Dislocation transmutation by tension twinning in magnesium alloy AZ31

Authors:

Fulin Wang and Sean R. Agnew

Email address:

fw8yc@virginia.edu (Fulin Wang); agnew@virginia.edu (Sean R. Agnew)

Affiliation:

Department of Materials Science and Engineering, University of Virginia, Charlottesville, VA
22904, U.S.A.

Corresponding author:

Fulin Wang

Abstract

The interaction between dislocations and $\{10\bar{1}2\}\langle\bar{1}011\rangle$ twins appears to play an important role in the strain hardening behavior of Mg. Detailed transmission electron microscopy study was performed to investigate the concept of dislocation “transmutation” across twin boundaries. A previously proposed dislocation transmutation reaction is confirmed. For $(10\bar{1}2)$ twins, the transmutation reactions involve $[a_1]$ or $[a_3]$ matrix dislocations resulting in $\langle c \pm a_2 \rangle$ dislocations, which populate the vicinity of the twin boundary. No other $\langle c + a \rangle$ slip systems are observed within the twins, despite the fact that the $\langle c \pm a_2 \rangle$ dislocations have similar or lower resolved shear stress, as compared to other slip systems. This suggests the $\langle c + a \rangle$ slip mode is source limited, since the observed slip systems are the only ones that can result from transmutation. The formation of a unit $\langle c + a \rangle$ dislocation in the twin is proposed to involve two consecutive reactions, **necessitating dislocation pile-up in the matrix**, and the associated dislocation configurations are evaluated in terms of elastic strain energy considerations. **Dislocation reactions are proposed which could explain the presence of basal stacking faults of either I_1 or I_2 type inside twin. At the low stress levels typical of $\{10\bar{1}2\}\langle\bar{1}011\rangle$ twinning dominated flow of textured polycrystals, the observed $\langle c \pm a_2 \rangle$ dislocations are most likely sessile. However, this could serve as a source mechanism for later deformation, and as a forest hardening mechanism against other slip systems. The interfacial reaction products could result in a dragging effect on twin boundary advancement, and establish a basis for subsequent rapid hardening.**

Keywords: A. Dislocations; A. Twinning; B. Polycrystalline material; C. Electron microscopy.

1. Introduction

Deformation twinning is an important mechanism in hexagonal structured metals, because of its ability to accommodate the strain along the c axis of the crystal. Among all the twinning systems, the relatively low shear of the $\{10\bar{1}2\}$ mode and high mobility of the twin boundary make it the most frequently observed (e.g., El Kadiri, Barrett, Wang and Tome, 2015). For metals whose c/a ratio is smaller than $\sqrt{3}$, such as Mg, Ti, Zr and Be, $\{10\bar{1}2\}$ twinning results in extension along the c axis, it is therefore commonly termed extension, tension or tensile twinning (Yoo, 1981).

When profuse tension twins are activated, some materials exhibit tension-compression asymmetry, including a change of concavity in the flow curve and a rapid hardening regime (Reed-Hill, 1973; Kelley and Hosford Jr., 1968). In addition to normal dislocation-dislocation interaction (Taylor hardening a la Kocks and Mecking (Kocks and Mecking, 2003)), the hardening due to deformation twinning was classically attributed to two effects (Kalidindi, 2001; Proust, 2009): the composite strengthening effect associated with reorienting the twinned crystal from a soft to a hard orientation, and twin-dislocation interaction by considering twin boundary as obstacles to subsequent slip and twinning deformation (Serra et al., 2002). Recently, a theory for latent hardening through dislocation transmutation by twinning was developed (El Kadiri and Oppedal, 2010), which hypothesizes that a rapid increase in the dislocation density or type inside twins is responsible for the larger hardening rate of the twin than that observed in the matrix. According to the dislocation transmutation theory, Oppedal et al. (2012) introduced in their model a fitting parameter called the “twin storage factor” that allows the twins harden more than the parent and showed its dominant effect on the increased hardening rate. Ma et al. (2012) showed that the incorporation of dislocation transmutation theory into latent hardening of twin-dislocation will more accurately simulate the texture and stress-strain data of magnesium alloy AM30, and suggested that at least the upper-half of the rapid hardening regime in the flow curve should be characterized by slip dominance within the twins.

Dislocation transmutation refers to the change of slip plane and Burgers vector of a dislocation when incorporated into the twin. Dislocation reactions at the $\{10\bar{1}2\}$ twin boundary have been anticipated by Price (1961) and Yoo and Wei (1966) based primarily on geometric analysis. Tomsett and Bevis (1969a) were the first to study the transmutation of dislocations

using the transmission electron microscope (TEM), and they showed the consistent appearance of $\langle a \rangle$ dislocations in the matrix and $\langle c + a \rangle$ dislocations in the $\{10\bar{1}2\}$ twin, as suggested by the previous geometric analysis; thus, the transmutation concept was confirmed. Morozumi et al. (1976) observed $\langle c + a \rangle$ dislocations near and within the $\{10\bar{1}2\}$ twins in Mg. Lay and Nouet (1994) studied the interaction of slip dislocations with a $(01\bar{1}2)$ twin in Zn, but did not observe the $\langle a \rangle \rightarrow \langle c + a \rangle$ transmutation reaction. However, they only examined dislocation movement and penetration of the twin boundary in situ, under the influence of electron beam heating in the TEM. Due to the weak stress field resulting from differential heating of the beam, their observations of dislocation interactions were limited to direct transfer of $\langle a \rangle$ type dislocations whose Burgers vectors were parallel to the common axis of matrix and twin. Bhattacharyya et al. (2009) explored the origin of the dislocations within both tensile and compressive twins in textured Zr. Rather than invoking the transmutation argument, the c -type dislocations inside the tensile twin were hypothesized to be the remnants of dislocation interactions related to the formation of the twin itself. So far, the experimental evidence of dislocation transmutation has not been explored thoroughly, and no systematic explanation of the dislocation structure observed within twins has been advanced.

The current work explores the dislocation transmutation reaction at the boundaries of $\{10\bar{1}2\}$ twins in a conventional Mg alloy using a TEM. The origin of the dislocations inside twin will be studied by focusing on the defect structure in the vicinity of twin boundary. We seek to understand the configuration and process of transmutation reactions. Finally, the potential operation of the dislocations inside the twin will be discussed in terms of the stress state.

2. Experimental procedures

2.1 Sample preparation

The studied material is a commercially rolled plate of magnesium alloy AZ31B in the H24 temper (strain hardened and partially annealed), with an average grain size of $\sim 15 \mu\text{m}$ (Wu et al., 2010). The nominal composition of the alloy is 3 wt% Al, 1 wt% Zn, and balance Mg. The rolled plate possesses a preferred crystallographic orientation with the $[c]$ axis of the crystals roughly parallel to the normal direction (ND) of the plate. The plate was subsequently

compressed along the rolling direction (RD) at room temperature and individual samples were strained to various levels (0.5, 1, 2, 4 and 8%). For TEM characterization, the samples from the plateau region in the flow curve (Figure 1a), up to 2.0% strain, were examined. Thin sections with the foil normal parallel to the transverse direction (TD) and ND respectively were cut from the cuboidal compression samples using a diamond saw (Figure 1c).

Figure 1. (a) The flow curves of the rolled plate of AZ31, whose initial texture is shown in (b). RD-C denotes compression along the rolling direction, and RD-T denotes tension along the rolling direction. The flow curves and pole figures are reproduced from (Wu et al., 2010). (c) The disks for TEM characterization were machined from the TD and ND planes of the RD-C sample.

The two foil orientations provide two perspectives for the characterization of the $(10\bar{1}2)$ twin: in the TD sample, the basal planes in the matrix and the twin, as well as the twinning plane itself are all close to edge-on (the crystallographic plane is perpendicular to the plane of the foil); in the ND sample, the basal plane in most matrix grains is close to face-on (the crystallographic plane is parallel to the plane of the foil). The thin sections were mechanically ground to a

1
2
3
4 thickness of ~250 μm and then punched into 3 mm disks. The disks were further thinned by 1200
5
6 grit SiC paper to ~150 μm . Thinning to electron transparency was achieved by electropolishing
7
8 in a solution of 500 ml methanol, 100 ml butyl cellosolve, 11.16 g magnesium perchlorate and
9
10 5.3 g lithium chloride, at c.a. 100 V, 20 mA and -50°C maintained by a solution of liquid
11
12 nitrogen and methanol within a Fischione double-jet polisher. After perforation, the disks were
13
14 rinsed in ethanol.

15 16 17 18 2.2 Characterization 19 20

21 TEM observations were performed on a JEOL 2000FX TEM with a LaB_6 cathode
22
23 operating at 200 kV. A double tilt sample stage was used to achieve various sample orientations.
24
25 The Burgers vectors of dislocations were determined based on diffraction contrast analysis
26
27 criteria (Fultz and Howe, 2001; Williams and Carter, 2009) by judging their visibility or
28
29 invisibility in bright-field (BF) images and axial dark-field (DF) images under different two
30
31 beam diffraction conditions. Weak-beam dark-field (WBDF) imaging was employed in areas of
32
33 relatively large distortion due to, e.g. dense defect content. Simulated selected area electron
34
35 diffraction (SAED) patterns and the stereographic projections used to navigate orientation space
36
37 were generated by the software, Desktop Microscopist.

38 In specific cases, the TEM thin foil was characterized by an electron backscattered
39
40 diffraction (EBSD) type technique known as transmission Kikuchi diffraction (TKD) (Keller and
41
42 Geiss, 2012; Trimby, 2012) to quantitatively obtain the orientation of individual crystals with
43
44 respect to the sample coordinate system. For this purpose, the rolling direction was marked on
45
46 the edge of TEM foil with a notch. EBSD was performed on a FEI Quanta 650 scanning electron
47
48 microscope (SEM) equipped with AZtecHKL software.
49
50

51 52 3. Twinning relationship and dislocation transmutation reactions 53 54

55 The crystallographic frames of the two lattices (twin and matrix) were established based
56
57 on the orientation relation investigated comprehensively by many authors (e.g. (Bevis and
58
59 Crocker, 1969; Partridge, 1967)), and more recently reviewed by Christian and Mahajan (1995).
60
61 With the twinning plane $K_1 = \{10\bar{1}2\}$ and the shear direction $\eta_1 = \langle \bar{1}011 \rangle$, it is a compound
62
63
64
65

twin according to the categorization in Christian and Mahajan (1995). Throughout this work, the observed extension twins are arbitrarily indexed as $(10\bar{1}2)$ with the direction $a_2 = [1\bar{2}10]$ being the common axis of matrix and twin. The crystallographic relationship is depicted in Figure 2 in both real space (a) and reciprocal space (b). The mirror symmetry of the matrix and twin and the characteristic $\sim 86^\circ$ rotation of the c axis of $\{10\bar{1}2\}$ twinning is revealed in the diffraction pattern from the common zone axis $[a_2]$. This diffraction pattern is accessible in the TD samples in this work and was used to confirm that the twins were of the $\{10\bar{1}2\}$ extension type. It should be noted that in the thin TEM foil, double diffraction commonly occurs, i.e. the (0001) diffraction spots are excited, and this effect is present in the simulated pattern (Figure 2b).

Figure 2. The $180^\circ/\eta_1$ rotation relationship connecting the matrix and the twin is shown schematically in (a). (b) is a simulated diffraction pattern of the two lattices with $[a_2]_{m,t}$ being the zone axis. The black dots represent the matrix spots, the red squares the twin spots. The reciprocal vectors (0002) and $(10\bar{1}0)$ are drawn in the pattern for the matrix and the twin respectively. The real space twinning plane K_1 is determined from the common diffraction spot.

Based on the crystallographic frames of the matrix and the twin lattices and using the correspondence matrix developed by Niewczas (2010), a vector in either lattice can be transformed into the other, and the dislocation transmutation reactions were formulated as in Table 1. Presently, focus was laid on the reactions involving the transmutation of $\langle a \rangle$ dislocations from the matrix, and the reactions fall into two types: I. the transmutation of the common $[a_2]$ dislocations from the plane of $(0001)_m$ to $(10\bar{1}0)_t$, and II. the transmutation of $[a_1]$ or $[a_3]$ on the $(0001)_m$ plane into $\langle c \pm a_2 \rangle_t$ on the $(10\bar{1}0)_t$ plane. The basal planes in the

matrix and the indicated $(10\bar{1}0)_t$ prismatic planes in the twin are closely aligned (Yoo and Wei, 1966). For type II reactions, a residual dislocation is left on the twin boundary with the magnitude of $0.0643[\bar{1}011]$, which is the twinning dislocation b_t , also known as the twinning disconnection $b_{-2/-2}$ (Serra, Bacon and Pond, 1988). It should be emphasized that the $\langle c + a \rangle_t$ dislocation which results from this mechanism contains the particular $\langle a \rangle$ direction that is common to the matrix and twin, i.e. $[a_2]$.

Table 1. Dislocation reactions across a $(10\bar{1}2)$ twin boundary, the twinning relation is described by 180° rotation about the twinning direction, η_1 . $b_t = \frac{3-\gamma^2}{3+\gamma^2}\langle\bar{1}011\rangle = 0.0643\langle\bar{1}011\rangle$ (Serra, Bacon and Pond, 1988), with $\gamma = 1.624$ for Mg. The subscripts m and t denote the matrix and the twin respectively.

I.	$2 \times \frac{1}{3}[\bar{1}2\bar{1}0]_m \rightarrow 2 \times \frac{1}{3}[\bar{1}210]_t$	$2 a_{2m} \rightarrow -2 a_{2t}$
II.	$2 \times \frac{1}{3}[\bar{1}\bar{1}20]_m \rightarrow 0.0643[\bar{1}011] + \frac{1}{3}[\bar{1}2\bar{1}3]_t$	$2 \times a_{3m} \rightarrow b_t + [c + a_2]_t$
	$2 \times \frac{1}{3}[\bar{2}110]_m \rightarrow 0.0643[\bar{1}011] + \frac{1}{3}[\bar{1}2\bar{1}3]_t$	$2 \times -a_{1m} \rightarrow b_t + [c - a_2]_t$

4. Experimental results

In the rolled material where the c -axes of the crystals are roughly parallel to the ND of the plate, deformation along RD reveals a pronounced tension-compression asymmetry, which is related to tension twinning (Figure 1a). Whereas basal slip is active in grains with orientations which deviate from the prominent texture component, compression along RD will activate tension twinning, though it is unable to accommodate tensile strain along RD, which will generally require prismatic slip. During compression, tension twinning is preferentially activated as early as $\sim 0.3\%$ plastic strain, preceded by $\langle a \rangle$ dislocation slip on the basal plane in matrix (Brown et al., 2005; Agnew, Brown and Tome, 2006; Wang, Wu, Tome and Wang, 2012). After tension twinning is activated, basal and prismatic $\langle a \rangle$ slip systems remain the dominant slip systems in matrix (e.g., Clausen et al., 2008). The twinning-dominated compression is often characterized by a plateau in the flow curve, followed by a rapid strain hardening effect which coincides with the exhaustion of twinning, and ultimately, strain hardening slows as slip-based deformation occurs inside twins (e.g., Muransky et al., 2010). Basal slip activity is dominant

when twin volume fraction is low, and it drops in favor of pyramidal slip as twin grows. Viscoplastic self-consistent (VPSC) (Proust et al., 2009) and elastoplastic self-consistent (EPSC) (Juan et al., 2014) polycrystal models predicted that pyramidal slip activity inside twin commences near the end of the plateau regime (~4% strain), and increases rapidly before reaching steady state at higher strains. In a previous study, we examined some of the dislocation structures near the $\{10\bar{1}2\}$ twin boundary in both matrix and twin at 2% RD compressed material (Wang and Agnew, 2015). While the matrix contains mostly $\langle a \rangle$ dislocations and rarely $[c]$ -containing dislocations, abundant $[c]$ -containing dislocations were surprisingly common in the twin, even at such low stress level, and it was thus suggested that they are the product of dislocation transmutation reactions.

In this study, samples compressed along RD up to 2% true strain were selected, in order to pursue the origin of the $[c]$ -containing dislocations and the activity of pyramidal slip systems inside tension twins.

4.1 Dislocations near the $(10\bar{1}2)$ twin boundary from the perspective of ND

Characterization was performed firstly from the ND perspective, where the basal plane in the matrix and the prismatic plane in the twin are close to face-on orientation, and the twinning plane is inclined (Figure 3b). This orientation allows the “plan-view” examination of the planes on which the dislocation transmutation occurs. Dictated by the twinning relationship, the $[0001]_m$ and $[10\bar{1}0]_t$ zone axes are only $\sim 4^\circ$ away from each other. Since the characteristic $[\bar{1}2\bar{1}0]$ diffraction pattern (Figure 2b) is not available in the ND sample, the $(10\bar{1}2)$ twin was identified by comparing the $[0002]_m$ and $[10\bar{1}0]_t$ diffraction patterns of the two crystals and their common $(1\bar{2}10)$ diffraction spots.

In all the dark field (DF) images in Figure 3, the matrix is on the right side and the twin on the left side. Since the $(10\bar{1}2)$ twinning plane is 43.2° inclined to the basal plane, twin boundary appears as a wide band as delineated in Figure 3(a) below the DF image. With a common diffraction vector $g = (\bar{1}2\bar{1}0)$ operating simultaneously in both matrix and twin, the $\langle a \rangle$ and $\langle c + a \rangle$ dislocations are visible, but pure $[c]$ dislocations are invisible according to the invisibility criterion. Thus, Figure 3(a) presents the scenario of $\langle a \rangle$ dislocations in the matrix and $\langle c + a \rangle$ dislocations in the twin, corresponding to the type II reaction in Table 1. The straight lines of bright contrast in the range of the twin boundary have line directions parallel to the line

of intersection of twinning plane and basal plane. They represent the defects on the twin boundary, such as twinning disconnections with edge character.

In Figure 3(a), within the range of the inclined twin interface, the contrasts of the dislocations from both crystals are projected into the image. Thus, the sample was oriented such that two-beam diffraction operated in one crystal but no diffracted beam was strongly excited in the other crystal. In this way, the dislocations can be isolated to either the matrix or the twin. As shown in Figure 3(c) and (e), the diffraction vector of the twin $g_t = (1\bar{2}12)$ and that of the matrix $g_m = (1\bar{1}00)$ respectively reveal the contrast of dislocations in each crystal. The selected dislocation segments, for consideration, are sketched schematically in Figure 3(d), and a critical junction is indicated by an arrow in Figure 3(d), which connects one dislocation segment in the twin and two segments in the matrix.

Figure 3. Dislocations in the matrix and the twin adjacent to a $(10\bar{1}2)$ twin boundary (TB) presented in DF images. Image (a) was obtained with the common diffraction vector for the

matrix and the twin, the area highlighted by the dashed box outline is magnified for close inspection. The orientations of the two lattices are shown schematically in (b). Images (c) and (e) were obtained with two beam conditions for only the twin or the matrix, respectively. The selected dislocations are sketched schematically in (d). The dislocation segment AA' in (a), (c) and (d) is highlighted to guide the eye.

The orientation of the matrix allows using the diffraction vectors $(0\bar{1}10)$, $(\bar{1}010)$ and $(1\bar{1}00)$ to identify the three types of $\langle a \rangle$ Burgers vectors according to the invisibility criterion (Table 2). The corresponding images are shown in Figure 4. At the lower left part of all three images, the twin crystal appears dark and void of dislocation contrast because these sample orientations do not result in any strongly excited diffraction beam in the twin. Inside the matrix, dislocations of all three types of Burgers vector are present, and because the basal plane on which the dislocations lie on is roughly parallel to the foil surface, dislocations appear long before they terminate at a dislocation junction, an internal boundary, or the foil surface. The dislocation segments indicated by the red arrows have the Burgers vector of $[a_1]$. Some are connected with the twin boundary, and include the segments identified as being connected with the $\langle c + a \rangle$ dislocation segment AA' in the twin (Figure 3d and e).

Table 2. The $g \cdot b$ values of $\langle a \rangle$ dislocations with selected diffraction vectors from the zone axis of $[0001]$. The corresponding figures (a), (b) and (c) in Figure 4 for each g vector are indicated.

Mode	ZA	[0001]		
	$b \setminus g$	$(0\bar{1}10)$, (a)	$(\bar{1}010)$, (b)	$(1\bar{1}00)$, (c)
$\langle a \rangle$	$1/3[11\bar{2}0]$	-1	-1	0
	$1/3[1\bar{2}10]$	1	0	1
	$1/3[\bar{2}110]$	0	1	-1

Figure 4. Two-beam DF images of dislocations in the matrix from the area shown in Figure 3. The corresponding diffraction vectors are under the images with arrows indicating the directions. Some dislocation segments are annotated by their Burgers vectors (a_1 , a_2 or a_3). The red arrows in (b) and (c) indicate the same dislocation segments which are invisible in (a).

For the dislocations in the twin, it is determined with the diffraction vector $(\bar{1}\bar{2}\bar{1}0)$ that their Burgers vector is of $\langle c + a \rangle$ type, because the c -type dislocations would be invisible (Figure 3a). The exact determination of the Burgers vectors necessitates the diffraction vectors from the zone axes $[10\bar{1}0]$ and $[11\bar{2}0]$ (Table 3), and the corresponding two-beam BF and DF images are shown in Figure 5. The images are from the same area as Figure 3(c), and the dislocation segment AA' is indicated in Figure 5(f). Between the images obtained with the first three diffraction vectors, the $\langle c + a \rangle$ dislocations are visible with sharp contrast in Figure 5(a, f) and (b, g), they are of diffuse contrast in Figure 5(c, h). Between the images obtained with the last two diffraction vectors, dislocations are defined by clearer and sharper contrast in Figure 5(d, i) than in Figure 5(e, j). They are completely invisible in Figure 5(j). Since images (e) and (j) were obtained at the same two-beam condition with $+g$ (BF) and $-g$ (DF) respectively, dislocations are expected to display the same visibility in both images. This distinction is attributed to residual contrast, which is more apparent in BF mode than in DF mode. The visibility of the dislocations inside twin at the selected diffraction conditions suggests that their Burgers vector is $\frac{1}{3}[1\bar{2}13]$, or $[c - a_2]$ (Table 3).

Table 3. The $g \cdot b$ values of $\langle c + a \rangle$ dislocations with selected diffraction vectors from the zone axes of $[10\bar{1}0]$ and $[11\bar{2}0]$. The visibility of the dislocations in Figure 5 at each g vector is indicated in the last row.

	ZA	$[10\bar{1}0]$			$[11\bar{2}0]$	
Mode	b \ g	$(\bar{1}2\bar{1}0)$	$(1\bar{2}12)$	$(1\bar{2}1\bar{2})$	$(\bar{1}101)$	$(1\bar{1}0\bar{1})$
$\langle c + a \rangle$	$1/3[11\bar{2}3]$	-1	1	-3	1	-1
	$1/3[1\bar{2}13]$	2	4	0	2	0
	$1/3[\bar{2}113]$	-1	1	-3	0	-2
	$1/3[11\bar{2}\bar{3}]$	-1	-3	1	-1	1
	$1/3[1\bar{2}1\bar{3}]$	2	0	4	0	2
	$1/3[\bar{2}11\bar{3}]$	-1	-3	1	-2	0
Visibility*		v	v	d	v	i

* v: visible, d: diffuse, i: invisible.

Figure 5. Two-beam BF and DF images of the dislocations in the same region of the twin shown in Figure 3. The five diffraction vectors employed are shown below each column of BF and DF images. As explained in the text, the contrast reveals the AA' dislocation to be $1/3[1\bar{2}13]$, or $[c - a_2]$. The faint parallel fringes aligned diagonally from the upper left to lower right of the images in (d, e, i, j) are examples of the numerous basal faults within the twins.

Additionally, the straight and parallel extinction fringes are observed in the interior of the twin in Figure 5, they are characteristic of stacking faults (SFs). These SFs are parallel to the

1
2
3
4 trace of the basal planes, they are visible in Figure 5 because the high tilt employed to achieve
5 the two-beam conditions has oriented the basal plane at a large deviation from the initial edge-on
6 orientation (e.g. as in Figure 3). Tomsett and Bevis (1969b) suggested that the basal SF inside
7 the (10 $\bar{1}$ 2) twin can be formed as a result of dislocation transmutation.
8
9

10
11 Different from the curved morphology of the dislocations in the matrix, the dislocations
12 in the twin are mostly rectilinear. As shown in Figure 6, the $[c - a_2]$ dislocations in the twin
13 were observed using two-beam conditions from two zone axes, the dislocation segments i, ii and
14 iii are straight for most of their length. (The dislocation segment iii (AA') is the same one as in
15 Figure 3(c).) Segments i and ii clearly belong to one dislocation line, and the connection between
16 them is blunt in Figure 6(a) and sharp in (b), indicating that the line direction of the connection is
17 parallel to the beam direction $[11\bar{2}0]$. Segments i and iii are approximately parallel in both
18 Figure 6(a) and (b), thus they have the same line direction. The line directions of segments i and
19 ii were determined by trace analysis (Edington, 1976), and the analysis for segment i is shown in
20 Figure 6(c) as an example. Procedurally, it is preferential to use images obtained at three non-
21 coplanar zone axes, in order to gain three projections of the dislocation line and a quantitative
22 sense of uncertainty. But mathematically, images taken from two zone axes suffice for the
23 determination. Using the conventional stereological techniques, the line direction of dislocation
24 segments i and iii is determined to be $[4\bar{7}3\bar{9}]$.
25
26
27
28
29
30
31
32
33
34
35
36

37 As shown in Figure 6(c), the line direction $[4\bar{7}3\bar{9}]$ of segments i and iii is very close to
38 the periphery of the stereographic projection, and is 113° from the Burgers vector $[1\bar{2}13]$. It
39 suggests that segments i and iii are primarily of edge character and remain on the (10 $\bar{1}$ 0) plane
40 after transmutation. Following the same procedures, dislocation segment ii was determined to
41 have the line direction $u = [01\bar{1}\bar{3}]$, which is distant from the periphery of the stereographic
42 projection and is 163° from the Burgers vector $[1\bar{2}13]$. Thus, this dislocation segment is of
43 primarily screw character and has probably undergone cross-slip after transmutation. The cross
44 product of the line direction $u = [01\bar{1}\bar{3}]$ and Burgers vector $b = [1\bar{2}13]$ were used to determine
45 a possible glide plane normal. And this analysis suggests (11 $\bar{2}$ 1) as a potential glide plane. The
46 preferred glide planes of $\langle c + a \rangle$ dislocations reported in literature include the first-order
47 pyramidal planes $\{1\bar{1}01\}$ and second-order pyramidal planes $\{1\bar{2}12\}$ (Sandloebes et al., 2013;
48 Obara, Yoshinga and Morozumi, 1973; Li and Ma, 2009). It is possible that the observed
49 dislocation has undergone wavy slip involving a combination of these planes (Geng et al., 2015).
50
51
52
53
54
55
56
57
58
59
60
61
62
63
64
65

Figure 6. Stereological analysis for dislocation line direction determination. (a) and (b) are images of the $\langle c + a \rangle$ dislocations in the twin obtained at two-beam conditions from two zone axes. (c) presents the results from a trace analysis for dislocation segment i, conducted in the

[10 $\bar{1}$ 0] stereographic projection. The determined line directions for segment i, ii and iii are shown in the twin crystal lattice (d). The orientation of the lattice is consistent with Figure 3(b).

With this complete crystallographic knowledge of the $[c - a_2]$ dislocations in the twin, the connection of the segment AA' on the twin boundary was confirmed. It was presented in Figure 3 that the segment AA' is connected with two dislocation segments in the matrix on the twin boundary. The segments ii and iii (AA') terminate within the range of the twin boundary in the image, thus they intersect the boundary within the foil thickness (Figure 6a, b, d). Segment i, on the other hand, terminates within the interior of the twin, away from the twin boundary fringes. Thus, it terminates at the foil surface before reaching the twinning plane.

In conclusion, a dislocation transmutation reaction comprised of two $[a_1]$ dislocation segments in the matrix and one $[c - a_2]$ dislocation in the twin was observed. The reaction is consistent with the type II reaction in Table 1. The junction of the associated dislocations on the twin boundary was confirmed by stereological analysis.

4.2 Dislocations near the (10 $\bar{1}$ 2) twin boundary from the perspective of TD

In the ND sample, since the basal plane in the matrix and prismatic plane in the twin on which the reaction occurs, are roughly parallel to the foil surface, only a limited number of $\langle c + a \rangle$ dislocations were observed through the thin foil thickness. In contrast, characterization of the transmutation reaction on multiple layers was realized in the TD sample, where the basal, prismatic and twinning planes are all nearly edge-on. Figure 7 presents a grain scale picture of a (10 $\bar{1}$ 2) twin and the associated dislocation structure. When the matrix diffraction vector $g_m = (10\bar{1}0)$ is used for imaging, the close alignment with $g_t = (0002)$ of the twin enables the observation of only $\langle a \rangle$ containing dislocations in the matrix and $[c]$ containing dislocations in the twin. In the matrix, dislocations are well aligned with the basal plane trace, so they are most likely lying on the basal plane. Inside the (10 $\bar{1}$ 2) twin, the dislocations are either $\langle c + a \rangle$ or $[c]$. It is readily revealed that there are abundant dislocations near the left twin boundary, and the boundary deviates from the (10 $\bar{1}$ 2) plane trace indicated by the red dashed line, whereas in other areas of the twin, dislocations appear only occasionally. It is also note-worthy that at the two-beam condition of $g_t = (0002)$, there is contrast on the basal plane inside the twin (Figure 7b),

suggesting that the defects on the basal plane have a $\langle c \rangle$ component. They are possibly $\langle c + a \rangle$ dislocations or dissociation products lying on the basal plane, e.g. the I_1 fault, which has been suggested to be intimately connected with $\langle c + a \rangle$ dislocations (Sandloebes et al., 2012; Sandloebes et al., 2013; Agnew, Capolungo and Calhoun, 2015; Wu and Curtin, 2015).

Figure 7. (a) A $(10\bar{1}2)$ twin in a grain, whose grain boundary (GB) is delineated by black lines. The BF images were obtained with the same diffraction condition $g_m = (10\bar{1}0)$ of the matrix from consecutive areas and were montaged. The dashed red line indicates the plane trace of the $(10\bar{1}2)$ twinning plane. The area (b) was imaged with $g_t = (0002)$, in WBDF mode. The twin boundary is not only curved, it also has a serrated appearance.

Closer examination of the left twin boundary in Figure 7(b) is presented in Figure 8. In the matrix (Figure 8a-c), a train of dislocations is lying on the basal plane, potentially in a pile-up configuration directed to a point on the twin boundary, as indicated by the red arrow. At the orientation where $g = (10\bar{1}0)$ is excited (Figure 8a), the basal plane is close to edge-on, and the dislocations appear short and sharply curved; when $g = (0\bar{1}11)$ is excited (Figure 8c), large deviation from the edge-on orientation of the basal plane reveals long and only slightly bowed appearance of the same set of dislocations. The Burgers vector of these dislocations was

determined to be $[a_3]$ due to their visibility in Figure 8(a) and (c) and invisibility in (b) (Table 4, the rows for Mode $\langle a \rangle$).

Figure 8. Dislocations in the matrix (a), (b) and (c), and in the twin (e)-(g), (i)-(l) imaged with various diffraction vectors. All micrographs show the same area, and the red arrows in the images point at the same location to guide the eye. The orientations of the matrix and twin are shown schematically in (d), with only the basal and prismatic planes plotted for clarity. Selected features of the area are sketched in (h), where the curved lines represent dislocations and the double dashed lines represent SFs.

For the dislocations inside the twin, they are visible with $g = (000\bar{2})$ and invisible with $g = (\bar{1}010)$ (Figure 8e,i and f,j respectively), confining the possible Burgers vectors to $\langle c + a \rangle$ and $[c]$ (Table 4). They are of weaker contrast with $g = (10\bar{1}\bar{1})$ and invisible with $g = (11\bar{2}0)$ (Figure 8g,k and l respectively). Therefore, their Burgers vector is determined as $[c]$. The

stronger diffraction contrast in BF images with (0002) than with (10 $\bar{1}\bar{1}$) is consistent with the larger $g \cdot b$ value of the former than the latter.

Table 4. The $g \cdot b$ values of $\langle a \rangle$ dislocations in the matrix and $\langle c \rangle$ containing dislocations in the twin with selected diffraction vectors. The corresponding image numbers in Figure 8 for each g vector are indicated.

	ZA	$[\bar{1}2\bar{1}0]$			$[\bar{1}100]$	$[\bar{2}110]$
Mode	b \ g	(0002)	(10 $\bar{1}0$)	($\bar{1}011$)	(11 $\bar{2}0$)	(0 $\bar{1}11$)
Dislocation in the matrix		(b)	(a)			(c)
$\langle a \rangle$	$1/3[11\bar{2}0]$	0	1	1	2	-1
	$1/3[1\bar{2}10]$	0	0	0	-1	1
	$1/3[\bar{2}110]$	0	-1	-1	-1	0
Dislocation in the twin		(e, i)	(f, j)	(g, k)	(l)	
$\langle c + a \rangle$	$1/3[11\bar{2}3]$	2	1	0	2	0
	$1/3[1\bar{2}13]$	2	0	1	-1	2
	$1/3[\bar{2}113]$	2	-1	2	-1	1
	$1/3[11\bar{2}\bar{3}]$	-2	1	-2	2	-2
	$1/3[1\bar{2}1\bar{3}]$	-2	0	-1	-1	0
	$1/3[\bar{2}11\bar{3}]$	-2	-1	0	-1	-1
$\langle c \rangle$	[0001]	2	0	1	0	1

Besides the $[c]$ dislocations in the twin, fringes parallel to the basal plane are visible in Figure 8(f), (g) and (l), characteristic of SFs. At the diffraction condition of $g = (0002)$, which reveals defects with a $[c]$ component, there is also contrast on the basal plane (Figure 8e and i). However, the contrast is discontinuous and diffuse if one compares the fringes which appear at the same locations imaged with other diffraction vectors. Although the identity of these defects was not determined definitively, it is reasonably suggested that some are of I_2 (Shockley) type and those visible with $g = (0002)$ are most likely I_1 type.

In terms of the distribution of these $[c]$ dislocations and basal SFs in the twin, the faults appear in stacks, with each group of SFs separated by about 200 nm (e.g. Figure 8j and k, Figure 9), and the mostly straight $[c]$ dislocations connect these groups of SFs. This combination of a $[c]$ and a dissociated $\langle a \rangle$ dislocation is one of the $\langle c + a \rangle$ decomposition configurations originally proposed by Bevis and Tomsett (1969b) and recently revealed by molecular dynamics

simulation (Wu and Curtin, 2015). Figure 9 presents the dislocations in the twin from a nearby area imaged at two-beam conditions from two zone axes $[\bar{1}2\bar{1}0]$ and $[\bar{2}110]$. Most dislocations in this field of view have a $[c]$ Burgers vector, but the segment in the middle of the images is identified as having a $[c + a_2]$ Burgers vector, whose diffuse contrast with $g = (01\bar{1}\bar{1})$ (Figure 9c) is ascribed to residual contrast. The observations of $[c]$ dislocations from these two perspectives reveal that most dislocations remain rectilinear and perpendicular to the basal plane, which is conveniently delineated by the SFs in Figure 9(b). This morphology means that the line direction of the $[c]$ dislocations in the twin is close to the $[0001]$ direction, i.e., they are mostly of screw type.

To summarize this section, a train of $[a_3]$ dislocations in the matrix with $[c]$ dislocations and basal SFs in the twin were observed in the close vicinity of each other. According to the dislocation transmutation reactions listed in Table 1, the $[a_3]$ dislocation in the matrix can transmute into $[c + a_2]$. The resultant dislocation may further dissociate into $c + I_2$ SF or into a basal I_1 SF bounded by two c -containing partial dislocations. These reactions are consistent with the observation, although the precise identity of the SFs in the images remains to be confirmed.

Figure 9. Defect structure inside the $(10\bar{1}2)$ twin imaged with different diffraction vectors from two zone axes (indicated as beam direction B).

4.3 Defect structure inside the $(10\bar{1}2)$ twin

In order to develop a collective understanding of the defect structure inside the twin as a result of dislocation transmutation, the $(10\bar{1}2)$ twins in differently oriented grains were characterized. Figure 10 shows two grains near the hole of a TEM sample sectioned from the TD plane. The external directions RD and ND were marked during the sample preparation to relate the crystal orientation to the loading direction. TEM analyses of dislocations were subsequently performed within the twins T1 in Grain 1 and T2 & T3 in Grain 2.

Figure 10. (a) Two grains near the hole of the electropolished TEM foil, both including tension twins. The large image was obtained by secondary electron signals, and the images for the two grains were band contrast maps obtained by EBSD scan. (b) The IPF map of Grain 1, the lattice orientations of the matrix (G1) and the twin (T1) are shown below the map. (c) the IPF map of Grain 2, the lattice orientations of the matrix (G2) and the two twins (T2, T3) of the same variant are shown to the right of the map. The IPF map is color coded by the RD of the sample.

Figure 11 presents the defects inside the $(10\bar{1}2)$ twin T2 in Grain 2. The images at three different two-beam conditions suggest that the defect structure in this twin is qualitatively consistent with the local observations in the previous sections: the twin contains basal stacking faults and $[c]$ containing dislocations, and the dislocations preferentially populate the vicinity of the twin boundary. The diffraction vector $(10\bar{1}0)$ yields $[a_2]$, $\langle c \pm a_2 \rangle$ and $[c]$ dislocations invisible, and the vectors (0002) and $(11\bar{2}0)$ give contrast to $[c]$ -containing dislocations and $\langle a \rangle$ containing dislocations respectively (Table 4). A collective comparison between the visibility of the dislocations at three conditions determines that most inclined non-basal dislocations are $\langle c \pm a_2 \rangle$, while some are pure $[c]$. For the $\langle a \rangle$ containing dislocations on the basal plane, as shown at the $(11\bar{2}0)$ condition (Figure 11c), it is noticed that some are visible at the (0002) condition, indicating they are $[c]$ -containing as well. As revealed by the local observations in the TD sample in the previous section (Figure 8), the $[c]$ -containing dislocation segments on the basal plane may be related to faults, namely the bounding partials of the I_1 SF. On the other hand, it is also possible that these segments are edge-type perfect $\langle c + a \rangle$ dislocation lying on the basal plane. An example is shown in the magnified view Figure 11 (c.1), where a dislocation semi-loop has its both ends connected to the dislocation segments on the basal plane.

Whereas most non-basal dislocations in the twin T2 within Grain 2 are $\langle c \pm a_2 \rangle$, those in T3 in Grain 2 and T1 in Grain 1 are mainly $[c]$ dislocations. Figure 12 includes the images of T3 in Grain 2 obtained at two conditions. At the (0002) diffraction condition, many visible $[c]$ -containing dislocations are straight and perpendicular to the basal plane, and they are invisible at the condition of $(11\bar{2}0)$. It is therefore determined that they are pure $[c]$ dislocations. In the meantime, this twin contains some $\langle c \pm a_2 \rangle$ dislocations, based on their visibility in both the (0002) and $(11\bar{2}0)$ diffraction conditions. An example is shown in Figure 12(a.1). The $\langle c \pm a_2 \rangle$ dislocation is not perpendicular to the basal plane, and has its both ends connected to the twin boundary, which is a strong indication that it originates from the dislocation transmutation

reaction. Additionally, it should be noted that this morphology is very similar to the local observation obtained on ND plane as in Figure 6, if it is inspected with the electron beam in the horizontal direction of the image.

Figure 11. Defects inside T2 in Grain 2 from Figure 10(c). The twin was characterized with diffraction vectors (a) $(10\bar{1}0)$, (b) (0002) and (c) $(11\bar{2}0)$ respectively. (c.1) highlights a dislocation feature from (c). In all the images, the basal plane is roughly horizontal. The red arrows point at the same locations in the twin at different imaging conditions to guide the eye.

Finally, in much narrower twins, only basal stacking faults were observed (Figure 13). In (Tomsett and Bevis, 1969b), such basal SFs inside a tension twin were identified as I_2 and were attributed to the decomposition of the transmuted $\langle c + a \rangle$ dislocation and the subsequent dissociation of $\langle a \rangle$ dislocation into Shockley partial dislocations. While it is possible that some are I_2 faults formed by this reaction, or other reactions, such as dissociation of $[a_2]$ dislocations. However, some of the faults give contrast for the $g = (0002)$ diffraction condition, suggesting that they are of I_1 type. Definitive conclusion with respect to the type of basal stacking faults necessitates the Burgers vector determination of the bounding partial dislocations on twin interface with the assistance of image simulation, which is beyond the scope of the present work. Regardless of being I_2 or I_1 type, the basal stacking fault is confirmed to be a typical kind of defect structure inside the tension twin, their possible origins will be discussed subsequently.

Figure 12. Defects inside T3 in Grain 2 from Figure 10(c). The twin was characterized with diffraction vectors (a) (0002) and (b) (11 $\bar{2}$ 0) respectively. (a.1) highlights a dislocation feature from (a). In all the images, the basal plane is roughly horizontal. The red arrows point at the same locations in the twin at different imaging conditions to guide the eye.

Figure 13. DF image obtained with $g = (0004)_m$ showing basal stacking faults in a narrow (10 $\bar{1}2$) twin.

5. Discussion

The current work aims to clarify dislocation transmutation across the (10 $\bar{1}2$) tension twin boundary by TEM characterization. The incorporation and transmutation of basal [a_1] and [a_3] dislocations from the matrix has been observed. From the perspective of the sheet ND, two [a_1] dislocations from the matrix formed a junction on the twin boundary with a [$c - a_2$] dislocation in the twin, the junction was confirmed by stereological analysis of the dislocation segments. From the perspective of TD, a train of [a_3] dislocations in the matrix were observed to impinge upon the boundary where primarily [c] dislocations and basal stacking faults (as well as an occasional $\langle c + a \rangle$) were observed in the adjacent twin. Both observations accord with the type II reactions in Table 1.

The $\langle c + a \rangle$ dislocations inside the (10 $\bar{1}2$) twins preferentially populate the vicinity of twin boundary. At the low global stress level of about 100 MPa, they are generally not expected to be activated in this alloy with this grain size (e.g., Agnew, Brown and Tome, 2006). Therefore, the generation of $\langle c + a \rangle$ dislocations is attributed to dislocation transmutation. According to the

crystallographic derivation and experimental observation (especially the collective invisibility at the two-beam condition of $(10\bar{1}0)$), these $\langle c + a \rangle$ dislocations in the $(10\bar{1}2)$ twin are exclusively $\langle c \pm a_2 \rangle$, not any other variant. Therefore, the $(10\bar{1}2)$ twin provides a suitable location to study the behavior of a specific set of $\langle c \pm a_2 \rangle$ dislocations. In the following, the process of dislocation transmutation across twin boundary, the origin of $[c]$ dislocations and the operation of the $\langle c + a \rangle$ dislocations inside twin are discussed.

5.1 The process of dislocation transmutation

In the type II dislocation transmutation formulae in Table 1, two $\langle a \rangle$ dislocations are required in order to produce a unit $\langle c + a \rangle$ dislocation. According to Frank's rule (which is reasonably applied for Mg, given the near elastic isotropy) there is a penalty of $\Delta b^2 = 1.66a^2$, with a being the lattice parameter of Mg. Obviously, this reaction is energetically unfavorable. If the reaction is carried out in two steps successively, each time one unit $\langle a \rangle$ dislocation is incorporated and transmutes into one half $\langle c + a \rangle$ dislocation,

$$\langle a \rangle \rightarrow \frac{1}{2}\langle c + a \rangle + \frac{1}{2}b_t$$

Frank's rule yields $\Delta b^2 = -0.0848a^2$, revealing a slight energetic incentive for this reaction. When the transmutation of a second $\langle a \rangle$ dislocation of the same Burgers vector occurs, another $\frac{1}{2}\langle c + a \rangle$ is generated, which may associate with the first $\frac{1}{2}\langle c + a \rangle$ to form a perfect $\langle c + a \rangle$ dislocation in the twin.

Regarding the incorporation and transmutation of one unit $\langle a \rangle$ dislocation, two possible scenarios and procedures are proposed. The first scenario, illustrated in Figure 14 (a-d), shows a mobile dislocation in the matrix moving towards the twin boundary. When the $\langle a \rangle$ dislocation reaches the twin boundary (TB), transmutation reaction occurs (Figure 14 c and d), resulting in $\frac{1}{2}\langle c + a \rangle_t$ and $\frac{1}{2}b_t$ on the TB. While the exact interface structure is better addressed by atomistic simulation (e.g., Serra and Bacon (1996) and El Kadiri, Barrett, Wang and Tome (2015)), what is of interest at present is the behavior of the $\frac{1}{2}\langle c + a \rangle_t$ dislocation. When the second basal dislocation approaches, it is subject to a repulsive force from the $\frac{1}{2}\langle c + a \rangle_t$ dislocation due to their very similar Burgers vector. Consequently, a pile-up configuration would be required to force the combination of the two repulsive dislocations. On the other hand, if the $\frac{1}{2}[c + a]$

dislocation is repelled and able to glide into the twin with a fault trailing on its glide plane (Yoo and Wei, 1966), the stress required of the pile-up is somewhat dissipated.

Figure 14. The incorporation of one $\langle a \rangle$ dislocation in the matrix and its transmutation to $\frac{1}{2}(c + a)_t$ at twin boundary. (a-d) depict the scenario of mobile dislocation in the matrix moving towards the stationary twin boundary. The dashed line indicates the basal plane trace in the matrix, which is approximately the prismatic plane trace in the twin. (e-h) depict the scenario of mobile twin boundary moving towards the stationary dislocation, realized by the glide of a twinning dislocation b_t on twinning plane.

The second scenario, illustrated in Figure 14(e-h), describes a mobile twin boundary moving towards the stationary $\langle a \rangle$ dislocation. It is conventionally held that the twin boundary moves by the glide of twinning dislocations on the twinning plane (Serra and Bacon, 1996). When the TB finally meets the $\langle a \rangle$ dislocation in the matrix after successive glide of twinning dislocations, the $\langle a \rangle$ dislocation is incorporated and transmutes to $\frac{1}{2}(c + a)_t$ and $\frac{1}{2}b_t$ on the TB (Figure 14 g and h). During further advancement of the TB, the transmuted $\frac{1}{2}(c + a)_t$ dislocation may either move along with the TB or detach, leaving a fault in its wake. In either case, again a pile-up structure would be necessary to force the second matrix dislocation to be incorporated by the TB rather than being repelled, due to the same reason as discussed in the first scenario. In fact, the relative movement of the matrix dislocations and TB remain virtually the same between the two scenarios in Figure 14.

In both scenarios, successive transmutation reactions generate a large number of twinning dislocations. On one hand, their glide along the twinning plane as well as the basal/ prismatic

facets on the twin interface results in TB movement (El Kadiri, Barrett, Wang and Tome, 2015). On the other hand, abundant defects that populate the twin boundary vicinity, including the dislocation pile-ups in the matrix, the trailing faults, and perfect $\langle c + a \rangle$ and $\langle c \rangle$ dislocations inside twin, could impede the glide of twinning dislocations on twin boundary and therefore contribute to twin hardening. It is suggested that, if the twinning dislocations accumulate locally, they may contribute to the formation of steps on the TB with large height. This is consistent with the observation in Figure 7(b), where the twin boundary that is accompanied by abundant $[c]$ -containing dislocation not only is curved but also exhibits serrated appearance.

Regarding the necessity of a pile-up of matrix dislocations to force the combination of two repulsive dislocations, the present TEM observations show that basal dislocations appear to have formed a pile-up structure in front of the twin boundary in the TD sample (Figure 8a and c). No obvious evidence of pile-ups was observed in the ND sample (Figure 4). The dislocation pile-up in question has been modelled recently using Discrete Dislocation Dynamics (DDD) (Fan et al., 2015), where the initial $\frac{1}{2}\langle c + a \rangle$ dislocation is assumed to be sessile on the TB. As discussed above, it is also possible that the $\frac{1}{2}\langle c + a \rangle$ dislocation detaches TB and creates a fault on its glide plane, which was suggested by Yoo and Wei (1966) to be prismatic plane because of its close alignment with the basal plane in matrix. In hcp metals, the faults associated with $\langle c + a \rangle$ dissociation are reported to be more probable on the pyramidal planes (Numakura, Minonishi and Koiwa, 1990; Ghazisaeidi, Curtin and Hector Jr., 2014) and basal plane (Sandloebes et al., 2013; Agnew, Capolungo and Calhoun, 2015; Wu and Curtin, 2015), and no stable stacking fault was found on the $\{10\bar{1}0\}$ prismatic plane using the Lennard-Jones model (Bacon and Liang, 1986). On the second order pyramidal $\{11\bar{2}2\}$ plane, the dissociation of $\langle c + a \rangle$ into two $\frac{1}{2}\langle c + a \rangle$ dislocations enclosing a stable stacking fault was predicted in Zr (Morris et al., 1997) and more recently in Mg (Ghazisaeidi, Curtin and Hector Jr., 2014).

In the following, the elastic strain energies of possible configurations of the dislocations involved in the transmutation reaction is considered. The twinning dislocation b_t is neglected in the subsequent stress and energy calculation, which seems valid because the interfacial step created by $\frac{1}{2}b_t$ is small, and its stress field is weak due to the small Burgers vector ($0.076a$ as compared to a of a unit basal dislocation). Furthermore, the $\frac{1}{2}b_t$ will be repelled by the dislocations in matrix and twin and, hence, likely glide away on the twin boundary (Fan et al., 2015).

Figure 15 presents a model for the energetic evaluation. The matrix $\langle a \rangle$ dislocations of the same Burgers vector pile up in front of the twin boundary. The pile-up is maintained by the external stress τ and comprises N dislocations up to a distance L away from the twin boundary. Since the zeroth dislocation is on the twin boundary, it has transmuted and has the Burgers vector of $\frac{1}{2}\langle c + a \rangle$ (Figure 15c), a step is also created on TB as in Figure 14 (d) and (h). This represents the configuration of a sessile $\frac{1}{2}\langle c + a \rangle$ dislocation on the twin boundary. If the $\frac{1}{2}\langle c + a \rangle$ dislocation can glide into the twin with a fault trailing on its glide plane, until the 1st dislocation reaches the twin boundary, a new pile-up configuration comprising $(N-1)$ dislocations with a length L' is formed (Figure 15d). According to (Hirth and Lothe, 1982; Eshelby, Frank and Nabarro, 1951), if one reasonably assumes the Mg alloy is elastically isotropic (Capolungo, Beyerlein and Wang, 2010), the length L is correlated with the number of dislocations N under the same loading τ ,

$$L = \frac{2NA}{\tau}$$

where $A_s = \mu b/2\pi$ for screw and $A_e = \mu b/2\pi(1 - \nu)$ for edge dislocations, b is the magnitude of the Burgers vector, μ is the shear modulus and ν is the Poisson's ratio; the latter two materials constants take the value of 16.5 GPa and 0.035 respectively for this simple analysis. For the mixed dislocations of the present case,

$$A = A_s \cos \phi + A_e \sin \phi$$

ϕ is the angle between the Burgers vector and line direction, which is 60° for $\langle a \rangle$ and 58.4° for $\frac{1}{2}\langle c + a \rangle$.

For state (b), the energy of the system is the energy of the pile-up, which is treated as one super dislocation with the Burgers vector of Nb ,

$$E_b = \frac{Gb_{\langle a \rangle}^2 N^2}{4\pi} \left(\cos^2 60^\circ + \frac{\sin^2 60^\circ}{1 - \nu} \right) \left(\ln \frac{R}{r_0} + 1 \right)$$

R/r_0 is the ratio of the cut-off radius and the core radius of dislocation, and the value of $\ln \frac{R}{r_0} + 1 = 12$ is taken, representing $R/r_0 \approx 6 \times 10^4$. It can be seen from the equation that the total energy depends only on the number of dislocations N in the pile-up.

For state (d), the zeroth dislocation will be at equilibrium when the repulsive force due to the pile-up and the attractive force due to the fault are equal. As a first approximation, the

$\frac{1}{2}\langle c + a \rangle$ dislocation is assumed to be on the prismatic plane, the 4° difference between the basal plane in the matrix and the prismatic plane in the twin is neglected, since the width of the fault w is expected to be much smaller than the length of the pile-up L . The energy of the fault led by the $\frac{1}{2}\langle c + a \rangle$ dislocation takes the value of the fault energy on the second order pyramidal plane $\gamma = 328 \text{ mJ/m}^2$ (Yang et al., 2013), since that on the prismatic plane has not been reported in the literature yet. Note that the difference between the present calculation and one which examines $\frac{1}{2}\langle c + a \rangle$ partial dislocation emission from the boundary on a second order pyramidal plane is only different by a small geometrical factor. The force equilibrium is

$$\sigma_{pile-up} \cdot b_{\frac{1}{2}[c+a]} = \gamma$$

and $\sigma_{pile-up} = \tau \cdot \sqrt{\frac{2(N-1)A}{\tau \cdot w}}$ near the tip of the pile-up, where $w \leq L'$. Note that the pile-up comprises $(N-1)$ dislocation in state (d) (Figure 15).

Figure 15. (a) Dislocation lines and Burgers vectors in the matrix and the twin. (b)-(d) The incorporation and transmutation of the zeroth and first dislocations. The pile-up dislocations are depicted schematically in front of the twin boundary (TB). The wavy line in (d) indicates the fault created by the $\frac{1}{2}\langle c + a \rangle_t$ dislocation.

A calculation with an external stress $\tau = 100$ MPa and a large number of dislocations in the pile-up $N = 10$ yields that the length of the pile-up L is 236 nm, and the equilibrium fault width w is 1.85 nm, so the aforementioned approximations related to the relative lengths w and L are valid. The energy of the system is the sum of the energy of the pile-up, fault energy and energy of the $\frac{1}{2}\langle c + a \rangle$ dislocation,

$$E_d = \frac{Gb_{\langle a \rangle}^2(N-1)^2}{4\pi} \left(\cos^2 60^\circ + \frac{\sin^2 60^\circ}{1-\nu} \right) \left(\ln \frac{R}{r_0} + 1 \right) + w \cdot \gamma$$

$$+ \frac{Gb_{\frac{1}{2}[c+a]}^2}{4\pi} \left(\cos^2 58.4^\circ + \frac{\sin^2 58.4^\circ}{1-\nu} \right) \left(\ln \frac{R}{r_0} + 1 \right)$$

The energy of the states (b) and (d) in Figure 15 are evaluated as a function of the external stress τ for different number of dislocations in the pile-up N . For a given number of dislocations in the pile-up, the energy of the state (b) is constant regardless of the external stress, because the length of the pile-up varies with stress, but the energy of the ensemble depends only on the number of dislocations. The energy of state (d) increases with increasing stress, since the increasing stress tends to increase the width of the fault led by the $\frac{1}{2}\langle c + a \rangle$ dislocation in the twin. The comparison of the energy between the two states reveals that, in the range of plausible stress (≤ 4 GPa) and for a given number of dislocations ($N \leq 20$) in the pile-up, the state (d) has lower overall energy than the state (b). Assuming that the dislocation core energies are not a strong function of applied stress, the energy difference is on the order of 10^{-8} J/m (~ 20 eV/b).

Finally, the above calculation suggests that, at the first step of the dislocation incorporation and transmutation, the $\frac{1}{2}\langle c + a \rangle$ dislocation is likely to leave the twin boundary (or be left by it) with a trailing fault in the twin (Figure 16a and b). Such configuration necessitates a pile-up of dislocations in the matrix in front of the TB to maintain the force balance. With increasing number of dislocations in the pile-up or increasing external stress, the second matrix dislocation will be incorporated and transmuted. At this point, the second $\frac{1}{2}\langle c + a \rangle$ partial dislocation would recover some energy as it erases the fault created by the first partial and form a perfect $\langle c + a \rangle$ dislocation (Figure 16c). Note that if the $\frac{1}{2}\langle c + a \rangle$ partial dislocation is effectively sessile within the twin, motion of the TB could increase the fault length even further than suggested by the force balance computed above, rendering the subsequent formation of a perfect $\langle c + a \rangle$ even more energetically favorable. **After passing this critical step, it is reasonably**

hypothesized that subsequent motion of the TB will force continued “zipping up” of the two repulsive $\langle a \rangle$ dislocations into a perfect $\langle c + a \rangle$ dislocation at the junction shown in Figures 16 (c) & (d). The resulting picture is consistent with the observations, such as those presented in Figure 3.

Figure 16. The successive incorporation and transmutation of the first two basal dislocations from the matrix at twin boundary. The area filled by parallel red lines in (b) and (d) represents a stacking fault. Figure (d) presents the detailed configuration of dislocations junction as highlighted by dashed circle in (c).

Admittedly, the model may not capture all the critical kinetic and interfacial structural details. Previous results from Serra and Bacon (1996) and El Kadiri, Barrett, Wang and Tome (2015) showed that a basal dislocation only decomposes into interfacial defects when incorporated by the $\{10\bar{1}2\}$ twin boundary, and that slip is not transferred from one crystal to another. The current model is not considered in disagreement with the preceding work. Rather, it provides additional possibilities, which are consistent with the experimental evidence, and may be tested by additional atomistic modeling, perhaps with distinct interatomic potentials and boundary conditions.

5.2 The $\langle c \rangle$ dislocations and basal stacking faults inside twin

In some of the twins surveyed, not only perfect $\langle c + a \rangle$ dislocations, but also $[c]$ dislocations and basal stacking faults were observed, sometimes even exclusively. In the work of Tomsett and Bevis (1969b), based on the coexistence of basal dislocation in the matrix and I_2 SF in the twin, and the premise of $\langle a \rangle$ dislocation transmuting into $\langle c + a \rangle$ dislocation, they reported the decomposition of the resultant $\langle c + a \rangle$ dislocation in the twin into $\langle c \rangle$ and $\langle a \rangle$ dislocations. Further dissociation of the $\langle a \rangle$ dislocation into two Shockley partial dislocations gives rise to the formation of I_2 SF. A general expression of the dissociation is

$$\frac{1}{3}\langle \bar{1}2\bar{1}3 \rangle \rightarrow \langle 0001 \rangle + \frac{1}{3}\langle \bar{1}2\bar{1}0 \rangle \rightarrow \langle 0001 \rangle + \frac{1}{3}\langle 01\bar{1}0 \rangle + \frac{1}{3}\langle \bar{1}100 \rangle$$

Assuming elastic isotropy and ignoring the core contributions, the dissociation of $\langle c + a \rangle \rightarrow \langle c \rangle + \langle a \rangle$ results no reduction in elastic strain energy for a dislocation line. This reaction was proved possible recently by molecular dynamics simulations (Wu and Curtin, 2015) based on a reduction in dislocation total energy, despite being a smaller reduction than that of other possible decomposition reactions. Further, the atomistic simulations showed that this decomposition is the dominant one at high loads. The current results involve a different scenario where the $\langle c + a \rangle$ dislocation assumes a specific configuration, e.g. both ends terminating at twin boundary or connecting with dislocation segments on the basal plane (e.g. Figure 6, Figure 11, Figure 12). The approximate geometry is depicted in Figure 17, and the energy evaluation using Frank's rule proves that even this simple decomposition is favorable without resorting to considerations of partial dislocations:

$$\langle c + a \rangle^2 \times (2l + w) > c^2 \times (2l + w) + a^2 \times w$$

Only the elastic self-energy of dislocation is considered here using the abstract lengths of dislocation segments; this simple analysis ignores the core energy and the interaction energy between segments. Recall that the observed $\langle c \rangle$ dislocations within the twins were observed to lie on the prismatic plane with the majority of the length perpendicular to the basal plane. This suggests the $\langle c \rangle$ dislocations have limited mobility, at least for the loading scenario under consideration.

Figure 17. A semi-looped $\langle c + a \rangle$ with both ends on the twin boundary (TB). Part of the dislocation dissociates into $\langle c \rangle$ and $\langle a \rangle$ dislocations with length l and width w .

An alternative mechanism was proposed by Westlake (1961) that a $\langle c \rangle$ dislocation can be formed by the transmutation of a $\langle 10\bar{1}0 \rangle$ dislocation. Following the notations in this work, by adding the two different reactions of type II in Table 1, it gives

$$\frac{1}{3}[\bar{2}110]_m + \frac{1}{3}[\bar{1}\bar{1}20]_m \rightarrow \frac{1}{2}b_t + \frac{1}{6}[1\bar{2}13]_t + \frac{1}{2}b_t + \frac{1}{6}[\bar{1}2\bar{1}3]_t$$

In a concise form, the transmutation is

$$[\bar{1}010]_m \rightarrow b_t + [0001]_t$$

Therefore, we identify that this mechanism is a variant of the basic transmutation reactions as listed in Table 1. Procedurally, this transmutation is realized by the incorporation of two different dislocations, in the current notation $[a_1]$ and $[a_3]$, and the two dislocations react either in the matrix or in the twin. In the former case, when the two basal dislocations form a junction, the third dislocation connected to the junction has the Burgers vector of $[\bar{1}010]_m$ based on the continuity of dislocation (Hirth and Lothe, 1982). If the line direction of the third dislocation is parallel to the twin boundary, it is of edge type. The subsequent transmutation of the $[\bar{1}010]_m$ results in $[0001]_t$. In the latter case, the two basal dislocations would undergo transmutation sequentially, resulting in $\frac{1}{2}[c + a_2]_t$ and $\frac{1}{2}[c - a_2]_t$, respectively. The subsequent reaction of the two yields a pure $\langle c \rangle$ dislocation in the twin. While the concurrent presence of both $\langle c \rangle$ dislocations and basal SFs in our results is in agreement with the reaction of $\langle c + a \rangle$ decomposition, it does not exclude the possibility of the mechanism of Westlake (1961). In fact, the reaction of two different $\langle a \rangle_m$ or $\langle c + a \rangle_t$ dislocations might be easier in terms of dislocation interaction than the reaction of two dislocations of the same Burgers vector as discussed in the previous section. The two different $\langle a \rangle_m$ vectors, $[-a_1]$ and $[a_3]$, are 60° to each other, and the

two $\langle c + a \rangle_t$ vectors, $[c + a_2]$ and $[c - a_2]$, are 63.2° to each other. These dislocations are subject to smaller repulsive forces than the case of two dislocations of the same Burgers vector.

Rather than forming an I_2 stacking fault from the decomposition of $\langle c + a \rangle$ dislocation, according to (Stohr and Poirier, 1972), a possible non-planar dissociation of $\langle c + a \rangle$ dislocation can result in an I_1 stacking fault on the basal plane:

$$\frac{1}{3}\langle\bar{1}2\bar{1}3\rangle \rightarrow \frac{1}{6}\langle 02\bar{2}3\rangle + \frac{1}{6}\langle\bar{2}203\rangle$$

It was recently proven as a low energy dissociation product of $\langle c + a \rangle$ dislocations by elasticity considerations (Agnew, Capolungo and Calhoun, 2015) and molecular dynamics simulations (Wu and Curtin, 2015), though it requires climb of the leading partial dislocation into the basal plane. Notably, this faulted configuration is expected to be sessile. In the current study, two types of SF were observed and discriminated using the (0002) two-beam diffraction condition. Those became invisible at that condition were presumably of I_2 type and those remained visible were presumably of I_1 type.

Finally, if transmutation of $[a_2]$ occurs, although not captured in the current observations, the product inside twin is $[a_2]$ (the type I reaction in Table 1). The subsequent dissociation of $[a_2]$ inside twin into two Shockley partial dislocations will create a SF on basal plane. The balance between the repulsive force of the two bounding partials and the attractive force of the stacking fault energy renders an equilibrium fault width on the order of 20 nm (23.9 nm from isotropic continuum elasticity and 16.7 nm from ab initio modeling (Yasi et al., 2009)). However, it is suggested that if the two bounding partials are attached to opposing twin boundaries, a larger fault width may be realized. This is similar to the emission of partial dislocation from grain boundary with a trailing fault in nanocrystalline metals, e.g. Al (Yamakov et al., 2002). And this may be a possible explanation for the traversing basal stacking faults in narrow twins (Figure 13, the twin width is 260 nm).

5.3 Effect of stress state on the $\langle c + a \rangle$ dislocations inside twin

Abundant $\langle c + a \rangle$ dislocations were observed in the $(10\bar{1}2)$ twins, in material loaded into the plateau regime of the RD compression flow curve. In light of the prediction by polycrystal plasticity modeling that pyramidal slip inside twin commences at the end of the plateau (~4%

macroscopic strain) (Proust et al., 2009; Juan et al., 2014), it is of interest to study the stress state imposed on these $\langle c + a \rangle$ dislocation, in order to assess their activity.

In a suitably oriented grain under external loading, the occurrence of deformation twinning strongly modifies the stress and strain state of the surrounding (Lebensohn and Tome, 1993). The twin thusly experiences a back stress from the surrounding, counteracting with the forward stress that promotes twin boundary advancement. If the sum of the resolved shear stress (RSS) on the twin shear system of the applied stress and twin internal stress (one of whose components is back stress as will be introduced below) is zero, or at least smaller than the friction stress of moving a twinning dislocations, the twin is at equilibrium. In the meantime, potential plasticity in the surrounding is able to relax the constraint on the twin and reduce the magnitude of back stress (Barnett et al., 2013), so further twin growth is allowed. Relaxation is induced by dislocation slip and twinning in not only the parent grain but also the suitably oriented neighboring grain. For instance, twins in the neighboring grains can be initiated to maintain strain compatibility at grain boundaries, and in this manner twinning spreads progressively over the sample (Barnett, Nave and Ghaderi, 2012; Wang et al., 2014). In Figure 10 (a), the twins T1 in G1 and T2 in G2 are accompanied by twins in the adjacent grain, forming twin pairs, but the twin T3 in G2 is not. It is expected that the internal stress in these twins is modified accordingly.

The exact determination of the net effective stress inside twin necessitates grain scale experimental characterization (Aydinler et al., 2009) or simulation, considering various extents of plastic relaxation of the stress. In the present work, the effective stress is calculated at two limiting cases. The first case assumes that the elastic internal stress of the twin is completely relaxed by plastic deformation in the surroundings. Therefore, the twin interior is subject to only the external applied stress. The second case assumes that the elastic internal stress of twin is preserved, without plastic relaxation from the surrounding grains. In this case, the effective stress inside twin is the superposition of the applied stress and internal stress. The studied material of this work is at the applied stress of ~100 MPa, which is much smaller than the macroscopic yield stress (more than 200 MPa as taken from the RD tension flow curve in Figure 1a). At this stage, plasticity in the surrounding is unlikely to relax all the back stress acting on a twin (Barnett et al., 2013). So the effective stress in most twins should be within the limiting bounds.

In order to calculate the internal stress, the twin is treated as an elastic inclusion of oblate spheroidal shape ($a_1 = a_2 > a_3$), and the elastic constants are assumed to be the same for the twin and the matrix, based on the fact that magnesium is nearly elastically isotropic. In the spatial coordinate system of x_1 parallel to the shear direction η_1 , x_2 perpendicular to the twinning plane K_1 and x_3 perpendicular to the other two axes, the deviation of the stress, internal stress σ_{ij}^i , within the twin from the external applied stress σ^0 stress can be calculated as (Lebensohn and Tomé, 1993)

$$\sigma_{ij}^i = C_{ijkl} (S_{klmn} \varepsilon_{mn}^p - \varepsilon_{kl}^p)$$

where C_{ijkl} is the stiffness tensor of Mg, and S_{klmn} is the Eshelby tensor, relating the eigenstrain to the actual total (elastic and transformation) strain of the inclusion when imbedded in a body (Eshelby, 1957; Kuykendall et al., 2012). In the above equation, both tensors are transformed to the same set of spatial coordinate axes x_1 , x_2 and x_3 . Analytical expressions of the Eshelby tensor S for inclusion in an isotropic body have been derived by (Mura, 1982) for particular inclusion shapes, with its components being functions of the inclusion shape and the elastic constants of the matrix. The exact components of S are not presented here for the interest of space. ε^p is the plastic transformation associated with twin. In the spatial coordinate system, it takes the form of

$$\varepsilon_{mn}^p = \frac{1}{2}s(\delta_{m1}\delta_{n2} + \delta_{m2}\delta_{n1})$$

where s is the characteristic twinning shear ($s = 0.13$ for tension twinning in Mg), and δ_{ij} is the Kronecker delta function. From the above two equations and based on the traits of the Eshelby tensor, for an oblate spheroidal twin in a given matrix, the only variable that influences the stress components is the twin aspect ratio ($q = \text{width}/\text{diameter}$), and the internal stress is uniform in the case of ideal elasticity. In the spatial coordinate system of the twin and with the approximated elastic isotropy, the internal stress takes the simple form of

$$\sigma^i = \begin{pmatrix} 0 & \sigma_{12} & 0 \\ \sigma_{12} & 0 & 0 \\ 0 & 0 & 0 \end{pmatrix}$$

The only non-zero stress component σ_{12} acts on the twinning plane (normal to x_2) along the shear direction (parallel to x_1), and is often referred to as the back stress.

In what follows, calculations were performed for twins inside the matrix at two ideal orientations under a fixed applied uniaxial compression of 100 MPa. The twins have aspect ratio

q ranging from 0.01 to 0.2, covering most of the twins experimentally observed in our material, e.g. from 0.10 to 2 μm twins inside grains of 10 μm in diameter. This is representative of the situation of twins at different stages of growth in the textured material from the plateau regime of the RD compression flow curve, as evident by Figure 10. As shown in Figure 18 (a) and (e), the two orientations are referred to as $[10\bar{1}0]$ compression and $[11\bar{2}0]$ compression, respectively, based on the alignment of the crystallographic direction with the uniaxial sample compression axis y_s . For $[10\bar{1}0]$ compression, the twinning system $(10\bar{1}2)[\bar{1}011]$ is preferentially activated, the twin crystal has its c -axis almost parallel to the compression axis with a $\sim 4^\circ$ deviation arising from the twin-matrix relation, and the $[a_2]$ axis perpendicular to the compression axis; for $[11\bar{2}0]$ compression, the twinning system $(10\bar{1}2)[\bar{1}011]$ and $(01\bar{1}2)[0\bar{1}11]$ are equally stressed, the angles between the compression axis and the twin $[c]$ and $[a_2]$ axes are approximately 30° and 60° respectively. For consistency, only the $(10\bar{1}2)$ twin variant is considered. The pyramidal $\langle c \pm a_2 \rangle$ and basal and prismatic $[a_2]$ slip systems inside the twin are of special interests, based on the observed dislocation transmutation reactions.

In the first limiting case, the twin interior is subject to only the external applied stress, and the stress is invariant to twin aspect ratio. For $[10\bar{1}0]$ compression, all six pyramidal slip systems inside twin experience very similar RSS due to the symmetry of the slip systems with respect to the loading direction. The RSS on pyramidal $\langle c \pm a_2 \rangle$ slip systems is 44.5 MPa, and 46.8 or 41.8 MPa on others. Since $[a_2]$ is perpendicular to the stress axis, it experiences zero RSS on either basal or prismatic plane. For $[11\bar{2}0]$ compression, the pyramidal $[c - a_2]$ is stressed the most among all the six systems with a RSS of 41.6 MPa and the pyramidal $[c + a_2]$ the least with a RSS of 2.7 MPa. In contrast to the former orientation, the $[a_2]$ experiences a much higher RSS of 43.2 MPa on basal plane, but only 2.8 MPa on prismatic plane due to the $\sim 4^\circ$ deviation of the prismatic plane from the parallel position to the compression axis. At both matrix orientations, the external loading of 100 MPa yields a less than 50 MPa RSS on all six pyramidal slip systems, they are very unlikely active at this stress level (Proust et al., 2009; Juan et al., 2014).

In the second limiting case, dislocations inside twin are subject to not only the external applied stress but also the internal stress of twin, and the effective stress σ_{eff} is given by

$$\sigma_{eff} = \sigma^0 + \sigma^i$$

The applied stress, internal stress and effective stress are firstly resolved to the twinning shear system, and the RSS is plotted with respect to twin aspect ratio for two orientations as in Figure 18 (b) and (f). The RSS of the above three stresses represent respectively the twin forward stress, back stress and the net effective shear stress. The applied stress is independent of the twin aspect ratio and results in a RSS of 49.4 MPa and 37.4 MPa for the two orientations respectively. On the other hand, the internal stress increases in magnitude with increasing q but is independent of the matrix crystal orientation. The sign of the RSS of the internal stress is negative, indicating that it opposes the twin shear. The RSS associated with the net effective stress exhibits two striking features. First, the effective shear stress on the twinning system is zero or close to zero at a very low aspect ratio $q \sim 0.01$. For twins with larger aspect ratios, apart from plastic accommodation of the twinning shear, the effective shear stress on the twin shear system is negative, i.e., it would cause the twin to shrink. The only reason twins are able to thicken beyond the equilibrium aspect ratio is due to plastic accommodation within the surrounding and the twin. Second, the effective shear stress within purely elastic twins reaches a high magnitude exceeding 600 MPa at $q = 0.2$. To be clear, the calculation at the limiting case of full elasticity is only for the purpose of illustrating the rank order of the effective RSS values experienced by various slip systems. Focus should be laid to the range of low q values, where plastic relaxation in the surrounding may only be at small scale and the calculated stress values in the plots are realistic.

When the applied stress is superimposed with the internal stress, the six pyramidal $\langle c + a \rangle$ slip systems have distinct RSS values (Figure 18c and g), as compared to the case of considering only the applied stress. The RSSs on all the slip systems increase monotonically with twin aspect ratio, but some start from negative values, indicating that the stress is in the reversed direction of the defined Burgers vector. Since the slip activity of interest is affected by the magnitude of the RSS, rather than the direction, the absolute values $|RSS|$ are employed for plotting.

For both matrix orientations, the magnitude of RSS on the transmuted $\langle c \pm a_2 \rangle$ dislocations is higher than the others only at low q values, but becomes the lowest for all larger q values among six pyramidal systems. The RSS curves of $[c + a_2]$ and $[c - a_2]$ overlap at the orientation of $[10\bar{1}0]$ compression (Figure 18c), due to the crystal symmetry with respect to the stress, so do those of $[c + a_1]$ and $[c - a_3]$, $[c - a_1]$ and $[c + a_3]$. At the orientation of $[11\bar{2}0]$

compression (Figure 18g), $[c + a_2]$ and $[c - a_2]$ systems have distinct values, yet still remain the lowest.

Figure 18. When compression load 100 MPa is applied along y_s to the two matrix orientations (a) and (e), $(10\bar{1}2)[\bar{1}011]$ twinning is activated, the twinning plane and shear direction are projected onto the basal plane of each matrix crystal as indicated by the blue lines. The RSS on

different systems are plotted as a function of twin aspect ratio q in (b, c, d) and (f, g, h) for the two orientations respectively. (b) and (f): RSS on the twin shear system due to the applied stress, internal stress and effective stress; (c) and (g): the magnitude of RSS on pyramidal $\langle c + a \rangle$ slip systems due to the effective stress; (d) and (h): the magnitude of RSS on basal and prismatic $\langle a \rangle$ slip systems due to the effective stress.

Since the non-zero component of the twin internal stress is along the twin shear direction on twinning plane, which is perpendicular to the $[a_2]$ direction, the RSS on $[a_2]$ dislocation is invariant to q , it is only affected by the external loading. The $[a_2]$ dislocation on basal plane experiences zero RSS at the orientation of $[10\bar{1}0]$ compression (Figure 18d), but the highest RSS for most twin aspect ratios at the orientation of $[11\bar{2}0]$ compression (Figure 18h) among all three basal slip systems. The prismatic $[a_2]$ slip system has zero RSS at the former orientation and only a small value of 2.8 MPa at the latter one.

The above two limiting cases establish the bounds for effective stress state inside twin. Although the magnitude of the stress in the second limiting case, which assumes full elasticity, may be lower in reality due to plastic relaxation, the results are able to provide some general insights of the dislocation behavior. In both limiting cases, the magnitude of the RSS on the transmuted $\langle c \pm a_2 \rangle$ dislocations is not high enough to render them active. However, the TEM characterization shows that the $(10\bar{1}2)$ twins contain exclusively $\langle c \pm a_2 \rangle$ dislocations among all 6 possible symmetrically equivalent variants. This simultaneously suggests that the $\langle c \pm a_2 \rangle$ dislocations are the result of dislocation transmutation across the twin boundary and that the operation of $\langle c + a \rangle$ dislocations is source limited. With increasing applied stress in subsequent straining, $\langle c \pm a_2 \rangle$ dislocations would be activated and become a dominant slip system as predicted by polycrystal plasticity models (Proust et al., 2009; Juan et al., 2014). Furthermore, acknowledging the fact that the current investigation is conducted in samples from the plateau region of the flow curve, toward the onset of rapid hardening, the transmuted $\langle c + a \rangle$ dislocations act as forest dislocations inside twin and will strongly contribute to latent hardening, due to the strong interactions of the pyramidal $\langle c + a \rangle$ dislocation with basal and prismatic $\langle a \rangle$ dislocations (Bertin et al., 2014). Therefore, the observed defect structures inside twins establish a basis for subsequent rapid hardening.

6. Conclusion

In the present work, Mg alloy AZ31B plate was compressed into the plateau region of the flow curve and samples were obtained to study the possibility of dislocation transmutation across the $(10\bar{1}2)$ twin boundary. The character and configuration of dislocations within the twin and in the adjacent matrix were determined using TEM. Reactions were confirmed which involve $[a_1]$ and $[a_3]$ matrix dislocations that “transmute” into $[c - a_2]$ and $[c + a_2]$ dislocations, respectively, in the $(10\bar{1}2)$ twin. Collective observation of a number of twins in different grains further showed that the $(10\bar{1}2)$ twins contain only $\langle c \pm a_2 \rangle$ among all six possible $\langle c + a \rangle$ dislocations, and dislocations form a particular distribution inside twin, preferentially populating the vicinity of the twin boundary. The formation of a unit $\langle c + a \rangle$ dislocation in the twin is proposed to involve consecutive incorporation and transmutation of two matrix dislocations of the same Burgers vector. This process necessitates dislocation pile-up in the matrix in front of the twin boundary, and the associated dislocation configuration was evaluated in terms of elastic strain energy.

Stress analyses were performed inside twin at two limiting bounds. It was shown that $\langle c \pm a_2 \rangle$ dislocations are only moderately stressed in the plateau regime of the RD compression flow curve, and they experience either similar or lower effective resolved shear stress (RSS) as compared to other $\langle c + a \rangle$ dislocations. Their exclusive presence suggests that the $\langle c + a \rangle$ slip mode is source limited and that the transmutation concept provides an obvious source mechanism, which could be activated at later stages of deformation. It is hypothesized that the $\langle c \pm a_2 \rangle$ dislocations are effectively sessile at this stress level. As evidence of this fact, note that they remain only in the vicinity of twin boundary and not uniformly distributed throughout the twin. Further, it is suggested that some of the interfacial defects introduced by transmutation may induce a dragging effect on twin boundary advancement. The abundant $\langle c \pm a_2 \rangle$ dislocations (and faults) resulting from the transmutation mechanism will also contribute to hardening within the twin itself, during subsequent deformation. Finally, it is noted that the observed $\{10\bar{1}2\}$ twin provides a suitable location for future study of $\langle c + a \rangle$ dislocation behavior at a rather low global applied stress of about 100 MPa in a common alloy system.

Acknowledgments

The present work was sponsored by the National Science Foundation grant number CMMI 1235259 (Mary Toney). The authors would like to acknowledge L. Wu and P.K. Liaw in University of Tennessee for providing the samples. H. El Kadiri and C.D. Barrett (Mississippi State University), J. El Awady (Johns Hopkins University), and W.D. Curtin (École polytechnique fédérale de Lausanne) are gratefully acknowledged for fruitful discussions. We gratefully acknowledge the journal reviewers who identified a number of opportunities to improve upon the analysis which we had performed initially.

References

- Agnew, S.R., Brown, D., Tome, C.N., 2006. Validating a polycrystal model for the elastoplastic response of magnesium alloy AZ31 using in situ neutron diffraction. *Acta Materialia* 54, 4841-4852.
- Agnew, S.R., Capolungo, L., Calhoun, C.A., 2015. Connections between the basal II “growth” fault and $\langle c+a \rangle$ dislocations. *Acta Materialia* 82, 255-265.
- Aydiner, C.C., Bernier, J.V., Clausen, B., Lienert, U., Tomé, C.N., Brown, D.W., 2009. Evolution of stress in individual grains and twins in a magnesium alloy aggregate. *Physics Review B* 80, 024113.
- Bacon, D. and Liang, M., 1986. Computer simulation of dislocation cores in h.c.p. metals I. Interatomic potentials and stacking-fault stability. *Philosophical Magazine A* 53, 163.
- Barnett, M., Nave, M.D., Ghaderi, A., 2012. Yield point elongation due to twinning in a magnesium alloy. *Acta Materialia* 60, 1433-1443.
- Barnett, M., Stanford, N., Ghaderi, A., Siska, F., 2013. Plastic relaxation of the internal stress induced by twinning. *Acta Materialia* 61, 7859-7867.
- Bertin, N., Tome, C.N., Beyerlein, I.J., Barnett, M.R., Capolungo, L., 2014. On the strength of dislocation interactions and their effect on latent hardening in pure Magnesium. *International Journal of Plasticity* 62, 72-92.
- Bevis, M. and Crocker, M., 1969. Twinning modes in lattices. *Proceedings of the Royal Society of London A* 313, 509-529.

- Bhattacharyya, D., Cerreta, E., McCabe, R., Niewczas, M., Gray III, G., Misra, A. and Tome, C.N., 2009. Origin of dislocations within tensile and compressive twins in pure textured Zr. *Acta Materialia* 57, 305-315.
- Brown, D., Agnew, S.R., Bourke, M., Holden, T., Vogel, S. and Tome, C.N., 2005. Internal strain and texture evolution during deformation twinning in magnesium. *Materials Science and Engineering A* 399, 1-12.
- Capolungo, L., Beyerlein, I. and Wang, Z., 2010. The role of elastic anisotropy on plasticity in hcp metals: a three-dimensional dislocation dynamics study. *Modelling and simulation in materials science and engineering* 18, 1-16.
- Christian, J. and Mahajan, S., 1995. Deformation twinning. *Progress in Materials Science* 39, 1-157.
- Clausen, B., Tome, C.N., Brown, D.W., Agnew, S.R., 2008. Reorientation and stress relaxation due to twinning: modeling and experimental characterization of Mg. *Acta Materialia* 56, 2456-2468.
- Edington, J., 1976. *Practical electron microscopy in materials science*. New York: Van Nostrand Reinhold Co.
- El Kadiri, H. and Oppedal, A., 2010. A crystal plasticity theory for latent hardening by glide twinning through dislocation transmutation and twin accommodation effects. *Journal of the Mechanics and Physics of Solids* 58, 613-624.
- El Kadiri, H., Barrett, C., Wang, J. and Tome, C.N., 2015. Why are {10-12} twins profuse in magnesium. *Acta Materialia* 85, 354-361.
- Eshelby, J., Frank, F. and Nabarro, F., 1951. The Equilibrium of Linear Arrays of Dislocations. *Philosophical Magazine* 42, 351-364.
- Eshelby, J., 1957. The determination of the elastic field of an ellipsoidal inclusion, and related problems. *Proceedings of the Royal Society of London A* 241, 376-396.
- Fan, H., Aubry, S., Arsenlis, A. and El-Awady, J., 2015. The role of twinning deformation on the hardening response of polycrystalline magnesium from discrete dislocation dynamics simulations. *Acta Materialia* 92, 126-139.
- Fultz, B. and Howe, J., 2001. *Transmission electron microscopy and diffractometry of materials*. New York: Springer.

- Geng, J., Chisholm, M.F., Mishra, R.K., Kumar, K.S., 2015. An electron microscopy study of dislocation structures in Mg single crystals compressed along [0001] at room temperature. *Philosophical Magazine* 95, 3910-3932.
- Ghazisaeidi, M., Curtin, W., Hector Jr., L., 2014. First-principles core structures of $\langle c+a \rangle$ edge and screw dislocations in Mg. *Scripta Materialia* 75, 42-45.
- Hirth, J. and Lothe, J., 1982. *Theory of dislocations*. New York: Wiley.
- Juan, P.-A., Berbenni, S., Barnett, M.R., Tome, C.N., Capolungo, L., 2014. A double inclusion homogenization scheme for polycrystals with hierarchical topologies: application to twinning in Mg alloys. *International Journal of Plasticity* 60, 182-196.
- Kalidindi, S.R., 2001. Modeling anisotropic strain hardening and deformation textures in low stacking fault energy materials. *International Journal of Plasticity* 17, 837-860.
- Keller, R. and Geiss, R., 2012. Transmission EBSD from 10 nm domains in a scanning electron microscope. *Journal of Microscopy* 245, 245-251.
- Kelley, E. and Hosford Jr., W., 1968. Plane-strain compression of magnesium and magnesium alloy crystals. *Transactions of the Metallurgical Society of AIME* 242, 5-13.
- Kocks, U. and Mecking, H., 2003. Physics and phenomenology of strain hardening: the FCC case. *Progress in materials science* 48, 171-273.
- Kuykendall, W.P., Cash, W.D., Barnett, D.M., Cai, W., 2012. On the existence of Eshelby's equivalent ellipsoidal inclusion solution. *Mathematics and Mechanics of Solids* 17, 840-847.
- Lay, S. and Nouet, G., 1994. Interaction of slip dislocations with the (01-12) twin interface in zinc. *Philosophical Magazine A* 70, 1027-1044.
- Lebensohn, R. and Tome, C.N., 1993. A study of the stress state associated with twin nucleation and propagation in anisotropic materials. *Philosophical Magazine A* 67, 187-206.
- Li, B. and Ma, E., 2009. Pyramidal slip in magnesium: Dislocations and stacking fault on the $\{10\bar{1}1\}$ plane. *Philosophical Magazine* 89, 1223-1235.
- Ma, Q., El Kadiri, H., Oppedal, A.L., Baird, J.C., Li, B., Horstemeyer, M.F., Vogel, S.C., 2012. Twinning effects in a rod-textured AM30 Magnesium alloy. *International Journal of Plasticity* 29, 60-76.
- Morozumi, S., Kikuchi, M. and Yoshinaga, H., 1976. Electron microscope observation in and around $\{1-102\}$ twins in magnesium. *Trans. JIM* 17, 158-164.

- Morris, J., Scharff, J., Ho, K., Turner, D., Ye, Y. and Yoo, M., 1997. Prediction of a {1122} hcp stacking fault using a modified generalized stacking-fault calculation. *Philosophical Magazine A* 76, 1065-1077.
- Mura, T., 1982. *Micromechanics of defects in solids*. Hague: Martinus Nijhoff Publishers.
- Muransky, O., Barnett, M.R., Luzin, V., Vogel, V., 2010. On the correlation between deformation twinning and Luders-like deformation in an extruded Mg alloy: In situ neutron diffraction and epsc.4 modelling. *Mater. Sci. Eng.: A*, 527(6), 1383 – 1394.
- Niewczas, M., 2010. Lattice correspondence during twinning in hexagonal close-packed crystals. *Acta Materialia* 58, 5848-5857.
- Numakura, H., Minonishi, Y. and Koiwa, M., 1990. Atomistic study of $1/3\langle 11-23 \rangle\{101-1\}$ dislocations in hcp crystals. II. Motion of the dislocations. *Philosophical Magazine A* 62, 545-556.
- Obara, T., Yoshinga, H. and Morozumi, S., 1973. $\{11-22\}\langle 1123 \rangle$ Slip system in magnesium. *Acta Metallurgica* 21, 845-853.
- Oppedal, A., El Kadiri, H., Tome, C., Kaschner, G., Vogel, S., Baird, J. and Horstemeyer, M., 2012. Effect of dislocation transmutation on modeling hardening mechanisms by twinning in magnesium. *International Journal of Plasticity* 30-31, 41-61.
- Partridge, P., 1967. The crystallography and deformation modes of hexagonal close-packed metals. *Metallurgical Reviews* 12, 169.
- Price, P., 1961. Nucleation and growth of twins in dislocation-free zinc crystals. *Proceedings of the Royal Society of London* 260, 251-262.
- Proust, G., Tome, C.N., Jain, A., Agnew, S.R., 2009. Modeling the effect of twinning and detwinning during strain-path changes of magnesium alloy AZ31. *International Journal of Plasticity* 25, 861–880.
- Reed-Hill, R., 1973. Role of deformation twinning in determining the mechanical properties of metals. In R. Reed-Hill (Ed.), *The inhomogeneity of plastic deformation* (pp. 285-311). Metals Park, OH: American Society for Metals.
- Sandloebes, S., Friak, M., Neugebauer, J. and Raabe, D., 2013. Basal and non-basal dislocation slip in Mg–Y. *Materials Science and Engineering A* 576, 61-68.

- Sandloebes, S., Friak, M., Zaefferer, S., Dick, A., Yi, S., Letzig, D., Pei, Z., Zhu, L.-F., Neugebauer, J. and Raabe, D., 2012. The relation between ductility and stacking fault energies in Mg and Mg-Y alloys. *Acta Materialia* 60, 3011-3021.
- Serra, A., Bacon, D.J., Pond, R.C., 1988. The crystallography and core structure of twinning dislocations in H.C.P. metals. *Acta Metallurgica* 36, 3183-3203.
- Serra, A., Bacon, D.J., 1996. A new model for {10-12} twin growth in hcp metals. *Philosophical Magazine* 73, 333-343.
- Serra, A., Bacon, D.J., Pond, R.C., 2002. Twins as barriers to basal slip in hexagonal-close-packed metals. *Metallurgical and Materials Transactions A* 33, 809-812.
- Stohr, J. and Poirier, J., 1972. Etude en Microscopie Electronique du Glissement Pyramidal {11-22}<11-23> dans le Magnesium. *Philosophical Magazine* 25, 1313-1329.
- Tomsett, D. and Bevis, M., 1969a. The incorporation of basal slip dislocations in {10-12} twins in zinc crystals. *Philosophical Magazine* 19, 129-140.
- Tomsett, D. and Bevis, M., 1969b. The formation of stacking faults in {10-12} twins in zinc as a result of slip dislocation-deformation twin interactions. *Philosophical Magazine* 19, 533-537.
- Trimby, P., 2012. Orientation mapping of nanostructured materials using transmission Kikuchi diffraction in the scanning electron microscope. *Ultramicroscopy* 120, 16-24.
- Wang, F., Sandloebes, S., Diehl, M., Sharma, L., Roters, F., Raabe, D., 2014. In situ observation of collective grain-scale mechanics in Mg and Mg-rare earth alloys. *Acta Materialia* 80, 77-93.
- Wang, F. and Agnew, S., 2015. Dislocation-twin interactions in magnesium alloy AZ31. In A. S. M. V. Manuel (Ed.), *Magnesium Technology 2015* (pp. 139-144). Hoboken, NJ, USA: John Wiley & Sons, Inc.
- Wang, H., Wu, P., Tome, C.N. and Wang, J., 2012. Study of lattice strains in magnesium alloy AZ31 based on a large strain elastic-viscoplastic self-consistent polycrystal model. *International Journal of Solids Structures* 49, 2155-2167.
- Westlake, D., 1961. Twinning in zirconium. *Acta Metallurgica* 9, 327-331.
- Williams, D. and Carter, C., 2009. *Transmission electron microscopy: a textbook for materials science*. Springer.

- 1
2
3
4 Wu, L., Agnew, S., Ren, Y., Brown, D., Clausen, B., Stoica, G., Wenk, H.R. and Liaw, P.K.,
5
6 2010. The effects of texture and extension twinning on the low-cycle fatigue behavior of
7
8 a rolled magnesium alloy, AZ31B. *Materials Science and Engineering A* 527, 7057-7067.
9
10 Wu, Z., Curtin, W.A., 2015. The origin of high hardening and low ductility in magnesium.
11
12 *Nature* 526, 62-67.
13
14 Yamakov, V., Wolf, D., Phillpot, S.R., Mukherjee, A.K., Gleiter, H., 2002. Dislocation processes
15
16 in the deformation of nanocrystalline aluminium by molecular-dynamics simulation.
17
18 *Nature Materials* 1, 45-49.
19
20 Yang, Z., Chishom, M., Duscher, G., Ma, X. and Pennycook, S., 2013. Direct observation of
21
22 dislocation dissociation and Suzuki segregation in a Mg–Zn–Y alloy by aberration-
23
24 corrected scanning transmission electron microscopy. *Acta Materialia* 61, 350-359.
25
26 Yasi, J.A., Nogaret, T., Trinkle, D.R., Qi, Y., Hector Jr., L.G., Curtin, W.A., 2009. Basal and
27
28 prism dislocation cores in magnesium: comparison of first-principles and embedded-
29
30 atom-potential methods predictions. *Modelling and Simulation in Materials Science and*
31
32 *Engineering* 17, 055012.
33
34 Yoo, M., 1981. Slip, twinning, and fracture in hexagonal close-packed metals. *Metallurgical*
35
36 *transactions A* 12A, 409-418.
37
38 Yoo, M. and Wei, C., 1966. Growth of deformation twins in zinc crystals. *Philosophical*
39
40 *Magazine* 14, 573-587.
41
42 Yoo, M. and Wei, C., 1966. Application of anisotropic elasticity theory to the plastic
43
44 deformation in hexagonal zinc. *Philosophical Magazine* 13, 759.
45
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60
61
62
63
64
65