Microstructure Evolution of Extruded Mg–6Gd Alloy Under 175 °C and 150 MPa

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Abstract

The tensile creep behavior of extruded Mg–6Gd alloy, having the tensile yield strength of ~ 110 MPa at 175 °C, has been investigated under 175 °C and 150 MPa. In this study, the extruded Mg–6Gd sample exhibits the total tensile strain of $\sim 10.5\%$ after the creep time of 1100 h, and the fast plastic strain of $\sim 4.6\%$ at the beginning of the creep test. The microstructure result suggests that the dislocation deformation is the main deformation mode during creep, and the grains with orientation close to $\langle 0001 \rangle \parallel \text{ED}$ disappear after creep. The creep process containing a low creep strain has no effective promotion for the precipitation compared with the aging process without strain. The origination of creep crack is related to the formation of precipitate-free zone during creep. The work offers an important implication to research the microstructure evolution under an applied stress in a weak aging response Mg alloy.

Keywords Mg–Gd alloy · Texture · Creep · Precipitates · Crack

1 Introduction

The equilibrium solid solubility of Gd in Mg is relatively high, and the Mg–Gd system is therefore regarded as an ideal one for developing high-strength and light-weight alloys via precipitation hardening [1–10]. How to improve the precipitation response of Mg–Gd alloys as much as possible becomes an interesting and important topic in recent years. Firstly, Mg–Gd binary alloys with high Gd content above 15 wt% are found to have obvious age-hardening response [11]. However, the excessive high Gd content inevitably leads to the increase in alloy density and

the waste of RE resource. Secondly, later researches indicated that the age-hardening response of Mg–Gd alloys can be improved by using other proper RE partly instead of Gd [3, 11–13]. Thirdly, the alloying with non-RE elements can also be found to regulate and improve precipitation response of Mg–Gd alloys [6–8]. For example, Nie et al. [7] reported that additions of 2 wt% Ag and 1 wt% Zn in combination can lead to a substantial enhancement of the age-hardening response of the cast Mg–6Gd–0.6Zr alloy, which is attributed to the formation of a dense and uniform distribution of nano-scale basal precipitate plates in matrix.

Dislocation-induced precipitation is another method for improving the precipitation response. Druzhkov et al. [14] have reported that cold deformation prior to aging makes coherent γ' particles nucleate preferentially on edge dislocations in cold-worked Fe–Ni-based aged alloys, when the lattice misfit between the γ' phase and fcc γ matrix is greater than 0.05%. Čížek et al. [15] found that the dislocation density is an important parameter which influences the precipitation process in the Mg–15 wt% Gd alloy. Although the precipitation in Mg–Gd-based alloys has attracted wide attention [11, 16–20], the tensile stress and strain intervention to regulate precipitation response in a low Gd-containing binary Mg alloy is rarely reported. This work investigates the microstructure evolution of extruded

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Mg–6Gd alloy during creep. The creep stress higher than yield stress is applied in this paper, whose aim is to elucidate the effect of constant external stress on microstructure of extruded Mg–6Gd alloy during creep.

2 Experimental

The Mg–6Gd (wt%) alloy was prepared from high-purity (99.9%) Mg and Mg–30Gd (wt%) master alloy. The studied alloy was casted into a steel mold after melting in an electric furnace at 750 °C, and the ingot had the dimensions of 90 mm in diameter and 220 mm in height. The cast alloy was machined into a cylinder of 100 mm in height and 85 mm in diameter, and then homogenized at 500 °C for 24 h. The homogenized material was directly extruded at 500 °C with the ram speed of 0.6 mm/s and extrusion ratio of 28:1, followed by quenching in water. The directly extruded specimens were denoted as E samples. Tensile test of the extruded Mg–6Gd (E sample) alloy was conducted in a MTS 810 material test system at room temperature and 175 °C with the initial strain rate of 10^{-3} /s. Tensile creep tests were performed using a LZW-100G creep machine at the same temperature of 175 °C and creep stress of 150 MPa. Creep specimens with the gauge dimensions of 5 mm in diameter and 50 mm in length were cut from the extruded sample. Creep strains were measured using extensometer which was connected directly onto the gauge length of the specimens, and the creep direction was parallel to the extrusion direction. The creep sample started to fracture when the creep time reached 1100 h, and this crept sample was called as E + C sample.

Microstructure was characterized by an OLS3000 optical microscope (OM), a FEI NOVA400 scanning electron

microscope (SEM) and a Zeiss Libra 20FE transmission electron microscope (TEM). Specimens for OM and SEM observation were etched in a mixed solution (2 g picric acid + 5 ml distilled water + 5 ml acetic acid + 4 ml nitric acid + 85 ml alcohol). The thin foil samples for TEM observation were prepared by low-energy ion beam thinning. The deformation behaviors of crept sample were investigated by an Oxford HKL Channel 5 electron backscattered diffraction (EBSD) detector operating in a FEI Nano 400 SEM, and the observation surfaces were parallel to the extrusion direction (ED).

3 Results and Discussion

3.1 Microstructure of Extruded Sample Before Creep

Figure 1 shows the microstructure of E sample. The E sample is mainly comprised by the equiaxed grains, implying that dynamic recrystallization has occurred during the extrusion process. There are also a few elongated and large grains in the E sample pointed by the white arrow in Fig. 1a. In the equiaxed grain observed by TEM (Fig. 1b), a few dislocations can be observed, and the dark zone in the image suggests that the lattice distortion induced by the dislocations exists in the E sample. Furthermore, there is no particle in the interior and boundaries of equiaxed grains, which suggests that no dynamical precipitation occurs during extrusion.

Fig. 1 Microstructure of the E sample: **a** OM image along the cross section; **b** TEM image

3.2 Deformation Behavior During Tensile Test and Creep

Figure 2 shows the tensile stress–strain curves of E sample. The yield stress points of the samples at room temperature and 175 °C are ~ 120 MPa and ~ 110 MPa, respectively. In fact, we design the stress parameter according to the consideration that the dislocation should be formed prior to the precipitation during tensile creep, and therefore the creep stress (150 MPa) more than the yield stress (110 MPa) is chosen. When the E sample is tensile-tested at 175 °C, the serration arises in the tensile curve, which suggests that the reaction between the dislocation and the solute diffusion or the fine precipitate occurs [21]. Figure 3 shows the tensile creep curve of the E sample at 175 °C/150 MPa. The E sample exhibits the total tensile strain of $\sim 10.5\%$ after the creep time of 1100 h, and the fast plastic strain of $\sim 4.6\%$ can be obtained at the beginning of creep test. The plastic deformation occurs since

Fig. 2 Tensile stress–strain curve of the extruded Mg–6Gd (E sample) alloy at room temperature and 175 °C with a strain rate of 10^{-3} /s

Fig. 3 Creep curve of E sample under 175 °C and 150 MPa

150 MPa of the creep stress is higher than 110 MPa of the yield stress.

3.3 Texture Evolution During Creep

Figure 4 shows the misorientation angle distribution derived from EBSD data of the E and E + C samples. There are a large amount of high-angle boundaries with the misorientation above 20° in the E sample, and their proportion is about 81.11% calculated from Fig. 4a, and just a few low-angle boundaries exist in the E sample. However, the low-angle boundaries with the misorientation below 5° predominate in the E + C sample, and their proportion is as high as 79.5% calculated from Fig. 4b. Although the dislocation morphology in the E + C sample is not observed in our present work, the finding of profuse low-angle boundaries in the E + C sample suggests that dislocation deformation is still the main deformation mode [22, 23].

Figure 5a and b shows the inverse pole figures (IPFs) from EBSD analysis of the E and E + C samples containing only the recrystallized grains, respectively, and the reference direction is parallel to ED. The E sample exhibits a weak texture component $\langle 11\bar{2}1 \rangle \parallel \text{ED}$ with the maximum value of 1.82 (Fig. 5a), which is similar to that in other extruded Mg–RE alloys [24, 25]. The E + C sample also shows a weak texture component $\langle 11\bar{2}1 \rangle \parallel \text{ED}$ with the maximum value of 2.35 (Fig. 5b). The result suggests that there is little change in texture intensity, and no change happens in texture type due to the low strain of $\sim 10.5\%$ during creep. Figure 5c, d shows the IPF maps of E sample and E + C sample, respectively, and the reference direction is parallel to transverse direction (TD). The average grain size of E sample is $\sim 10.8 \mu\text{m}$, and it is $\sim 11.1 \mu\text{m}$ for the E + C sample, obtained from the EBSD data. This suggests that there is no grain growth and dynamical recrystallization during creep due to the relatively low creep temperature. Figure 6 shows the IPF maps of the E and E + C samples containing both recrystallized grains and deformed grains, and the reference direction is parallel to ED. The deformed or elongated grains have an orientation close to $\langle 10\bar{1}0 \rangle \parallel \text{ED}$ in both samples, which suggests that these are recovered grains inherited from the extrusion process, with the typical deformed orientation. However, for recrystallized grains, there are many grains with orientation close to $\langle 0001 \rangle \parallel \text{ED}$ in the E sample, while grains with orientation close to $\langle 0001 \rangle \parallel \text{ED}$ disappear in the E + C sample. Grains with other orientations like $\langle 10\bar{1}0 \rangle \parallel \text{ED}$ or $\langle 11\bar{2}0 \rangle \parallel \text{ED}$ almost do not change. The result indicates that a more clear analysis of texture component close to $\langle 0001 \rangle \parallel \text{ED}$ orientation can be obtained in IPF maps referred to ED (Fig. 6) compared with those referred to TD (Fig. 5). The Kikuchi band contrast map of the E + C sample is shown in Fig. 7.

Fig. 4 Misorientation angle distribution graphs derived from EBSD of **a** E sample, **b** E + C sample

Fig. 5 EBSD-IPFs of the recrystallized grains referring to the ED for **a** E sample, **b** E + C sample; IPF maps of **c** E sample, **d** E + C sample, and the reference direction is parallel to TD. The vertical direction is parallel to ED

Fig. 6 IPF maps derived from EBSD of **a** E sample, **b** E + C sample containing the recrystallized grains and deformed grains, and the reference direction is parallel to ED. The vertical direction is parallel to ED

Some twins are visible in partial grains, especially tensile twins (red lines), and no double twin is observed in this research [26–28]. Combined with the previous literature

[29–32], the result suggests that the grains with orientation close to $\langle 0001 \rangle \parallel \text{ED}$ in the E sample would be deformed by twinning during creep.

Fig. 7 Kikuchi band contrast map derived from EBSD for the E + C sample, and the vertical direction is parallel to ED

3.4 Precipitation During Creep

Figure 8a shows the microstructure of the peak-aged condition sample with an electron beam parallel to $[0001]_{\text{Mg}}$, which is aged at 175 °C for 570 h. Figure 8b shows the microstructure of the E + C sample in the interior of grain with an electron beam parallel to $[0001]_{\text{Mg}}$. A high density and uniform distribution of nano-scale precipitates is formed in both the peak-aged condition sample and the E + C sample, which suggests that the precipitation is not influenced by the creep stress. Most of the fine precipitates (Fig. 9a) are confirmed to be the orthorhombic β' precipitates according to the index of Fast Fourier Transform (FFT) in Fig. 9b. Besides β' precipitates, a few fcc β_1 nano-scale precipitates are also found in grain interior of the E + C sample. Figure 10a and b shows the HAADF-STEM images revealing the brighter precipitate connected with the fine β' precipitates with an electron beam parallel to $[0001]_{\text{Mg}}$. These brighter precipitates are confirmed as fcc β_1 phase according to the index of FFT in Fig. 10c. In

summary, the high density of nano-scale prismatic precipitates can precipitate during both aging and creep. The results suggest that there is no effective promotion for the precipitation by the creep process containing a low creep strain compared with the aging process involving only a high temperature. However, A remarkably high tensile yield strength of 445 MPa is obtained in a Mg–14Gd–0.5Zr (wt%) alloy when this alloy is prepared by the combined processes of hot extrusion, cold rolling and aging [19]. This improvement in strength is associated with a denser distribution of precipitates. The rolling process with a high reduction in thickness can increase markedly the dislocation in matrix, which can enhance the number density of the precipitates during aging. However, for the creep, the constant stress is applied at a high temperature, which is different from rolling at room temperature. The relative low dislocation density caused by the low strain during the creep process should be responsible for the barely unchanged precipitation. Also, both the different

Fig. 8 HAADF-STEM images of microstructure of **a** sample in peak-aged condition, **b** E + C sample. The electron beam is parallel to the $[0001]_{\text{Mg}}$

Fig. 9 HAADF-STEM images of the E + C sample: **a** microstructure in the interior of grain containing the fine β' precipitates, **b** corresponding FFT of the precipitate contained in red square zone in **(a)**. The electron beam is parallel to the $[0001]_{\text{Mg}}$

Fig. 10 HAADF-STEM images of the E + C sample showing β_1 phase: **a** brighter β_1 precipitate connecting with the fine β' precipitates; **b** high-magnified image of β_1 precipitate, **c** corresponding FFT of the precipitate contained in site of red square in **(b)**. The electron beam is parallel to the $[0001]_{\text{Mg}}$

deformation mechanism and dislocation type may influence the precipitation compared creep with the cold rolling process.

Microstructure feature near grain boundaries of the E + C sample is also examined by HAADF-STEM as shown in Fig. 11. The formation of dense and uniform precipitates in grains during creep is confirmed again. Furthermore, an obvious precipitate-free zone (PFZ) along the grain boundaries can be formed during creep process (pointed by red arrows in Fig. 11a). Besides, the relatively large stable Mg_5Gd phase particles can also be observed along the grain boundary, which is produced during creep process due to a higher diffusion rate of Gd in Mg grain boundary compared with in Mg matrix. Figure 11b shows the PFZ morphology with asymmetrical distribution along the grain boundary, which maybe is due to the atomic diffusion under stress condition. Figure 11c and d shows

the microstructure image near the grain boundary zone and the corresponding EDS mapping of Gd element. The result reveals that there is segregation of Gd at the grain boundary and confirms the presence of lower Gd content in the PFZ interior, which is similar to the previous observations [33].

3.5 Crack of Crept Sample

As for the PFZ phenomenon around grain boundaries, it is harmful to the mechanical properties of Mg alloys. As shown in Fig. 12, the crack originates along the grain boundaries of the recrystallized grains, which is influenced by the PFZ microstructure due to its lack of compounds and the low content of Gd element in solid solution of the PFZ interior (Fig. 11). How to inhibit or control PFZ in precipitation-hardenable Mg alloys is another interesting topic.

Fig. 11 HAADF-STEM image of the E + C sample showing the region of grain boundaries: **a** low-magnified, **b** high-magnified images; **c** microstructure image near the grain boundary, **d** corresponding EDS mapping of Gd element

Fig. 12 SEM images of the E + C sample near the fracture: **a** crack morphology mainly in the zone of the elongated grain, **b** crack morphology mainly in the zone of recrystallized grains, **c** a high magnification of the crack morphology along the grain boundaries of recrystallized grains

4 Conclusion

The tensile creep behavior of extruded Mg–6Gd alloy has been investigated under 175 °C and 150 MPa. In this study, the extruded Mg–6Gd sample exhibits the fast plastic strain of $\sim 4.6\%$ at the beginning of creep test and the total tensile strain of $\sim 10.5\%$ after the whole creep

time of 1100 h. The result suggests that the dislocation deformation is the main deformation mode during creep, and grains with orientation close to $\langle 0001 \rangle \parallel \text{ED}$ disappear after creep. The creep process has little effect on the precipitation compared with the aging process without strain due to the relatively low strain at high temperature. The

origination of creep crack is related to the formation of precipitate-free zone (PFZ) during creep.

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