Effect of frequency on fatigue crack growth behavior of magnesium alloy AZ61 under immersed 3.5 mass% NaCl environment

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\textbf{Abstract}

Fatigue crack growth test of AZ61 magnesium alloy was carried out under immersed NaCl environment at frequencies of 15, 5 and 0.5 Hz under a stress ratio of 0.1. In order to investigate the effect of frequency on fatigue crack growth behavior in detail, additional tests at frequencies ranged from 15 to 0.01 Hz were conducted under a constant $\Delta K$ of 3.25 MPa m$^{1/2}$. Effect of frequency was clearly observed in low $\Delta K$ region, where fatigue crack growth rate decreased with decreasing frequency. Crack closure would be a dominant factor for the frequency effect observed under immersed NaCl environment at frequencies ranged from 15 to 0.5 Hz. However, fatigue crack growth rates at frequencies lower than 0.05 Hz were higher than those at frequencies higher than 0.5 Hz. The accelerated fatigue crack growth rates at frequencies lower than 0.05 Hz would be attributed to the corrosion attack at the crack tip.

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1. Introduction

Magnesium alloys are the lightest metal with a relatively high specific strength and stiffness, good machinability, excellent damping capacity and good recyclability. Therefore, magnesium alloys are very attractive as structural materials in order to achieve high performance and energy saving of machines and structures. Recently, there have been increasing interests in the applications of magnesium alloys in transportation industries. The main objective is to reduce the weight of vehicles that eventually leads to the energy saving and reduction of pollution.

However, the principal drawback of magnesium alloys as a structural material is their low corrosion resistance. Since many mechanically loaded parts for automobile, etc. are often subjected to prolong cyclic stresses under corrosive environment, it is very important to understand fatigue behavior of magnesium alloys under corrosive environment. Many researchers reported the deleterious effects of corrosion on fatigue behavior of magnesium alloys \cite{1-7}. Sajuri et al. \cite{4} found that fatigue strength of AZ61 magnesium alloy was reduced significantly in the high cycle region under high humidity environment. In addition, Bhuiyan et al. \cite{8} reported that fatigue life of AZ61 magnesium alloy under 5 wt% NaCl and CaCl$_2$ environments was greatly reduced when the applied stress amplitude was lower than the fatigue limit under low humidity environment. Significant reduction of fatigue life below the fatigue limit in the air was also reported by Nan et al. \cite{9} by continuously dripping 3% NaCl solution on AZ31 specimen surface during the fatigue test. All of them showed that crack was initiated from corrosion pits. Small corrosion pits were formed on the specimen surface and grew up to the critical size where stress concentration was high enough to cause the crack to initiate. Subsequently, fatigue crack was initiated and then growth from bottom of the critical pit. Since crack initiation occurred at the bottom of pit, the depth of pit was considered as the critical pit size. Sajuri et al. \cite{4} reported that the critical pit size was as small as 5 $\mu$m when AZ61 magnesium alloy was tested at stress amplitude of 145 MPa under high humidity environment. The critical pit size was found to be decreased with increasing stress amplitude. On the other hand, Nan et al. \cite{9} showed that fatigue crack was initiated from a pit with critical size of 9 $\mu$m for AZ31 magnesium alloy tested at stress amplitude of 90 MPa in 3% NaCl solution. Since in all cases fatigue cracks were initiated and then propagated from the pit, fatigue crack initiation life was found to be equal to the life of formation and growth of the corrosion pit to a critical size. The ratios of crack initiation life to the total of corrosion fatigue life were reported as low as 10%.
Fig. 1. Microstructure of extruded AZ61 magnesium alloy: (a) cross-section perpendicular to extrusion direction (average grain size = 15 μm) and (b) cross-section parallel to extrusion direction (average gain size; longitudinal direction (along the extrusion direction) = 16 μm, transverse direction = 14 μm).

[9], 30% [8] and 50% [4], where corrosion pit formation was more accelerated in NaCl environment.

It was reported that the corrosion fatigue process is composed of several stages: formation and growth of pit, crack initiation from the pit and its propagation [10]. However, fatigue fracture process only consists of crack initiation and propagation stages before final failure under non-corrosive environment. It is known that, crack initiation life in high cycle fatigue region under non-corrosive environment is late in fatigue life and more than 70% of the total fatigue life [11,12]. Therefore, under corrosive environment, fatigue crack propagation life becomes more dominant, since crack initiation life significantly reduces due to pit formation. However, most of the works in corrosion fatigue of magnesium alloys have been focused on fatigue strength, fatigue life and cyclic deformation, but there have been very limited works on fatigue crack propagation behavior under corrosive environment. Therefore, it is of primary importance to understand the effect of corrosive environment on fatigue crack propagation (FCP) behavior of magnesium alloys.

In the presence of corrosive environment, the loading frequency can influence fatigue crack growth behavior of metallic materials [13–19]. For example, a low frequency may promote synergistic effect between corrosion and mechanical loading, which may generate an acceleration of fatigue crack propagation [20–22]. However, this tendency is not always observed in some particular systems [14–17]. Meanwhile, fatigue crack growth (FCG) testing is generally carried out at the load frequency higher than that under service conditions in order to avoid the extraordinarily long testing time. This might provide inappropriate results and data for the real applications, since time-dependent corrosion process at the crack tip may affect crack propagation behavior.

Therefore, in the present study, fatigue crack growth tests of extruded AZ61 magnesium alloy were carried out in a wide range of frequencies from 15 to 0.01 Hz under immersion in 3.5 mass% NaCl solution. Furthermore, fatigue crack growth tests were also carried out under low humidity (relative humidity of 55%) without the effect of corrosive environment for comparison. The fatigue crack growth behavior was discussed based on the crack closure measurements and fracture surface analysis and observations.

2. Experimental procedures

2.1. Material

The material used in the present study was an extruded AZ61 (Mg–6%Al–1%Zn) magnesium alloy. The dimensions of as-received extruded plate were 3.0 mm × 70 mm × 1000 mm. The chemical composition and mechanical properties are summarized in Tables 1 and 2, respectively. Microstructures in the perpendicular and parallel directions to extrusion direction are shown in Fig. 1. As seen from the figure, the microstructures in both directions were not much different. The average grain size determined by the intercept method was about 15 μm.

2.2. Specimen

Fatigue crack growth (FCG) tests have been performed using 3 mm thick compact tension (CT) specimens. Details of the specimen configuration are according to ASTM E-647-95a [23], as illustrated in Fig. 2.

Prior to fatigue crack growth tests, all the specimens were polished using 600, 800, 1000, 1200 and 1500 grit emery papers and followed by 6 and 3 μm diamond paste. The specimens were then cleaned and rinsed with ethanol in an ultrasonic cleaner, and dried in air. The fatigue crack propagation direction was perpendicular to the extruded direction. The specimens were pre-cracked to a length of 9.8 mm in air. For fatigue crack growth tests under immersed 3.5 mass% NaCl environment (hereafter noted as immersed NaCl environment), a final 1.0 mm increment of pre-cracking was con-
ducted in the corrosive environment of the present study as recommended by ASTM E-647-95a [23].

2.3. Fatigue crack growth test procedure

The fatigue crack growth tests were carried out under immersed NaCl environment. The test in a controlled laboratory air at a temperature of 20 °C under relative humidity of 55% (low humidity environment) was also performed for comparison purpose. Fatigue crack growth tests were carried out using a servo-hydraulic fatigue machine equipped with a load cell of 1 kN. The specimens were subjected to cyclic load with sinusoidal waveform. The stress ratio (\(R = P_{\text{min}}/P_{\text{max}}\)) applied was 0.1. Fatigue crack growth tests to obtain full crack growth curve were conducted at test frequencies of 15, 5 and 0.5 Hz. In order to investigate the effect of frequency on the fatigue crack growth rate under wide range of frequency, additional constant \(\Delta K\) tests were carried out at test frequencies of 15, 10, 5, 0.5, 0.05 and 0.01 Hz under \(\Delta K = 3.25\) MPa m\(^{1/2}\). General corrosion on the specimen surface was not significant and did not influence on the specimen thickness even after fatigue crack growth tests at low frequency.

The schematic diagram of experimental setup for fatigue crack growth test under immersion in 3.5 mass% NaCl solution is shown in Fig. 3. The NaCl solution used in the present study was a 3.5 mass% mixture of sodium chloride (NaCl) salt and distilled water corresponding approximately to the composition of artificial seawater. The pH of the solution was maintained in a range from 6.5 to 7.2. The specimens were immersed in a pool of NaCl solution in a chamber attached to the fatigue machine. The solution was flow at constant flow rate of 0.3–0.5 l/min from a reserved tank to the chamber. The solution was continuously aerated with air bubble in the reserved tank. An amount of 1400 ml solution per day was recirculating during the fatigue crack growth tests by using a pump. New solution was used for every specimen. The temperature of the solution was controlled at 25 °C ± 2.

The crack length during the fatigue crack growth test under low humidity environment was measured by using a computer controlled elastic compliance technique with an accuracy of 1 μm and also monitored by a traveling optical microscope. Stress intensity factor range, \(\Delta K\) was calculated according to ASTM standard E-647 [23]. On the other hand, only crack length measurement by the compliance technique was applied for the fatigue crack growth tests under immersed NaCl environment.

\(\Delta K\)-decreasing and \(\Delta K\)-increasing tests under load range control condition were conducted to obtain a complete curve of FCG by using a single specimen for each frequency. Fatigue crack growth tests were first conducted by \(\Delta K\)-decreasing test under a load shedding technique until threshold stress intensity factor range, \(\Delta K_{\text{th}}\) was obtained. The threshold stress intensity factor range, \(\Delta K_{\text{th}}\) was defined as the \(\Delta K\) at which no crack growth was observed for at least \(10^6\) cycles. The decreasing load step was below 10% of the previous load. The experiments were then continued by \(\Delta K\)-increasing test under constant load range. In additional constant \(\Delta K\) tests to investigate crack growth behavior in much wider range of frequency from 15 to 0.01 Hz, load range, \(\Delta P\) was decreased as crack length increased in order to maintain a constant \(\Delta K\) value of 3.25 MPa m\(^{1/2}\). The tests were repeated at least three times for each frequency. The fatigue crack growth rate data points for each frequency were obtained by averaging the test results. During FCG test, crack closure load was determined by using an unloading elastic compliance method [24], where a strain gage was mounted on the back face of CT specimen. In order to understand the fatigue crack propagation mechanism in each environment, fracture surfaces were observed under a scanning electron microscope (SEM). Corrosion products on fracture surface of the specimen tested under immersed NaCl environment were analyzed by using an energy dispersive spectroscopy (EDS).

3. Results and discussion

3.1. Fatigue crack growth behavior

3.1.1. Fatigue crack growth test

Relationships between fatigue crack growth rate, \(da/dN\), and stress intensity factor range, \(\Delta K\), for frequencies of 15, 5 and 0.5 Hz under immersed NaCl environment are shown in Fig. 4. Fatigue crack growth curve obtained under low humidity (RH 55%) environment is also shown in the figure for comparison. As seen from the figure, fatigue crack growth curves under immersed NaCl environment were almost similar at high \(\Delta K\) region. No effect of test frequency could be observed in this region. The fatigue crack growth rate under low humidity environment was lower than those under immersed NaCl environment in this region. However, the

<table>
<thead>
<tr>
<th>Table 3</th>
<th>(\Delta K_{\text{th}}) values for each test conditions.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test environment</td>
<td>Test frequency (Hz)</td>
</tr>
<tr>
<td>Low humidity</td>
<td>5</td>
</tr>
<tr>
<td>Immersed NaCl</td>
<td>15</td>
</tr>
<tr>
<td>Immersed NaCl</td>
<td>5</td>
</tr>
<tr>
<td>Immersed NaCl</td>
<td>0.5</td>
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</table>
The effect of corrosive environment on fatigue crack growth behavior can be inversely observed at low $\Delta K$ region, where fatigue crack growth rates under immersed NaCl environment were lower than those obtained under low humidity environment. Furthermore, effect of test frequency could be observed in this region, where fatigue crack growth rates decreased with decreasing frequency. The threshold stress intensity factor range, $\Delta K_{th}$, for the frequency of 15 Hz was about 1.60 MPa m$^{1/2}$, while the test frequencies of 5 Hz and 0.5 Hz showed relatively higher $\Delta K_{th}$-values of 1.95 and 2.10 MPa m$^{1/2}$, respectively. On the other hand, the $\Delta K_{th}$-value under low humidity environment was about 1.20 MPa m$^{1/2}$, which was lower than those under immersed NaCl environment. The $\Delta K_{th}$-values for each test are summarized in Table 3.

Effective stress intensity factor range, $\Delta K_{eff}$, was determined based on the crack closure measurements. In order to understand effect of test frequency under immersed NaCl environment, the fatigue crack growth curves were rearranged by using effective stress intensity factor range, $\Delta K_{eff}$, as shown in Fig. 5. As seen from the figure, fatigue crack growth curves arranged by $\Delta K_{eff}$ were almost merged into one curve regardless of test environments and frequencies except the threshold region. This indicates that the difference in FCG rates for different environments and frequencies except the threshold region was mainly attributed to the crack closure effect. However, in the threshold region, the crack growth curves under immersed NaCl environment did not coincide with that under low humidity environment, which may suggest that corrosive environment affects and accelerates fatigue crack growth near threshold region. As can be seen from Fig. 5, the $\Delta K_{th}$ under NaCl immersed environment is lower than that under low humidity environment.

**Table 4**

<table>
<thead>
<tr>
<th>Test frequency</th>
<th>15 Hz</th>
<th>10 Hz</th>
<th>5 Hz</th>
<th>0.5 Hz</th>
<th>0.05 Hz</th>
<th>0.01 Hz</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fatigue crack growth rate (m/cycle)</td>
<td>$1.36 \times 10^{-8}$</td>
<td>$1.87 \times 10^{-8}$</td>
<td>$3.46 \times 10^{-8}$</td>
<td>$3.94 \times 10^{-8}$</td>
<td>$5.86 \times 10^{-8}$</td>
<td>$9.18 \times 10^{-8}$</td>
</tr>
</tbody>
</table>
environment for all frequencies tested. This implies that a fatigue crack under NaCl immersed environment could propagate at lower load amplitude, $\Delta P$, than that under low humidity environment.

3.1.2. Constant $\Delta K$ tests under wide range of frequency

For further investigation of frequency effect, fatigue crack growth tests at a constant $\Delta K$ of 3.25 MPa m$^{1/2}$ were carried out under wider range of frequency. The results obtained are shown in Fig. 6 and Table 4. From the figure, similar levels of fatigue crack growth rates with some scatter were found in the higher frequency region, while they were rather high at frequencies lower than 0.05 Hz.

From the crack closure measurements, relationship between crack closure ratio, $U = \Delta K_{\text{eff}} / \Delta K$, and frequency is shown in Fig. 7. From the figure, it was found that crack closure was more significant in lower frequencies.

Based on the results shown in Figs. 6 and 7, the results are plotted in the relationship between crack growth rate and effective stress intensity factor range, as shown in Fig. 8, where the scatter band of the crack growth curves for the frequencies from 15 to 0.5 Hz

![Fracture surfaces under NaCl environment](image)

Fig. 9. Fracture surfaces under low humidity environment at low and high $\Delta K$ values. Crack growth direction is from left to right.

![Fracture surfaces under NaCl environment](image)

Fig. 10. Fracture surfaces under immersed NaCl environment: (a1–a3) tested at a frequency of 15 Hz, (b1–b3) tested at a frequency of 5 Hz and (c1–c3) tested at a frequency of 0.5 Hz. Crack growth direction is from left to right.
shown in Fig. 5 is schematically indicated in the figure. As can be seen from the figure, the crack growth rates for 0.05 and 0.01 Hz were out from the scatter band and became higher with decreasing frequency.

From the foregoing results and discussion, it is suggested that there are two factors for influencing fatigue crack growth behavior. One is crack closure due to corrosion product: corrosion products will be more significantly formed on the crack surfaces under lower frequencies with longer soaking time and then result in significant crack closure. The other is acceleration of crack growth due to corrosion attack: longer soaking time of lower frequency will induce the acceleration, which is also found in threshold region under higher frequencies as seen from Fig. 5. Detailed mechanism of acceleration of crack growth due to corrosion attack is not always clear and further investigation will be required.

3.2. Fracture surface observations

3.2.1. Under low humidity (RH 55%) environment

For further investigation, fracture surfaces were observed by using an SEM. SEM micrographs of fracture surfaces tested under low humidity (RH 55%) environment are shown in Fig. 9. As seen from the figure, fracture surface at low ΔK region showed quasi-cleavage facet appearance. This suggests that quasi-cleavage fracture plays a role in fatigue crack propagation at low ΔK region. On the other hand, wavy shearing marks with slip deformation were dominantly observed on the fracture surface at high ΔK region. The presence of shearing mark indicates that slip is the main fracture mechanism at high ΔK region. As discussed in the previous investigation [25], the transition of fracture mechanism from quasi-cleavage fracture to slip deformation occurs when the maximum crack tip plastic zone size, \( R_y \) (\( R_y = 14.4 \mu m \); where \( R_y = 1/6\pi(K_{max}/\sigma_y)^{1/2} \)) is approximately equal to the grain size (15 μm).

3.2.2. Under immersed NaCl environment

Fracture surfaces observed just after the fatigue crack growth test under immersed NaCl environment are shown in Fig. 10. As seen from the figure, fracture surfaces at lower ΔK values were fully corroded for all frequencies. Fracture surfaces at higher ΔK values were less corroded compared to those observed at lower ΔK region. On the other hand, fracture surfaces became less corroded as frequency increased for all ΔK values. Consequently, similar fracture surface appearance to that under low humidity environment could be observed for the specimens tested at higher ΔK region under higher frequency of 15 Hz. These results show that the longer exposure time due to low ΔK with very low crack growth rate as well as low frequency results in more corrosion products on the fracture surfaces. These fracture surface observations did not contradict with the crack growth behavior shown in Fig. 4. In higher ΔK region, fatigue crack growth curves for each frequency almost coincided with each other due to less corrosion products and then similar crack closure level. In lower ΔK region, fatigue crack growth
Fig. 12. EDS analysis of fracture surface tested at a frequency of 0.5 Hz and \( \Delta K = 2.33 \text{ MPa m}^{1/2} \) under immersed NaCl environment: (a) element counts and (b) element mapping.

Fig. 13. Cross-sectioned through the fracture surface. Corrosion product layer developed on the fracture surface of the specimen tested at a frequency of 0.5 Hz and \( \Delta K = 2.33 \text{ MPa m}^{1/2} \) under immersed NaCl environment.

curves depended on frequency: fatigue crack growth rate became high with increasing frequency, which might correspond to less corrosion products and then less crack closure effect at higher frequency.

Fracture surfaces for the constant \( \Delta K = 3.25 \text{ MPa m}^{1/2} \) test are shown in Fig. 11. As seen from the figure, fracture surfaces became more corroded as the frequency decreased. Especially, at frequencies of 0.05 and 0.01 Hz, corroded and more flat surface without slip deformation marks could be observed. This may be induced by corrosion attack at the crack tip, which may result in the acceleration of crack growth.

The result of EDS analysis of the fracture surface for the specimen tested at \( \Delta K = 3.25 \text{ MPa m}^{1/2} \) and a frequency of 0.5 Hz under immersed NaCl environment is shown in Fig. 12. It was suggested from the figure that the corroded surface would be covered with condensed NaCl and Mg(OH)$_2$ products. The formation of corrosion products layer on the fracture surface was confirmed by SEM observation on the cross-sectioned through the fracture surface as shown in Fig. 13.

4. Conclusions

In order to understand effect of frequency on fatigue crack growth behavior of AZ61 magnesium alloy under corrosive environment, fatigue crack growth tests were carried out under immersed NaCl environment at various frequencies. From the results obtained, the following conclusions are summarized:

1. The effect of corrosive environment on fatigue crack growth behavior could be clearly observed in low \( \Delta K \) region, which was characterized by the fatigue crack growth rates lower than those obtained under low humidity environment without the effect of corrosive environment. The increase in fatigue crack growth resistance under immersed NaCl environment was attributed to the crack closure effect.

2. The effect of frequency was clearly observed in low \( \Delta K \) region, where fatigue crack growth rates decreased with decreasing frequency. The fatigue crack growth curves at frequencies ranged from 15 to 0.5 Hz were merged into one curve with some scatter when the curves were arranged by \( \Delta K_{eff} \). Therefore, the crack closure would be a dominant factor for the frequency effect under immersed NaCl environment.

3. From the results of constant \( \Delta K = 3.25 \text{ MPa m}^{1/2} \) crack growth tests in wider range of frequency from 15 to 0.01 Hz under immersed NaCl environment, the fatigue crack growth rates at frequencies ranged from 15 to 0.5 Hz were in the scatter band of the crack growth curves arranged by \( \Delta K_{eff} \). However, fatigue crack growth rates at frequencies lower than 0.05 Hz were higher than those at frequencies higher than 0.5 Hz. The corrosion attack at the crack tip would be contributed to the accelerated fatigue crack growth rates at frequencies lower than 0.05 Hz.

References