Fatigue behavior of AZ31 magnesium alloy produced by solid-state recycling

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Mechanical properties of AZ31 Mg alloy produced from machined chips by the solid-state recycle method were compared to those of the reference alloy which was produced from an as-received AZ31 Mg alloy block under the same conditions with the recycled alloy. Tensile properties of the recycled alloy were comparable to those of the reference alloy, however, the recycled alloy exhibited poorer fatigue resistance than the reference alloy.

1. Introduction

Magnesium alloys are currently the lightest alloys used as structural metallic alloys, and Mg products have been applied for structural uses such as automobile parts and electric appliance cases [1, 2]. It is required not only to improve mechanical properties, but also to develop useful recycling process for their greater applicability. Some recycling processes such as remelting [3], electrorefining [4] and vacuum distillation [5] have been developed.

Recently, "solid-state recycling" by hot extrusion has been proposed as a new recycling method [6–11]. In the recycling process, metal scraps are directly recycled by hot extrusion without remelting process. The recycled alloys exhibit excellent mechanical properties such as high strength [10] and superplasticity [12, 13]. This is attributed to grain refinement by hot extrusion. However, in certain commercial applications, it is required to investigate fatigue properties of the recycled alloys. In the present paper, fatigue properties of AZ31 Mg alloy produced by the solid-state recycle method are investigated and its fatigue properties are compared to those of the reference alloy.

2. Experimental procedure

Machined chips with the average dimensions of 12 mm × 1.9 mm × 80 μm of AZ31 (Mg-3 mass%Al-1 mass%Zn-0.5 mass%Mn) were used as scraps. The scraps were filled into a rectangular container with the cross-sectional dimensions of 50 mm × 30 mm and then extruded at 673 K with the extrusion ratio of 6:1 in air. For comparison, a reference alloy was processed by extruding an as-received AZ31 Mg alloy block under the same conditions as used for a recycled alloy produced from the scraps. The cross-sectional dimensions of the extrusions were 50 mm × 5 mm. After annealing at 704 K for 7.2×10^4 s, the extrusions were rolled at 673 K to the rolling ratio of 80%. The rolling direction was perpendicular to the extrusion direction.

Tensile specimens with 10 mm gage length, 5 mm gage breadth and 1 mm gage thickness were machined. Tensile tests were carried out at room temperature with an initial strain rate of 1.7×10^{-3} s^{-1} where the angle between the tensile direction and the rolling direction was 0 degrees. Fatigue tests were carried out under $R = 0.1$ using an electro servohydraulic fatigue testing machine operating at a frequency of 20 Hz in laboratory air at room temperature. The configuration of fatigue specimen is shown in Fig. 1. The specimen was prepared by polishing its surface to a mirror-like finish with emery papers and diamond pastes. The angle between the loading direction and the rolling direction was 0 degrees. The microstructure of the recycled alloy and the reference alloy was observed by optical microscopy. The SEI (Secondary Electron Image) and images of oxygen and manganese were detected by EPMA (Electron Probe Micro-Analyzer).

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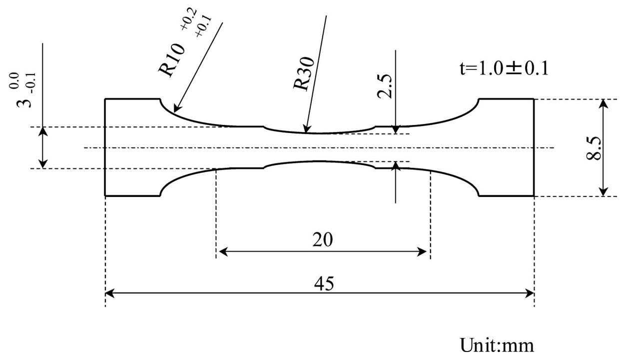

Figure 1 The configuration of fatigue specimen.

TABLE I The ultimate tensile strength, 0.2% proof stress and elongation by tensile tests at room temperature for the recycled AZ31 Mg alloy and the reference AZ31 Mg alloy

Meterial	Ultimate tensile strength (MPa)	0.2% Proof stress (MPa)	Elongation to failure (%)
Recycled alloy	295	281	6
Reference alloy	301	280	8

3. Results

Microstructure of the specimens before testing is shown in Fig. 2. The grain size was 13.3 μm for the recycled alloy and 15.2 μm for the reference alloy, respectively. Thus, the grain size of the recycled alloy was almost the same as that of the reference alloy. Also, twins were observed in both the recycled alloy and the reference alloy. Dotted black lines were found only in the recycled alloy. The origin of black lines is related to the oxide surfaces of the scraps [12]. An analysis by glow discharged mass spectrometry revealed that the oxygen concentration was 0.3 mass% for the recycled alloy and 1.4×10^{-3} mass% for the reference alloy, respectively, in the previous work [12]. Thus, the oxygen concentration in the recycled alloy was approximately two hundred times higher than that in the reference alloy.

The results by tensile tests at room temperature are listed in Table I. It is of interest to note that the strength of the recycled alloy was comparable to those of the reference alloy.

The S–N diagram is shown in Fig. 3. It is accepted that the fatigue limit does not often exist in non-ferrous alloys, thus fatigue tests were carried out until 10^7 cycles. No fatigue failure occurred at $\sigma_a = 80$ MPa for the recycled alloy and at $\sigma_a = 90$ MPa for the reference alloy, where σ_a is the stress amplitude. It is evident from Fig. 3 that the fatigue resistance in the recycled alloy is inferior to that in the reference alloy. It should be noted that tensile properties of the recycled alloy are comparable to those of the reference alloy; however, the recycled alloy exhibits poorer fatigue resistance than the reference alloy. Also, inspection of Fig. 3 reveals that on the subject of the stress at which fracture occurs when the fatigue life is the same, the difference between the recycled alloy and the reference alloy tends to increase with decreasing stress.

4. Discussion

Microstructure near the fracture surface of the specimens after fatigue testing is shown in Fig. 4, where fatigue tests were carried out at $\sigma_a = 120$ MPa for the recycled alloy and at $\sigma_a = 95$ MPa for the recycled alloy. Cracks with 10–100 μm were found in the recycled alloy. However, no such cracks were formed in the reference alloy. Clearly, the poorer fatigue resistance in the recycled alloy is attributed to significant crack formation, as shown in Fig. 4a. Horstemeyer *et al*. [14] showed that the

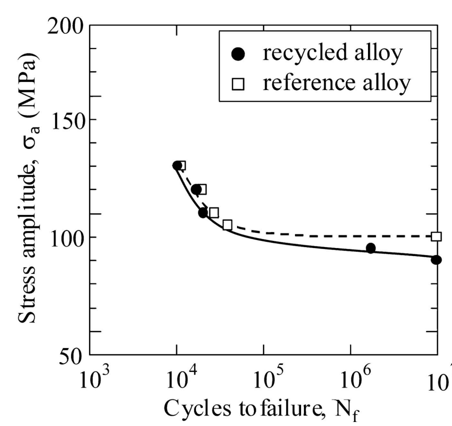

Figure 3 The S–N diagram for the recycled AZ31 Mg alloy and the reference AZ31 Mg alloy.

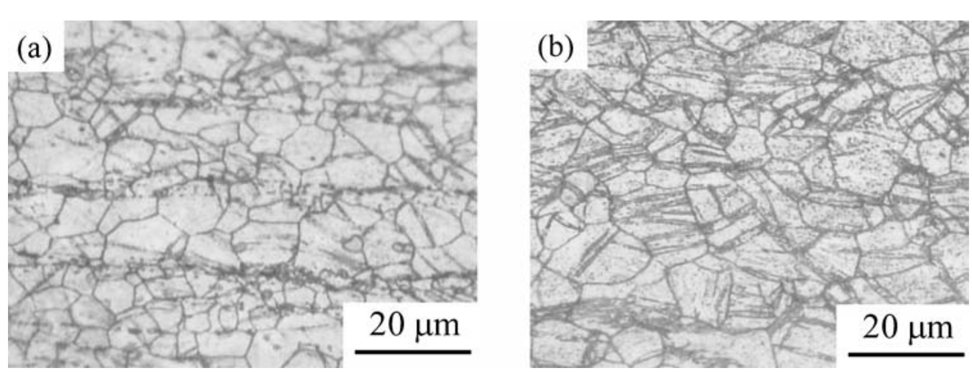

Figure 2 Microstructure of the specimens before testing, (a) the recycled AZ31 Mg alloy and (b) the reference AZ31 Mg alloy.

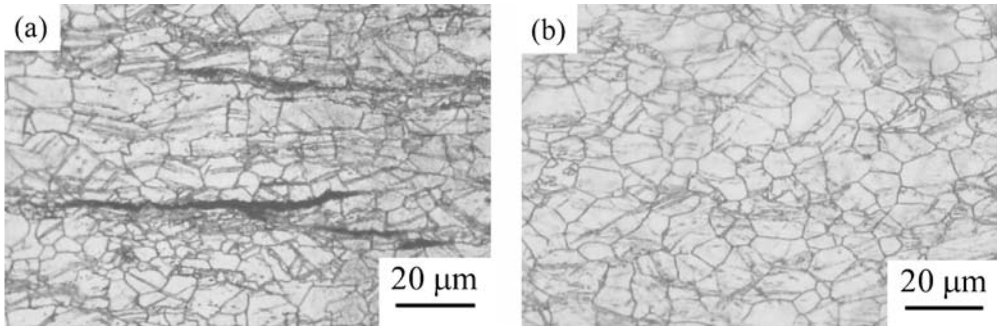

Figure 4 Microstructure near the fracture surface of the specimens after fatigue testing, (a) the recycled AZ31 Mg alloy and (b) the reference AZ31 Mg alloy.

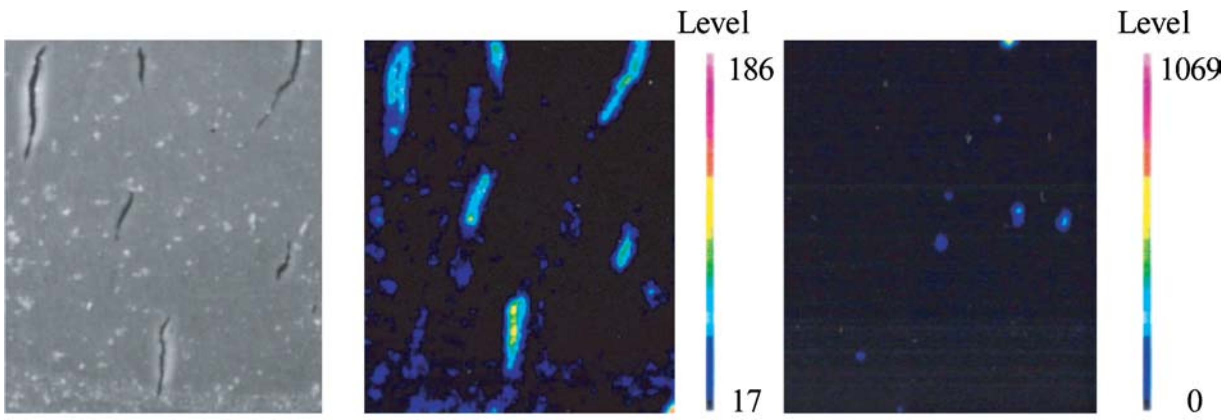

Figure 5 The results of analysis by EPMA at the portion near the fracture surface of the recycled alloy specimen after fatigue testing at $\sigma_a = 85$ MPa, (a) SEI (Secondary Electron Image), (b) oxygen image and (c) manganese image.

inclusion size has a first-order influence on the performance and larger inclusions have a tendency to form cracks earlier due to more widespread plasticity. Therefore, it is suggested that coarse oxide contaminants existed in the recycled alloy.

Fig. 5 shows the SEI (Secondary Electron Image) and images of oxygen and manganese by EPMA at the portion near the fracture surface of the recycled alloy specimen after fatigue testing at $\sigma_a = 85$ MPa. It should be noted that the portions of high oxygen concentration are in agreement with the ones of crack formation, whereas crack are not formed at the portions of high manganese concentration corresponding to Al-Mn precipitates [15]. Clearly, the oxide contaminants stimulated crack formation, resulting in poorer fatigue resistance in the recycled alloy. In other literatures [16, 17], fatigue crack propagation resistance for Mg alloys has been improved by the grain boundary and precipitates. In the present investigation, however, the oxide contaminants reduced fatigue resistance.

Another important result in the present investigation is that harmful effect by the oxide contaminants is larger in a lower stress range than in the higher stress range, as shown in Fig. 3. Tokaji *et al.* [18] showed that cracks initiate at surface in a high stress range, on the other hand, crack initiation occurred at subsurface in a low stress range. Also, tensile testing at room temperature–723 K revealed that difference in elongation between the recycled alloy and the reference alloy increased with increasing temperature, that is, with decreasing stress [19]. These indicate that effect of metallurgy factors such as inclusions and contaminants becomes significant upon reducing the stress [20]. This is likely to be responsible for the larger harmful effect of the oxide contaminants on the fatigue resistance in a lower stress range.

5. Conclusions

Fatigue properties of AZ31 Mg alloy produced by the solid-state recycle method were investigated and its fatigue properties were compared to those of the reference alloy. The results are concluded as follows.

1. The strength of the recycled alloy was comparable to that of the reference alloy, however, the recycled alloy exhibited poorer fatigue resistance than the reference alloy.
2. The poorer fatigue resistance in the recycled alloy is attributed to significant crack formation resulting from the existence of oxide contaminants.
3. On the subject of the stress at which fracture occurs when the fatigue life is the same, the difference between

the recycled alloy and the reference alloy increased with decreasing stress. This indicates that harmful effect of the oxide contaminants on the fatigue resistance becomes significant upon reducing the stress.

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