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Corrosion Fatigue Cracking of Low Alloy Steel in High Temperature Water

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Fatigue crack growth test of low alloy steel was performed in high temperature water. Test parameters were dissolved oxygen content, loading frequency and R-ratio (P_{min}/P_{max}). Since the sulfur content of the steel was low, there were no environmentally assisted cracks (EAC) in low dissolved oxygen(DO) water. At high DO, the crack growth rate at R = 0.5 tests was much increased due to environmental effects and the crack growth rate depended on loading frequency and maximized at a critical frequency. On the other hand, R = 0.7 test results showed an anomalous decrease of the crack growth rate as much different behavior from the R = 0.5. The main reason of the decrease may be related to the crack tip closure effect. All the data could be qualitatively understood by effects of oxide rupture and anion activity at crack tip.

Keywords : *low alloy steel, corrosion fatigue, loading frequency, r-ratio, oxide-induced crack closure.*

1. Introduction

Interests in corrosion fatigue of pressure vessel steels arose initially from overlay cladding failures in BWR pressure vessels, notably stress corrosion cracking of cladding in a Japanese BWR[1] and thermal fatigue of pressure vessel steels at the feedwater nozzles of several BWRs.[2),3)] Since then, although the causes and remedies for the initial problems with the cladding are well understood and can easily by avoided, there has nevertheless been continued interest in characterizing EAC properties in order to arrive at an improved flaw evaluation procedure for pressure vessel.

EAC of low alloy steels and their weldments has been internationally coordinated by a group known as the 'International Cyclic Crack Growth Rate' (ICCGR) group.[4)] The results of this inter-laboratory activity have shown that there are several variables acting separately or synergistically to control the extent of environmental cracking in corrosion fatigue or slow strain rate tests on low alloy steels. These are (1) steel sulfur content, (2) water flow rate, and (3) water chemistry, especially oxygen and sulfur anion concentrations.

There are, however, morphology, water chemistry, flow rate, loading ratio(R-ratio) and frequency, which can confuse the general trend with steel sulfur content. In addition, the high R tests have become important to provide a data under low frequency and low ΔK. As yet the effect of R-ratio has not been studied in a systematic method at low frequencies. In this study, the experiments of corrosion fatigue crack growth were performed to understand the cracking process and the effect of test parameters, especially high R-ratio with loading history.

2. Experimental procedures

2.1 Material

The SA508 Cl.3 used in this study was a forged vessel steel of 10 inch thickness. In as-received condition, the vessel steel had been austenized at 880 ℃ for 7 hr and water quenched, then tempered at 655 ℃ for 9 hr followed by air cooling. Due to low sulfur content (0.002 wt%), there are a few globular inclusions scattered.

2.2 Experiment of fatigue crack growth rate

The experimental equipment for the test has been described in the previous works.[5)] In the present study, the comparable data of fatigue crack growth rate were supplemented in argon and air environments. In addition, the test at higher R-ratio were performed in air and water environments. The argon environment was made by purging high purity argon gas at the pressure of 400kPa.

2.3 Observations of fracture surface

After each test, the specimen was broken in liquid nitrogen, and then fractographic study was performed through scanning electron microscope (SEM). Some specimens were sectioned by a slow diamond cutter. The sectioned area was polished and observed to characterize the growing features of crack. Also the crack length was corrected after the test to compare with the *in-situ* measurement.

3. Results and discussion

3.1 Results of the R = 0.5 test

3.1.1 The trends of crack growth rate

In argon and air environment, the fatigue crack growth rate of SA508 alloy followed Paris' law as shown in Fig. 1(a). In the applied load range, the crack growth rate increased by two or three times at 288 °C than that at room temperature in air environment. While crack growth rate at 288°C in argon environment decreased to lower than that in room temperature air. This trend of crack growth rate may result from reduced oxidation rate which depends on the temperature and oxygen pressure.

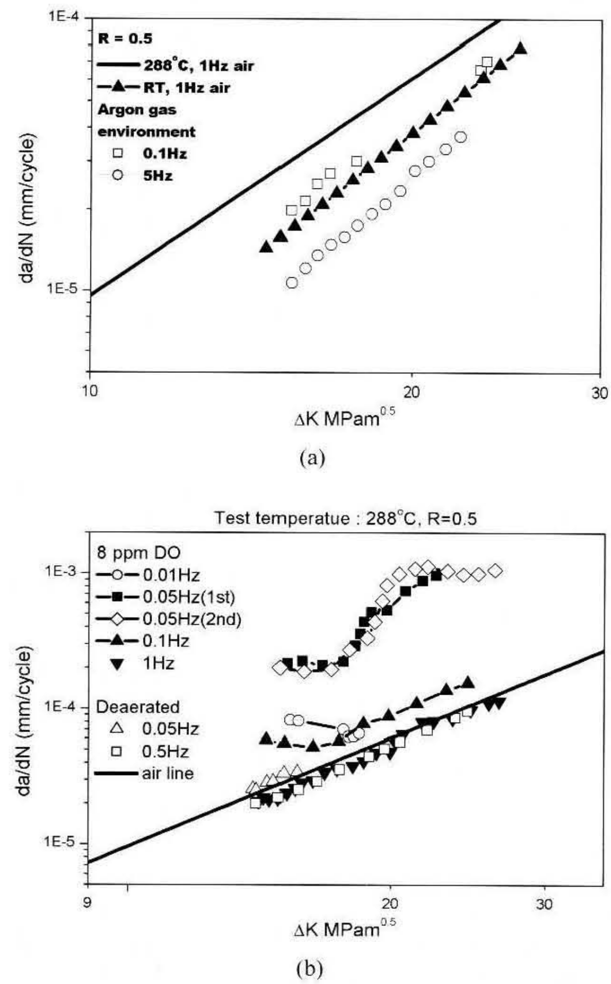


Fig. 1. Fatigue crack growth rate of SA 508 Cl.3 (a) in argon and air environment, (b) in water environment

Crack growth rate in water environment depended on the dissolved oxygen (DO) content and the loading frequency as shown in Fig. 1(b). Crack growth rate was much enhanced at high DO content, while there was little effect at low DO. The crack growth rate increased with decreasing frequency up to a critical frequency in the range of 0.01 Hz and 0.05 Hz, and the crack growth rate was diminished and became similar to that in air environment at 1Hz.

3.1.2 Fractography results

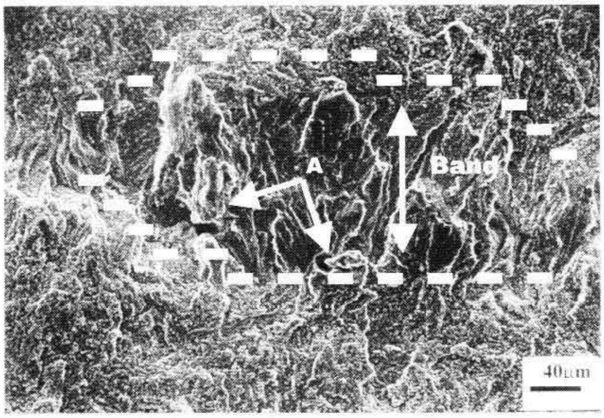
On the fracture surface there existed bands where oxide scale property was differentiated between inside and outside of the bands as shown in Fig. 2(a). The bands started at holes which would be dissolution sites of inclusions as indicated by arrow of A in the figure. Moreover near hole there was brittle facet region as shown in Fig. 2(b).

While the bands were not formed at low DO content and/or at high loading frequency although the holes were found as shown in Fig. 3. Since the trace of sulfur and Mn was detected by surface analysis on the bands with EDX in the band region and oxygen content was less than that of outside the band region,[5] the hole in the band would be a source of sulfur inclusion and oxide composition was affected by dissolution of the inclusion.

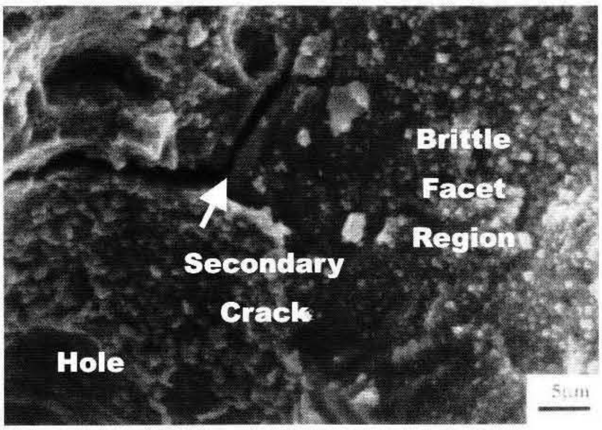
According to the detailed investigation of the oxide removed surfaces of fatigued specimens,[5] fractographic features depended on ΔK and also on test conditions such as, loading frequency and DO. The fracture features were classified by magnitude of stress intensity factor range and effective strain rate, for the case of specimen tested at 0.05 Hz and R = 0.5 in 288°C aerated water. At the site away from sulfide the brittle cracks were formed around microvoid much more at high ΔK than at low ΔK. At the sites around sulfide which would be a large inclusion in low alloy steel, brittle cracks were formed regardless of ΔK, besides the area of the brittle cracks was dependent on ΔK.

The observation of the sectioned area was shown in Fig. 4. There were several microcracks in crack tip yielding zone, and some microcracks around inclusion or precipitate were linked to the macrocrack with brittle manner. In addition, microcracks were formed by strain localization on slip bands where the arrow A indicated.

Above features of cracking may imply to a certain extent



(a)



(b)

Fig. 2. Fracture surface tested in high temperature water at air saturated condition;(a) band formation, (b) brittle facet and secondary intergranular crack at hole

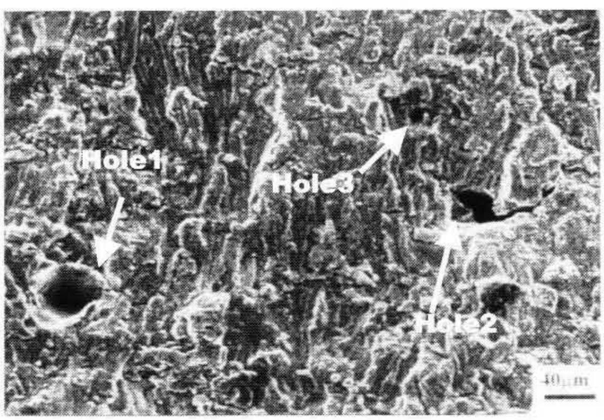


Fig. 3. Fracture surface tested in high temperature water at DO free condition

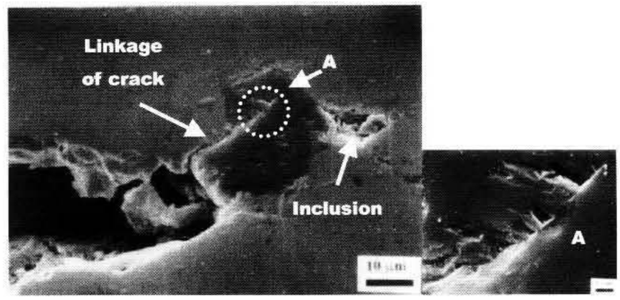


Fig. 4. Observation of sectioned area

the characteristics of hydrogen assisted cracking that brittle micro-cracks ahead of the main crack were enhanced due to hydrogen at crack tip plastic zone.[6),7)] Moreover the present cracking was equivalent to the hydrogen induced cracking mechanism proposed by Hanninen and et al.[7)] from the fractographic evidence.

3.1.3 The effects of MnS, DO content and loading frequency

Major parameters affecting on cracking could be MnS, dissolved oxygen and loading frequency. These parameters determine the crack tip conditions as interacting with each other. As shown in fractography results, dissolved MnS inclusion is a source of sulfur ion at crack tip which remains with forming oxide scale at the band in oxygenated water. At the high-oxygenated water, an activity of sulfur ion at crack tip should be higher than at oxygen free water since there is a potential drop between crack mouth and tip.[8)] The activity of sulfur at crack tip is the main rate determining parameter, since it controls the rate of metal dissolution and hydrogen evolution. This could be the cause that crack growth rate increases with the dissolved oxygen content.

However, the crack growth rate of 1 Hz in aerated water was not enhanced and the band around dissolution site was not found although the potential drop may exist. This implies that loading frequency affects the sulfur activity at crack tip and environmental effect was nearly diminished at 1 Hz. In addition, crack growth was less enhanced at 0.01 Hz condition than at 0.05 Hz. It seemed there is a critical frequency between 0.01 Hz and 0.05 Hz where the crack growth rate is maximized. Effect of loading frequency could be resulted because loading frequency affects fracture rate of oxide film, a pumping velocity of crack surfaces and a diffusion time of sulfur ion.[8)] Oxide film may act as a rate-determining parameter of metal dissolution and hydrogen absorption,[6)] which makes it difficult to differentiate experimentally between the mechanisms.

3.2 The results of the R = 0.7 test

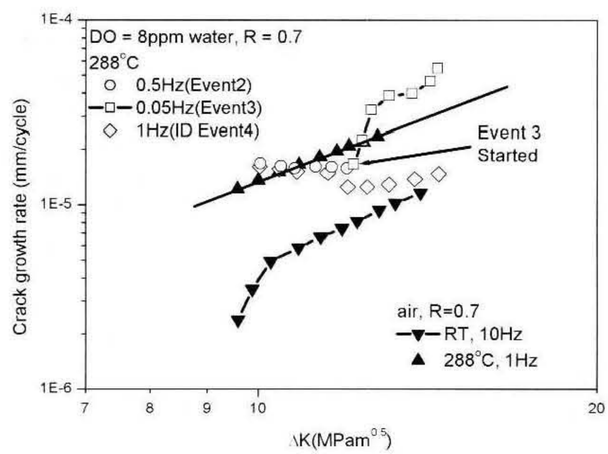
3.2.1 The trends of crack growth rate

The history of R = 0.7 tests are summarized in Fig. 5(a). For the case of specimen A, there was too little crack growth for long time (about 30 days; about 12000 cycles) to be resolved by DCPD method with the initial loading condition of ΔK of 9 $MPa \cdot m^{0.5}$ and 0.05 Hz (Event 1). After the loading frequency changed to 0.5 Hz (Specimen B), the crack grew(Event 2). As the loading frequency changed to 0.05 Hz (Event 3) from the 0.5 Hz after the crack increment where ΔK was about 12 $MPa \cdot m^{0.5}$, there was an enhancement of the crack growth. For the case of specimen C, the crack grew (Event 4) as the test started with the same ΔK of Event 1 but different loading frequency, although there was little crack growth in Event 1.

Fig. 2 shows the change of crack growth rate with stress intensity factor range for the tests. The fatigue crack growth results of air test conducted at room temperature and 288°C are included in the figure as data that any environmental growth rate enhancement in water can be identified.

R-ratio ($R = P_{min} / P_{max}$)	Specimen ID	Loading Frequency	Remarks
R = 0.7, ΔK_{start} : 9 ($MPa \cdot m^{0.5}$)	A	0.05Hz	Event 1 : No crack growth; ΔK = 9 $MPa \cdot m^{0.5}$
	B	→0.5Hz	Event 2 : Crack growth; ΔK = 9 ~ 12 $MPa \cdot m^{0.5}$
		→0.05Hz	Event 3 : Crack growth; ΔK = 12 ~ 14.5 $MPa \cdot m^{0.5}$
	C	1Hz	Event 4 : Crack growth; ΔK = 9 ~ 14.5 $MPa \cdot m^{0.5}$

(a)



(b)

Fig. 5. (a) Loading history of the R = 0.7 test, (b) Fatigue Crack growth rate of SA 508 Cl.3 at R = 0.7 in air and water environment

From the figure, there was little noticeable enhancement of the crack growth rate and the rate of crack growth was lower than that of 288°C air test in some conditions. The trends were slightly different from the those tested with R = 0.5 where the crack growth rates in water were enhanced by environmental effects or were similar at least.

It may be difficult to reach a firm conclusion with present results, because of the limited data. In addition, the tested ΔK range of R = 0.7 test was different from that of R = 0.5 test. To evaluate the effect at the same ΔK range, the specimen size for R = 0.7 test should be larger according to the ASTM criteria.[9] In the limited situations, some evident features were observed as following;

– At the low frequency of 0.05 Hz and the low ΔK of 9 $MPa \cdot m^{0.5}$, the crack growth rate was so low to be negligible in the R = 0.7 test;

– At the lower than ΔK of about 12 $MPa \cdot m^{0.5}$, there was little difference between the crack growth rates of 0.5Hz and 1Hz although the lowering trend of 1 Hz data existed in the load range;

– At the higher than ΔK of about 12 $MPa \cdot m^{0.5}$, the crack growth rate of 1Hz much decreased, meanwhile that of 0.05Hz much increased.

3.2.2 Discussion about R = 0.7 test

The trends of R = 0.7 test probably can be understood by considering the protective film rupture rate and the anion activity at crack tip at the similar manner of the R = 0.5 test as discussed above. At higher ΔK of than 12 $MPa \cdot m^{0.5}$, the crack growth rate decreased as the loading frequency increased. This could be explained by the anion activity at crack tip which can be favored in the low loading frequency.

P. M Scott et al.[10] mentioned crack growth at a low cyclic frequency could not be sustained below a critical value of ΔK independent of the applied R-ratio, and at higher cyclic frequencies apparent threshold for fatigue crack propagation was reduced to values normally observed in inert or only mildly oxidizing environments. From the Sluys et al, for each frequency, a ΔK threshold is present below which EAC growth does not exist.[11] Those are agreeable to the present result regarding Event 1 where the crack could not propagate due to the condition of the low loading frequency and the low ΔK. Moreover, at the same loading frequency and higher ΔK (Event 3), the crack growth was enhanced.

However there was an unordinary decrease of the crack growth rate in a ΔK region of R = 0.7 test, the crack growth rate in water environment was even lower than that in air. Although a closure load was not able to be measured by the system of present study, the crack closure would be a possible reason of the decrease due to oxide growth on the crack surface. The closure reducing a cracking driving force probably ensued if the oxide growth rate is relatively higher than crack growth rate and/or the oxide is thick enough to be similar to the crack tip opening displacement.

From the report of Katada et al.[12] in higher 200 ppb DO, there was a slight decrease of crack growth rate with increasing ΔK and they postulated the decrease at high DO might be caused by the reduction of effective ΔK due to the oxide-induced crack closure since the quantity of the corrosion products tend to increase in higher DO. Their loading condition was R = 0.2 and 0.0167 Hz. Their assumption would be possible due to their low R value of 0.2 which results in low crack tip opening displacement, although the crack growth rate was fast. However, because of relatively high crack opening displacement and fast propagation rate, there might be little effect of crack closure in the R = 0.5 tests of the present study.

A significant effect of prior history was observed by Sluys et al.[13]. When the high crack growth rate could not be sustained in frequency decreasing-experiments below some critical frequency level and when increasing-frequency experiments do not attain either the same high growth rates or do so only after substantial delays.[13] Although they did not mention the role of crack closure, it could be expected that some cyclic loadings should be substantial to overcome the closure at the increasing-frequency experiment, if crack closure ensued at the decreasing-frequency experiment.

On the other hand, considering the trend of the rates at the higher ΔK than of 12 $MPa \cdot m^{0.5}$ which showed that the rate of 1 Hz water test was much lower than that of 0.05 Hz water, this can not be understood by the effect of oxide induced closure and oxide rupture rate itself. The difference between the results of 0.05 Hz and 1 Hz could be related to dependence of anion activity on pumping effect of loading frequency. It is expected that anion activity could be enough to enhance the crack growth rate of 0.05 Hz due to its low pumping rate, while for the case of 1Hz it could be limited by relatively high pumping rate.

4. Summary

1. Features of cracking may imply the characteristics of hydrogen assisted cracking that brittle micro-cracks ahead of the main crack were enhanced due to hydrogen at crack tip plastic zone

2. There was a critical frequency where crack growth rate was maximized. The critical frequency ranged from 0.01 to 0.05 Hz in the R = 0.5 test over the tested ΔK range. In the R = 0.7 test, it was difficult to evaluate such critical loading frequency because the crack growth rate much depended on ΔK.

3. The crack closure problem was negligible at the R = 0.5 but had much effects at R = 0.7.

Acknowledgments

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