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Deformation twinning in a Mg–Al–Gd ternary alloy containing precipitates with a long-period stacking-ordered (LPSO) structure

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A magnesium alloy containing the Mg–Al–Gd long-period stacking-ordered (LPSO) platelet precipitates parallel to the basal plane was found to deform by *c*-axis tension twinning on $\{11\bar{2}1\}$, whose passage causes the bending of the LPSO platelets, when Mg grains are oriented favorably for extension along the *c*-axis during plane-strain compression at room temperature. The bending of the LPSO platelets was found to be caused by the deformation twinning equivalent to $\{11\bar{2}1\}$ twinning in the Mg matrix.

Keywords: Magnesium alloys; Deformation structure; Twinning; Transmission electron microscopy (TEM)

Recently, magnesium alloys containing Mg–TM (transition-metal)–RE (rare-earth) ternary precipitates with long-period stacking-ordered (LPSO) structures have attracted considerable attention as promising lightweight structural materials because of the simultaneous achievement of high strength (~600 MPa) and good ductility (~5%) [1]. It is usually reported that these excellent mechanical properties are achieved only after extrusion at high temperatures above 350 °C [2]. Although grain refinement of the Mg matrix in the vicinity of bent LPSO platelet precipitates as a result of recrystallization has been considered as a possible reason for the observed high strength and good ductility [3,4], the detailed mechanisms behind this have largely remained unsolved. The lack of knowledge on fundamental properties of the LPSO phase, such as crystal structure, thermal stability and deformation mechanisms, is largely responsible for this. Hagihara et al. [5,6] were the first to make a systematic study on the deformation mechanisms of the LPSO phase (Mg–5 at.% Zn–7 at.% Y) with the use of directionally solidified (DS) ingots consisting of LPSO platelets with their platelet faces (parallel to the basal plane) nearly parallel to the growth direction. They concluded that basal slip is by far the easiest slip system operative in most orientations while deformation bands are formed when the compression axis is almost parallel to the basal plane so that the operation of basal slip is suppressed. They further concluded from transmission electron microscopy (TEM) observations that the deformation bands they observed are actually kinks formed by numerous basal dislocations accumulated perpendicularly to the basal planes in a wall, as in the classical explanation for the kink formation in hexagonal close-packed (hcp) metals by Hess and Barrett [7]. Hagihara et al. inferred that the kink formation is responsible for the bending of LPSO platelet precipitates observed in hot-extruded Mg alloys [5]. However, the rotation angles between two crystalline regions separated by a kink boundary in Figs. 14 and 15 of Ref. [5] are as large as 30–60°, which requires a basal dislocation every few basal planes in the kink wall. On top of that, it is very difficult to see each individual dislocation in the kink wall in Figs. 14 and 15 of Ref. [5]. These findings raise a serious question as to whether or not all these kink bands are indeed formed by the accumulation of basal dislocations, which moved on closely spaced atomic

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planes, in the wall perpendicular to the basal plane. In the present study, we investigate deformation microstructures of a Mg–Al–Gd alloy containing a ternary LPSO phase after plane-strain compression deformation at room temperature, in order to gain insight into the mechanisms behind the bending of LPSO platelets during hot extrusion, excluding any high-temperature effects such as dynamic recrystallization. In our recent studies [8,9], the crystal structure of the LPSO phase in the Mg–Al–Gd system has been found to be based on the stacking of six-layer structural blocks (18*R*-type) as described by order–disorder (OD) theory [8–10], and generally possesses a one-dimensional disordered nature along the stacking direction.

An ingot of a Mg–Al–Gd ternary alloy with a nominal chemical composition of Mg–1.5 at.% Al–6.5 at.% Gd was produced by high-frequency induction-melting mixtures of high-purity Mg, Al and Gd in a carbon crucible with a lid in vacuum. The ingot was heat-treated at 525 °C for 64 h. Rectangular specimens for plane-strain compression tests measuring 2.2 mm × 2.3 mm × 5.5 mm (in the *x*, *y* *z* directions, respectively, in Fig. 1a) were cut from the heat-treated ingot by electric discharge machining, and were polished mechanically and then electrolytically in a solution of perchloric acid, n-butyl alcohol and methanol (1:30:130 by volume) with 0.2 M of LiCl at −55 °C. Plane-strain compression tests were performed at a nominal strain rate of $5.5 \times 10^{-4}\ s^{-1}$ at room temperature with a channel die similar to that designed by Chin et al. [12]. As seen in Figure 1a, the compression axis was set parallel to the *z* axis, while the channel die was placed to suppress specimen deformation in the *y* axis, allowing specimen extension only in the *x* direction. Compression deformation was made until the reduction measured in the *z*-axis reaches ∼15% with the specimen wrapped in polytetrafluoroethylene film to reduce friction with the channel die and compression jig. Deformation microstructures were examined by scanning electron microscopy (SEM), TEM and scanning transmission electron microscopy (STEM). Specimens for TEM/STEM observations were cut perpendicular to the *y*-axis of the specimen, polished mechanically and then ion-milled with 5 (initial) or 2 (final) keV Ar ions.A SEM backscattered-electron image of Figure 1b depicts a deformation structure of a Mg grain that is located on the cross-section perpendicular to the *y* direction in Figure 1a. For this particular Mg grain, the compression axis is approximately parallel to $[25\ \bar{5}\ \overline{20}\ 1]$, which is almost parallel to the basal plane. The *x*, *y* and *z* axis directions are indicated in Figure 1b. Many thin platelet precipitates of the LPSO phase, which appeared as bright bands in Figure 1b with platelet faces parallel to the basal plane of Mg, are observed to be bent frequently, and appear as deformation kinks after hot extrusion [4]. It is noteworthy in Figure 1b that the bending angles observed for LPSO platelets are almost identical. Orientation mapping by electron backscattered diffraction (EBSD) clearly indicates that the bending of LPSO platelets is formed as a result of the passage of deformation bands, as seen in Figure 1c. There are two sets of deformation bands: one with the boundary parallel to $(11\bar{2}1)$ and the other with the boundary parallel to $(\overline{11}21)$. Each of these two sets of deformation bands has an identical orientation, exhibiting a particular orientation relationship with the other areas (designated arbitrarily as the matrix). Orientation analysis by EBSD for the Mg grain revealed that the matrix and the deformation bands with boundary planes parallel to $(11\bar{2}1)$ and $(\overline{11}21)$ are both approximately in a 34° rotation relationship around $[\bar{1}100]$. This is further confirmed by TEM observations, as shown in Figure 1d. A TEM bright-field image of Figure 1d clearly indicates that the bending of LPSO platelets is always accompanied by the passage of deformation bands with the boundary plane parallel either to $(11\bar{2}1)$ or to $(\overline{11}21)$. In addition, many dislocations are observed to be generated in the Mg phase in both the matrix and deformation bands. Diffraction analysis by

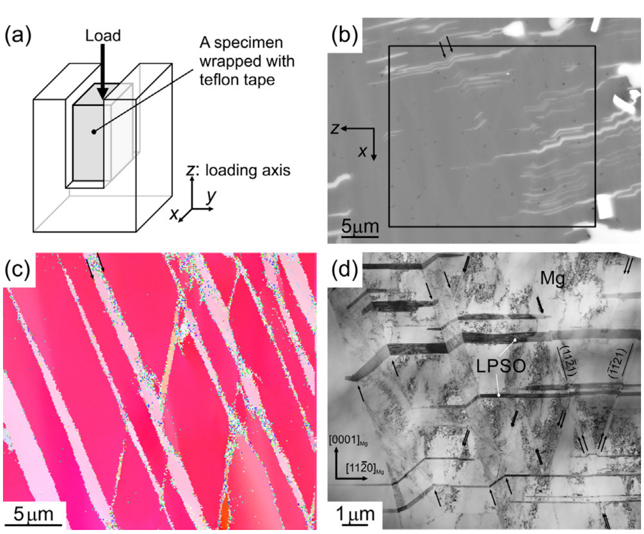

Fig. 1. (a) Geometric configuration of plane-strain compression and (b–d) deformation microstructure of Mg–Al–Gd ternary ingot deformed in plane-strain compression. (b) SEM backscattered electron image, (c) EBSD orientation map and (d) TEM BF image. Arrows in (b) and (d) indicate boundaries between matrix and twinned regions.

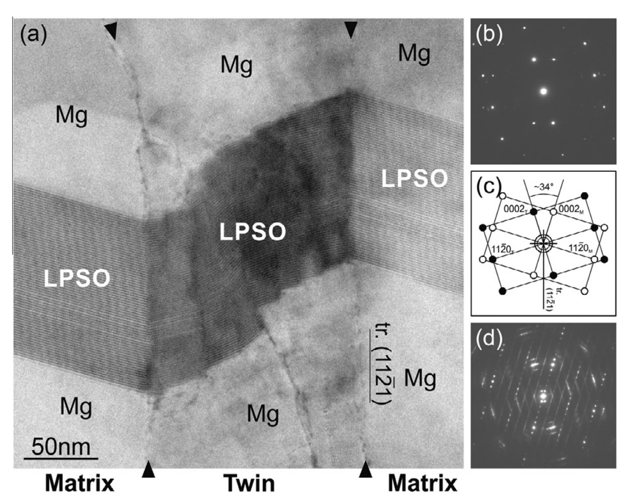

Fig. 2. (a) BF-STEM image of a deformation twin and (b–d) SAD patterns taken from (b) Mg phase and (d) LPSO phase. (c) Schematic illustration of the SAD pattern of (b).