Corrosion fatigue behavior of extruded magnesium alloy AZ61 under three different corrosive environments

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Corrosion fatigue process of extruded AZ61 magnesium alloy has been investigated under three different corrosive environments: (a) high humidity environment (80% relative humidity), (b) 5 wt% NaCl solution environment, and (c) 5 wt% CaCl₂ solution environment. The fatigue strength drastically reduced under the three different corrosive environments: the reduction rates of fatigue limit under high humidity, NaCl and CaCl₂ environments were 0.22, 0.85 and 0.77, respectively. The drastic reduction in fatigue limit under corrosive environments resulted from pit formation and growth to the critical size for fatigue crack nucleation. It is suggested that the NaCl environment enhances pit formation and growth more than the CaCl₂ environment, due to the high Cl⁻ concentration and low pH value. It is also found that the corrosion pit grows during fatigue cycles and the fatigue crack starts when the pit reaches a critical size for crack nucleation. The critical size is attained when the stress intensity factor range reaches the threshold value. The ratio between pit growth life to fatigue crack nucleation and total fatigue life was about 30%.

1. Introduction

Magnesium alloys are now increasingly used for casings of electronic products, vibrating plates of vibration test machines, wheels, etc., wherever the weight is the major concern. This is attributed to their inherent superior properties such as very low density, high specific strength, excellent castability and machinability, good damping capacity and so on. The demand for use of magnesium alloys as structural materials in automobile industry has increased in recent years. The use of magnesium alloys in automotive reduces the total weight of the component up to 20%. At the same time this will substantially reduce the fuel consumption and the CO₂ emissions. In order to apply the magnesium alloy to high strength structural components in automobile, aerospace and other industries, the characterization of fatigue properties is highly required.

Corrosion fatigue may be defined as the combined action of an aggressive environment and a cyclic stress leading to premature failure of metals by cracking [1,2]. Several surveys of corrosion failures in different industries have shown that 20–40% of the failures experienced have been due to corrosion fatigue [3,4]. Since many mechanically loaded parts are often subjected to prolonged cyclic stresses in an active medium, it is of significant importance to study the corrosion fatigue characteristics of magnesium alloys.

Wet argon has been found to decrease the fatigue limit of cast magnesium alloy AZ91D and AM60B [5,6]. It should be noted that most of these studies were performed on die cast materials, which include casting defects such as pores. It is known that the pores significantly affect the fatigue strength as stress concentrators, and also that their size, number and shape depend on the fabrication process and condition. Therefore, it is important to investigate the fatigue behavior of pore-free materials such as extruded materials to understand basic fatigue behavior of magnesium alloys. Hilpert and Wagner [7] examined the fatigue behavior of extruded high strength AZ80 magnesium alloy under a spray of aqueous NaCl solution. The fatigue life was considerably reduced at stresses below the fatigue limit under ambient air, while no significant effect of NaCl solution on fatigue life was found at high stress amplitudes. Unigovski et al. [8] carried out the corrosion fatigue tests of extruded AZ31 and AZ91D magnesium alloys under aggressive environment as well as die cast AM60 alloy and reported that extruded alloys showed a significantly longer fatigue life in both air and NaCl solution in comparison with die-cast alloys. Sajuri et al. [9] studied the fatigue behavior of extruded AZ61 magnesium alloy under tension–compression loading and reported that the fatigue behavior of AZ61 magnesium alloy was highly sensitive to the humidity under the ambient environment. However, number of
research works on corrosion fatigue of magnesium alloys has been still limited. Detailed corrosion fatigue processes and effects of frequency, stress ratio, etc. have not yet been fully understood. Systematic and basic study on corrosion fatigue of magnesium alloys has been highly requested.

As corrosive environments, both NaCl and CaCl₂ environments are used, where NaCl environment can simulate the seashore atmosphere and CaCl₂ is a deicing agent for traffic roads. So, it is important to know the corrosion fatigue behavior of CaCl₂ environment as well as NaCl environment. Therefore, in the present study, as the first step of the systematic study, for understanding basic corrosion fatigue processes of magnesium alloys, fatigue tests of extruded AZ61 magnesium alloy were carried out under three different corrosive environments: (a) high humidity (80% relative humidity (RH)), (b) sprayed 5 wt% NaCl solution environment, and (c) sprayed 5 wt% CaCl₂ solution environment. The extruded material was selected for avoiding the influence of casting defect on fatigue behavior.

2. Experimental procedure

2.1. The material

The material used in the present study was an extruded AZ61 magnesium alloy. The chemical composition and the tensile properties of the AZ61 magnesium alloys are shown in Tables 1 and 2, respectively. The microstructure of the as-received extruded AZ61 magnesium alloy bar (16 mm in diameter) was observed under a digital microscope, as shown in Fig. 1. The grain structures were uniform and equi-axed. Sajuri et al. [10] observed the microstructure of extruded AZ61 magnesium alloys in three different planes: longitudinal, transverse and 45° to extrusion direction and concluded that no difference in grain structures among the three different planes was observed. The specimen in 45° direction from the extruded direction showed lower tensile strength compared to those in the longitudinal and transverse directions, whereas the fatigue strength of the specimen in the longitudinal direction was higher compared to those for the specimens in the transverse and 45° directions. Moreover, they found that texture introduced by extrusion does not significantly influence on fatigue crack growth behavior.

2.2. The choice of the specimen

Preliminary fatigue tests were performed on the specimens with 6 mm gauge diameter and 40 mm gauge length as per ASTM E 466 under high humidity (HH) condition (relative humidity was about 80%), which resulted in failure of the threaded end region of specimen, as shown in Fig. 2. Also evidence of corrosion products was found at the failure location. Such failure has not been observed in other structural materials like steels, aluminum alloys and so on. In the present experiment, the magnesium alloy specimen was coupled with stainless steel grips. Since magnesium is ranked the lowest among the metals in the electromotive force (emf) and galvanic series, galvanic corrosion due to potential differences between magnesium alloy and stainless steel could be induced in the magnesium alloy specimens. The combination of low corrosion resistance and high notch sensitivity of magnesium alloy might have promoted the observed failure not in the gauge part but in the threaded region of specimen. In order to avoid the failure in the threaded region, systematic modification of specimen

<table>
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<th>Table 1</th>
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<tr>
<td>Chemical composition of the AZ61 magnesium alloy used (mass%)</td>
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<tr>
<td>Al</td>
</tr>
<tr>
<td>5.95</td>
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<th>Table 2</th>
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<tr>
<td>Mechanical properties of the AZ61 magnesium alloy used</td>
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<tr>
<td>Yield stress, (\sigma_{0.2}) (MPa)</td>
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<td>AZ61</td>
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Fig. 1. Microstructure of as-received AZ61 magnesium alloy.

Fig. 2. Specimen with 6 mm gauge diameter failed from the thread.
geometry (in terms of decreasing gauge diameter and corresponding gauge length) was carried out. It was finally found that the specimen with 3 mm gauge diameter and 6 mm gauge length provided fatigue failure in the gauge part. Therefore, this specimen geometry showed in Fig. 3 was adopted for all the fatigue tests in the present study.

The round bar fatigue specimens with threaded ends were machined from the as-received round bar. After machining, the specimens were polished in the loading direction with 280–800 grit emery papers in laboratory air and with 1000–1500 grit emery papers under kerosene oil to prevent corrosion of the specimen surface during polishing process. After all polishing processes were completed, the specimens were cleaned by ethanol in an ultrasonic cleaner.

2.3. Fatigue test

Tension–compression fatigue tests were conducted on a servohydraulic fatigue testing machine using a sinusoidal waveform with a frequency of 20 Hz and a stress ratio of −1 under three different corrosive environments; (a) high humidity environment (80% RH), (b) sprayed 5 wt% NaCl solution environment with a pH value of 6.59 (noted as NaCl environment) and (c) sprayed 5 wt% CaCl₂ solution environment with a pH value of 8.07 (noted as CaCl₂ environment). The fatigue tests for obtaining the basic fatigue properties of AZ61 magnesium alloy with no influence of corrosion were carried out under low humidity environment (at 16–20 °C with 35–40% RH). In order to examine the influence of humidity, fatigue tests were conducted under high humidity (80% RH) in a specially designed chamber which can control the relative humidity level ranging from 30% to 80% RH. Fatigue tests under the NaCl and CaCl₂ environments were conducted in a 31.5 × 26.5 × 17 cm³ chamber by spraying the solution with a flow volume of 1.6 ml/h at 0.1 MPa air pressure, where the temperature of solution was 25.1 °C and the temperature inside the chamber was 25.4 °C. The fatigue test was continued up to complete failure of the specimen, while the test was stopped when the specimen did not fail up to 10⁷ cycles.

Specimen surfaces and fracture surfaces of the specimens were observed in detail by using a scanning electron microscope (SEM) to investigate the pitting process and the fracture surface morphology under three different corrosive environments.

3. Results and discussion

3.1. S–N curve

The relationships between stress amplitude and number of cycles to failure under three different corrosive environments are shown in Fig. 4. The result under low humidity condition (35–40% RH), where no corrosion fatigue took place, is also indicated in the figure. The extruded AZ61 magnesium alloy indicated a fatigue limit of 135 MPa under the low humidity condition. The fatigue limit was reduced to 105 MPa in the high humidity environment. However, no significant difference in fatigue life between low and high humidity conditions was observed at stress amplitudes higher than the fatigue limit under the low humidity. A drastic reduction in fatigue limit was observed in the NaCl and CaCl₂ environments. In the NaCl environment, the fatigue limit was 20 MPa, while it was 30 MPa in the CaCl₂ environment. The NaCl environment is more detrimental for the fatigue limit of the AZ61 magnesium alloy compared to the CaCl₂ environment, while
the difference of fatigue limit was not much significant. The effect of corrosive environment on fatigue strength was evaluated by using the reduction rate of fatigue limit (RRFL), as shown in Table 3, where RRFL is given as

\[
RRFL = \frac{\sigma_{LH} - \sigma_{CE}}{\sigma_{LH}}
\]

where \(\sigma_{LH}\) is the fatigue limit under low humidity condition and \(\sigma_{CE}\) is that under corrosive environment.

It can be seen from the table that the NaCl environment reduces the fatigue limit four times and the CaCl2 environment reduces it 3.5 times more than the high humidity (80% RH) environment. It has been reported that Chloride ions promote rapid attack of magnesium in neutral, aqueous solutions. The corrosion rate increases rapidly with increasing Cl⁻ concentration [11]. Extruded magnesium alloys with 3–8% Al and 0.5–0.8% Zn are susceptible to film-form corrosion and pitting corrosion in aqueous chloride solutions depending on chloride concentration [12]. In the present study, in 5% NaCl solution the Cl⁻ concentration is 0.85 mEq/L (millequivalents per liter), where as in CaCl2 solution it is 0.45 mEq/L, indicating that the Cl⁻ concentration in NaCl is almost double that compared to the Cl⁻ in CaCl2. This high Cl⁻ concentration in NaCl solution makes the NaCl environment more detrimental than the CaCl2 environment. On the other hand, it is also reported that corrosion fatigue life of magnesium alloys depends highly on the pH values of the solution [13]. The pH value of the medium has an important impact on the corrosion morphology of magnesium alloys. Magnesium is very resistant to corrosion by alkalis of the pH exceeds 10.5 which corresponds to the pH of a saturated Mg(OH)₂ [14]. The corrosion rate of AZ91 ingot and die cast is high in acidic solutions (pH 1–2) as compared to that in neutral and highly alkaline solutions (pH 4.5–12) [15]. Based on these study we may speculate that since the pH (6.59) of NaCl is lower than the pH (8.07) of CaCl2, the NaCl is more corrosive than the CaCl2 environment. Therefore, on the basis of the above discussion, we can say that due to the combine effect of the Cl⁻ concentration and the pH value, NaCl environment is more detrimental than the CaCl2 environment.

By using Eq. (1), similar results have been obtained on AZ280 in NaCl solution (RRFL = 73%) [7], extruded AZ31 and AM50 magnesium alloys in NaCl solution (RRFL = 34% and 27%, respectively) [8], extruded AZ61 under high humidity (RRFL = 27%) [9], cast AZ91E in 3.5% NaCl aqueous environment (RRFL = 83%) [16], electro polished AZ31 and AZ80 in NaCl environment (RRFL = 27% and 81%, respectively) [17]. From these comparisons, it seemed that, fatigue strength under NaCl environment of high strength magnesium alloys like AZ91E, AZ80 degraded more than those of low strength magnesium alloys like AZ31, while the fatigue data for magnesium alloys specially under NaCl environment fell in a wider scatter band (RRFL = 27–83%).

### 3.2. Fractographic observations

SEM fractographs for the specimen tested at stress level of 165 MPa under low humidity condition (36–40% RH) are shown in Fig. 5. The crack nucleation region is shown by a rectangular mark in Fig. 5a and is shown at high magnification in Fig. 5b. The crack nucleation region (Fig. 5b) shows no existence of foreign particles such as inclusions and also no defect on the specimen surface. The crack nucleation region was relatively flat with transgranular fracture, which was commonly observed in smooth specimens of other structural metals. Fig. 6a and b shows SEM fractographs for the specimen tested under high humidity condition (80% RH) at stress amplitude of 155 MPa (higher than the fatigue limit under low humidity condition). The crack nucleation region revealed the similar features as found under low humidity condition. However, under high humidity environment, as the stress amplitudes become lower than 135 MPa (the fatigue limit under low humidity condition), the failure of the specimen was attributed due to the formation of corrosion pit. Fig. 6c and d shows the presence of corrosion pit in the crack nucleation region on the specimen surface tested under high humidity condition (80% RH) at stress amplitude of 115 MPa. Fig. 7 shows fracture surfaces of the specimen tested under the NaCl environment at stress levels of 130 MPa (Fig. 7a and b) and 50 MPa (Fig. 7c and d). High magnification of the crack nucleation site revealed the existence of corrosion pit. Moreover, the specimen surfaces and the fracture surfaces tested under the NaCl environment were covered with corrosion products. Fig. 8 shows fracture surfaces of the specimens tested

![Fig. 5. SEM fractographs showing: (a) overview of the fracture surface and (b) high magnification of the crack nucleation region tested at 165 MPa under the low humidity condition.](image-url)
Fig. 6. SEM fractographs showing: (a) overview of the fracture surface and (b) high magnification of the crack nucleation region tested at 155 MPa; (c) overview of the fracture surface and (d) corrosion pit at the crack nucleation point on the specimen surface tested at 115 MPa under the high humidity condition.

Fig. 7. SEM fractographs showing: (a) overview of the fracture surface and (b) existence of corrosion pit at the crack nucleation site tested at 130 MPa; (c) overview of fracture surface and (d) existence of corrosion pit at the crack nucleation site tested at 50 MPa under the NaCl environment.
at 110 MPa (Fig. 8a and b) and 50 MPa (Fig. 8c and d) under the CaCl₂ environment. As can be seen from the figure, corrosion pits were also found at the crack nucleation site.

3.3. Pit growth behavior

Since NaCl environment is more detrimental than CaCl₂ environment (as shown in Table 3), the pit growth behavior was observed under this environment by performing interrupted fatigue test. At a high stress amplitude of 120 MPa, the isolated single pit was found to form and grow, as shown in Fig. 9. The size of pit found on the specimen surface was about 7.5 µm at 10,000 cycles (28% of total life), which increased to 24 µm at 30,000 cycles (83% of total life). Therefore, the isolated pit is formed and grows to a critical size for starting a fatigue crack at high stress level. Similar pit growth behavior under high humidity environment was reported on AZ61 alloy [9], where the stress level was rather high. It is known that extruded magnesium alloy indicates metallographic anisotropy. However, clear effect of anisotropy on pit geometry was not observed in the present experiment. In order to investigate the interactive action of cyclic loading and corrosive environment, one of the specimen was placed without loading under NaCl environment.
environment up to the time equivalent to 432,510 cycles. It was found that without loading under NaCl environment, few corrosion pits nucleated and grew on the specimen surface, the average size of which was about 28 μm. On the other hand, under simultaneous action of cyclic loading and NaCl environment, at 80 MPa one of specimens failed after 305,540 cycles which corresponds to the closest life duration of that without cyclic loading and the pit size at the fracture origin was about 70 μm, as shown in Fig. 10. These observations suggested that formation and growth of corrosion pits under corrosive environment were significantly enhanced by cyclic loading. In order to compare the pit growth behavior at low stress amplitudes, a similar interrupted fatigue test under NaCl environment at a low stress amplitudes of 50 MPa was conducted. Fig. 11 shows the surface observations of the specimens interrupted at various loading cycles. As can be seen from the figure that, after 50,000 cycles (2.8% of total life), corrosion pits were found on the specimen surface (Fig. 11a). The number of corrosion pits was increased with increasing number of cycles. However, the formation sites of corrosion pits were not uniform but localized in some area, as seen from Fig. 11b. Coalescence of corrosion pits in the localized area was found to form a large pit as further increase of cycles, as found in Fig. 11c and d. Therefore, pit formation process under NaCl environment at low stress amplitudes is considered as follows: multiple corrosion pits nucleated in some localized area on the specimen surface, number of pits increased with increasing number of cycles and finally localized dense pits coalesced to form a large corrosion pit. This large coalesced pit could become a fatigue crack starter at very low stress amplitude.

### 3.4. Fatigue crack nucleation life

Fatigue crack nucleation life after crack nucleation from a pit can be predicted based on the Paris equation modified by the threshold stress intensity factor range, as follows:

\[
\left(\frac{da}{dN}\right)_{\text{crack}} = C(\Delta K^m - \Delta K_m^m)
\]

\[
\Delta K = \sigma \Delta \sqrt{\pi a}
\]

\[
N_p = \int_{a_0}^{a_f} \frac{1}{C(\sigma \Delta \sqrt{\pi a})^{m} \bar{a}^2 - C\Delta K_m^m} \, da
\]

From Ref. [18] C and m has been taken as 2.0 × 10^{-10} m cycle^{-1} and 3.0.

Therefore, the crack nucleation life \(N_i\) can be obtained as follows:

\[
N_i = N_f - N_p
\]

By using Eq. (4), the crack propagation life was evaluated as 211,137 cycles at stress amplitude of 80 MPa under NaCl environment. Therefore, the crack nucleation life at this stress amplitude was 94,403 cycles (using Eq. (5)). Therefore, the crack nucleation life is expected to be almost 30% of total fatigue life \(N_i\), which is rather low compared to the reported crack nucleation life (about 50% of the total fatigue) [9,18].

### 3.5. Relationship between stress amplitude and critical pit size

It can be considered that the pit size observed at the fracture origin is the critical pit size for fatigue crack nucleation. Mechanical stress is a major factor for the pit formation and growth under corrosive environment as seen from Fig. 10. Therefore, once a fatigue crack starts from the pit, stress around the pit is released and further pit growth is not significant. The pit size was defined as the radius of semi-circular crack [19], which had the equivalent area to the pit observed at the crack nucleation site. From the detailed fractographic observations, the relationships between stress amplitude and critical pit size under the NaCl and the CaCl₂ environments are shown in Fig. 12. As can be seen from the figure, the relationship under the NaCl environment is almost a straight line for all stress amplitudes tested except for stress amplitudes higher than 130 MPa, which indicates the so-called Kitagawa-Takahashi diagram for explaining small crack behavior. The straight line gives the threshold stress intensity factor range as 0.8 MPa√m. Therefore, this linear relationship suggests that a fatigue crack starts from a pit when the stress intensity factor range reaches the threshold value. A similar hypothesis has been proposed by Hoeppner [20]. The relationship under the CaCl₂ environment is also almost a straight line in the range of stress amplitudes lower than 100 MPa, which also gives the threshold stress intensity factor range of 0.8 MPa√m. However, at stress amplitudes higher than 100 MPa, the relationship shows a deviation.
from the linear line, which also indicates the so-called Kitagawa–Takahashi diagram similar to that under the NaCl environment. The similar tendency is observed under high humidity condition, as indicated in Fig. 12.

In the small crack region, applying the El Haddad equation [21–23] which is given as

$$\Delta K_{th} = x \Delta K_{th} \sqrt{\pi(a + a_0)}$$

(6)
where \( a_0 \) is a constant known as “an effective crack length” and the value of \( a_0 \) can be obtained by

\[
a_0 = \frac{1}{\pi} \left( \frac{\Delta K_{th}}{\sigma_e} \right)^2
\]

(7)

In the small crack problem without influence of corrosion, \( \sigma_e \) has been taken as the fatigue limit \( \sigma_{ow} \) as found in the El Haddad equation. This can be interpreted in the following way: the \( \sigma_{ow} \) gives the lowest stress, below which fatigue failure (crack nucleation and propagation) cannot occur without pre-existence of small crack (or defect). In the present study, \( \sigma_e \) is considered as the lowest stress, above which the corrosion pit is not the crack nucleation site and has no influence on fatigue crack nucleation. If the fatigue strength without influence of pit \( \sigma_e \) is assumed as 140 MPa under high humidity environment, 200 MPa under NaCl environment and 160 MPa under the CaCl₂ environment and \( z \) as 0.67 for semicircular crack, the relationship between stress amplitude and critical pit size in the small crack region can be obtained by the El Haddad equation, as shown in Fig. 12. It can be found from the figure that the data points under the three different corrosive environments follow the El Haddad equation. It is also suggested that the NaCl environment enhances pit formation and growth more than the CaCl₂ environment, since the fatigue strength without influence of corrosion pitting is higher under the NaCl environment, as seen from Fig. 12.

From the foregoing discussion, it is found that the corrosion pit grows during fatigue cycles and the fatigue crack starts when the pit reaches a critical size for crack nucleation. The critical size is attained when the stress intensity factor range reaches the threshold value not only for long crack region but also for small crack region, where the El Haddad correction has to be taken.

![Fig. 13. S–N curves for steel, aluminum alloy and magnesium alloy (a) under ambient air and NaCl environment. (b) Normalized S–N curves for steel, aluminum alloy and magnesium alloy.](image-url)
3.6. Comparison of corrosion fatigue strength

Corrosion fatigue strengths of structural materials other than magnesium alloy are available because of importance of corrosion fatigue problems. For example, Lin et al. [24] studied the fatigue behavior of 7050-T73 high strength aluminum alloys in both laboratory air and 3.5% NaCl solution environment with a frequency of 20 Hz and reported that the fatigue strength of the alloy in NaCl environment was reduced to 67.4% relative to the fatigue strength in the low humidity environment. Tokaji et al. [25] also studied the fatigue behavior of low alloy steel (Cr–Mo) under both laboratory air and 3% NaCl environment with a frequency of 19 Hz and reported a large reduction of fatigue strength (50–80%) under NaCl environment. The present work on magnesium alloy also shows a similar trend of reduction of fatigue strength under corrosive environment, as shown in Fig. 13a. In all the cases, the reduction in fatigue strength under NaCl environment was attributed to the formation of corrosion pits. Since the materials plotted in Fig. 13a has different ultimate tensile strengths (σ_b), the stress amplitude was normalized by the ultimate tensile strength of each material. The resultant normalized plot is shown in Fig. 13b. It is found from the figure that under NaCl environment, S–N curves for all three materials fall in the same data band. This means that the difference in the corrosion fatigue strength shown in Fig. 13a primarily results from the difference of ultimate strength, despite the fact that the pit formation and growth behavior and threshold stress intensity factor of each material are different.

4. Conclusions

Fatigue tests were carried out under three different corrosive environments and also in air to understand the basic corrosion fatigue processes of AZ61 magnesium alloy. From the results obtained, the following conclusions are summarized:

1. The fatigue limit under low humidity (36–40% relative humidity) with no effect of corrosion was obtained at 135 MPa and the ratio of fatigue limit to tensile strength was about 0.42. Sajuri et al. [9] also found that the ratio of fatigue limit to tensile strength was about 0.45 under low humidity environment.
2. The fatigue limit was reduced to 105 MPa under high humidity (80% RH). The reduction rate of fatigue limit (RRLF) due to high humidity was 0.22, which indicates that the fatigue strength of AZ61 magnesium alloy is highly sensitive to the humidity level of ambient environment.
3. The fatigue limits under the NaCl and CaCl_2 environments were 20 and 30 MPa, respectively. The RRLF values under the NaCl and CaCl_2 environments were 0.85 and 0.78, respectively.
4. The drastic reduction in fatigue limit under corrosive environments resulted from pit formation and growth to the critical size for fatigue crack nucleation. Corrosion pit formation and growth were attributed to the interactive action of mechanical loading and corrosive environment. It was found that at low stress amplitudes under corrosive environments, multiple corrosion pits nucleated on the specimen surface, grew and then coalesced to form a large corrosion pit. On the other hand, an isolated pit was formed and grew to the critical size at higher stress amplitudes. The ratio between pit growth life to fatigue crack nucleation and total fatigue life was about 30%, which is similar to that reported in Refs. [9,18].

References

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