



## Effect of stress ratio on high cycle fatigue properties in Mg-6Zn-1Mn alloy

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### ABSTRACT

This work investigated the effect of loading mode on high cycle fatigue properties in Mg-6Zn-1Mn (ZM61) alloys. Extruded, T5 treated and double-aged ZM61 alloys were used in this study. High cycle fatigue tests were carried by a servo-hydraulic fatigue testing system under zero-tension load ( $R = 0$ ) and fully reversed tension-compression load ( $R = -1$ ), respectively. The results showed that all the three alloys exhibited more outstanding high cycle fatigue strength under zero-tension load than under tension-compression load. By observing microstructures of post-fatigued specimens, a large number of low angle grain boundaries appeared in zero-tension load specimens, but  $\{10\bar{1}2\}$  twins were found in tension-compression load specimens. The variations of fatigue properties in zero-tension load and tension-compression load were related to deformation mechanisms under different load conditions. The fatigue deformation mechanism in Mg-6Zn-1Mn alloy under zero-tension load was mainly dislocation slip, while under tension-compression load, the activation of  $\{10\bar{1}2\}$  twinning in compression half cycles resulted in the deteriorative fatigue strength.

### 1. Introduction

Magnesium (Mg) alloys are one of the lightest materials among metals. In recent years, Mg alloys have attracted great interests in structural applications, especially in aeroplanes and ground vehicles for which weight saving is extremely important [1,2]. Compared with casting Mg alloys, wrought Mg alloys do not have casting defects and have favorable mechanical properties. These advantages make wrought Mg alloys more promising for using as engineering components. When Mg alloys are used as load-bearing parts, they inevitably undergo dynamic loads and fatigue failure may occur. To ensure the safety and reliability in service, revealing the high cycle fatigue (HCF) behavior of wrought Mg alloys is significant and necessary [3,4].

Due to their hexagonal close-packed (HCP) lattice, Mg alloys have limited slip systems to accommodate imposed plastic deformation. In addition, after extrusion or rolling, most grains predominately orient with their basal planes paralleling to the processing direction, inducing a strong texture in wrought Mg alloys.  $\{10\bar{1}2\}$  extension twins can be activated readily when Mg alloys are applied an extension paralleling to the c-axis or a contraction perpendicular to the c-axis of the principal components. These characters lead to a significant tension-compression yield asymmetry of most wrought Mg alloys [5]. In recent years, some

studies investigated the HCF behavior and fatigue deformation mechanisms of different Mg alloys [6–14]. The HCF performance of Mg alloys can be influenced by two aspects: one is materials itself such as alloying elements, surface treatment, heat treatment and texture, the other is service conditions [15–17]. In the application of Mg alloys, some components are used under compression load such as aircraft seat components. Since their unique deformation mechanisms under the conditions of tension load and compression load, the fatigue deformation mechanisms of Mg alloys are complicated [15]. As a consequence, the effects of load types on HCF properties in Mg alloys should be taken into account.

Mg-6Zn-1Mn alloy (ZM61) is a recently developed, cost-effective and high-strength wrought Mg alloy [18]. In order to apply ZM61 alloy as load-bearing parts, it is important to comprehensively understand HCF properties under different load conditions. The extruded, T5 treated and double-aged ZM61 alloy are used in present work. Fatigue tests are conducted under two typical load types of zero-tension load ( $R = 0$ ,  $R$ : minimum cyclic stress/maximum cyclic stress) and fully reversed tension-compression load ( $R = -1$ ). Fatigue crack initiation mechanisms have also been discussed.

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## 2. Materials and methods

### 2.1. Materials

The ZM61 Mg alloy ingots used in this study were melted in a steel crucible under atmosphere protection with a gas of argon at 700 °C in a resistance furnace. The melt was refined by flowing argon for 15 min and then held at 700 °C for 30 min. The ingots were semi-continuous casted into bars with the diameter of 85 mm at 720 °C with a speed of 90 mm/min. The chemical composition was 5.57 wt% Zn, 0.61 wt% Mn, 0.01 wt% Fe and the balance Mg measured by the X-ray fluorescence spectrometer (Shimadzu XRF1800). Before extrusion, the ingots were homogenized at 330 °C for 24 h to eliminate macro segregation. Extrusion was conducted at a container temperature of 350 °C with a reduction ratio of 16. The extruded bar was directly aged at 180 °C for 16 h (hereafter, denoted as T5 alloy). Based on our previous work, the maximum age hardening response of ZM61 alloy could be obtained by double aging treatment [19]. Before double aging treatment, the extruded alloys were solution-treated at 420 °C for 2 h followed by hot water (~ 60 °C) quenching, then the alloys were pre-aged at 90 °C for 24 h and secondarily aged at 180 °C for 16 h (hereafter, denoted as double-aged alloy).

### 2.2. Test procedures

Cylindrical tensile specimens with a diameter of 5 mm and a gauge length of 35 mm were used for tensile strength measurements. The compressive strength was obtained by using cylindrical specimens with a diameter of 8 mm and a height of 12 mm. The tensile and compressive tests were performed at a constant strain rate of  $2 \times 10^{-3} \text{ s}^{-1}$ , the yield stress was determined as the 0.2% offset. Static mechanical testing specimens were machined with the loading axis paralleling to extrusion directions.

Cylindrical fatigue specimens were used for HCF tests with a gage diameter of 8 mm according to Chinese Standard GB/T 3075-2008 [20]. To avoid the influence of machining on the fatigue results, the surfaces of all fatigue specimens were mechanically polished by emery paper of grit 1200 and then buff-polished to a mirror-like surface. HCF tests were performed by using an MTS servo-hydraulic load frame (MTS Landmark Servo-hydraulic Test System) under zero-tension ( $R = 0$ , denoted as ZT) load and fully reversed tension-compression ( $R = -1$ , denoted as TC) load (For fatigue cycling, the load ratio,  $R$ , is the ratio of the minimum stress to the maximum stress in the cycle. Tension is taken as positive, compression as negative.). The specimens' dimensions and load waveform are shown in Fig. 1. All fatigue tests were performed at a frequency of 40 Hz at room temperature (about 25 °C) with relative humidity level of 40–50%. The stress amplitude and number of cycles to failure were recorded for each specimen. The specimens that did not fail after  $10^7$  cycles were noted as run out.

### 2.3. Microstructures analyses

Samples for metallography were ground with 1200 grit SiC paper and then etched with acetic picral (1 g picric acid, 8 ml ethanol, 1 ml acetic acid) for 15–25 s. The optical micrographs (OM) were observed by OLYMPUS OLS4000. With the help of ImageJ, the average grain sizes were determined by analyzing the optical micrographs with the mean linear intercept method. Fractographic analyses were carried out by using a Scanning Electron Microscope (SEM, Tescan VEGA 3). The macrotexture was measured by X-ray diffraction (XRD, Rigaku D/MAX-2500PC) with Cu-K $\alpha$  radiation at 40 kV and 30 mA and a scan rate of  $0.03^\circ \text{ s}^{-1}$  in a  $2\theta$  range of 10–90°. After fatigue tests, some typical post-fatigued specimens were observed by optical microscope, EBSD and transmission electron microscopy (TEM; FEI TECNAI G2 F20). Optical micrographs were taken on the section parallel to the gage length near the fracture surface, specimens for TEM were cut on the cross section

paralleling to fracture surface and about 3 mm away from fracture surface. The sketch of specimens' location and preparation is shown in Fig. 2. EBSD measurements were carried out within a JEOL JSM-7800F field emission gun SEM that operated at 20 kV acceleration voltage with an emission current of 3.0 nA. Oxford Instruments Naordlys Nano EBSD system with Channel 5 data acquisition and analysis software were used for data acquisition and analysis.

## 3. Results

### 3.1. Microstructures

The microstructures of ZM61 alloy under different conditions are shown in Fig. 3. As can be seen in Fig. 3a and b, the optical micrographs of extruded and T5 alloys are almost the same. The extruded and T5 treated alloy show an inhomogeneous grain structure consisting of both coarse and fine equiaxed grains with the size ranging from 2  $\mu\text{m}$  to 40  $\mu\text{m}$ . Typical necklace structure can be seen in extruded and T5 alloys. These regions are easily seen in the case of low extrusion ratio [21]. As a result of grain growth in solution treatment, the microstructures of double-aged alloy are more homogeneous and much coarser (grain size is about 40  $\mu\text{m}$ .) than that of extruded alloy and T5 treated alloy.

### 3.2. Texture

The {0002} and {10 $\bar{1}$ 0} pole figures of extruded, T5 treated and double-aged alloys are shown in Fig. 4. A typical extruded texture of Mg alloys is observed in three alloys, {0002} basal planes of three alloys mostly parallel to the extrusion direction [22]. T5 treatment has little effect on texture of ZM61 Mg alloy. It is noteworthy that though grain coarsen significantly during solution treatment, the texture type and texture intensity of double-aged alloy are slightly modified. As reported by Dogan [23], both deformation treatment and recrystallization can modify texture of Mg alloy. However, in this work there is little variation in texture intensity between the extruded and fully recrystallized alloy (double-aged alloy). It implies that the texture modification in ZM61 Mg alloy mainly results from recrystallization.

### 3.3. TEM microstructures

Fig. 5 shows TEM bright field micrographs of ZM61 alloys under different heat treatments conditions. There are three kinds of precipitates in ZM61 alloy subjected to aging: spherical Mn phase particles, rod-shaped  $\beta'_1$  and plate-shaped  $\beta'_2$  phases, the orientation relationships for these two precipitate phases are  $[0001]_{\beta'_1} // [1\bar{1}20]_{\alpha}$  and  $(11\bar{2}0)_{\beta'_1} // (0001)_{\alpha}$ ,  $[11\bar{2}0]_{\beta'_2} // [10\bar{1}0]_{\alpha}$  and  $(0001)_{\beta'_2} // (0001)_{\alpha}$ , respectively [24,25]. In order to compare the evolution of precipitates, all the TEM images were taken along the  $[2\bar{1}\bar{1}0]$  Mg direction. Bright field image of double-aged alloy is shown with higher resolution for its fine and high density precipitates. As shown in Fig. 5a, a few fine spherical particles could be found in extruded alloy, these precipitates are mainly Mn particles. Many rod-shaped precipitates can be found in T5 and double-aged alloys. The rod-shaped precipitates in double-aged alloy are finer and denser than that in T5 alloy. The high density of precipitates in double-aged alloy may result in excellent tensile strength.

### 3.4. Static mechanical properties

Table 1 shows the tensile and compressive properties of extruded, T5 and double-aged alloys. The stress-strain curves of three alloys can be seen in Supplementary material. Three important trends can be found from this table. First, aging treatment (both T5 and double aging treatment) enhances tensile yield strength (YTS) and ultimate tensile strength (UST). Though the grains became coarser in double-aged treatment, double-aged alloy shows the highest tensile strength among

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