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Author(s)	Hagihara, Koji; Li, Zixuan; Yamasaki, Michiaki et al.
Citation	Materials Letters. 2018, 214, p. 119-122
Version Type	AM
URL	https://hdl.handle.net/11094/89904
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Strain-rate dependence of deformation behavior of LPSO-phases

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Abstract: This is the first report clarifying the influence of the strain rate on the deformation behavior of Mg-based long-period stacking ordered (LPSO) phases with 14H, 18R, and 10H structures. The flow stress by basal slip showed a weakly positive or negligible strain-rate dependence, while the flow stress accompanied by the formation of deformation kink bands showed a unique negative strain-rate dependence. These results give the first experimental evidence on the recent proposal that Zn and Y atoms segregate at the kink band boundaries and hinder their migration, from the viewpoint of the mechanical properties.

Keywords: Metals and alloys; Structural; Magnesium alloys; Deformation kink band; Cottrell atmosphere

1. Introduction

Recently, the long-period stacking ordered (LPSO) phase has received considerable attention as a possible strengthening phase in Mg alloys [1–3]. Mg/LPSO two-phase alloys show superior mechanical properties compared with conventional Mg alloys. Thus, practical applications using these alloys are greatly expected. The formation of a deformation band in addition to basal slip has been reported as a predominate deformation mode in the LPSO-phase. By the crystallographic analysis, the deformation band was identified to be the deformation kink band, in which the basal dislocations are aligned perpendicular to the slip plane [4-6]. However, the role of this deformation kink band on the mechanical properties of the LPSO-phase has not been sufficiently clarified, and the factors controlling the formation and migration behaviors are not known yet. To clarify them, the strain-rate dependence of the deformation behavior of the LPSO-phase as a function of the tested temperature and alloy composition was examined in this study.

2. Experimental procedure

Three alloys with compositions of $Mg_{85}Zn_7Y_8$, $Mg_{85}Zn_6Y_9$, and $Mg_{75}Zn_{10}Y_{15}$ (at%) were examined. Their mother ingots were prepared by induction melting, and directional solidification (DS) was conducted using a vertical Bridgman furnace in an Ar-gas atmosphere at a growth rate of 10 mm/h in a carbon crucible. Details regarding the preparation of DS crystals were previously described in [4,5]. The $Mg_{85}Zn_7Y_8$ crystal was then annealed at 525 °C for 3 days to increase the volume fraction of the 14H LPSO-phase. The crystal structure and microstructure were examined by transmission electron microscopy (TEM). From the obtained DS crystal, rectangular specimens with dimensions of 2×2×5 mm^3 were cut by electrical discharge machining for compression tests. Two loading orientations were selected for the tests to examine the orientation dependence of the deformation behavior, as detailed later. Compression tests were performed in the temperature range of 20–500 °C in vacuum. The nominal strain rate was 1.67×10^{-4} s^{-1} for the initial deformation but was alternatively varied to 1.67×10^{-3} s^{-1} and vice versa during the test to examine the strain-rate dependence of the flow stress.

3. Results and discussion

It is known that the stacking sequence of the close-packed planes in the LPSO-phase varies depending on its composition [4–6]. Figs. 1(a–c) show the selected-area electron diffraction (SAED) patterns of the DS crystals. From these TEM observations, the $Mg_{85}Zn_6Y_9$ and $Mg_{75}Zn_{10}Y_{15}$ DS crystals were confirmed to predominately consist of the 18R- and 10H-type LPSO-phases, respectively, as previously reported [4,5]. Regarding $Mg_{85}Zn_7Y_8$, the as-DS-grown crystal predominately consisted of the 18R phase. However, after annealing this crystal at 525 °C for 3 days, the volume fraction of the 14H phase was greatly increased and became the predominant constituent phase, similar to that reported for $Mg_{88}Zn_5Y_7$ [6]. The $Mg_{85}Zn_7Y_8$ alloy annealed at 525 ºC and the as-DS-grown $Mg_{85}Zn_6Y_9$ and $Mg_{75}Zn_{10}Y_{15}$, which predominantly contained the 14H, 18R, and 10H LPSO-phases, were used for mechanical testing.

The texture developed in the DS crystals was previously reported in detail [4,5]. Figs. 1(d–f) show the typical microstructures in the DS crystals. In all DS crystals, the LPSO-phase grains showed plate-like shapes with interfaces parallel to (0001), and the grains were well-aligned as the plate-like interface became parallel to the growth direction. From these DS crystals, rectangular specimens with two different loading orientations were prepared: one with the loading direction parallel to the growth direction (0°-orientation) and another inclined 45° from the growth direction (45°-orientation). Thus, in the 0°-oriented specimen, the basal plane in most of the grains was aligned almost parallel to the loading axis, and in the 45°-oriented specimen, the basal plane in some of the grains was inclined ~45° from the loading orientation.

Figs. 2(a, b) show the temperature dependence of the yield stress and the typical deformation microstructures of the DS crystals, which were partly reported in [5]. As shown in Fig. 2(a), the yield stress for both loading orientations showed a large difference for all LPSO-phases because of the operation of different deformation modes in these orientations, as shown in Fig. 2(b). In the 45° orientation, introduction of slip traces parallel to the grain boundaries was observed in all the specimens. This indicates that the basal slip is operative, resulting in a low yield stress. However, in the 0°-orientation, the plastic deformation occurred mostly via kink bands formation since the operation of basal slip was mostly prohibited owing to the small Schmid factor. In the three DS crystals, a difference in the operative deformation mode was not observed at temperatures below 400 °C, although a higher occurrence of nonbasal slip, i.e., $\{10\bar{1}0\}$ prismatic slip [7], was observed for $Mg_{85}Zn_7Y_8$ above 400 °C, similar to that reported for annealed $Mg_{88}Zn_5Y_7$ [6].

Figs. 3(a–c) show the typical stress–strain curves from compression tests. At the points indicated by arrows, the strain rate, $\dot{\varepsilon}$, was abruptly changed by a factor of ten to examine the variation in the flow stress, $\Delta\sigma$. As common features observed for all DS crystals, the stress–strain curves showed relatively smooth morphologies for deformation in the 45°-orientation, while small serrations were observed in the 0°

orientation, especially for the $Mg_{85}Zn_7Y_8$ crystal at 200–400 °C. To discuss the variation in the strain rate sensitivity (SRS) of the flow stress in detail, Figs. 3(d, e) show the temperature dependence of the SRS factor, S, defined as follows:

$$S = \frac{1}{T}\left(\frac{\Delta\sigma}{\Delta ln\dot{\varepsilon}}\right) \qquad (1)$$

Positive and negative S values mean that the flow stress increases or decreases when increasing the strain rate, respectively. The temperature dependence of the SRS for the flow stress showed different features for the two loading orientations. In the 45°-orientation, S was weakly positive or negligibly small at low temperatures for $Mg_{85}Zn_6Y_9$ and $Mg_{75}Zn_{10}Y_{15}$. Similar features were observed for $Mg_{85}Zn_7Y_8$, but a weak negative dependence was observed at around 300 °C. At temperatures above 400 °C, S showed large positive values for all crystals. In the 0°-orientation, on the other hand, S was weakly negative for all crystals for temperatures of 20–300 °C, followed by a drastic increase to positive values. The magnitude of the "negative" S factor at intermediate temperatures was much larger for $Mg_{85}Zn_7Y_8$ compared to those for $Mg_{85}Zn_6Y_9$ and $Mg_{75}Zn_{10}Y_{15}$.

One of the plausible origins for the negative SRS is the occurrence of dynamic strain aging during deformation; the so-called Portevin–Le Chatelier (PLC) effect. Indeed, the formation of a Cottrell atmosphere, i.e., the segregation of Y and Zn atoms around the basal dislocation and deformation kink band boundary, was recently reported by high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) observation [8-10]. In particular, Hu et al. observed the line-like concentration profile of segregated Zn/Y atoms as a trace for escape of a kink band boundary from the Cottrell atmosphere [8]. The present results demonstrate the first evidence obtained by mechanical testing suggesting the occurrence of the PLC effect during deformation. For the individual operation of basal dislocations, the drag force caused by the Cottrell atmosphere may not be sufficiently strong to manifest the significant serration on the stress–strain curves in most of the tested conditions at 45°-orientation. However, in the motion of the kink band boundary, which is formed by the collective arrangement of basal dislocations perpendicular to the (0001) slip plane, the effect must be amplified. That is, the serration observed in the stress–strain curves for deformation in the 0°-orientation is the manifestation of pinning effect by the dragging atmosphere evolution along the kink and boundary and the subsequent escape from it. The present results suggest that the segregation of Zn and Y atoms at the kink band boundaries hardens the LPSO-phase by hindering their motion, as previously proposed from TEM observation results [8–10].

In the present results, the extent of the negative SRS was measured to be stronger for $Mg_{85}Zn_7Y_8$ (14H) than those for $Mg_{85}Zn_6Y_9$ (18R) and $Mg_{75}Zn_{10}Y_{15}$ (10H). In LPSO-phases, Y and Zn atoms are known to exist on particular stacking planes (chemically modulated planes) and form $L1_2$-like clusters [11]. Kimizuka et al. reported that as the number of $L1_2$-like clusters increases, the system undergoes a continuous evolution into a highly ordered densely packed one while maintaining a high degree of six-fold symmetric order, which is mainly attributable to an entropic effect [12]. This was experimentally confirmed in Figs. 1(a–c), as the intensity of the ordered spot increased as the Y content increased, which is derived from the development of the in-plane ordered arrangement of the $L1_2$-like cluster. In other words, the loose binding of $L1_2$-like clusters in the low-Zn/Y-containing LPSO phase might be related to an enhancement in the occurrence of the PLC effect. Further studies are required to elucidate the details.

4. Conclusion

The flow stress of the LPSO phase during deformation accompanied by the formation of deformation kink bands was found to show a negative strain-rate dependence. This result strongly suggests that the dynamic segregation of Y and Zn occurs at the kink band boundaries and it inhibits their migration, affecting the mechanical properties of the LPSO phase.

Acknowledgement

This work was supported by the Light Metals Educational Foundation of Japan and by Cross-Ministerial Strategic Innovation Promotion Program (SIP) from the NEDO, Japan.

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Figure captions

Fig. 1 (a–c) SAED patterns observed along $[11\bar{2}0]$ in the TEM and (d–f) typical microstructures observed for the longitudinal section along the growth direction in the DS crystals. (a, d) $Mg_{85}Zn_7Y_8$ annealed at 525 °C for 3 days, (b, e) as-DS-grown $Mg_{85}Zn_6Y_9$, and (c, f) as-DS-grown $Mg_{75}Zn_{10}Y_{15}$. The rows of the ordered diffraction spots and/or streaks are indicated by arrows in Fig. 1(a–c).

Fig. 2 (a) Temperature dependence of the yield stress in the DS crystals and (b) typical deformation microstructures observed in the specimens deformed at 300 °C.

Fig. 3 Typical stress–strain curves from compression tests of (a) 14H-$Mg_{85}Zn_7Y_8$, (b) 18R-$Mg_{85}Zn_6Y_9$, and (c) 10H-$Mg_{75}Zn_{10}Y_{15}$ DS crystals. (d,e) Corresponding variations in strain rate sensitivity (SRS) factor as a function of temperature, measured for the flow stress at ~1% plastic strain in the (d) 0°- and (e) 45°-orientations.