms of internal and external hydrogens at the crack tip and (ii) the increase rate for internal hydrogen shows a tendency of saturation around $f = 0.0001$ Hz.

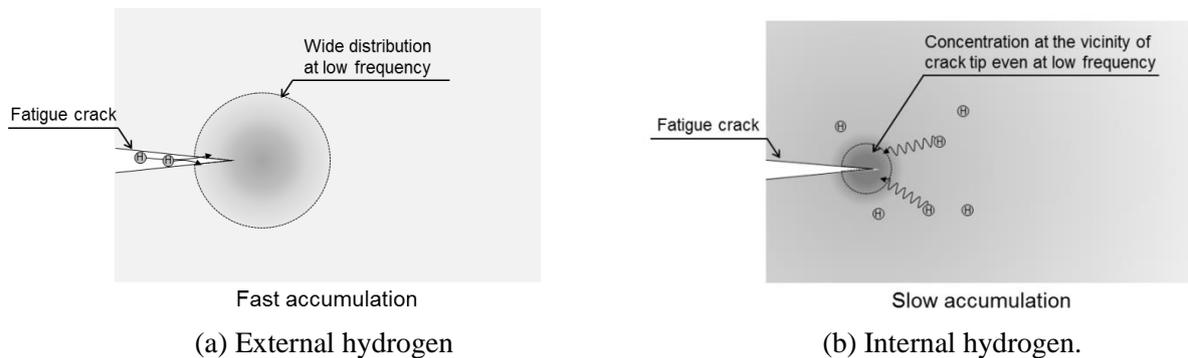


Figure 7. Different mechanisms of hydrogen accumulation at crack tip between internal and external hydrogens.

4. Conclusions

The frequency dependence of the crack growth rate due to hydrogen was investigated by conducting four-point bending fatigue tests with internal circulation of hydrogen-charging solution in a pipe specimen. The conclusions are summarized as follows:

1. The crack growth rate was increased by continuous hydrogen-charging in the range of a frequency from 0.0002 Hz to 1 Hz, which became pronounced with decrease in frequency.
2. The frequency at which the degree of increase showed a peak was much lower for internal hydrogen than for external hydrogen.
3. The different effects of internal and external hydrogen on frequency dependence of crack growth rate could be explained by considering the different mechanisms of hydrogen accumulation at the crack tip.

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