Scanning probe microscope observations of fatigue process in magnesium alloy AZ31 near the fatigue limit

Z.Y. Nan, S. Ishihara *, T. Goshima, R. Nakanishi

Department of Mechanical Engineering, Toyama University, 3190 Gofuku, Toyama 930-8555, Japan

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Abstract

In this study, fatigue crack initiation and propagation behavior of magnesium alloy AZ31 within a crystal grain near the fatigue limit were investigated in detail using a scanning probe microscope (SPM). At a stress amplitude slightly higher than the fatigue limit, cracks are arrested by the grain boundary for an extended period.

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Keywords: Magnesium alloy; Scanning probe microscope; Fatigue; Crack initiation; Crack propagation

1. Introduction

Magnesium alloys are very attractive as structural materials, because they are extremely light, possessing excellent specific tensile strength, good stiffness and vibrational absorption [1]. However, magnesium alloys have not been used extensively until recently because of their vulnerability to corrosion and their high cost. Now, due to their energy and weight saving characteristics, magnesium alloys are considered to be good candidates as materials in, for example, auto parts, portable personal computers and telephones. When considering the use of magnesium alloys as structural materials, a thorough understanding of the fatigue characteristics is necessary.

The fatigue process of smooth metallic materials can be divided into two periods—crack initiation and propagation periods. Much research has been done on comparatively long fatigue crack propagation characteristics. It has been demonstrated that long fatigue crack propagation behavior can be accurately evaluated using linear elastic fracture mechanics. However, the behavior of small fatigue crack initiation and propagation, especially of the crystal grain order in the early stage of fatigue life has not yet been well documented.

The authors studied the material characteristics of extruded magnesium alloy AZ31, and showed that the material consists of two kinds of lamellar structures, namely phases A and B. The latter, phase B, further consists of the crystal grain, phase C. During the fatigue process, micro-cracks initiate at the early stage of fatigue life from both the top and bottom edges of the crystal grain (phase C) [2].

In this study, the crack propagation process within a grain size, initiating from the edge of the crystal grain (phase C) was investigated using a scanning probe microscope, SPM. By utilizing the SPM, the material structure can be studied with a resolution of 0.1 nm [3–5], and three-dimensional material deformations could also be investigated. Special attention was paid to describing how the crack initiated and propagated within a crystal grain (phase C) and how the crack propagation was arrested at the grain boundary under stress amplitude near the fatigue limit.

2. The material and experimental methods

2.1. The material

The magnesium alloy AZ31 used in the present experiment was obtained by extruding a billet with a diameter of 88.9 mm into a round bar 19 mm in diameter. Its chemical composition and mechanical properties are listed in Tables 1 and 2, respectively. Fig. 1 shows a photograph of the material microstructure of the plane which is parallel to the extrusion direction.
A schematic illustration is appended to the figure on the right side of the figure for descriptions. As seen from the photograph, the microstructure consists of two types of lamellar structures, the white part (phase A) and the black part (phase B). Both lamellar structures are layered alternately and parallel to the extrusion direction. In addition, a separate crystal structure (phase C) can be observed inside the B phase. Analysis of the composition of each phase was done [2] using Electron Probe Micro Analyzer (EPMA, Shimadzu, EPMA-1500) and X-ray diffraction equipment (Rigaku, RINT2200/PC/K). The EPMA plane analysis revealed that the white part (phase A) is pure magnesium, the black part (phase B) is the intermetallic compound (Mg–Al–Zn system), and the C phase is the intermetallic compound (Mg–Al system). Identification using X-ray diffraction showed that phases B and C were Mg₃₂(Al, Zn)₄₉ and Mg₁₇Al₁₂, respectively. The representative dimensions of the material microstructure, such as the widths of phases A and B and the grain diameter of phase C were measured using an optical microscope. Table 3 shows the results of the X-ray diffraction analysis, the representative dimensions and the micro-hardnesses for phases A, B, and C. The micro-hardness of each phase was measured using a micro-hardness tester (Akashi: HM-102). \( W_A \) and \( W_B \) in the table show the width of phases A and B, respectively, whereas \( d_i \) is the grain size of phase C. According to this table, the hardness of phases B and C are \( H_V = 93 \) and \( H_V = 83 \), respectively. These hardnesses are clearly greater than that of phase A, \( H_V = 61 \). We can also see that the average sizes of the microstructures \( (W_A, W_B, d_i) \) are 40, 24, 20 \( \mu \)m, respectively.

### 2.2. Experimental method

Fatigue tests were performed using specimens with a configuration (stress concentration factor = 1.04) as shown in Fig. 2. The specimen was machined from a round bar 19 mm in diameter. The specimen was prepared for the experiment by polishing its surface to a mirror-like finish with emery paper and diamond paste. Fatigue tests were performed using a cantilever-type rotating bending fatigue machine under laboratory conditions with a room temperature of about 298 K and 63–73% humidity. The stress cyclic speed was 30 Hz. Temperature and humidity were not strictly controlled during the experiment. The replicas of the specimen surfaces were collected during the fatigue process by interrupting the test at a fixed number of cycles in order to investigate fatigue crack initiation and growth behavior. Stress cyclic speed for successive observations of the specimen surface was 10 Hz. In order to study the effect of specimen microstructure on fatigue crack initiation and propagation behavior, parts of the experiments were carried out after exposing the microstructures of the specimen using an etching solution. To investigate crack initiation and propagation mechanisms at the atomic level, the replicas obtained through successive observations during the fatigue process were studied using the scanning probe microscope (SHIMADZU: 9500J2).

### Table 1

**Chemical composition of the material (wt%)**

<table>
<thead>
<tr>
<th></th>
<th>Al</th>
<th>Zn</th>
<th>Mn</th>
<th>Fe</th>
<th>Ni</th>
<th>Cu</th>
<th>Si</th>
<th>Ca</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2.98</td>
<td>0.97</td>
<td>0.004</td>
<td>0.007</td>
<td>0.005</td>
<td>0.002</td>
<td>0.02</td>
<td>0.05</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

### Table 2

**Mechanical properties of the material**

<table>
<thead>
<tr>
<th></th>
<th>Yield strength</th>
<th>Tensile strength</th>
<th>Elongation</th>
<th>Young’s modulus</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>200 MPa</td>
<td>275 MPa</td>
<td>11%</td>
<td>45 GPa</td>
</tr>
</tbody>
</table>

### Table 3

**Composition and vickers hardness of the microstructure**

<table>
<thead>
<tr>
<th>Structure</th>
<th>Composition</th>
<th>Vickers hardness</th>
<th>Typical size (( \mu )m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Mg</td>
<td>61</td>
<td>( W_A = 40 )</td>
</tr>
<tr>
<td>B</td>
<td>Mg₃₂(Al,Zn)₄₉</td>
<td>93</td>
<td>( W_B = 24 )</td>
</tr>
<tr>
<td>C</td>
<td>Mg₁₇Al₁₂</td>
<td>83</td>
<td>( d_i = 20 )</td>
</tr>
</tbody>
</table>
3. Experimental results

3.1. $S$–$N$ curve

Fig. 3 shows the $S$–$N$ curve for the AZ31 magnesium alloy obtained using a stress ratio of $R = -1$ and a stress cyclic speed of 30 Hz. As seen from the figure, at the stress amplitude of 122.5 MPa the fatigue life is about $8.5 \times 10^4$ cycles, while at 120 MPa, only 2.5 MPa less than the former, the fatigue life becomes $5.14 \times 10^7$ cycles, about 600 times longer than the former. As a result, a characteristic sharp bend can be seen in the $S$–$N$ curve.

3.2. Successive SPM observations of the fatigue process near the fatigue limit

In order to determine why fatigue life is greatly affected by a slight change (2.5 MPa) in the stress amplitude, the successive observations of the specimen surfaces during the fatigue process were conducted at stress amplitudes of 120 and 122.5 MPa using a scanning probe microscope. Fig. 4 shows the results of the successive observations of the fatigue process at a stress amplitude of 120 MPa using a scanning probe microscope. In the figure, the arrows are appended at the crack tips to show their exact locations. The vertical bars with grey tones and profiles on the right sides of the submicrographs show the height density distributions for the areas investigated. The successive SPM observations in Fig. 4 show that at $4.9 \times 10^6$ cycles of Fig. 4(a), crack initiation could not be observed. However, in Fig. 4(b) at $7.34 \times 10^6$ cycles, a crack is seen to have been initiated in phase C from both the upper and lower vertical ends of the crystal grain. Through a cyclic stressing of $2.45 \times 10^7$ cycles, the crack developed slowly to 22.5 μm in length which was identical to the grain size. At that point, crack propagation was arrested for an extended period by the grain boundary (Fig. 4(d)). Through a stress cycling of $5.14 \times 10^7$ cycles, the crack finally broke through the grain boundary, and it grew to 61 μm in length (Fig. 4(e)) followed by rapid specimen failure. The above results using the SPM are similar to those obtained using an optical microscope [2].
At a stress amplitude of 122.5 MPa, (although the figures were omitted) a crack was generated in the crystal grain of the C phase and propagated into the adjacent B phase. Such a characteristic is common to that observed at a stress amplitude of 120 MPa. However, at a stress amplitude of 122.5 MPa, the crack easily crossed the grain boundary and expanded.

During fatigue process of AZ31 magnesium alloy just above the fatigue limit, it was observed that only one crack initiated and propagated, then followed by a final failure of the specimen as shown in Fig. 4. At the higher stress amplitudes, several cracks were observed to initiate during fatigue process, but their coalescences had no significant effect on the specimen failure. These observations in the present AZ31 magnesium alloy are different from those in the cast magnesium alloy reported by Eisenmeier et al. [6]. These authors reported that many cracks initiate from the cast defects during fatigue process and crack coalescences among them shorten the fatigue life.

In fatigue studies of magnesium alloy AZ61 [7], it was observed that a crack was initiated from a corrosion pit in air with a comparatively high humidity. However such a result was not observed in the current study.

4. Crack growth mechanism near the fatigue limit

Fig. 5 shows the microscopic deformation behavior of the specimen surface near the crack when it initiates and propagates within phase C. As an example, the figure shows the case of a stress amplitude of 120 MPa. The successive observations were done using the scanning probe microscope. The white portion and the arrows in Fig. 5(a) indicate the C phase and the crack-tip respectively. The vertical bar with grey tone shows the height density distribution for the area shown in Fig. 5(a). As seen from the figure, the crack is being generated from the both of the vertical ends of the C phase. A microscopic height of the specimen surface along the segment AB drawn in the figure was measured, and an example of its result is shown in Fig. 5(b). As seen from this figure, the left portion of the crack is higher than the right portion of the crack. This observation suggests that the crack is generated by the out of plane slip of the crystal.

Fig. 6 shows the height difference between the right and left portions of the crack at segment AB as a function of the number of stress cycles. The points indicated by (b)–(e) in the figure correspond to those in Fig. 4. We can see that the crack width increases slightly as it approaches point (c) in the figure, and afterwards it levels off at point (d) with a width of 1 µm. However, from point (d), the crack width increases rapidly followed by specimen failure.

On the basis of the above changes in the crack width (Fig. 7) as well as the height difference between right and left portions of the crack (Fig. 6), crack initiation and microscopic crack propagation mechanisms will be considered.
The interval from (b) to (c) in Figs. 6 and 7 seems to indicate the period during which the crack propagates within the crystal grain (phase C) in a mixed mode (modes I and III, as shown schematically in Fig. 8(a)). In addition to these modes, mode II is also thought to be included but only partially in the crack deformation. Under such conditions, it seems reasonable, therefore, to assume that both the width and the height differences of the crack increase with the number of stress cycles. At point (b), the two cracks initiated from the top and bottom of the grain coalesce after which the combined crack propagates before being arrested by the grain boundary of the C phase (point (c)). In the arrested crack, the change in width (mode I type crack opening displacement) is insignificant. However, the difference between the heights of the right and left portions of the crack increases vigorously through mode III type deformation, i.e., out of plane slip, as shown in the schematic illustration of Fig. 8(b). Therefore, the interval from (c) to (d) in Figs. 6 and 7 indicates the period during which many dislocations pile up at the grain boundary due to out of plane slip produced by the cyclic stress. When, due to cyclic stress, the accumulated energy at the crack tip reaches a critical value sufficient to break the grain boundary, the crack breaks through the grain boundary and then propagates into the adjacent B phase.

Point (d) in both Figs. 6 and 7 seems to indicate the point at which the accumulated energy at the grain boundary exceeds the critical value. When the crack breaks through the grain boundary and starts to propagate into the adjacent B phase, the accumulated energy generated by the out of plane slip at the grain boundary is suddenly released, and the height difference between the right and left portions of the crack will decrease rapidly as shown in the schematic illustration in Fig. 8(c). Simultaneously, the crack width may increase rapidly due to the initiation of mode I type crack propagation.

5. Conclusions

Fatigue crack initiation and propagation behavior of magnesium alloy AZ31 within a crystal grain near the fatigue limit were investigated in detail using a scanning probe microscope. The results obtained are summarized as follows:

(1) Fatigue cracks initiate from both the upper and lower vertical ends of the crystal grain (phase C) in the early stages of fatigue life.
(2) The initiated crack propagates within a crystal grain in a mixed mode, modes I and III.
(3) At a stress amplitude slightly higher than the fatigue limit, cracks are blocked and arrested by the grain boundary for an extended period. During this period, the height difference between the right and left portions of the crack increases vigorously through mode III type deformation, i.e., out of plane slip. When, due to cyclic stress, the accumulated energy at the crack tip reaches a critical value sufficient to break the grain boundary, the crack breaks through the grain boundary and then propagates into the adjacent B phase.
(4) When the crack breaks through the grain boundary and starts to propagate into the adjacent B phase, the energy accumulated at the grain boundary by the out of plane slip is released, and this is followed by a sudden decrease in the height difference between the right and left portions of the crack. Simultaneously, the crack width increases rapidly due to the initiation of mode I type crack propagation.

![Fig. 8. Schematic illustration of crack propagation behavior within a crystal grain (phase C): (a) b–c in Figs. 6 and 7, (b) c–d in Figs. 6 and 7 and (c) d–e in Figs. 6 and 7.](image-url)
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References