Detwinning in Mg alloy with a high density of twin boundaries

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Abstract
To investigate the role of preexisting twin boundaries in magnesium alloys during the deformation process, a large number of {10-12} tensile twins were introduced by a radial compression at room temperature before hot compressive tests with both low and high strain rates. Unlike the stable twins in Cu-based alloys with low stacking fault energies, {10-12} twins in Mg alloy are extremely unstable or easy to detwin through {10-12}-{10-12} re-twinning. As a result, non-lenticular residual twins and twin traces with misorientation of 5°–7° were present, as confirmed by electron backscatter diffraction. The extreme instability of the twins during compression indicates that both twin and detwinning require extremely low resolved shear stresses under our experimental conditions.

Keywords: magnesium alloys, forging, twinning, detwinning, electron backscattering diffraction (EBSD)

1. Introduction
Twinning and detwinning in Mg alloys during cyclic deformation processes is an important subject, which is related to either the deformation behavior [1–5] or anelastic recovery behavior of Mg alloys [6, 7]. Lou et al [8] revealed that twinning occurs during compression of AZ31B Mg alloy; detwinning occurs under the subsequent tension when the compression is relaxed. Caceres et al [6] analyzed the twinning–detwinning behavior of magnesium alloys by carrying out cyclic tensile tests. The results indicated that hysteresis curves of Mg alloys are closely related to the cyclic motions of the twin boundary during the cyclic process. In a model proposed by Li and Enoki [9–12], detwinning of Mg alloys during unloading was ascribed to the internal stress arising from the resistance of the matrix to macroscopic changes in the twinned volume. When the external stress was withdrawn, internal stress could result, at least partly, in the opposite effect, namely, deformation by detwinning. In other words, detwinning of Mg alloy can be treated as a twinning process with a reverse twin variant. These results also imply that twins in Mg alloys are not stable because of the extremely low resolved shear stress for detwinning. In contrast, the result obtained by Lu et al [13] indicated that pure copper samples with a high density of nanoscale Σ3 twin boundaries showed substantially higher tensile stresses due to the effective blockage of dislocation motion by numerous coherent twin boundaries. This is consistent with the results of Miura et al [14] where in both pure Cu and Cu–Al alloys, with average grain sizes from 8.4 to 176.0 μm, it was found that twin boundaries are almost equivalent to grain boundaries in terms of their effect on the strength, because many dislocations pile up against the stable twin boundaries during deformation. It should be noted that unlike the stable twins in Cu alloy due to its extremely low stacking fault energy, the twins in Mg alloy, especially those formed by {10-12} twinning, are extremely unstable, which can easily shrink or grow even under an extremely low stress [6]. Nevertheless, no systematic research
has ever been carried out from a microscopic viewpoint on the detwinning behavior of Mg alloy.

Hence, in the present research, in order to evaluate the role of a pre-existing twinning interface in the subsequent deformation process, the detwinning activity during hot compression was investigated in twin-containing AZ31B alloy using electron backscattering diffraction (EBSD) analysis. The results are very helpful in explaining the role of the twin boundary during deformation for not only Mg alloys but also other metallic materials with the hexagonal close-packed crystalline structure.

2. Experimental details

Cylindrical samples of AZ31 alloy (8 mm in diameter and 12 mm in height) were cut from an as-received hot-extruded rod (8 mm diameter, Osaka Fuji Corporation) followed by an annealing. The initial microstructure, shown by the inverse pole figure (IPF) and image quality (IQ) maps in figure 1, is free of twinning. It reveals a mean grain size of approximately 25 μm with a strong basal texture, in which most of the basal planes are aligned parallel to the extrusion direction (ED) (figure 1(c)).

Before the hot compressive test, the sample was pre-stressed at room temperature by application of radial stress (about 50 MPa) normal to the lateral surface of the sample using custom-designed equipment (figure 2). Prior to compression, annealing on all samples was carried out at 250 °C.
for 5 min in order to remove the dislocations induced by the radial compression without destroying the twinning density. Hot compressive tests at 250 °C along the ED were carried out on both the radially compressed and as-received samples in a vacuum using a computer-aided Thermecmaster-Z hot-forging simulator with a strain rate of $10^{-3}$ s$^{-1}$. In these tests, it was confirmed that no further deformation twinning occurs during the deformation process; thus the activity of the pre-existing twins during the subsequent deformation process can easily be clarified. A strain rate of $10^{-1}$ s$^{-1}$ was also selected for comparison. The specimens were heated to the target temperature at a rate of 5 °C s$^{-1}$ by high-frequency induction. As soon as the samples were compressed to the final strain level, they were quenched to room temperature with high-pressure He (0.4 MPa) to freeze the high-temperature microstructure.

Microstructural analysis of the centers of the compressed samples was carried out using an EBSD instrument equipped with data acquisition software (TSL-OIM 5.0). The textures of the samples were evaluated by plotting the pole figures, where all samples were scanned by the EBSD equipment over a fixed area (540×540 μm) with a step size of 0.5 μm.

3. Results and discussion

3.1. Radially compressed microstructure

The microstructure after radial compression at room temperature is illustrated in figure 3 by IPF maps plotted for observation directions both along the ED and perpendicular to the ED. A significant microstructure refinement due to the formation of twin boundaries was observed: the mean grain size of the sample including twin boundaries was estimated to be approximately 12 μm, which is half that of the as-received sample. In the matrix grains, many near-lenticular twins are observed dispersed either in the interiors of the grains or along the grain boundaries, as indicated by black lines. They are all identified as {10-12} <10-11> tensile twins by EBSD. In contrast to the twinning with group rotations of the c-axis during uniaxial compression along the ED [12], under radial compression the twinning is observed to occur with rotations of the c-axis almost within the plane perpendicular to ED. As a result, no noticeable variation in the overall texture of the sample is observed, as indicated by the (0001) pole figure in figure 3(c). For a more detailed analysis of these
special twins, four numbered grains $M_1$, $M_2$, $M_3$, and $M_4$; their corresponding twins $T_1$, $T_2$, $T_3$, and $T_4$ were selected, as indicated in figure 3(b). The orientations of the $c$-axes before and after twinning are indicated in figure 3(d) by the (0001) pole figure. The twinning variant was chosen so that the orientation of the $c$-axis was mostly within ±30° of the equator in the pole figure.

3.2. Stress–strain curves

For samples with and without radial compression, the true stress–true strain ($\sigma$–$\varepsilon$) curves obtained at 250 °C and strain rates of $10^{-3}$ s$^{-1}$ and 10 s$^{-1}$ are presented in figure 4. It should be noted that after a large number of $\{10\text{-}12\}$ twin boundaries was introduced, the radially compressed sample did not demonstrate significant increase in stress as reported by both Xin et al [15] and Furui et al [16], in contrast to the as-

Figure 4. Stress–strain curves of the as-extruded sample and radially compressed sample obtained at 250 °C and strain rates of $20$ s$^{-1}$ and 0.001 s$^{-1}$, respectively.

Figure 5. (a) IPF map and (b) corresponding (0001) pole figure of the radially compressed sample after hot compression at 250 °C and 10 s$^{-1}$ to a true strain of 0.02.

Figure 6. IPF maps of the radially compressed sample after hot compression at 250 °C and $10^{-3}$ s$^{-1}$ to true strains of (a) 0.02, (b) 0.04, and (c) 0.08.
received sample. As found in most research on Mg alloys without prior radial compression [17–20], the flow curves obtained at a strain rate of $10 \text{s}^{-1}$ exhibit yield points at around 62 MPa for both samples, followed by strong work hardening reflected as a concave shape. Similarly, the flow curves of the samples obtained at a low strain rate demonstrate comparable stress levels; after both samples yield at a lower point (about 35 MPa), clear work softening is observed.

3.3. Microstructure evolution

During compression at $10 \text{s}^{-1}$, the twin that occurred during the prior radial compression can no longer be observed, even after the strain reaches 0.02, as shown in figure 5(a). In contrast, a fresh twin with a group rotation of $c$-axes into the ED occurs extensively. As a result, a significant increase in the maximum value of the (0001) plane density along the ED in the pole figure occurred as shown in figure 5(b).

Figure 6 shows IPF maps illustrating the microstructures of radially compressed samples subsequently subjected to compression at $10^{-3} \text{s}^{-1}$ along the ED to strains of 0.02, 0.04, and 0.08. With increasing strain, the twins formed during the radial compression disappear gradually, indicating the occurrence of detwinning similar to that occurring at the higher strain rate, but with a much lower rate. No dynamic recrystallization is observed at these strain levels; in addition the texture did not vary significantly, which is quite similar to those of both the as-received (figure 1) and the radially compressed samples, as shown in figures 7(a)–(c), except for the slightly decreased (0001) plane density. The corresponding misorientation angle distributions in the range of $2^\circ$ to $100^\circ$ at these strains are plotted in figure 8. Two separate boundary misorientation peaks at angles of $5^\circ$–$7^\circ$ and $86^\circ$–$88^\circ$ were observed. It is also clear from the radial axis distributions of the peak boundaries in the insets in figure 8 that these peaks share a $<2-1-10>$ rotation axis. In addition to the decrease in the intensity of the [10-12] twinning peak ($86^\circ$–$88^\circ$/<2-1-10>) with strain, a gradual increase in the fraction of $5^\circ$–$7^\circ$/<2-1-10> low-angle boundaries (LABs) was clearly observed, implying that the twin boundaries were possibly replaced by these LABs after detwinning.

Figure 9 shows the magnified microstructures after hot compression ($10^{-3} \text{s}^{-1}$) to a true strain of 0.02 as IPF and IQ maps. Partial detwinning is observed clearly at this strain...
level. In contrast to the detwinning induced by reverse loading, which left residual lenticular twins without twinning traces, as observed by Caceres et al [6] and Yasutomi and Enoki [21], the detwinning behaves very inhomogeneously under the present conditions and seems to initiate not from the tips but in the interiors of preexisting twins. Therefore, the residual twins observed in the present research are much more irregular; distinct twin boundary traces are observed after detwinning, as indicated by the white lines in the IPF map (figure 9(a)) and the contrast in the IQ map (figure 9(b)). A large number of granular grains finer than 1 μm that are sporadically distributed along the twin traces or in the interior of the detwinned region are observed to roughly follow the original twinning relationship with the detwinned area, but they finally disappear together with the residual twins at higher strain levels (figure 6(c)). From the abovementioned results, it is clear that the twin boundary is extremely unstable under the conditions investigated here and that detwinning tends to take place at a low strain level.

Typical twinning traces in figures 9(a), (b) are selected and numbered from a to h, and the resulting point-to-point misorientation profile across these traces indicates that most of the detwinning leads to LABs with misorientation angles of 5°–7° (figure 9(c)). To analyze these twinning traces in more detail, the misorientation angle distributions of the boundaries in figure 8(a), which dominantly include both the residual twin boundaries and the twinning trace boundaries, were plotted in figure 9(d). The rotation axis distributions of the prominent peaks are also included. Like those in figure 8, the results indicate two separate boundary misorientation peaks in the ranges of 5°–7° and 86°–88°, with their overall number fraction above 95%. From the corresponding rotation axis distributions in figure 9(d), both of these peaks are observed to have the same rotation axis, <2-1-10>. On the basis of these results, it is confirmed that the detwinning occurring under the conditions investigated here proceeds through formation of {10-12}-{10-12} re-twin boundaries [22–24], leaving twinning traces with misorientation angles of 5°–7°.

3.4. Detwinning

Both twinning during radial compression and detwinning in the subsequent hot compression are schematically described in figure 10. In figure 10(a), position (1) denotes the orientation of the initial parent grain in the as-received sample, with its c-axis roughly normal to the ED. When {10-12} twinning occurs during the radial compression, reorientation...
by approximately 87° occurs within the plane perpendicular to the ED of the extruded sample from (1) to (2), because this twinning variant has the highest Schmid factor. During the subsequent compression along the ED, there are two possible twinning variants for detwinning: reorientation of the c-axis forward by another 87° following \((2) \rightarrow (1)\), which is the same twinning variant that occurs during radial compression, and reorientation in the reverse direction to the original position following \((2) \rightarrow (1)\). Detwinning through the \((2) \rightarrow (1)\) route will give rise to a complete disappearance of the twin boundary or no twinning trace, like that during reverse loading [21, 25]. In contrast, detwinning through the \((2) \rightarrow (3)\) route actually proceeds through \((10-12)-(10-12)\) re-twinning which will be accompanied by a transition of the twin boundaries (87°) into LABs with misorientation angles of about 6° (figure 8).

Three different regions were selected and indicated in figure 9(a) by characters \(M, T,\) and \(D\) to denote the parent grain, the twinned region formed under the radial compression, and the detwinned region, respectively. For the parent grain \(M\), as shown schematically in figures 10(c) and (d), the chosen twinning variant exhibits shear along the red arrow around the \(a\)-axis with a rotation of 87° to produce the \((10-12)-(10-11)\) twin \((T)\) during the radial compression. In the subsequent compression along the ED, detwinning proceeds through double twinning in the interior of twin \(T\) with exactly the same twinning variant, resulting in twinning traces with misorientation angles of 5°–7° with respect to the matrix \(M\) (the angle between \(c_M\) and \(c_T\)) after the double rotation of the c-axis around the same axis \(a\).

The Schmid factors and the corresponding critical resolved shear stresses (CRSSs) for various deformation modes in the interior of the parent grain \(M\) and in the interior of twin \(T\) were calculated based on the EBSD measurements as tabulated in table 1. The Schmid factors of the non-basal slips in both \(M\) and \(T\) are above 0.45, much higher than those of the basal slip systems (0.274 for \(M\) and 0.099 for \(T\)). For the basal slip system, the Schmid factor in \(T\) decreased to close to 0.1 from the 0.27 Schmid factor in \(M\) because of reorientation of the grain by twinning, implying that the basal slip activity in \(T\) becomes lower while the non-basal slips activity is increased accordingly. The non-basal slip activity is also higher because of the substantially decreased CRSS of non-basal slip systems at elevated temperature [26–28]. For detwinning along the \((2) \rightarrow (1)\) route with the \((-1012)<-10-11>\) twinning variant or the \((2) \rightarrow (3)\) route with the reverse twinning variant \((-1012)<10-1-1>\), as indicated in figure 11, both Schmid factors are extremely close to 0 (table 1). The resolved stresses for detwinning at this strain level (2%) through the \((2) \rightarrow (1)\) and \((2) \rightarrow (3)\) double twinning processes are calculated to be about 1.55 MPa and –1.55 MPa, respectively, implying an extremely low possibility of detwinning activated directly by the external stress at such a low stress level, especially for route \((2) \rightarrow (3)\).

This is in conflict with most previously reported results [24–27], where twinning activity was mostly dominated by the Schmid factor. In contrast to the low activity of non-basal slip systems at room temperature, at 250 °C non-basal slip systems are considered to be strongly activated to accommodate the deformation due to the drastically decreased CRSS at higher temperature [29–32] (table 1). It is thus speculated that the activation of a large amount of non-basal slips in the interior of the twinning region possibly alters the stress field inside the twins region, with the result that activation of \((-1012)<10-1-1>\) double twinning becomes possible. This is also consistent with our present results, where at a low strain level a residual twinned granular structure remained along the preexisting twin boundaries after hot compression, as indicated by double arrows in figure 8(a). Therefore, at a higher strain rate, detwinning occurred at a...
Table 1. Schmidt factors of several deformation modes calculated in the $M$, $T$, and $D$ regions marked in figure 9(a) and the corresponding critical resolved shear stresses.

| Variant  | Basal slip  | Prismatic slip | Pyramidal slip | Double twin | Double twin |
|----------|-------------|----------------|----------------|-------------|-------------|
|          | $(0001)<11-20>$ | (1-101)<11-20> | $(11-22)<1-123>$ | (-1012)<10-11> | (-1012)<1-101> |
| $M$      | 0.274      | 0.467          | 0.447          | 0.0294      | -0.0294     |
| $T$      | 0.099      | 0.454          | 0.492          | 0.0294      | 3$^a$       |
| CRSS     | 0.5$^b$    | 13$^b$         | 13$^b$         | 3$^a$       | 3$^a$       |

$^a$ The values at ambient temperature [31].  
$^b$ Calculated based on the fact that the CRSS for non-basal slip is about 25 times of that for basal slip at 250 °C [28–31].

much smaller strain (figure 5) than at the lower strain rate, possibly because of the higher stress applied to the sample. In particular, the shear stress for detwinning indirectly applied by the activation of non-basal slips in the interior of the twinning greatly promoted the detwinning.

4. Conclusions

In order to investigate the role of preexisting twin boundaries in magnesium alloys in the deformation process, hot compressive tests were conducted with both low and high strain rates on samples in which a large number of {10-12} tensile twins were introduced by radial compression at room temperature before the hot compression. The results are summarized as follows:

(1) The {10-12} twins in Mg alloy are extremely unstable; detwinning through {10-12}→{10-12} re-twinning was confirmed by electron backscatter diffraction. In this double twinning process, the same twinning variant occurs as had proceeded to form the preexisting twin, resulting in non-lenticular residual twins and twinning traces with a misorientation of 5–7°.

(2) Both twinning growth and detwinning require extremely low resolved shear stress; for a Mg alloy with a large grain size, strengthening by introducing twin boundaries to refine the grain size is not as effective as reported before.

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