Influence of Grain Size and Aging on Fatigue Behavior of AZ-type Magnesium Alloys

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Abstract
In order to make clear the grain-size and precipitation dependence on the fatigue properties of wrought Mg-Al-Zn alloys in terms of dislocation theories, pure magnesium, AZ31 and AZ61 alloys with the average grain sizes ranging from 18 to 101µm were produced by the strain-annealing method. In AZ61 alloys, two kinds of specimens were prepared by solution treating at 683K and followed by T6 aging at 423K. Fatigue tests at room temperature were performed under the constant stress amplitude over a life range of 10^4 to 10^7 cycles. The fatigue crack propagation and fracture were also examined in each specimen. The grain-size dependence on the fatigue strength for pure magnesium and AZ31 alloys is uniformly confirmed at the whole life, whereas that of as-quenched AZ61 alloys decreases with an increase of the number of cycles and disappears beyond about 10^5 cycles. The fatigue limits of pure magnesium, AZ31 and aged AZ61 alloys increase with grain refining. The fatigue strength at 10^7 cycles for them conforms well to the Hall-Petch relation. The 10^7 cycle stress of AZ-type magnesium alloys is found to be proportional to the linear aluminum concentration. The results based on the microscopic observations indicate that the slip bands, the twin boundaries and the precipitates of $Al_{12}Mg_{17}$ as well as the grain boundaries have a significant effect on the fatigue fracture in AZ-type magnesium alloys.

1 Introduction

Magnesium alloys are greatly attractive as a super-light material in the next generation applications, owing to the excellent specific strength and the superior recycling ability among the conventional metals [1]. However, it is well known that they possess poor cold formability and low creep resistance as compared with steels, because the basal slip and/or twinning mainly occur during the deformation. In order to overcome these serious problems, we should carry out forming at the elevated temperature. On the other hands, it is also pointed out that the considerable improvement in the mechanical properties of magnesium alloys can be achieved by means of the grain refinement [2]. Therefore, the present authors [3-5] have investigated the validity of the Hall-Petch relation [6, 7] in pure magnesium and Mg-Al-Zn alloys to understand the grain-size dependence of polycrystalline metals containing twins or precipitates in detail. Indeed, many studies concerning the grain-size effect on the plastic and fracture behavior of these alloys have so far been performed, whereas the metallurgical data of the fatigue properties is not always sufficient, in particular that of wrought AZ-type magnesium alloys which are expected to be quite useful as structural materials [8].

In the present work, the change of fatigue behavior with an increase of the number of cycles at room temperature was confirmed for pure magnesium, AZ31, as-quenched and aged AZ61 alloys with various grain sizes. From the results based on the Hall-Petch analysis, the grain-size dependence on the fatigue strength was clarified in connection with the effects of the number of cycles, aluminum concentration and precipitation. Furthermore, the mechanisms of fatigue fracture for AZ-type magnesium alloys were examined by the microscopic observations of both crack propagation and fracture surface from the viewpoint of dislocation theories.

2 Experimental Procedures

Rolled sheets of magnesium (purity of 99.95%), commercial quality AZ31 (Mg-3.0%Al-1.0%Zn) and AZ61 (Mg-6.5%Al-0.6%Zn) alloys made by Osaka Fuji Industry Corp. were employed in the present study, where the composition of each material stands for mass percent. They were cut in the form of rectangles with the dimensions of 1×5×18mm in gauge length. A variety of grain sizes of the specimens was obtained by the strain-annealing method at 573, 623, 673 and 723K. The average grain sizes ranging from 18 to 101μm in pure magnesium and from 19 to 71μm in the AZ-type alloys were selected to examine the plastic and fatigue behavior (refer to Table 1). They were measured by the linear intercept method with a ruler on optical micrographs. In AZ61 alloys, two kinds of specimens were prepared by solution treating at 683K for 36ks and followed by aging at 423K for 864ks, so that Vickers hardness of as-quenched AZ61 alloys increased with an increase of the aging time at 423K and achieved the maximum value for 864ks (T6 aging treatment) [9].

Prior to tensile and fatigue tests, all specimens were electrolytically polished to remove the surface layer and reveal the grain boundaries by using anodic oxidation. The polishing solution for pure magnesium was consisted of 165ml HNO_3 and 335ml CH_3OH, whereas the mixed solution of 180ml $P_2O_5H_2O$ and 300ml C_2H_5OH was employed for the AZ-type alloys. First, the specimens were pulled in tension with the constant strain rate of $4.6\times10^{-4}s^{-1}$ by the Instron-type tester equipment. Subsequently, fatigue tests at room temperature were performed under the constant stress amplitude over a life range of 10^4 to 10^7 cycles. The testing machine made by Shimadzu Corp. was the Servo-pulser EHF-EG50kN-20L, where the smooth specimens were subjected to cyclic tensile loading-unloading at 40Hz. The fatigue crack propagation and the fracture surface in pure

Table 1 Average grain sizes of pure magnesium, AZ31 and AZ61 alloys.

Specimen	Grain size (μm)			
Mg	18	42	71	101
AZ31	19	26	30	33
AZ61	29	50	71	-

magnesium, AZ31, as-quenched and aged AZ61 alloys without a notch were also observed by using optical and electron microscopy.

3 Results and Discussion

3.1 Grain-size dependence of yield and fatigue strengths

The relationship between the yield strength and the inverse square root of grain size for all specimens was obtained from the results of tensile experiments. Taking advantage of the Hall-Petch plots, the present authors determined the slope coefficient of k and the characteristic constant of σ_0 which is the frictional stress of mobile dislocations on slip planes. The grain-size dependence on 0.2% proof stress of $\sigma_{0.2}$ for pure magnesium, AZ31, as-quenched and aged AZ61 alloys at room temperature is depicted in Fig. 1, where each symbol denotes the mean value of three measurements. The obtained graph enables us to clarify the Hall-Petch relation. Owing to the solid solution and precipitation strengthening, the σ_0 values for the $\sigma_{0.2}$ of AZ31 and AZ61 alloys increase in comparison with that of pure magnesium. However, it is noteworthy that the grain-size dependence of yielding on the k markedly decreases by the influence of precipitated particles in aged AZ61 alloys. The precipitates were identified as $Al_{12}Mg_{17}$ which was incoherent with the matrix phase by using the X-ray diffraction method [9].

Figs. 2 to 4 show the relationship between the stress amplitude and the number of cycles (*S-N* curves) for each specimen with various grain sizes. It is found that the grain-size dependence on the fatigue strength for pure magnesium and AZ31 alloys is uniformly confirmed over a life range of 10^4 to 10^7 cycles (see Figs. 2 and 3). The improvement of their fatigue properties with grain refining is in good agreement with the demonstration which Kamakura *et al*. [8] have shown in the extruded magnesium alloys. As a result, this fact suggests that the slip bands and

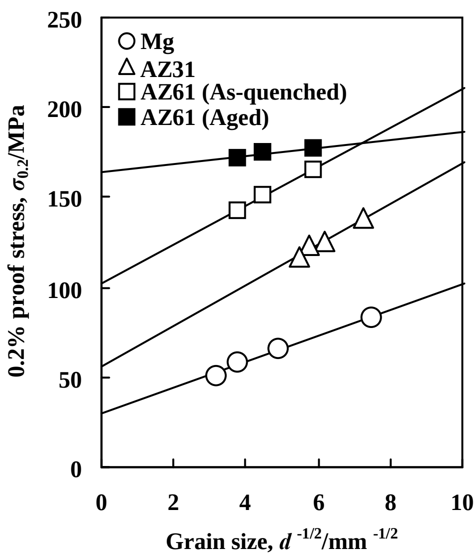

Fig. 1 Grain-size dependence on 0.2% proof stress of each specimen.

the twin boundaries as well as the grain boundaries have a significant effect on the fatigue fracture, because they appear preferably in the coarse grains of both specimens due to the local stress concentration occurring by the operation of a group of piled-up dislocations against the grain and twin boundaries under cyclic loading [10, 11]. However, the effect of grain size on the fatigue properties for as-quenched AZ61 alloys decreases with an increase of the number of cycles, and then disappears beyond about 10^5 cycles as shown in Fig. 4 (a). The grain-size independence at the region of long life of them is supposed to be associated with the early precipitation during fatigue tests. In aged AZ61 alloys with the T6 treatment, the effect of grain refinement on the fatigue strength is gradually seen with increasing the number of cycles (refer to Fig. 4 (b)).

On the other hand, the relationship between the number of cycles and the inverse square root of grain size for pure magnesium, AZ31 and AZ61 alloys can be approximately represented by each straight line as seen from Fig. 5, in which the

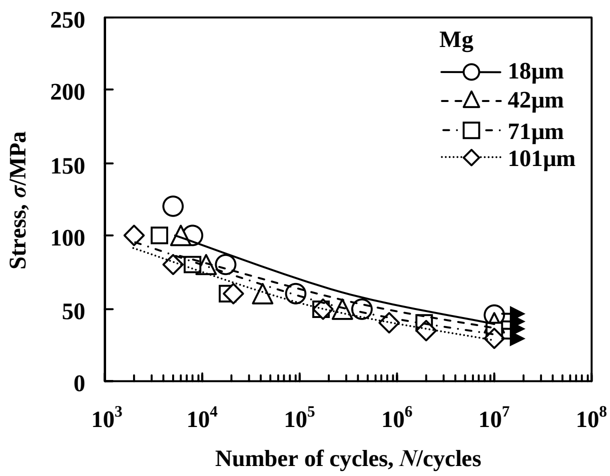

Fig. 2 *S-N* curves for pure magnesium with various grain sizes.

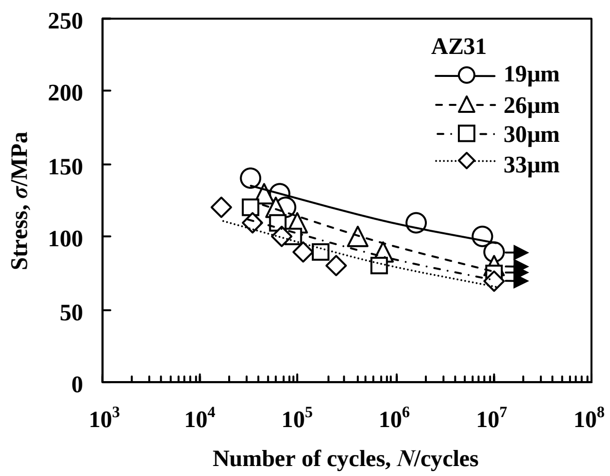

Fig. 3 *S-N* curves for AZ31 alloys with various grain sizes.

stress amplitude applied for them is 40, 100 and 90MPa, respectively. Based on the above Hall-Petch analysis, Fig. 6 shows the grain-size dependence on the fatigue strength at 10^7 cycles for all specimens. As in case of yielding, the 10^7 cycle stress for pure magnesium, AZ31 and aged AZ61 alloys is found to be proportional to the inverse square root of grain size. Table 2 gives the σ_0 and k values derived from the straight lines shown in Figs. 1 and 6, which are estimated by using the least square method. It is clear that the fatigue limits of the specimens excluding as-quenched AZ61 alloys increase with the grain refinement. Moreover, we may notice that the Hall-Petch slope in the fatigue properties of AZ31 alloys is the largest k value of 10.7MPa·mm$^{1/2}$. This fact is in good agreement with the case of yielding (see Table 2). Although the 10^7 cycle stresses and its k values for two AZ-type magnesium alloys are larger than those of pure magnesium, the strength at the fatigue limit of AZ61 alloys obviously decreases because of the precipitation with the peak aged hardness, especially in the coarse grain-sized specimens.

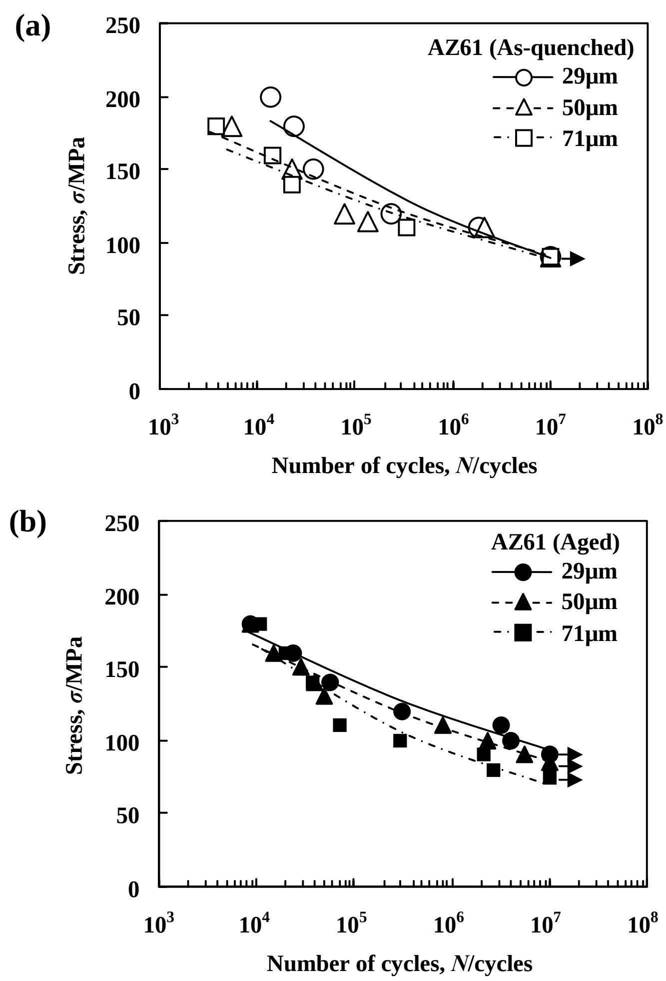

Fig. 4 *S-N* curves for two kinds of AZ61 alloys with various grain sizes: (a) As-quenched; (b) Aged.

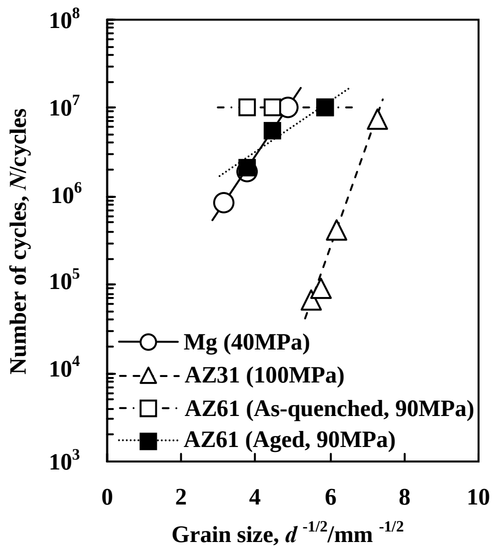

Fig. 5 Relationship between the number of cycles and the inverse square root of grain size for each specimen. The stress amplitude for them is 40, 100 and 90MPa, respectively.

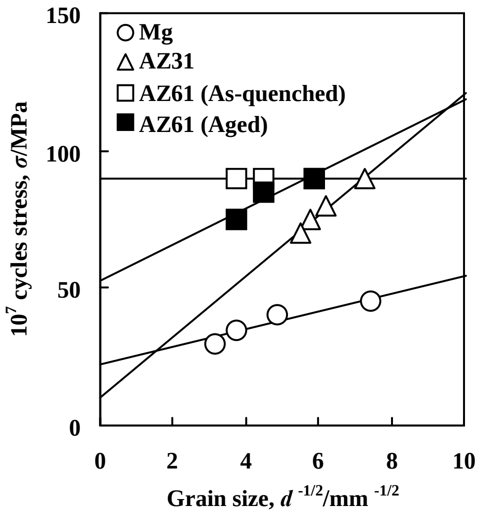

Fig. 6 Grain-size dependence on fatigue strength at 10^7 cycles for each specimen.

Table 2 The σ_0 and k values of the straight lines shown in Figs. 1 and 6.

Specimen	0.2% proof stress		10^7 cycle stress	
	σ_0 (MPa)	k (MPa·mm$^{1/2}$)	σ_0 (MPa)	k (MPa·mm$^{1/2}$)
Mg	30	7.2	22	3.2
AZ31	56	11.4	11	10.7
AZ61 (As-quenched)	102	10.8	90	0
AZ61 (Aged)	164	2.2	52	6.6

3.2 Effect of solute element concentration on fatigue life

The yield strength of alloys becomes greater than that of pure metals due to the interaction between moving dislocations and the solute atoms. Generally, it is considered that the shear stress of the solid solution of alloys increases with an increase of the solute element concentration. It is also reported that this strength is proportional to the linear or square root concentration of solute elements [12]. When the zinc content in our specimens is assumed to be about constant, Fig. 7 shows the aluminum concentration dependence on 10^7 cycle stress, together with the 0.2% proof stress for AZ-type magnesium alloys having the similar grain sizes ranging from 18 to 30μm. As well as the obtained results of the 0.2% proof stress, the relationship between the fatigue strength at 10^7 cycles and the linear aluminum concentration for AZ-type magnesium alloys can be represented by the straight line. It is noteworthy that the slope value of $6.7 MPa \cdot at\%^{-1}$ which implies the locking effect of dislocations on the fatigue behavior is found to be about half as large as that of 0.2% proof stress.

3.3 Observations of fatigue crack propagation and fracture surface

The fatigue fracture is roughly divided into two processes concerning the initiation of crack and the growth of small crack [13]. It is known that the micro-crack usually occurs at the early stage below about 10% in the whole fatigue life, which depends on the applied stress, the environment, the kind of materials and so on. In our specimens without a notch, we intermittently observed the propagation of large crack which finally results in the fatigue fracture. The typical growth path of fatigue crack in as-quenched AZ61 alloys with the grain size of 29μm is shown in Fig. 8, where the stress amplitude of 140MPa was applied over 2.8×10^4 cycles. It is seen that a large crack is initially formed in the edge part of them at an angle of about 45 degrees to the loading direction. Subsequently, the crack propagation in the specimens excluding aged AZ61 alloys occurs mainly along the grain

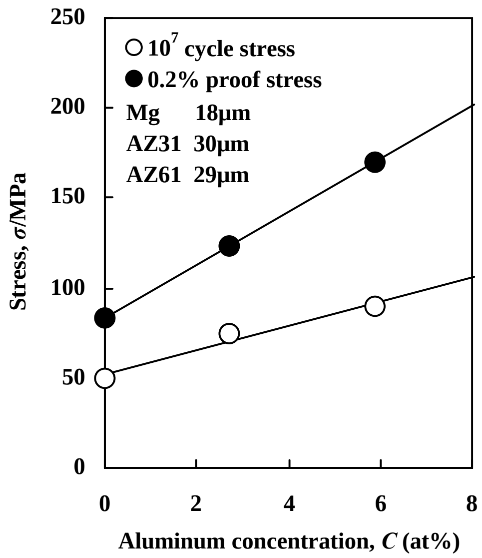

Fig. 7 Aluminum concentration dependence on 10^7 cycle stress and 0.2% proof stress for AZ-type magnesium alloys.

boundaries perpendicular to the loading axis, and rapidly leads them to the fatigue fracture. However, it seems that the large crack close to the coarse grains occasionally advances along the slip bands or the twin boundaries within the grains (see Fig. 8), which is supposed to cause a decrease in the growth resistance of crack as well as the interference against the propagation in the grain boundaries. According to the investigations by Kamakura *et al*. [8] and Yokobori [13], the initiation resistance of crack may also decrease due to the occurrence of the slip bands and twins. As described in the previous section 3.1, they appear easy in the coarse grains of the specimens, where the local stress concentration by the piled-up dislocations increasing with the grain size must effectively operate to increase the growth rate of crack. Consequently, it is considered that the grain refinement contributes to improvement in the fatigue strength of pure magnesium and AZ31 alloys, together with as-quenched AZ61 alloys less than about 10^5 cycles.
In aged AZ61 alloys, there is not the above relationship between the growth of large crack and the grain boundaries, especially at the short life within 10^5 cycles. Fig. 9 shows the fracture surface on the latter places of the crack propagation in

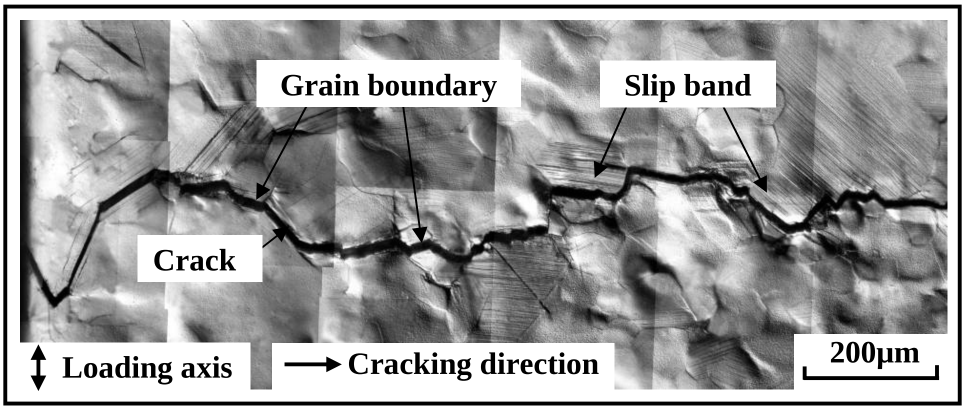

Fig. 8 Growth path of large fatigue crack in as-quenched AZ61 alloys with the grain size of 29μm under the stress amplitude of 140MPa and 2.8×10^4 cycles.

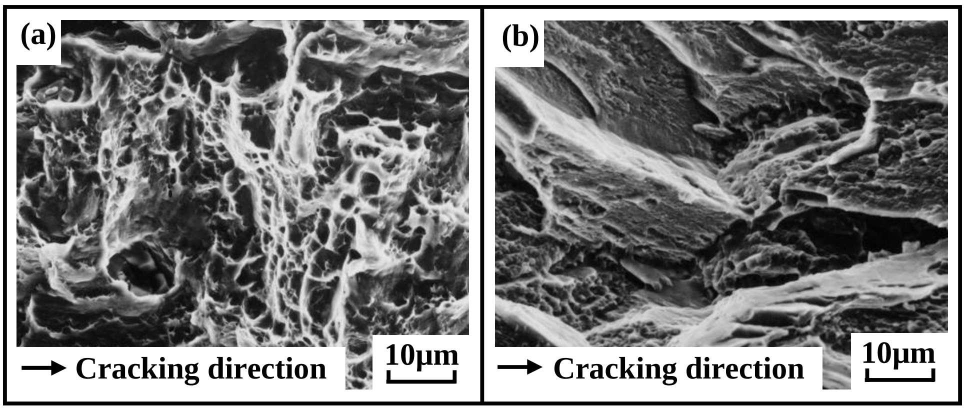

Fig. 9 Fracture surface of AZ-type magnesium alloys by SEM: (a) AZ31 alloys (110MPa, 4.2×10^4 cycles); (b) Aged AZ61 alloys (140MPa, 3.7×10^4 cycles).

AZ31 and aged AZ61 alloys by means of a scanning electron microscope. The stress amplitude applied for both specimens is 110 and 140MPa, respectively. Although the characteristic of the cyclic striation markings is not always clear in all specimens, the river pattern can be markedly observed in aged AZ61 alloys. It is also found that the fracture surface of aged AZ61 alloys obviously consists of the cleavage facets in comparison with that of AZ31 alloys (refer to Fig. 9 (a) and (b)). This fact implies that the typical brittle fracture occurs by the influence of the precipitated particles of $Al_{12}Mg_{17}$ in aged AZ61 alloys with the T6 treatment, resulting in a decrease of the grain-size dependence on the fatigue behavior at the shorter life. However, it is noteworthy that the fatigue strength at the region beyond about 10^5 cycles of the aged specimen increases with the grain refinement (see Fig. 4 (b)). This reason is supposed to be that the application of the lower stress amplitude enables the large crack to propagate along the slip bands, the twin and grain boundaries. From these results, it is considered that the effect of grain size on the fatigue properties of AZ61 alloys with the peak aged hardness depends mainly on the magnitude of externally applied stress. It remains to be seen if we can microscopically clarify the correlation of the grain boundaries and precipitates with the growth of crack at the fatigue limits of wrought AZ-type magnesium alloys in terms of dislocation theories.

4 Conclusions

The grain-size dependence on the fatigue strength for pure magnesium and AZ31 alloys is uniformly confirmed over a life range of 10^4 to 10^7 cycles. In two kinds of AZ61 alloys, the effect of grain size on the fatigue properties for the as-quenched specimen decreases with an increase of the number of cycles and disappears beyond about 10^5 cycles, whereas the fatigue limit of the aged specimen increases with the grain refinement. The relationship between the fatigue strength at 10^7 cycles and the inverse square root of the average grain size for pure magnesium, AZ31 and aged AZ61 alloys can be represented by each straight line. The 10^7 cycle stress of AZ-type magnesium alloys is found to be proportional to the linear aluminum concentration. On the basis of the microscopic observations of both fatigue crack propagation and fracture surface, it is confirmed that the slip bands, the twin boundaries and the precipitates of $Al_{12}Mg_{17}$ as well as the grain boundaries play a significant role on the fatigue fracture in AZ-type magnesium alloys.

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References

[1] M. Kohzu, Domestic or overseas trend in research and developement of magnesium alloy plastic processing, J Jpn Inst Light Metals 54 (2004) 493-498

(in Japanese).
[2] M. Mabuchi, K. Higashi, Strengthening mechanisms of Mg-Si alloys, Acta Mater 44 (1996) 4611-4618.
[3] N. Ono, K. Nakamura, S. Miura, Influence of grain boundaries on plastic deformation in pure Mg and AZ31 Mg alloy polycrystals, Mater Sci Forum 419-422 (2003) 195-200.
[4] N. Ono, R. Nowak, S. Miura, Effect of deformation temperature on Hall-Petch relationship registered for polycrystalline magnesium, Mater Letters 58 (2004) 39-43.
[5] N. Ono, M. Ueda, S. Miura, Mechanisms of plastic deformation in pure Mg and AZ31 Mg alloy polycrystals, Mater Sci Forum 488-489 (2005) 555-558.
[6] E.O. Hall, The deformation and ageing of mild steel, Proc Phys Soc London 64B (1951) 747-753.
[7] N.J. Petch, The cleavage strength of polycrystals, J Iron Steel Inst 174 (1953) 25-28.
[8] M. Kamakura, K. Tokaji, H. Shibata, N. Bekku, Grain refinement and improvement of fatigue strength due to controlled extrusion in magnesium alloys, J Soc Mat Sci Jpn 54 (2005) 245-250 (in Japanese).
[9] M. Ueda, Effect of grain boundaries on plastic deformation of Mg-Al-Zn system alloys, Master's Thesis Sojo Univ, Kumamoto, 2005 (in Japanese).
[10] J. Koike, T. Miyamura, Microscopic mechanisms of plastic deformation in plycrystalline magnesium alloys, J Jpn Inst Light Metals 54 (2004) 460-464 (in Japanese).
[11] Y. Nishimura, N. Ono, S. Miura, Etch pit observation of dislocations in early deformation of Cu-Al alloy and pure Mg polycrystals, Key Eng Mater 345-346 (2007) 25-28.
[12] J. Takamura, Zairyo-kyodo no kiso, Kyoto Univ Press, Kyoto, 1999, pp.136-138 (in Japanese).
[13] T. Yokobori, The theory of fatigue fracture of metals, J Phys Soc Jpn 10 (1955) 368-374.