Orientation Dependence on Bending Deformation Behavior of Pure Zinc Single Crystals*1

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Three-point bending tests were performed on pure zinc single crystals with different crystal orientations to investigate orientation dependence on bending deformation behavior. With basal planes parallel to the neutral planes, the specimens deformed due to basal slips and formed a gull shape after deformation. On the other hand, specimens whose neutral planes are perpendicular to the basal planes and neutral axes are perpendicular to [0001] deformed due to second order pyramidal (c + a) slips, {1012} twinning and basal slips within {1012} twins, displaying a V shape. The bending deformation behavior of pure zinc single crystals was found to show strong orientation dependence. Also, the bending deformation behavior of pure zinc single crystals was found to differ from that of pure magnesium single crystals when the neutral plane and neutral axis are respectively perpendicular to the basal planes and [0001]. [doi:10.2320/matertrans.MT-M2021253]

Keywords: basal slip, pyramidal slip, {1012} twin, serration, neutral plane, magnesium

1. Introduction

Zinc is most commonly used in the galvanization of iron and steel products because of its excellent corrosion resistance. On the other hand, the structural application of zinc is limited because its strength and formability are low. Recently, zinc has been reported to exhibit ideal physiological corrosion behavior as bioabsorbable stents1 and is an attractive metal for use in biomedical structural materials. Here, pure zinc reportedly has crystallographic textures after rolling and shows crystal orientation dependence in its tensile strength.5,6) Such anisotropic deformation results from the crystal structure of zinc: a hexagonal close-packed crystal structure with low symmetry. The main slip system and twinning type of zinc are {0001}(1120) basal slip7 and {1012} twin,8,9) respectively. However, basal slip never occur when the loading axis is perpendicular or parallel to the basal planes. Also, {1012} twins never occur geometrically when the loading direction in compression or tension is parallel or perpendicular to the basal planes. Therefore, when basal slips or {1012} twins do not occur, zinc is expected to show anisotropic deformation behavior. However, the number of studies on the mechanical properties of zinc remains limited.

Bending deformation is a key process to produce structural parts. Effects of textures on strength and elongation have been reported in bending deformation tests of two types of rolled zinc alloy sheets with different textures.10) Single crystals are suitable in order to understand the effect of crystal orientations on deformation behavior. Pure magnesium11) and Mg–Y12) alloy single crystals with an hcp structure have been reported to deform due to basal slips and form a gull shape in specimens whose neutral planes are parallel to their basal planes. On the other hand, they formed a V shape in specimens whose neutral planes are perpendicular to their basal planes, since {1012} twins were generated at the compression side. Thus, pure magnesium and Mg–Y alloy single crystals show strong dependences on crystal orientations in bending deformation. In magnesium, critical resolved shear stress (CRSS) for basal slip is the lowest, followed by CRSS for the {1012} twin. On the other hand, in zinc, CRSS for basal slip is the lowest, followed by CRSS for second order pyramidal (c + a) slip (SPCS).13) CRSS for {1012} twin in zinc is much larger than that for basal slip and SPCS. Also, shear directions for {1012} twinning in zinc and magnesium are opposite since the c/a ratio of zinc is larger than √3. Therefore, the bending deformation behavior of zinc is also expected to differ from that of magnesium. In this study, pure zinc single crystals with different orientations were applied to three-point bending tests to investigate orientation dependence on bending deformation behavior.

2. Experimental Procedures

Pure Zinc (99.999% purity) single crystals were made by the Bridgeman method. Also, pure magnesium (99.99% purity) single crystals were made by the same method for comparison. Single crystals were crystallographically analyzed using the X-ray back reflection Laue method and were cut into cuboid specimens using a non-distortion cutting machine with nitric acid. The cuboid specimens were chemically polished to be approximately 3 × 3 × 25 mm³ using chemical polishing solutions (HNO₃:H₂O₂:C₂H₅OH = 1:1:4 for pure zinc and HNO₃:H₂O₂:C₂H₅OH = 5:7:20 for pure magnesium). The specimens were thermal cyclic annealed in an argon atmosphere to remove dislocations and sub-grain boundaries induced from cutting and polishing. The polished specimens of pure zinc14) were annealed...
between 573 K and 673 K; they were held for 3.6 ks at 573 K and 673 K, with the rate of change between temperatures being $5.6 \times 10^{-3}$ K/s. A single thermal cyclic annealing time was 43.2 ks, and a total of 12 thermal cycles were applied. In pure magnesium,\textsuperscript{12} the polished specimens were annealed between 673 K and 723 K; they were held for 3.6 ks at 673 K and 723 K, with the rate of change between temperatures being $6.9 \times 10^{-3}$ K/s. A single thermal cyclic annealing time was 21.6 ks and a total of 8 thermal cycles were applied to pure magnesium.

Figure 1 shows schematic illustrations of four types of single crystalline specimens with different crystal orientations for bending tests. Kitahara \textit{et al.}\textsuperscript{11} and Oka \textit{et al.}\textsuperscript{12} employed specimens A and B whose neutral planes are parallel to (0001) but neutral axes are respectively [\(\frac{1}{2}\) 1\(\overline{1}\) 00] and [\(\frac{1}{2}\) 0\(\overline{1}\) 00], and specimens C and E whose neutral planes are perpendicular to (0001) but neutral axes are respectively [\(\frac{1}{2}\) 1\(\overline{1}\) 00] and [\(\frac{1}{2}\) 0\(\overline{1}\) 00]. In this study, specimens A, B, C and E were used for comparison. Three-point bending tests were carried out at room temperature. The support span \(L\) was 16 mm. The radii of the loading and supporting pins were \(R_1 = 2.0\) mm and \(R_2 = 2.5\) mm, respectively. The loading rate was \(1.67 \times 10^{-2}\) mm/s. Bending stress \(\sigma\) and bending strain \(\varepsilon\) were calculated from eq. (1) and eq. (2).

\[
\sigma = \frac{3PL}{2bh^2} \quad (1)
\]
\[
\varepsilon = \frac{6dh}{L^2} \quad (2)
\]

where \(P\) is the load, \(b\), \(h\), and \(d\) are the width, height, and displacement of the specimens, respectively. Table 1 shows specimen names and dimensions. Slip lines and twins were observed using a Nomarski differential interference contrast microscope and a CCD camera in order to investigate the deformation mechanism of the specimens during and after bending tests. In addition, the dislocations in pure zinc specimen B were also observed by the etch pit method after bending tests. Specimens were etched using N solution (C\(_2\)H\(_5\)OH:35%H\(_2\)O\(_2\):36%HCl:H\(_2\)O = 40:10:1:20),\textsuperscript{15,16} the pits correspond to basal and non-basal dislocations.

3. Results and Discussion

3.1 Bending stress-bending strain curves

Figure 2 shows typical bending stress-bending strain curves of pure zinc single crystals. The arrows in Fig. 2 indicate bending yield stresses \(\sigma_y\). In this study, \(\sigma_y\) is defined as bending stress when linear parts finish in bending stress-bending strain curves. \(\sigma_y\) of all specimens are shown in Table 1. Specimen A yielded at 2.1 MPa and two specimens B yielded at 1.3 and 1.5 MPa. After yielding, both specimens A and B showed low strain hardening. Bending tests were carried out until approximately \(\varepsilon = 30\%\), but neither specimen A nor B fractured. Specimen A showed serrations on its bending stress-bending strain curve at \(\varepsilon = 8\%\) or more. After yielding, both specimens C and E showed several large

**Table 1** Specimen names, dimensions and bending properties of four types of zinc (Zn) and magnesium (Mg) single crystalline specimens.

| Specimen | \(b\) (mm) | \(h\) (mm) | \(\sigma_y\) (MPa) | \(\tau_y\) (MPa) | \(\varepsilon_y\) (%) |
|----------|------------|------------|------------------|-----------------|------------------|
| Zn-A     | 3.02       | 2.84       | 2.14             | 0.11            | > 33.3           |
| Zn-B     | 2.79       | 3.26       | 1.45             | 0.10            | > 38.8           |
| Zn-C     | 1.94       | 2.94       | 1.29             | 0.08            | > 36.9           |
| Zn-D     | 3.03       | 3.16       | 28.0             | —               | > 20.9           |
| Zn-E     | 3.30       | 2.58       | 25.9             | —               | > 21.0           |
| Zn-F     | 3.29       | 2.86       | 20.3             | —               | > 24.4           |
| Zn-G     | 2.85       | 2.62       | 13.2             | —               | > 17.2           |
| Zn-H     | 2.84       | 2.98       | 11.7             | —               | 15.8             |
| Zn-I     | 2.69       | 3.23       | 17.4             | —               | > 29.4           |
| Mg-C     | 2.34       | 2.38       | 38.7             | —               | 6.35             |
| Mg-D     | 2.72       | 3.04       | 27.1             | —               | 6.18             |
| Mg-E     | 3.13       | 3.12       | 32.8             | —               | 6.12             |
| Mg-F     | 2.66       | 2.97       | 26.9             | —               | 12.5             |
| Mg-G     | 2.98       | 2.48       | 23.2             | —               | 10.8             |
stress drops and strain hardening with serrations at \( \varepsilon = 2\% \) or more followed by stress plateau regions. Neither specimen C nor E fractured until approximately \( \varepsilon = 20\% \). The serration range and the plateau stress after strain hardening of specimen E were smaller than those of specimen C. The above results show bending deformation behaviors depend greatly on crystal orientations in pure zinc.

### 3.2 Bending deformation behavior of specimens A and B

Figure 3 shows optical micrographs of compression areas in specimens A and B below the loading pin just after yielding. After yielding, slip lines parallel to the basal planes were observed in both specimens A and B, and thus they were found to yield due to basal slip. Pure magnesium single crystals were reported to show the same yield bending deformation due to basal slip.\(^{11}\) The Schmid factor (S.F.) for basal slip against the bending stress is 0 in both specimens A and B when the bending stress is parallel to the basal planes. Kitahara et al.\(^ {11}\) have proposed that yield stresses of specimens A and B in pure magnesium single crystals were controlled by shear stress \( \tau_y \) parallel to the basal planes, calculated from eq. (3).

\[
\tau_y = P/(2bh)
\]  

Here, the direction of the neutral axis, i.e., the direction of the shear stress, is parallel to [1100] in specimen A, and thus it tilts 30° from the basal slip direction, [1120]. Therefore, the value of \( P \cos 30° \) was used as the load for specimen A, and then \( \tau_y \) was calculated. The calculated \( \tau_y \) was 0.08 to 0.11 MPa; they are summarized in Table 1. The values were close to the reported CRSS for basal slip in pure zinc, ranging from 0.18 to 0.4 MPa.\(^ {8,9,17} \) Therefore, the yield stress of the pure zinc specimens A and B were found to be determined by the shear stress loading on the basal plane, similar to pure magnesium.

Figure 4 shows optical micrographs of specimens A and B after bending tests. Both specimens A and B formed a gull shape as shown in Fig. 4(a) and (d). Also, uniform slip lines due to basal slips were observed at both compression and tension sides between the supporting pins, as shown in Fig. 4(b) and (e). Also, twins occurred from the tension side to the compression side in only specimen A, as shown in Fig. 4(c); they were geometrically analyzed and identified to be [1012] twins. The same twins were in-situ observed at \( \varepsilon = 8\% \) or more with a CCD camera. Therefore, the serrations observed on the bending stress-bending strain curve of specimen A result from [1012] twinning. On the other hand, no [1012] twins occurred in specimen B. S.F. for [1012] twin against bending stress is 0.499 for specimen A and 0.374 for specimen B, suggesting that [1012] twins are less likely to occur in specimen B. Here, the bending stress required for [1012] twinning was calculated using CRSS for [1012] twin in pure zinc (23.5 to 31.85 MPa)\(^ {9} \) and S.F. for [1012] twin of specimen A against bending stress (0.499), resulting in 47.1 to 63.8 MPa. However, [1012] twins occurred at a bending stress of approximately 11 MPa in specimen A. The role of [1012] twinning on bending behavior of specimen A is quite limited since the area fraction of [1012] twins to the whole specimen was approximately 8%, though further investigation is required.

Figure 5 shows optical micrographs of etch pits in specimen B. Pits with a rounded rectangle and shallow bottom, as indicated by Pit ①, were observed below the loading pin as shown in Fig. 5(a). The pits correspond to basal dislocations\(^ {15} \) since they align along basal planes. Similar pits corresponding to basal dislocations were uniformly observed in the area between the supporting pins. On the other hand, a few lenticular pits with deep bottoms as indicated by Pit ② were also observed; they correspond to non-basal dislocation.\(^ {15} \) Here, the bending stress required for SPCS using CRSS for SPCS in pure zinc (3 to 10 MPa)\(^ {8,13,18-23} \) and S.F. for SPCS of specimen B against bending stress (0.418), result in 7.2 to 23.9 MPa. The calculated result showed that the flow stress of specimen B reaches the bending stress required for SPCS. Therefore, the pits as indicated by Pit ② were formed by SPCS; however, the number of the pits were smaller than that of the pits corresponding to basal dislocations (Pit ①). Therefore, specimen B mainly deformed due to basal slips. Figure 5(b) shows optical micrographs of etch pits in specimen B above the right supporting pin. The pits corresponding to basal
dislocation (Pit ①) piled up at the left side of the supporting pin, as indicated by the white dashed line. Specimens A and B of pure magnesium single crystals form a gull shape since tilt boundaries formed due to the pile-up of basal dislocations above the right and left supporting pins. Therefore, pure zinc also displayed a gull shape due to the pile-up of basal dislocations above the right and left supporting pins, similar to pure magnesium.

3.3 Bending deformation behavior of specimens C and E

Figure 6 shows optical micrographs of specimens C and E...
below the loading pin just after yielding; the observed planes were parallel to the basal planes. Tilted slip lines were observed on the basal planes: approximately 60° from \( \frac{1}{2} \overline{1}210 \) in specimen C (Fig. 6(a)) and approximately 90° from \( \frac{1}{2}1120 \) in specimen E (Fig. 6(b)). The observation results indicate that SPCS were activated in bending tests of both specimens C and E. Here, the bending stress required for SPCS was calculated using CRSS for SPCS in pure zinc (3 to 10 MPa)9,13,18 and S.F. for SPCS against bending stress (0.313 for specimen C and 0.418 for specimen E). Bending stresses required for SPCS was 9.6 MPa to 31.9 MPa for specimen C and 7.2 to 23.9 MPa for specimen E. These values are in agreement with \( \sigma_e \) of specimens C and E shown in Table 1. Thus, specimens C and E were found to yield due to SPCS. Hereafter, SPCS activated at yielding are referred to as SPCS1. These results show that the deformation mechanism of specimens C and E at yielding in pure zinc differs from those in pure magnesium.

Figure 7 shows optical micrographs of specimens C and E below the loading pin just after the first bending stress drop. Twins occurred from the tension side to the compression area. The twins were geometrically analyzed and identified to be \{1012\} twins. Thus, bending stress drop due to \{1012\} twin was found in specimens C and E. Here, the bending stress for \{1012\} twinning will be discussed. Ren et al.24 and Desinghegh et al.25 have reported that the neutral plane moves in three-point bending tests of rolled AZ31; the neutral plane moves to the tension side when the rolling plane is parallel to the direction of bending stress, while the neutral plane moves to the compression side when the rolling plane is perpendicular to the direction of bending stress. Also, they concluded that the neutral plane moves to the side where no \{1012\} twins occur since \{1012\} twins only occur at either the compression or tension side. Similar neutral plane movements during bending deformation have been reported in other magnesium alloys.26–28 In addition, Kitahara et al. and Oka et al. performed three-point bending tests of pure magnesium single crystals\(^{11}\) and magnesium alloy single crystals\(^{12}\) reporting that \{1012\} twins generated at the compression side and tips of \{1012\} twins reached the tension side across the center of the specimens. They evaluate bending stress at the compression side considering the
movement of the neutral plane and revealed the relationship between \(\sigma\) and the CRSS for \{10\(\overline{1}\)2\} twins. Since \{10\(\overline{1}\)2\} twins propagated across the center of the specimen in pure zinc in a way similar to what is seen in magnesium, bending stress was evaluated considering the movement of the neutral plane. Here, \{10\(\overline{1}\)2\} twinning occurs on the tensile side in pure zinc. Therefore, bending stress at the tension side in zinc was calculated using the equation (4) for bending stress at the tension side without considering the movement of the neutral plane obtained from eq. (1). \(N_t\) is the distance ranging from the bottom of the specimen to the neutral plane (the position of the tips of \{10\(\overline{1}\)2\} twins). \(x\) is the ratio of the movement of the neutral plane to \(h\). \(N_t\) can be expressed by \(N_t = 0.5h + xh\). Thus, the bending stress \(\sigma'_t\) at the tension side considering the movement of the neutral plane can be expressed by the following equation:

\[
\sigma'_t = MN_t/I = (PL/4)(12/b(0.5h + xh))^3(0.5h + xh)
\]

(4)

where \(M\) is the bending moment, and \(I\) is the area moment of inertia. \(\sigma_{\text{twin}}\) and \(\sigma'_{\text{twin}}\) were calculated from eq. (1) and eq. (4); they are shown in Fig. 8. \(\sigma'_{\text{twin}}\) is much different from \(\sigma_{\text{twin}}\). The values in gray in Fig. 8 show bending stress required for \{10\(\overline{1}\)2\} twinning calculated using CRSS for \{10\(\overline{1}\)2\} twin in pure zinc (23.5 to 31.85 MPa)\(^9\) and S.F. for \{10\(\overline{1}\)2\} of specimens C and E against bending stress (0.499 for specimen C and 0.374 for specimen E). Bending stress required for \{10\(\overline{1}\)2\} twinning ranged from 47.1 to 63.8 MPa in specimen C and from 62.8 to 85.2 MPa in specimen E, and these values are in good agreement with \(\sigma'_{\text{twin}}\) of specimens C and E. Therefore, the neutral plane in pure zinc was found to also move when \{10\(\overline{1}\)2\} twin occurred, similar to magnesium.

Figure 9 and Fig. 10 show optical micrographs of specimens C and E after bending tests. Specimens C and E displayed a V shape, resulting from activations of SPCS1 between the supporting pins and \{10\(\overline{1}\)2\} twins at the tension side. Also, tilted slip lines caused by SPCS2 were observed near \{10\(\overline{1}\)2\} twin boundaries; as tilted by approximately 0° from \{1100\} in specimen C (Fig. 9(b)) and by approximately 30° from \{1120\} in specimen E (Fig. 10(b)). Here, the slip
direction of SPCS2 differs from that of SPCS1 observed at yielding, indicating that different SPCS slip systems were activated at yielding. S.F. for SPCS2 against bending stress of specimens C and E are 0 and 0.104, respectively. H. S. Rosenbaum has performed bending tests of pure zinc single crystals with an indentation and reported that SPCS was activated near tips of \{10\overline{1}2\} twins for the stress relaxation near \{10\overline{1}2\} twin boundaries. Therefore, SPCS with small S.F. was also activated near \{10\overline{1}2\} twin boundaries by the local stress whose direction is different from the loading direction of bending stress.

In bending deformation of specimens C and E, not only slip lines but also \{10\overline{1}2\} twins were observed. The role of \{10\overline{1}2\} twins in bending deformation was investigated. Figure 11 shows changes in the area fraction of \{10\overline{1}2\} twins in specimens C and E during bending tests. The area fraction of \{10\overline{1}2\} twins in specimens C and E increased with increasing bending strain after yielding. Therefore, the serrations after yielding on the bending stress-bending strain curves of specimens C and E in Fig. 2 result from \{10\overline{1}2\} twinning. The serration of curves disappeared in specimens C and E at about 10% bending strain, and both bending stress and the area fraction of \{10\overline{1}2\} twins were almost constant. Therefore, specimens C and E deformed at \(\varepsilon = 10\%\) or more, not due to \{10\overline{1}2\} twins, but rather SPCS1, SPCS2, and basal slip within \{10\overline{1}2\} twins as shown in Fig. 9 and Fig. 10.

The area fraction of \{10\overline{1}2\} twins of specimen E was smaller than that of specimen C when bending stress was constant as shown in Fig. 11, while basal slips within \{10\overline{1}2\} twins in specimen E (Fig. 10(c)) were more activated than those in specimen C (Fig. 9(c)). Here, the crystal orientation within \{10\overline{1}2\} twins rotates approximately 90° due to \{10\overline{1}2\} twinning. Therefore, S.F. for basal slip within \{10\overline{1}2\} twins against bending stress was low (0.07) in specimen C and high (0.43) in specimen E. Thus, basal slips within \{10\overline{1}2\} twins in specimen E are activated at lower bending stress, the result being that flow stress for bending deformation in specimen E is small. In other words, in specimen E, \{10\overline{1}2\}
twins do not necessarily widely expand from the loading pin in the direction of the right and left supporting pins. The reason is that basal slips within \( f_{10/C2212} \) twins are easily activated. From the above, the serration range and the plateau stress on the bending stress-bending strain curve after strain hardening in specimen E were smaller than those in specimen C.

The bending deformation behavior of zinc was similar to that of pure magnesium\(^{11,12}\) in specimens A and B, but not in specimens C or E. In order to investigate the cause, pure magnesium single crystalline specimens with the same shape as pure zinc were prepared and their bending deformation behavior was investigated. Figure 12 shows typical bending stress-bending strain curves of specimens C and E of pure magnesium single crystals. Vertical arrows in Fig. 12 indicate \( \varepsilon_B \), the bending strain when cracks initiated. Pure magnesium also showed work hardening with serrations, similar to pure zinc. However, as shown in Table 1, no crack initiated in pure zinc specimens even when \( \varepsilon \) reached approximately 20% except for the Zn-E2 specimen, while cracks initiated in pure magnesium when \( \varepsilon_B \) ranged between 6 and 12%.

Figure 13 shows optical micrographs of specimens C and E in pure magnesium single crystals after bending tests. In pure magnesium, \( \{10\overline{1}2\} \) twins occurred on the compression side and widely expanded in the direction from the loading pin to the right and left supporting pins. Also, basal slips were activated within \( \{10\overline{1}2\} \) twins; the area fraction of \( \{10\overline{1}2\} \) twins in pure magnesium are shown in Fig. 11. In comparison with the same bending strain, the area fraction of \( \{10\overline{1}2\} \) twins in pure magnesium was about twice that in pure zinc, demonstrating that \( \{10\overline{1}2\} \) twins play large roles in bending deformation of pure magnesium. On the other hand, in pure zinc, SPCS was also activated between the supporting pins in addition to two deformation mechanisms of basal slips and \( \{10\overline{1}2\} \) twinning. Therefore, \( \varepsilon_B \) in pure zinc was larger than that in pure magnesium.

4. Conclusions

Pure zinc single crystals were applied to three-point bending tests to investigate the effect of crystal orientation on bending deformation behavior. The main results are summarized below.

(1) Bending deformation behavior of pure zinc showed a strong orientation dependence.

(2) Specimens A and B, whose neutral planes are parallel to (0001) and neutral axes are parallel to \([1\overline{1}00]\) and \([1\overline{1}20]\), yielded due to basal slips. Specimen A yielded at 2.1 MPa and two specimens B yielded at 1.3 and 1.5 MPa. Yield stresses were determined by shear stress
loading on the basal planes. Both specimens A and B showed 30% or more bending strain without cracking.

(3) Specimens C and E, whose neutral planes are perpendicular to (0001) and neutral axes are parallel to [1100] and [1120], yielded at 20.3–28.0 MPa and 11.7 to 17.4 MPa due to second order pyramidal (c + a) slip. After yielding, both specimens C and E deformed due to [1012] twinning and basal slips within [1012] twins, and showed 20% or more bending strain without cracking.

(4) Specimens C and E, whose neutral planes are perpendicular to (0001) and neutral axes are parallel to \( \frac{1}{2} \langle 221 \rangle \) and \( \frac{1}{2} \langle 11 \rangle \), yielded at 20.3–28.0 MPa and 11.7 to 17.4 MPa due to second order pyramidal (c + a) slip. After yielding, both specimens C and E deformed due to \( f_{10} \) twinning and basal slips within \( f_{10} \) twins, and showed 20% or more bending strain without cracking.

(5) Bending deformation behavior was similar to that of pure magnesium in specimens A and B, but not in specimens C or E. The difference was caused by the ease of second order pyramidal (c + a) slips in pure zinc.

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