Hydrogen Gas Effects on the Fatigue Crack Growth Behavior of Cr-Mo Steel CT Specimen in Extremely Low Rate Range

Koki TAZOE[1,a], Yasuji ODA[2,b] and Hiroshi NOGUCHI[2,c]

[1] Graduate School of Engineering, Kyushu University,
744 Moto-oka, Nishi-ku, Fukuoka 819-0395, Japan

[2] Kyushu University, Faculty of Engineering, Collaborative Research Scientist of AIST,
744 Moto-oka, Nishi-ku, Fukuoka 819-0395, Japan

[a] 2TE11332K@s.kyushu-u.ac.jp, [b] oda@mech.kyushu-u.ac.jp, [c] nogu@mech.kyushu-u.ac.jp

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Abstract. In order to investigate extremely slow fatigue crack growth characteristics of JIS SCM440 CT specimen in 9 MPa hydrogen gas environment, stress intensity factor range (ΔK) decreasing tests with in-situ observation were carried out. Fatigue crack growth rate (FCGR) in hydrogen gas did not show threshold behavior but FCGR in helium and air showed threshold behavior clearly. Fatigue crack in hydrogen gas showed sudden increased after a temporary stop in growth. The sudden increase in growth was induced by coalescing with a new micro-crack initiated in front of the main crack tip. The fractographic analysis showed the existence of intergranular facets. The intergranular facets were observed in all over the fracture surface. The amount of the intergranular facets in hydrogen gas decreased with decreasing the ΔK. However, facets were still observed in the extremely low rate region. On the other hand, there was no facet in fracture surface tested in helium and in air in the extremely low rate region. The formation of facets was supposed to be one of the causes of non-threshold behavior in hydrogen gas.

Introduction

It is well known that hydrogen degrades strength of material. In these days, a lot of researches have been carried out for developing the fuel cell vehicle. A lot of reports were made to the crack growth behavior in region of high ΔK and high FCGR in hydrogen gas environment. However, only few reports about the crack growth behavior in region of near ΔK_{th} and low FCGR, which is very important, were made because of the difficulty with testing in the high pressure hydrogen gas environment. According to the report of Cr-Mo steel tested at 50 Hz in low pressure hydrogen gas, ΔK_{th} in hydrogen went down and the ΔK_{th} was smaller than that in air. The report concluded that the reason was the effect of oxide-inducted closure [1] [2]. However, it was reported that hydrogen affected the slip behavior [3] [4]. In addition, as the pressure rises, the amount of hydrogen in material increases. If the result of low pressure environment is applied for evaluation of high pressure environment, closer to real machine, it will have possibility to give danger decision. So, it is important to investigate extremely slow fatigue crack growth characteristics and the mechanism of near ΔK_{th} in high pressure hydrogen environment. In this study, the effect of high pressure hydrogen environment to the fatigue crack growth characteristics of Cr-Mo steel, for storage cylinder of hydrogen station, is discussed with in-situ observation and SEM fractography.

Experimental procedure

Specimen. The metal used in this study was Cr-Mo steel JIS SCM440. Tables 1 and 2 show the chemical composition and mechanical properties. This steel was quenched at 1133 [K] for 2.5 hours and tempered at 733 [K] for 3 hours. Fig. 1 shows the geometry of CT specimen.

Testing. In order to investigate the extremely slow fatigue crack growth characteristics, ΔK decreasing tests based on ASTM standard were carried out. Fig. 2 shows the load variation in the ΔK decreasing test. After the pre-crack was introduced, C constant (= $1/\Delta K \times d\Delta K/da$) satisfied the requirement of ΔK decreasing test ($C > -0.08$ [mm^{-1}]). FCGR by in-situ observation was measured every 20 [μm] in the last 100 [μm] of load step at the observation surface. After the test, the crack length of the other side was measured and the ΔK was revised. In the region of large ΔK (> 8 [MPa $\sqrt{m}$]) FCGR was measured by the compliance method [5].

Conditions. The tests were conducted in 9 [MPa] high purity (99.999 %) hydrogen gas, 9 [MPa] high purity (99.999 %) helium gas and in air at room temperature. The reason why helium was selected is to exclude the effect of oxygen. Stress ratio (R = minimum load/maximum load) was 0.1. Cyclic loading frequency was 5 [Hz] (sine wave).

In-situ observation. The environmental testing machine has a chamber with a viewing window on side. In-situ observation was realized through the window with the microscope camera.

SEM observation. After the test, to investigate the vestige of hydrogen effect in fracture surface, SEM observations were carried out. To observe by SEM, the specimen with protection was cut carefully, soaked in liquid nitrogen and broken in the load direction.

Table 1. Chemical composition [wt. %].

C	Si	Mn	P	S	Cu	Ni	Cr	Mo
0.36	0.26	0.8	0.014	0.025	0.24	0.07	1.13	0.15

Table 2. Mechanical properties.

σ_Y [MPa]	σ_B [MPa]	δ [%]	φ [%]	HV
1180	1324	6.22	33.2	430

HV : Load 2 [kgf], Time 30 [sec]

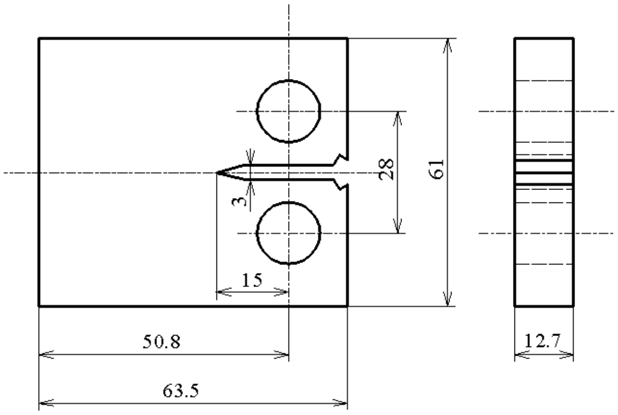


Fig. 1. Geometry of CT specimen.

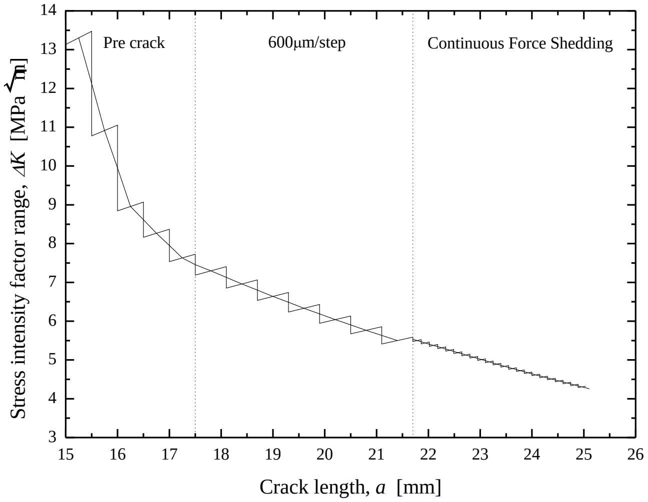
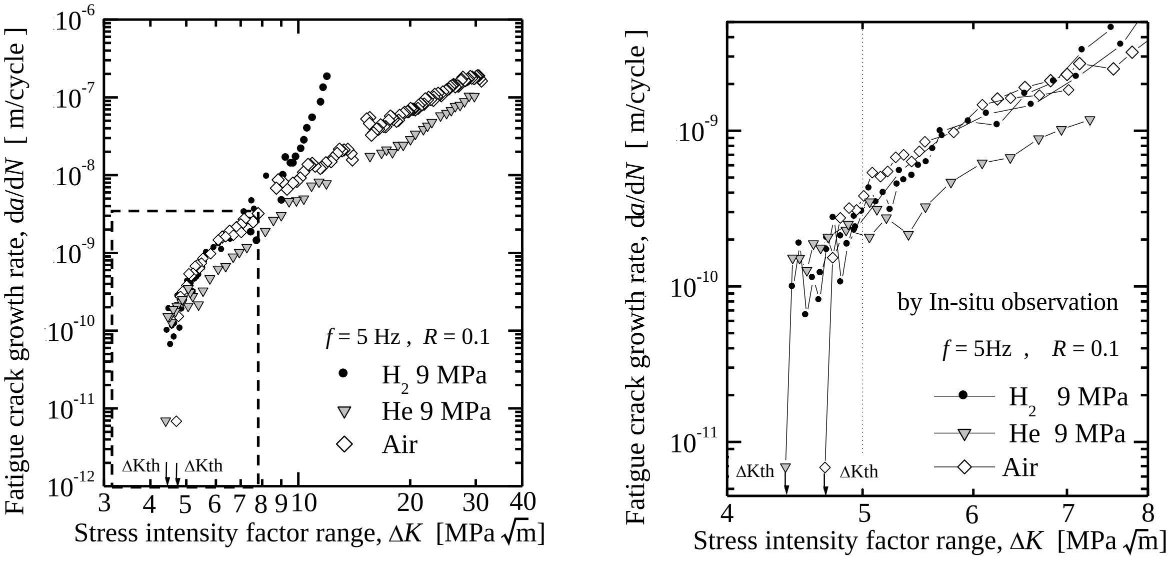


Fig. 2. Load variation in the ΔK decreasing test.

Result and discussion

Fatigue crack growth characteristics. Fig. 3 shows relationship between FCGR and ΔK. FCGR in hydrogen approached to the one in air at ΔK = 8 [MPa √m]. FCGR in helium and in air showed threshold behavior clearly at ΔK = 4.40 [MPa √m] and ΔK = 4.70 [MPa √m]. However, in hydrogen, FCGR did not show threshold behavior at ΔK_{th} in helium and the ΔK_{th} in air.

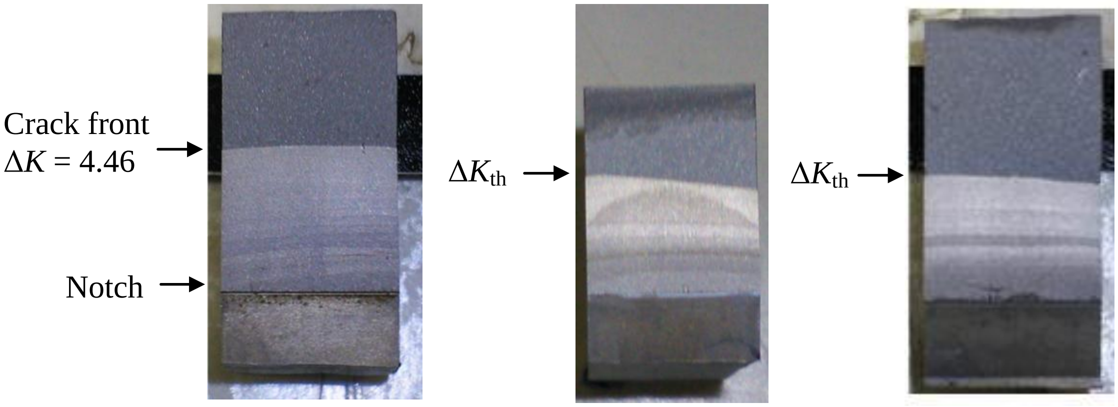


a) Whole

b) Enlargement of near ΔK_{th}

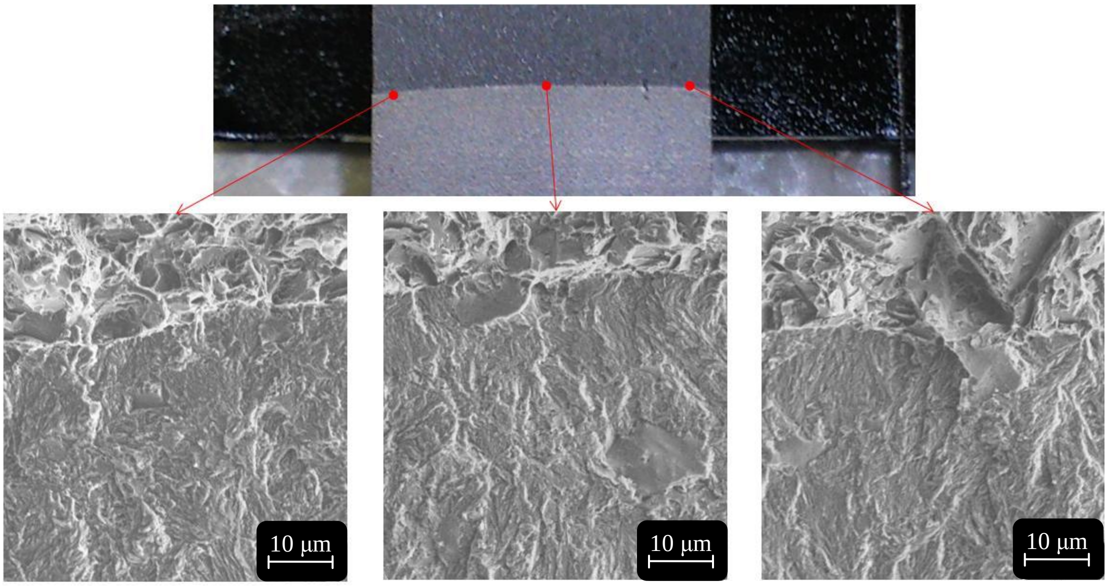
Fig. 3. Relationship between da/dN and ΔK.

Fracture surface. Fig. 4 shows fracture surfaces tested in hydrogen, helium and air. There are beach marks in the early load step region. The width of beach mark is almost the same. It shows that the difference of crack growth between near surface and center is very small. Fig. 5 shows SEM image of fracture surface in hydrogen. Figs. 5 a), b) and c) show almost the same pattern, so, there is no microscopic difference between near surface and center. Therefore, it is suitable to consider FCGR by in-situ observation as the average FCGR. Even more, there are intergranular facets in fracture surface in hydrogen. For convenience, in this paper the non facet area of fracture surface is called *matrix*. Then, with attention to intergranular facets, the microscopic fracture surface in hydrogen is compared with that in another environment. Fig. 6 shows the SEM image of fracture surface classified by FCGR and environment. In the region of 5×10^{-9} [m/cycle], there are facets not only in hydrogen but also in helium and in air. However, the amount of facets decreases with declining FCGR, and there is no facets in the area of 1×10^{-10} [m/cycle], near ΔK_{th}, in helium and air. On the other hand, facets are still observed clearly in 1×10^{-10} [m/cycle] in hydrogen. These results show that hydrogen still effects crack growth behavior in the extremely low rate range.



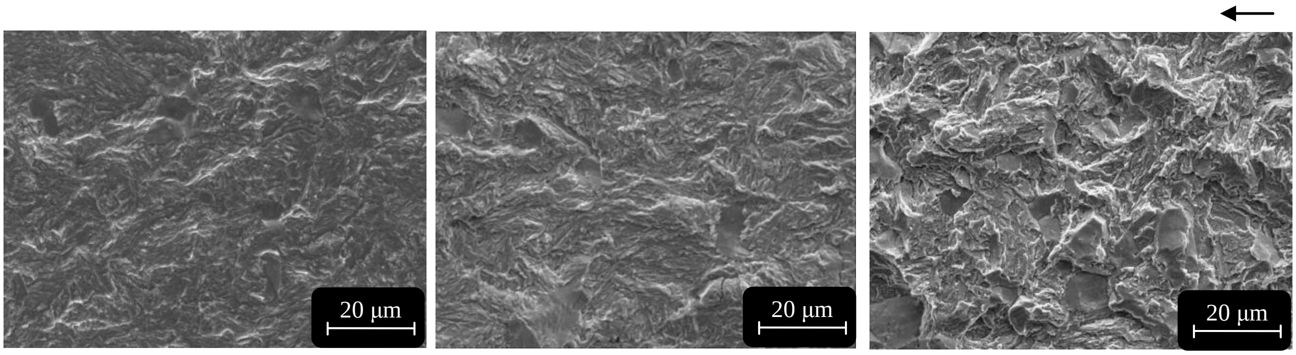
a) Hydrogen b) Helium c) Air

Fig. 4. Digital camera image of fracture surface.



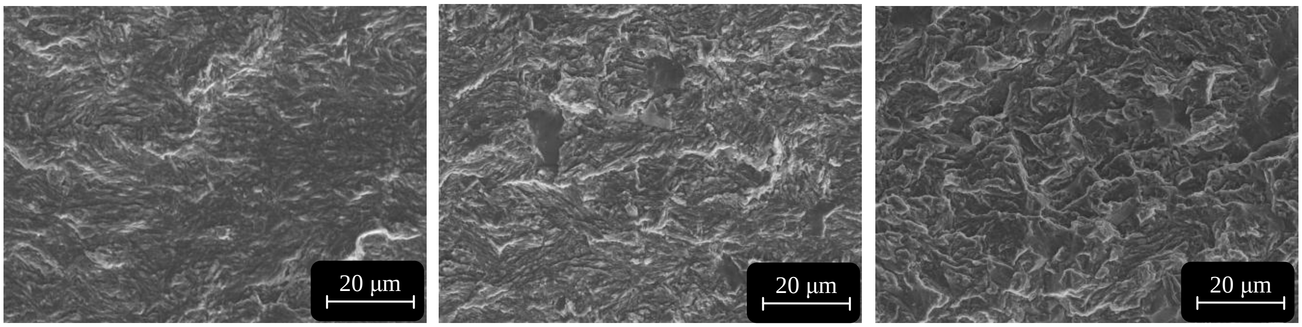
a) Observation side b) Interior c) Other side

Fig. 5. SEM image of fracture surface in hydrogen.



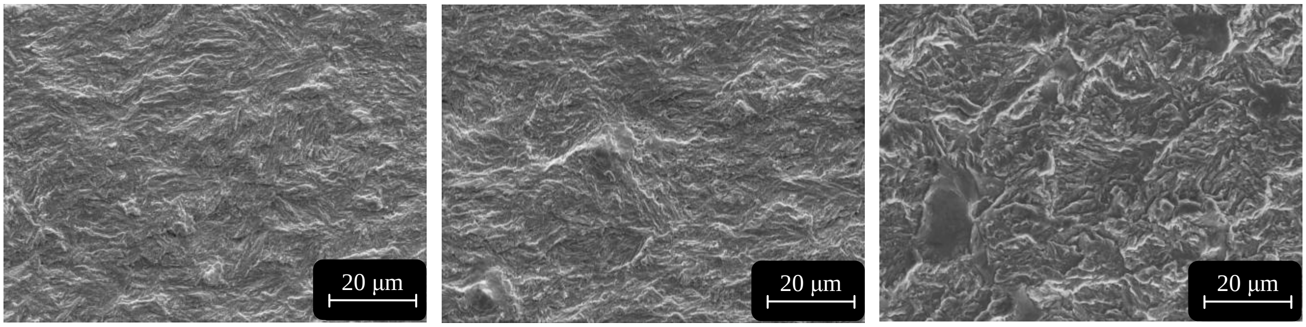
1×10^{-10} [m/cycle] 5×10^{-10} [m/cycle] 5×10^{-9} [m/cycle]

a) Hydrogen



1×10^{-10} [m/cycle] 5×10^{-10} [m/cycle] 5×10^{-9} [m/cycle]

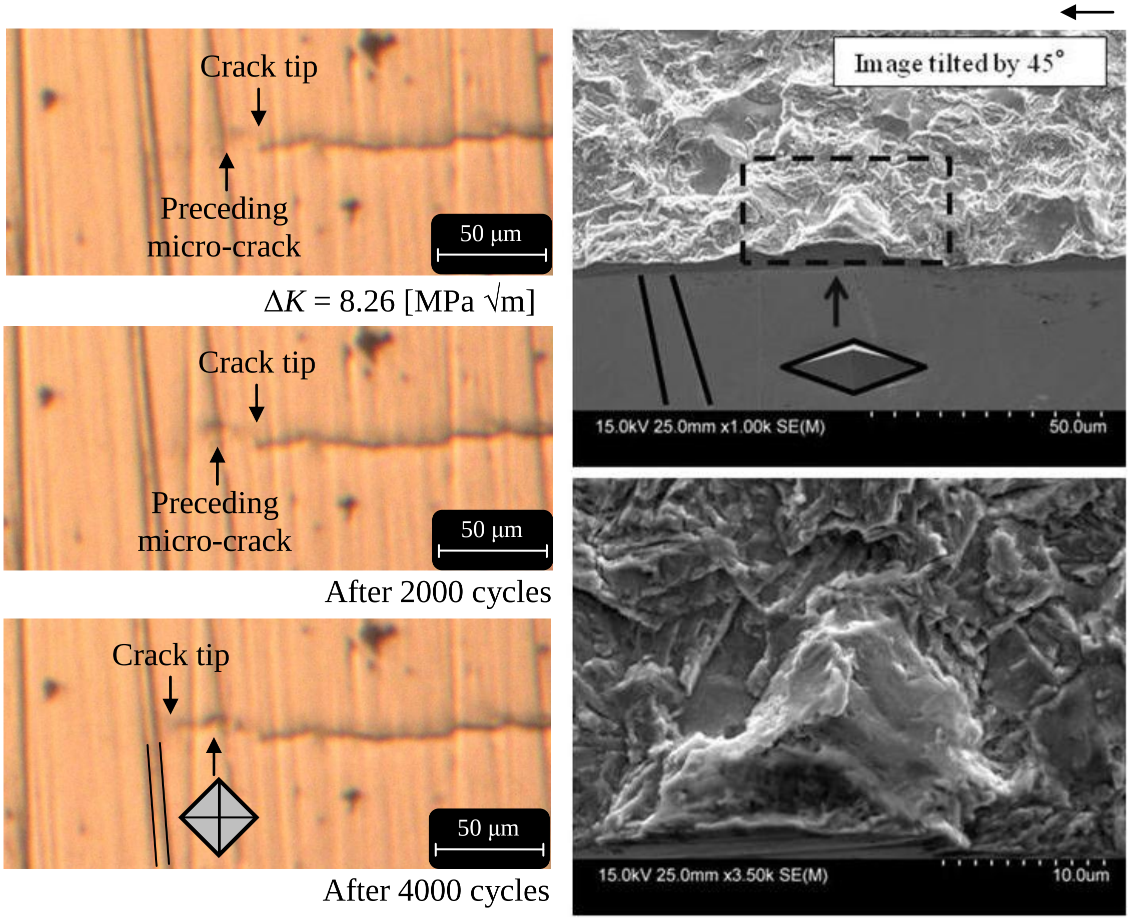
b) Helium



1×10^{-10} [m/cycle] 5×10^{-10} [m/cycle] 5×10^{-9} [m/cycle]

c) Air

Fig. 6. SEM image of fracture surface classified by FCGR and environment.

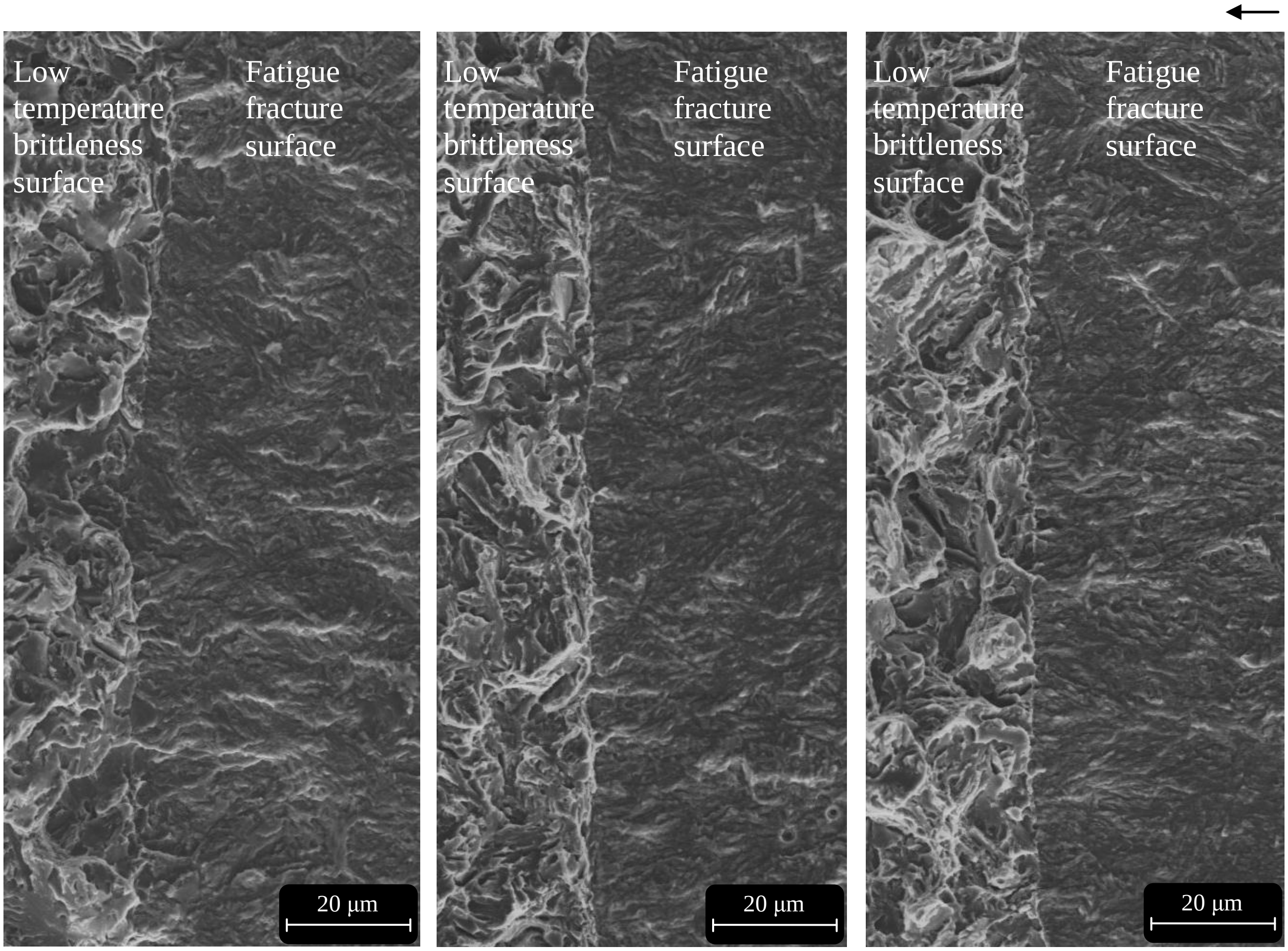


a) In-situ observation b) SEM image of fracture surface

Fig. 7. Image of preceding micro-crack in hydrogen.

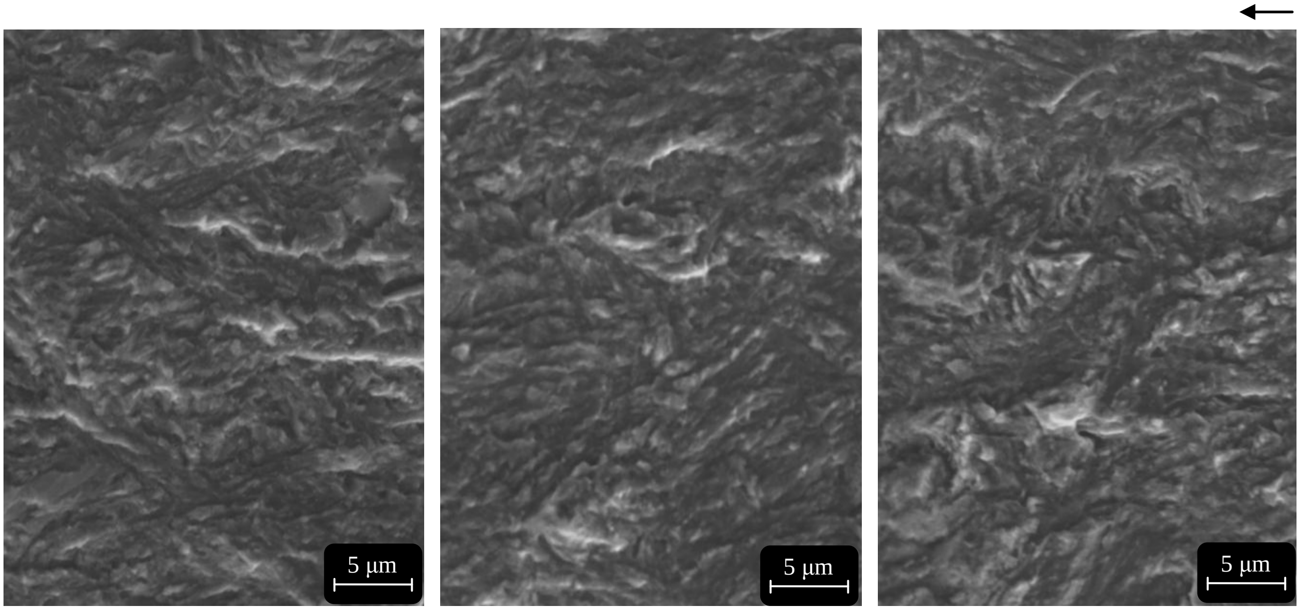
Preceding micro-crack in hydrogen. In the in-situ observation, preceding micro-crack was observed in hydrogen at ΔK = 8.26 [MPa √m]. Fig. 7 shows the image of the micro-crack. In the process of SEM observation, the point of the micro-crack was specified by the Vickers' indent and the patterns of grinding scratch. On the point of the micro-crack, facets are observed there. This result suggests that facet might form preceding micro-crack and be the starting point of fracture in extremely low rate range. Then, analysis on the crack front in hydrogen was done to find such trace. Figs. 5 b) and c) show the facets contacted with the crack front. Just like this, few facets contact with crack front, but none of these stride the borderline with low temperature brittleness surface. However, in hydrogen, it is reported that the grains forward the crack front is damaged before the intergranular fatigue crack propagation [6]. Therefore, some trace off damage might be forward the crack front. Specific analysis would be done in the future.

Matrix. Fig. 8 shows the SEM image of the crack front. In hydrogen, there are facets in extremely low rate range, however almost fracture surface is matrix. Also in hydrogen, main phenomenon in extremely low rate range occurred in matrix. Fig. 9 shows the SEM image of matrix each environment. In extremely low rate range, each matrix has similar form. However, it is reported that hydrogen atom around crack tip enhances the dislocation emission from crack tip [7]. So, there must be some distinguishing trait in matrix in hydrogen, it would be subject for the next time.



a) Hydrogen, $\Delta K = 4.46$ [MPa $\sqrt{\text{m}}$] b) Helium, ΔK_{th} c) Air, ΔK_{th}

Fig. 8. SEM image of crack front.



a) Hydrogen, $\Delta K = 4.46$ [MPa $\sqrt{\text{m}}$] b) Helium, ΔK_{th} c) Air, ΔK_{th}

Fig. 9. SEM image of matrix.

Conclusions

In order to investigate the extremely slow fatigue crack growth characteristics of JIS SCM440 CT specimen in 9 MPa hydrogen gas environment, following results are obtained.

1. (1) The FCGR in hydrogen did not show threshold behavior at ΔK_{th} in helium and the one in air.
2. (2) In hydrogen, the preceding micro-crack was observed in front of crack tip, and the intergranular facets were observed in fracture surface of the micro-crack.
3. (3) In hydrogen, the intergranular facets were observed in extremely low rate region.
4. (4) There is no facet in extremely low rate region in helium or air.
5. (5) The matrix patterns in hydrogen, in helium and in air did not have clear difference in extremely low rate region.

These results show that hydrogen still affects the fatigue crack growth in extremely low rate region. A research about the fatigue crack growth in the region of slower FCGR in hydrogen and analyzing the main factor and fracture mechanism in extremely low rate range would be done in the future.

Acknowledgements

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