NUCLEATION AND PROPAGATION OF $\{10\bar{1}2\}$ TWINS IN AZ31 MAGNESIUM ALLOY

A. Khosravani¹, D. Fullwood¹, B.L. Adams¹, T. Rampton¹, M.P. Miles², R.K. Mishra³

¹Mechanical Engineering, Brigham Young University, Provo, UT 84602

²Manufacturing Engineering Technology, Brigham Young University, Provo, UT 84602

³General Motors R&D Center, Warren, MI 48090

Keywords: Tensile twin nucleation, AZ31 magnesium alloy, High resolution Electron backscatter diffraction (HR-EBSD); Geometrically necessary dislocations (GNDs); Dislocation density.

Abstract

Nucleation and propagation of tensile twins in magnesium alloy AZ31 are investigated for a large number of twins at an early stage of their development. High-resolution electron backscatter diffraction (HREBSD) techniques are employed to give additional insights. Correlations with grain orientation, boundary misorientation and active slip systems are observed in the region of twins that arise at grain boundaries. Two types of twin are identified: 1) slip-assisted twins that nucleate at grain boundaries with no apparent influence from nearby twins, and 2) twin-assisted twins that result from twins propagating across a grain boundary. Twinning occurs in “hard” grains that cannot accommodate necessary contraction via $\langle a \rangle$ -type slip. Slip assisted twins nucleate at high-angle boundaries. Twin-assisted twinning occurs at low-angle boundaries. The distributions of grain boundary misorientation associated with each type of twin

nucleation are mapped, and a strong correlation between Schmid factor and the emerging twin variant is highlighted.

1 Introduction

Formability of Mg alloys at ambient temperature is generally poor due to an insufficient number of active slip systems in the HCP lattice [1-6]. According to the von-Mises criterion, five independent active slip systems are required for plastic deformation of polycrystalline materials. Hence deformation twinning plays an important role in plastic deformation of these alloys [6-10]. The most important available slip systems in magnesium alloys are basal $\langle a \rangle$ slip, prismatic $\langle a \rangle$ slip, pyramidal $\langle a \rangle$ (type I) slip and pyramidal $\langle c+a \rangle$ (type II) slip. Deformation studies on Mg and Mg alloy single crystals and polycrystals at room temperature have shown that the critical resolved shear stress (CRSS) for basal slip is significantly lower than for non-basal slip [1, 7, 8, 11-15]. However, for strain accommodation along the c-axis, either $\langle c+a \rangle$ slip and / or mechanical twinning is required [1, 8, 16]. In contrast to dislocations, which can move in two directions in the slip plane, twinning can occur in only one direction in the relevant plane; therefore, if the applied load is unfavorable for twin nucleation, the barriers to $\langle c+a \rangle$ slip activation may be overcome such that the hard-to-activate $\langle c+a \rangle$ slip mode plays an important role in deformation in order to fulfill the required five independent slip systems [1-3, 6].

Several researchers have observed activation of non-basal slip at room temperature. Koike et al [2, 3] showed that non-basal slip is needed in the vicinity of grain boundaries in order to maintain strain compatibility, and this compatibility stress causes nucleation of twins or activation of $\langle c+a \rangle$ slip systems; otherwise fracture would occur along grain boundaries. Keshavarz et al [11] observed the presence of prismatic slip within the grain far from grain boundaries and showed that non-basal slip could happen, not only at the grain boundary, but also

1
2
3
4 inside the grains. Agnew et al [13, 16], with experiments and simulations involving the ECAE
5
6 process, showed that in AZ31 and AZ80 the dominant slip system is basal; and also that there
7
8 exists a small portion of $\langle a \rangle$ prismatic and $\langle c+a \rangle$ pyramidal modes. However, they reported that
9
10 at room temperature prismatic slip is the most active non-basal slip system [16]. Beyerlein et al
11
12 [17-19] proposed that twin nucleation is strongly influenced by grain boundary structure and the
13
14 local stress state. By atomistic simulations and statistical analyses of experimental results, they
15
16 showed that twin pairs that join at a common location on the grain boundary were most strongly
17
18 connected to GBs with low misorientation angle.
19
20
21
22

23
24 These works (and many others referenced therein – particularly the review in [20]) begin to
25
26 paint a picture of the complex interactions between twin and slip activity. However, many gaps
27
28 still remain. The extensive study of relationships between grain boundary type and twin activity
29
30 by Beyerlein et al., for example, presents an after-the-event view of twin nucleation, based upon
31
32 inferences derived from analysis of fully developed twins. A more dynamic examination of
33
34 early-life twins is missing. Furthermore, a broader survey of related slip activity is both desirable
35
36 and possible due to recent developments in the field of electron backscatter diffraction (EBSD).
37
38 These advances provide not only higher spatial resolution, but also more information about
39
40 geometrically necessary dislocations (GND) near the scanned surface of a material [21, 22]. New
41
42 insights into slip activity of HCP materials have recently been reported [23, 24], giving hope that
43
44 similar advances may be possible in the study of Mg alloys. Furthermore, further insights into
45
46 slip twin and twin/twin interactions have recently been published [25-31].
47
48
49
50
51
52

53 In this study, we present the first extensive study (to our knowledge) of twins in the early
54
55 stages of propagation for a magnesium alloy. High-resolution EBSD (HR-EBSD) techniques are
56
57 used to incorporate resolved GND content (i.e. GND content resolved onto individual slip
58
59
60
61
62
63
64
65

systems), and inferred slip activity, into a study of the influence of microstructure and local slip activity on twinning events in magnesium alloy AZ31. An automated HR-EBSD technique is employed to map total and resolved dislocation density distributions at various stages of tensile twin formation. While the overall dislocation density measurements are generally admitted as accurate representations, the techniques for resolving dislocation density onto particular slip systems are currently being developed and validated by several groups. Nevertheless the results presented here give a consistent picture based upon current state of the art.

Correlations between tensile twin activity (specifically nucleation and propagation), local microstructure and GND activity are identified. In particular, the influence of specific grain and grain boundary types, and the activity of distinct GND slip systems on twin formation and growth are explored. The results provide more comprehensive physical insights into twin nucleation and propagation, beyond Schmid factor-based studies that have been reported in many previous works.

2 Materials and methods

2.1 Experimental procedure

The material investigated in this study is a rolled Mg alloy AZ31 sheet, which was annealed at 350 C for 1 h and cooled to room temperature in the furnace. The resultant microstructure is twin-free with equiaxed grains that predominantly have their c-axes close to normal with respect to the sheet surface, producing a strong basal texture. The average linear intercept grain size is ~10 μ m. A compression cube of 3mm $\times$ 4mm $\times$ 3mm was machined from the homogenized plate by using wire EDM to avoid any residual stress during cutting; then for EBSD scan preparation the sample was mounted in cold resin and polished. The sample preparation procedure was mechanical polishing followed by an OP-S colloidal silica slurry polish, which provides high

quality surface finishing for EBSD. The sample was etched with a solution of 60% ethanol, 20% distilled water, 15% acetic acid and 5% nitric acid, and the resin mounting was removed. Using a focused ion beam (FIB), platinum fiducial marks were deposited on the sample surface to provide an estimate of total strain in the region where EBSD scans were collected. The sample was compressed along the transverse to rolling direction (TD) up to 7% strain.

Automatic EBSD scans were performed using the FEI-Helios NanoLabTM 600i SEM equipped with OIMTM data acquisition software and a high-speed HikariTM camera. In order to get dislocation maps, the electron backscattered patterns (EBSPs) were saved for all the scan points and later they were post-processed. Regular square grid scans were used to examine a $100 \times 100 \mu m^2$ area with a 200 nm step size. The data with a confidence index < 0.07 were filtered out for texture analysis using TSL OIM software. For dislocation studies, several finer scans with a step size of 100nm were performed in the regions in which twin nucleation was observed.

2.2 HREBSD Methodology

Using high-resolution electron backscatter diffraction (HREBSD) techniques, near-surface GND tensors can be recovered from lattice distortion measurements. The principles and methods for determining GND density and resolving these values onto individual slip systems are described in [21, 32-34], building upon work by Nye and Kröner [35, 36].

The method of resolving dislocation content onto individual slip systems is non-unique; it is reported that the number of independent $\langle c+a \rangle$ slip systems is 5 (screw-type) [37, 38] and 8 prismatic and pyramidal- $\langle a \rangle$ systems [39] leading to 13 slip systems. Hence we obtain a set of 6 equations [40] with 13 unknowns. Furthermore, several of the Nye tensor components are not measurable by the surface methods used in this paper, potentially leads to errors in the reported

GND densities [34]; for example, in the quoted paper, the standard deviation for the error over a range of angles and dislocation types in Mg is approximately 30% for the ‘Pantleon’ approach used in this paper. This methodology, which utilizes seven components from the Nye tensor, is also much less sensitive to dislocation orientation than methods that only employ three components; for example, it can identify dislocations in the basal plane that have no components in the third column of the Nye tensor. For the step size of 200nm used for the detailed scans in this paper, the resolution limit is approximately 10^{14} m^{-2} [41]; hence this is the lower limit shown on those GND maps. It is also known that there is a strong dependency between measured dislocation density and scan step size [41, 42]; for step sizes of the order of a micron and less, the measured GND density can scale inversely with step size. However, in this paper the absolute density is less important than the ratios of densities on resolved slip systems; hence the step size choice is not critical. The GND calculations give consistent indications of trends, but not exact values of densities on individual systems using the current state of the art.

3 Results

Grain maps, Schmid factors and measured relative dislocation activity on different slip systems of a compressed sample are shown in Figs. 2 and 3 for two areas chosen for the local variety in Schmid factor. The IPF maps of the grains are shown in Fig. 2(a); schematic unit lattice cells are superimposed on the maps to provide a better visual sense of the crystal orientations. The Schmid factor maps of $\langle a \rangle$ basal, $\langle a \rangle$ prismatic, $\langle a \rangle$ and $\langle c+a \rangle$ pyramidal slips are plotted in Fig. 2 (b-e) respectively. Using the techniques described in the previous section, the relative activity of basal, prismatic, and pyramidal slip systems are mapped for the AZ31 sample at a strain of 0.5% in Fig. 3. This figure shows total dislocation density and resolved dislocation maps for $\langle 11\bar{2}0 \rangle$ (0001) basal, $\langle 11\bar{2}0 \rangle \{10\bar{1}0\}$ prismatic, $\langle 11\bar{2}0 \rangle$

1
2
3
4 {10 $\bar{1}1$ } pyramidal, and $\langle 11\bar{2}3 \rangle$ {11 $\bar{2}2$ } pyramidal slip systems respectively (both edge and
5
6 screw dislocation types, where relevant). While it is not expected that there will be a perfect
7
8 correlation between Schmid factor and active slip system since the CRSS values of the systems
9
10 are very different and the local stress state may be significantly different from the macro state,
11
12 the maps show a general correlation between Schmid factor and slip system activity for a given
13
14 system. In addition, these figures illustrate the sensitivity of HR-EBSD to a small amount of
15
16 strain.
17
18
19

20
21 While the total dislocation density and the total $\langle a \rangle$ and $\langle c+a \rangle$ dislocation densities are
22
23 reasonably homogeneous for the unstrained sample, as one might expect, the resolution of $\langle a \rangle$ -
24
25 type slip onto different slip systems shows a distinct pattern with certain grains having more
26
27 basal slip than others. This may not be expected in the unstrained sample, and may be an artifact
28
29 of the missing components in the Nye tensor, or the particular algorithm used to resolve slip at
30
31 this level. Nevertheless, the relative (rather than absolute) levels of $\langle a \rangle$ -type slip or $\langle c+a \rangle$ -type
32
33 slip are most important in this investigation, and these results appear to give a consistent picture
34
35 throughout the study.
36
37
38
39
40

41 In both scanned areas in Fig. 3, a slight increase in total dislocation density takes place after
42
43 the relatively small 0.5% strain step. More interestingly, different grain orientations and grain
44
45 boundaries respond differently to the applied strain. As observed in Fig. 3, the resolved
46
47 dislocation density maps are generally in good agreement with Schmid factor levels for each slip
48
49 system across various grains; for instance, in both areas the only two grains which have high
50
51 Schmid factor for basal slip demonstrate higher values in the $\langle a \rangle$ basal dislocation maps.
52
53 Inasmuch as the material has a strong basal texture, low $\langle a \rangle$ basal activity is detected in other
54
55 grains; for the same reason, negligible amounts of pyramidal $\langle a \rangle$ activity are present in both
56
57
58
59
60
61
62
63
64
65

1
2
3
4 scanned areas. Furthermore, the pyramidal $\langle a \rangle$ slip map has the poorest correlation with the
5
6 associated Schmid factor; i.e in both scanned areas, the grains with the highest Schmid factor for
7
8 pyramidal $\langle a \rangle$ slip don't show significant activity after 0.5% strain, while in each area a grain
9
10 with intermediate Schmid factor shows some pyramidal $\langle a \rangle$ activity after 0.5% strain. This may
11
12 be partially explained by the generally low measured activity of this slip system, making it
13
14 sensitive to optimization parameters in the algorithms that resolve the different activities, and
15
16 also sensitive to noise. Low activity of the pyramidal $\langle a \rangle$ slip system is in agreement with
17
18 previous experimental and simulated research in which basal $\langle a \rangle$, prismatic $\langle a \rangle$, and pyramidal
19
20 $\langle c+a \rangle$ slip were either observed or predicted [2, 3, 11, 13, 16].
21
22
23
24
25

26 For the purposes of determining the influence of slip activity on twinning, a simpler view of
27
28 GND content is to combine all $\langle a \rangle$ slip systems into one map and compare that with the total
29
30 $\langle c+a \rangle$ pyramidal slip activity. This is the approach taken in the central images of Fig. 3, and in
31
32 subsequent figures and analysis.
33
34
35

36 Figure 4(a) shows a wider view of the microstructure of the undeformed material,
37
38 illustrating its strong basal texture. The (0001) pole figure contours are spread more towards the
39
40 rolling direction (RD) than the transverse direction (TD). The $\{10\bar{1}0\}$ pole figure indicates no
41
42 preferred orientation for prismatic and pyramidal planes, and a-axes of the HCP lattice are
43
44 randomly oriented in the rolling plane. Figure 4(b) shows the inverse pole figure (IPF) map after
45
46 2.5% compression strain along TD (the regions shown in Figs 4a and 4b are not from the exact
47
48 same location, but are nearby and exhibit similar initial microstructures). Compression along TD
49
50 causes expansion along the ND (the predominant direction for c-axes), which is favorable for
51
52 tensile twin formation. The tensile twins modify the lattice orientation by 86° toward the load
53
54 direction from the orientation of the parent grain, which is evident from diminished basal texture
55
56
57
58
59
60
61
62
63
64
65

and the appearance of a component in the transverse direction on the (0001) pole figure.

Contraction of the peak along the RD direction is also consistent with the observed increase in basal dislocation activity (see [43], along with further data in [44]).

Figure 5(a) and 5(b) show different grain boundary types, quantified in terms of misorientation, superimposed upon Image Quality (IQ) maps of undeformed and 2.5% strained samples, respectively. In undeformed samples almost 60% of the boundaries have misorientations in the range of 13.2 to 39°, with 18.6% in the range of 39-53°. After 2.5% strain, 31 % of the boundaries have a misorientation of 80-90°, corresponding to tensile twin boundaries.

Experimental detection of small twin nuclei by EBSD is limited by the scan step size and poor IQ at the grain boundaries. It may happen that noise in highly deformed regions is confused for a twin, or a twin nucleus is overlooked as a result of choosing too large of a step size or due to low image quality; with carefully chosen microscope parameters we estimate a spatial resolution of approximately 200nm for the EBSD technique in this material. In order to reduce the probability of false positive or negative twin identification, points with low CI and isolated points of a given orientation were filtered out. If multiple neighboring scan points with high CI indicated a twin nucleus, then the presence of the twin was accepted.

Compressing the Mg samples by 2.5% strain causes tensile twins to form copiously across the microstructure, but at this strain level twin thickening is not prevalent and plenty of untwinned grains can be found. In the case of Fig. 5(b), 17% of the total scan area is twinned. Since at 2.5% strain, twinning has not been developed in all grains, it is possible to identify grains in which young tensile twins are present. These twins may form spontaneously at a grain boundary or nucleation may be triggered by another twin in a neighboring grain. In the rest of the

1
2
3
4 paper these two nucleation types are referred to as slip-assisted (a twin that is connected to only a
5
6 single grain boundary, and which does not lie adjacent to a twin in a neighboring grain) and
7
8 twin-assisted (a twin that lies adjacent, at a grain boundary, to a twin in a neighboring grain, that
9
10 may have influenced its formation, or vice versa). Figure 6 shows three tensile twins which
11
12 appear to have formed independently of other twins; they have formed at grain boundaries or
13
14 triple junctions and have not yet passed through the parent grain (i.e. they are slip assisted twins).
15
16 Each figure includes a total dislocation density map, $\langle a \rangle$ and $\langle c+a \rangle$ slip systems maps, and a
17
18 boundary misorientation map. As one can see in Fig. 6 more than one twin can nucleate in any
19
20 given grain at this deformation level. For instance, in regions 2 and 3 multiple twins have
21
22 nucleated, as indicated by T_1 , T_2 , T_3 and T_4 in the figures. We note that at high strain levels, the
23
24 slip-assisted twins have all propagated fully across grains and thickening of twins is more
25
26 prevalent.
27
28
29
30
31
32

33
34 It must be emphasized at this point that the EBSD approach taken in this paper is limited to
35
36 a 2D view of events. A nucleated twin may have developed some distance below the surface, and
37
38 may have even nucleated at a different grain boundary from the one observed on the surface, for
39
40 example. Nevertheless, it appears clear from our observations, and those of previous authors, that
41
42 nucleation generally occurs at grain boundaries (e.g. [45]). The consistent nature of the
43
44 observations for a large number of events in this study indicates that the observed trends and
45
46 correlations regarding nucleation and propagation events are valid.
47
48
49

50
51 Using the data from the scans, we wish to identify those grain boundaries more prone to
52
53 twin nucleation (slip-assisted twins), and those that allow propagation (twin-assisted twins).
54
55 Distributions of the misorientation angles for the grain boundaries that have been intersected by
56
57 at least one twin are plotted in Fig 7. The plot shows very good agreement with Beyerlein's
58
59
60
61
62
63
64
65

1
2
3
4 results in terms of the distribution of twins associated with a given grain boundary range [17-19].
5
6
7 However, this map does not distinguish between twins that are identified as slip assisted or twin
8
9 assisted. The plot is subsequently dissociated into two plots corresponding to each type of twin;
10
11 the data for the resolved twin type is a subset of the original data since it was determined
12
13 manually rather than using automated EBSD software. Figure 8 shows the distribution with
14
15 misorientation angle for 200 cases of twins that formed at grain boundaries, labeled as either
16
17 slip-assisted or twin-assisted. The grain boundary misorientations wherein twins nucleated
18
19 spontaneously (slip-assisted) all fall into the range of high angle boundaries, while the twin-
20
21 assisted GBs are all less than 40° .
22
23
24

25
26 In addition to the boundary type associated with twin nucleation, we have studied the
27
28 dislocation activity in the region of nucleation events. The total dislocation density maps show
29
30 higher activity of slip systems close to boundaries relating to nucleation events. Resolved
31
32 dislocation maps show more $\langle a \rangle$ slip contents in the vicinity of the high angle boundary in the
33
34 grains neighboring the twinning nucleation site (e.g. the grains labeled with a 'g' in Fig. 6). This
35
36 is almost universally the case in the slip-assisted twins studied, although in some cases the effect
37
38 was not as pronounced.
39
40
41

42
43 A further observation that can be generally made (once again, observed in almost all twins
44
45 to a greater or lesser extent) is that the twinned regions have lower dislocation densities than
46
47 parent grains. Taken at face value, the resolved dislocation maps reveal that the level of $\langle c+a \rangle$
48
49 dislocations are particularly reduced compared with $\langle a \rangle$ type; this is highlighted in region 3 of
50
51 Fig. 6, for example, which has a larger twin. The $\langle a \rangle$ slip map shows a homogenous distribution
52
53 inside and outside of the twins. Possible interpretations of this observation are discussed in
54
55 Section 4.1.
56
57
58

Knowing the orientation of each grain, as well as the loading direction, permits the calculation of Schmid factors pertaining to the six possible $\{10\bar{1}2\}$ twin variants: $(10\bar{1}2)[\bar{1}011]$, $(01\bar{1}2)[0\bar{1}11]$, $(\bar{1}102)[1\bar{1}01]$, $(\bar{1}012)[10\bar{1}1]$, $(0\bar{1}12)[01\bar{1}1]$, and $(1\bar{1}02)[\bar{1}101]$.

Theoretically, tensile $\{10\bar{1}2\}$ twins can be formed in six equivalent planes, with a specific shear direction. Twinning differs from slip activity by its polar nature; i.e. twins form in just one direction in the twin plane. Hence a shear vector is considered in the Schmid formula instead of a shear line; this leads to a negative Schmid factor for some of the twin variants. The twin variant with negative Schmid factor has no chance of being formed in the parent grain, even if it has a high absolute Schmid factor. In Fig. 9, the orientation of each of the G1, G2, and G3 grains shown in Fig. 6 are indicated in (0001) pole figures. The orientation of six possible twin variants with corresponding Schmid factors are plotted in the pole figure. The arrows indicate the twin variant formed during the experiment. In each of the twins studied in this paper, the variant with highest positive Schmid factor was selected for the first twin in a grain. If there was more than one twin, the variant with second highest positive Schmid factor was sometimes selected. This observation supports previous work in this area, concluding that twin variant selection generally correlates with Schmid factor (e.g. [46]).

We now wish to extract correlations relating to propagation of twins across various grain boundary types. The formation of a twin leads to shear in the grain, and when the twin grows and intersects grain boundaries, it could trigger twin nucleation in the neighboring grain. To investigate which grain boundaries are susceptible to twin propagation, a large number of twins were identified that “transmit” from one grain to another one (i.e. that neighbor a twin in the next grain). Three scan areas are shown in Fig. 10. The orientations of the grains are first shown via the inverse pole figure map, followed by the image quality map with grain boundary

1
2
3
4 misorientations highlighted; finally the total GND content, and resolved $\langle a \rangle$ and $\langle c+a \rangle$ contents
5
6 are shown. The orientations of G1, G2, G3, and G4 grains in Fig. 10 are plotted in (0001) pole
7
8 figures in Fig. 11 with the corresponding six possible twin variants in each grain. For twin-
9
10 assisted nucleation, twin variant selection still shows good agreement with Schmid factor; i.e. the
11
12 nucleated twin variant generally has the highest Schmid factor.
13
14

15
16 For each of the regions shown, twin growth in one grain results in twinning in neighboring
17
18 grains; however, twins appear to transmit from one grain into another through certain types of
19
20 boundaries. The higher angle grain boundaries (highlighted in yellow) are not associated with
21
22 twin propagation (for example, see the region labeled by 'gb1' in region 1). In contrast, a twin
23
24 can cause twinning in a neighboring grain when grain boundary misorientation is low (for
25
26 example, the boundaries labeled by gb2). Figure 12 shows distributions of misorientation angle
27
28 for these two boundary types - those through which twins transmit and those at which twins are
29
30 blocked, respectively. It is evident from Fig. 12 that the boundaries which allow the twins to pass
31
32 through have low misorientation while the misorientation angles for the boundaries which block
33
34 twin propagation are high angled. This is consistent with recent observations by Kacher and
35
36 Minor in HCP rhenium; they observed twin transmission at misorientations below 25° , and
37
38 blocking above 25° [28]. Interpretation of the result was tied to alignment of the shear vector for
39
40 the incoming and potential outgoing twins from the GB.
41
42
43
44
45
46
47

48 A higher total dislocation content is found at the twin tips in the scans of Fig. 10, which
49
50 shows mostly $\langle a \rangle$ type slip (notice the twins labeled T1 and T2). In general this higher GND
51
52 content is ahead of the crack tip, where there is also higher noise in the image quality map
53
54 associated with the forming twin. However, we have not seen a strong correlation between this
55
56 type of noise and calculated GND content; furthermore, similar observations are noted in [45].
57
58
59
60
61
62
63
64
65

This observation also parallels the previous observation of high $\langle a \rangle$ type GND content near boundaries that nucleate twins (i.e. the high $\langle a \rangle$ slip at the twin tip may affect nucleation in the adjoining grain).

The following three figures provide further examples of twin-assisted twin activity. Figure 13 highlights the blocking properties of high angle boundaries; a twin is blocked at a grain boundary even up to 7% strain. The boundary indicated by an arrow has a misorientation angle of 57° ; several twins form in the grain below the boundary but do not cross it. While we have used the term “block” to indicate that twins do not cross a boundary, one may also think of this phenomenon in terms of the lack of incentive for a twin to propagate into a neighboring grain. Since the grain above the boundary in question is oriented such that the c-axis is further from the normal axis than the surrounding grains (less red in the color version), its basal slip systems are oriented such that they may be able to accommodate extension in the ND direction (due to the constancy of volume effect) via basal slip, rather than requiring twin formation. This is discussed in more detail in the next section.

Figure 14 shows a larger scan, including several grains in which twinning (both nucleation and propagation) has occurred in some grains, but other grains are twin-free. In agreement with the trend highlighted by Fig. 8, the twins pass through boundaries whose misorientation is less than 40° , but they are not transmitted through high angle-misoriented boundaries (e.g. T1 twin in Fig. 14). Once again, slip-assisted twins form at the higher angle boundaries (T2 twins in Fig. 14). Again, as can be seen in the $\langle a \rangle$ and $\langle c+a \rangle$ dislocation maps, the grains with twins typically have higher $\langle c+a \rangle$ slip in the parent grain. This will be discussed in more detail below.

4 Discussions

Up to this point this investigation has purposefully focused on correlations between twin formation and the local phenomena of slip activity on particular planes, as well as grain boundary types. At this level, both slip- and twin-assisted twin nucleation appears to relate to dislocation concentration or pile-up at grain boundaries: (a) slip-assisted nucleation correlates with $\langle a \rangle$ type slip accumulation behind a high-angle boundary, and (b) twin-assisted nucleation may be triggered by dislocation concentration at the tip of a twin as it intersects low-angle grain boundaries. We now shift the discussion to the context of grain-level slip activity for a more complete picture of twin nucleation.

From this grain-level view the material has a strong basal texture, with the c-axis of the hexagonal grains pointing predominantly in the normal direction (ND) to the original sheet from which it was cut. The sample was compressed in the transverse direction (TD), and hence the material will tend to expand in other directions due to the constancy of volume effect. The sample can readily expand in the RD due to $\langle a \rangle$ -type slip (which has a low CRSS), but it cannot readily expand in the ND by slip, since the $\langle c+a \rangle$ systems, which facilitate slip in this direction, have high CRSS. From this point of view, “hard” grains (those that are resistant to slip in the ND direction) are defined as those grains that are oriented with the c-axis close to the ND direction. If a grain has a c-axis that is oriented away from ND, then $\langle a \rangle$ -type slip in that grain may accommodate extension in the ND, and the grain is termed “soft”, for the purpose of this paper.

4.1 Total and resolved dislocation densities

The evolution of dislocations maps in Fig. 3 shows activation of slip systems at low strain levels of 0.5 %. Once deformation starts, dislocation sources increase the dislocation content in the microstructure. Depending on boundary type, grain boundaries can act as a source for dislocations as well as a sink [47]. As is evident from Fig. 3, greater dislocation content is

1
2
3
4 observed at grain boundaries. Within the grains, dislocation content was compared with Schmid
5
6 factors on the available slip systems. In the Schmid factor calculation it was assumed that the
7
8 local stress state was identical to the global one, which is only an approximation. Nevertheless,
9
10 the resolved dislocation maps show good agreement with Schmid factor maps. Although the
11
12 CRSS for $\langle a \rangle$ basal slip is the lowest relative to other slip systems [2, 3, 7, 12, 13, 16], less
13
14 activation of $\langle a \rangle$ basal slip is observed in the two representative scans compared with $\langle a \rangle$
15
16 prismatic slip. This is the result of a strong basal texture where most of the grains are oriented
17
18 more favorably for prismatic rather than for basal slip. The $\langle a \rangle$ pyramidal slip type shows
19
20 negligible levels in both scan areas even though it has a relatively high Schmid factor; the same
21
22 result is observed after 2.5 % strain in other scans. The higher CRSS for $\langle a \rangle$ pyramidal slip
23
24 (compared to basal slip, as discussed above) is the most likely reason for the low observed levels
25
26 of $\langle a \rangle$ pyramidal slip.
27
28
29
30
31
32

33
34 Figure 14 illustrates a general trend indicating higher $\langle c+a \rangle$ content in grains that do not
35
36 twin. These grains are generally what we have termed soft grains; they can accommodate
37
38 deformation in the ND direction of interest via basal slip, but in order to accommodate macro-
39
40 scale compression in the TD direction they may need to undergo a small amount of $\langle c+a \rangle$ slip
41
42 and / or some compression twinning in order to provide the necessary c-axis compression. On the
43
44 other hand, the hard grains that are more prone to twinning to accommodate extension in the ND
45
46 can more easily accommodate the macro TD compression with $\langle a \rangle$ -type slip (specifically,
47
48 prismatic slip since the Schmid factor for basal slip would be small).
49
50
51
52

53
54 Considering the new twins, the relative dislocation activity in Fig. 6, Fig. 10, and Fig. 14
55
56 (and many other similar twins) indicates lower dislocation content inside the twin, with particular
57
58 reduction in $\langle c+a \rangle$ -type dislocations inside the twin. An accurate interpretation of this result is
59
60
61
62
63
64
65

not necessarily clear at this point. Several TEM studies have considered dislocation formation in twins of hexagonal materials and have noted the presence of residual $\langle c \rangle$ [45] or $\langle c+a \rangle$ [48, 49] dislocations within twins, while a-type dislocations are initially absorbed by the twinning process [45]. Later build-up of dislocations is also noted in twins (e.g. [29, 31]); for one thing, the reorientation of the lattice during the twinning process makes a-type slip more favorable during subsequent deformation [50]. These studies generally do not quantify dislocation content, and certainly do not consider GND content; furthermore, our observations are generally on young or partially formed twins with little chance for further slip within the twin. Hence further studies are warranted in this area. One explanation may be that the resultant lattice after twin formation contains fewer defects (noise), that were previously interpreted as $\langle c+a \rangle$ slip (i.e. the noise threshold is reduced, and the dislocation analysis is more accurate inside the twin than outside).

4.2 Correlation of twin nucleation sites with $\langle a \rangle$ slip systems

As is evident in Fig. 6 and 10, the activities of $\langle a \rangle$ and $\langle c+a \rangle$ slip types differ for different grains based on their orientations. Once deformation starts, slip system activities in each grain are generally based on Schmid factor and CRSS values for that type of slip system. The movement of dislocations is based on the stress field around the dislocation core and the magnitude of the Burgers vector. Lattice defects, such as twin and grain boundaries, vacancies, and stacking faults, can promote or impede dislocation movement. The split dislocation maps in Fig. 6 and Fig. 10 show concentration of $\langle a \rangle$ type slip at high angle grain boundaries. The lattices of the grains at the two sides of these boundaries have strong mismatch, which makes it difficult for dislocations to glide from one grain to another one [47]. Therefore accumulation of dislocations occurs at these boundaries.

1
2
3
4 The deformation mode of interest in this paper involves extension in the ND due to the
5
6 constancy of volume effect. This is facilitated in the soft grains via $\langle a \rangle$ -type slip, with
7
8 accumulation of dislocations at the boundaries with hard grains. To accommodate
9
10 incompatibility stress caused by basal slip systems at the grain boundary and to avoid grain
11
12 separation, either $\langle c+a \rangle$ slip (see Koike [3]) or twinning is required in the hard grains. The
13
14 misorientation distribution map in Fig. 5(c) shows that only 30% of the boundaries in the
15
16 undeformed material have misorientation above 39° , and yet, from Fig. 8, the vast majority of
17
18 slip-assisted twins form at these higher angle interfaces.
19
20
21
22

23
24 Once twins are formed, their boundaries also act as a barrier for dislocation movement [51].
25
26 As is evident in the split dislocation maps, higher $\langle a \rangle$ and $\langle c+a \rangle$ slip activity can be observed at
27
28 twin boundaries and twin tips. Upon further deformation, twin growth continues until it reaches
29
30 the other side of the parent grain. Depending on the boundary type that it then encounters, twins
31
32 can transmit to a neighboring grain or be suppressed. If the neighboring grain is also a hard
33
34 grain, then the extension due to the constancy of volume effect most likely requires twin
35
36 formation to be accommodated. Furthermore, the misorientation between the two grains is more
37
38 likely to be low since they both have a similarly aligned c-axes (the maximum misorientation
39
40 between two grains with aligned c-axes would be 30°). As it is shown in Fig. 10, when the grain
41
42 boundary has low angle misorientation (approximately $5-35^\circ$), the $\langle a \rangle$ slip concentration at the
43
44 twin tip potentially helps to instigate twinning in the neighboring grain. This is consistent with
45
46 studies such as that of Song and Gray [45], which notes the accumulation of a-type dislocations
47
48 near twin tips and the preferred nucleation of twins in regions of high a-type dislocation density.
49
50
51
52

53
54 In contrast to this mechanism, if the twin reaches a high angle boundary (above 35°), the
55
56 neighboring grain is more likely to be soft (as mentioned, the maximum misorientation between
57
58
59
60
61
62
63
64
65

two grains with perfectly aligned c-axes is 30°), and can accommodate the deformation via $\langle a \rangle$ -type slip. Hence, the $\langle a \rangle$ slip content at the twin tip does not cause twin nucleation in the neighboring grain. Therefore at the observed strain level the twins appear to be impeded by high angle boundaries.

[29, 31, 45, 48-50] The twin variant selection is highlighted in relation to Schmid factor in various grains in Fig. 6 and Fig. 10. Since the texture is a highly basal one, having a high Schmid factor of 0.45 for at least one of the twin variants is not surprising. Figure 9 and 11 show that tensile twin selection is governed by the Schmid factor. However, twin variants with lower Schmid factor still have some probability of being nucleated (i.e. in some cases the variant with the second highest Schmid factor appears).

5 Conclusions

The aim of this study was to investigate correlations relating to tensile twin nucleation / propagation using HR-EBSD techniques. Using this approach, total and, to a less certain extent, resolved dislocation densities can be derived from the EBSPs (electron backscattered patterns). The main results can be summarized as follows:

1. Tensile twinning generally obeys a Schmid factor criterion. The twin variant with higher Schmid factor is generally activated; however, the twin variant with second highest Schmid factor sometimes forms in the grain. Other statistical studies have shown similar results [17, 54, 55].
2. Slip-assisted tensile twin nucleation in polycrystalline AZ31 predominantly occurs in a grain which meets two requirements: the grain has a boundary with misorientation of higher than 39° and high activity of $\langle a \rangle$ slip systems present on the other side of that boundary in a neighboring grain. This is consistent with the view of a twin nucleating in a

1
2
3
4 hard (c-axis aligned with ND) grain, in order to provide compatible strain for a
5
6
7 neighboring soft grain that deforms by $\langle a \rangle$ -type slip; the $\langle a \rangle$ -type slip in the soft grain
8
9 accumulates at the high angle boundary with the hard grain, and leads to twin nucleation
10
11 in the hard grain in order to diffuse this incompatibility. The general observation of high
12
13 a -type dislocation at twin tips and in regions of twin nucleation is consistent with TEM
14
15 studies.
16
17

18
19 A certain interpretation of Beyerlein's results [17] might suggest that nucleation occurs at
20
21 low angle boundaries, rather than the observed high angle boundaries. However, given
22
23 that the distribution of overall grain boundary intersections with twins in the current study
24
25 agrees with that of Beyerlein's [17], the authors believe that the observation of twins in
26
27 their early stages (which was not possible in the Beyerlein study) is critical for accurate
28
29 correlations of nucleation with grain boundary types to be drawn.
30
31
32

- 33
34 3. At low strain, high-angle grain boundaries, in the range above 35° , are not susceptible to
35
36 twin transmission through them to the neighboring grain. This is likely to be a due to a
37
38 combination of factors. Perhaps the most straightforward view is that given by Kacher and
39
40 Minor, in which they show that the misalignment of shear vectors for the incoming and
41
42 outgoing twins at the GB is too high for transmission [28].
43
44

45
46 In contrast, tensile twins can propagate through low angle grain boundaries (5 - 35°
47
48 misorientation). This conclusion is in agreement with Beyerlein's study of 'fully-twinned'
49
50 material where it was postulated that the adjoining twin pairs predominantly occurred at
51
52 low angle grain boundaries. At these boundaries the alignment of the twin shear vectors is
53
54 more favorable. Dislocation concentration or pile-up at the incoming twin tip can produce
55
56 incompatibility stress at the grain boundary that motivates formation of another twin in the
57
58
59
60
61
62
63
64
65

neighboring grain. Furthermore, with a low angle GB, the neighboring grain is more likely to also be a hard grain, and susceptible to twin growth rather than slip deformation.

4. The young and partially formed tensile twins exhibit lower net GND content than their parents; particularly with lower $\langle c+a \rangle$ slip content inside the twin. While TEM studies may indicate lower a-type dislocation content initially (in line with our observations), several studies have also noted $\langle c \rangle$ or $\langle c+a \rangle$ dislocation content in new twins. One explanation for the observations in this paper is that various lattice defects are reduced during twin formation, leading to a lower noise level for dislocation content detection; hence the analysis of relative dislocation activity is simply more accurate inside the twin than outside.

In a similar manner, higher $\langle c+a \rangle$ slip in a grain correlates with those grains that do not twin. These grains generally have c-axes that are oriented away from ND (soft grains), hence in order to accommodate the macro-scale compression in the TD direction they may undergo a small amount of $\langle c+a \rangle$ slip. On the other hand, the grains that we have termed hard (the c-axis is aligned with the ND) are more prone to twinning to accommodate extension in the ND due to the constancy of volume effect, but can more easily accommodate the macro TD compression with $\langle a \rangle$ -type slip.

Acknowledgments

Research was supported by the National Science Foundation (NSF), under award #CMMI 0928923 and 1404771 (magnesium-specific data collection and analysis) and by U.S. Department of Energy (DOE), Office of Science, Basic Energy Sciences (BES), under Award #DE-SC0012587 (high-resolution EBSD and dislocation microscopy development). The authors

also gratefully acknowledge Dr. Anil Sachdev for providing microscopy and lab facilities at GM R&D. We are grateful to Mr. Bob Kubic at GM R&D for assistance with sample preparation.

REFERENCES

- [1] Sandlöbes S, Zaefferer S, Schestakow I, Yi S, Gonzalez-Martinez R. *Acta Materialia* 2011;59:429.
- [2] Koike J, Ohyama R. *Acta Materialia* 2005;53:1963.
- [3] Koike J, Kobayashi T, Mukai T, Watanabe H, Suzuki M, Maruyama K, Higashi K. *Acta Materialia* 2003;51:2055.
- [4] Agnew SR, Yoo MH, Tomé CN. *Acta Materialia* 2001;49:4277.
- [5] Wu L, Jain A, Brown DW, Stoica GM, Agnew SR, Clausen B, Fielden DE, Liaw PK. *Acta Materialia* 2008;56:688.
- [6] Agnew SR, Horton JA, Yoo MH. *Metallurgical and Materials Transactions A* 2002;33A:851.
- [7] Hong S-G, Park SH, Lee CS. *Acta Materialia* 2010;58:5873.
- [8] Jiang L, Jonas JJ, Luo AA, Sachdev AK, Godet S. *Materials Science and Engineering: A* 2007;445-446:302.
- [9] Martin É, Capolungo L, Jiang L, Jonas JJ. *Acta Materialia* 2010;58:3970.
- [10] Park SH, Hong S-G, Lee CS. *Scripta Materialia* 2010;62:202.
- [11] Keshavarz Z, Barnett MR. *Scripta Materialia* 2006;55:915.
- [12] Koike J. *Metallurgical and Materials Transactions A* 2005;36:1689.
- [13] Agnew SR, Mehrotra P, Lillo TM, Stoica GM, Liaw PK. *Acta Materialia* 2005;53:3135.
- [14] Choi SH, Shin EJ, Seong BS. *Acta Materialia* 2007;55:4181.
- [15] Lou XY, Li M, Boger RK, Agnew SR, Wagoner RH. *International Journal of Plasticity* 2007;23:44.
- [16] Agnew SR, Brown DW, Tomé CN. *Acta Materialia* 2006;54:4841.
- [17] Beyerlein IJ, Capolungo L, Marshall PE, McCabe RJ, Tome CN. *Philosophical Magazine* 2010;90:2161.
- [18] Beyerlein IJ, McCabe RJ, Tomé CN. *Journal of the Mechanics and Physics of Solids* 2011;59:988.
- [19] Wang J, Beyerlein IJ, Tomé CN. *Scripta Materialia* 2010;63:741.
- [20] Beyerlein IJ, Capolungo L, Marshall PE, McCabe RJ, Tomé CN. *Philosophical Magazine* 2010;90:2161.
- [21] Gardner CJ, Adams BL, Basinger J, Fullwood DT. *International Journal of Plasticity* 2010;26:1234.
- [22] Wilkinson AJ, Randman D. *Philosophical Magazine* 2010;90:1159.
- [23] Britton TB, Biroscas S, Preuss M, Wilkinson AJ. *Scripta Materialia* 2010;62:639.
- [24] Littlewood PD, Britton TB, Wilkinson AJ. *Acta Materialia* 2011;59:6489.
- [25] Yu Q, Wang J, Jiang Y, McCabe RJ, Li N, Tomé CN. *Acta Materialia* 2014;77:28.
- [26] Wang L, Yang Y, Eisenlohr P, Bieler TR, Crimp MA, Mason DE. *Metallurgical and Materials Transactions A* 2010;41:421.
- [27] Guo Y, Britton TB, Wilkinson AJ. *Acta Materialia* 2014;76:1.
- [28] Kacher J, Minor AM. *Acta Materialia* 2014;81:1.

- [29] Morrow BM, E.K.Cerreta, R.J.McCabe, C.N.Tomé. *Materials Science and Engineering A* 2014;613:365.
- [30] Kadiri HE, Kapil J, Oppedal AL, Jr. LGH, Agnew SR, Cherkaoui M, Vogel SC. *Acta Materialia* 2013;61:3549.
- [31] Kaschner GC, Tome CN, McCabe RJ, Misra A, Vogel SC, Brown DW. *Materials Science and Engineering A* 2007;463:122.
- [32] Fullwood D, Adams B, Basinger J, Ruggles T, Khosravani A, Sorensen C, Kacher J. *Microstructure Detail Extraction via EBSD: An Overview*. In: Barrabas R, Ice GE, editors. *Strains and dislocation gradients from diffraction*. Imperial College Press, 2014.
- [33] Britton TB, Wilkinson AJ. *Acta Materialia* 2012;60:5773.
- [34] Ruggles T, Fullwood D. *Ultramicroscopy* 2013;133:8.
- [35] Nye JF. *Acta Metallurgica* 1953;1:153.
- [36] Kroner E. *Ergebnisse der Angewandten Mathematik* 1958;5:1327.
- [37] Nogaret T, Curtin WA, Yasi JA, Jr LGH, Trinkle DR. *Acta Mater* 2010;58:4332.
- [38] Staroselsky A, Anand L. *International Journal of Plasticity* 2003;19:1843.
- [39] Groh S, Marin EB, Horstemeyer MF, Bammann DJ. *Modelling and Simulation in Materials Science and Engineering* 2009;17.
- [40] Pantleon W. *Scripta Materialia* 2008;58:994.
- [41] Kysar JW, Saito Y, Oztog MS, Lee D, Huh WT. *International Journal of Plasticity* 2010;26:1097.
- [42] Adams BL, Kacher J. *Computers, Materials and Continua* 2009;14:185.
- [43] Wang YN, Huang JC. *Materials Chemistry and Physics* 2003;81:11.
- [44] Khosravani A, Scott J, Fullwood D, Miles M, Mishra R. *Int J Plasticity* 2013;45:160.
- [45] Song SG, Gray I, G. T. *Acta Metallurgica et Materialia* 1995;43:2339.
- [46] Koike J. *Metall. Mater. Trans.A* 2005;36:1689.
- [47] John Price Hirth JL. *Theory of Dislocations*: McGraw-Hill, 1975.
- [48] Bhattacharyya D, Cerreta EK, McCabe R, M. Niewczas, III GTG, Misra A, Tome CN. *Acta Materialia* 2009;57.
- [49] Morrow BM, McCabe RJ, Cerreta EK, Tome CN. *Met Trans A* 2014;45:36.
- [50] McCabe RJ, Proust G, Cerreta EK, Misra A. *International Journal of Plasticity* 2009;25:454.
- [51] Yoo M. *Metallurgical and Materials Transactions A* 1981;12:409.
- [52] Huber J, Hatherly M. *Metal Science* 1979;13:5.
- [53] Huber J, M.Hatherly. *Materials Research and Advanced Technique* 1980;71:15.
- [54] Capolungo L, Marshall PE, McCabe RJ, Beyerlein IJ, Tome CN. *Acta Materialia* 2009;57:6047.
- [55] Morrow BM, R.J.McCabe, E.K.Cerreta, C.N.Tomé. *Materials Science and Engineering A* 2013;574:157.

(a)

(b)

(c)

(d)

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10

Figure 11

Figure 12

Figure 13

Figure 14

Table 1. Numbers of total and independent slip systems in magnesium

Slip system	Number of slip system	Number of independent slip system
Basal $\langle a \rangle$	3	2
Prismatic $\langle a \rangle$	3	2
Pyramidal $\langle a \rangle$ type I	6	4
Pyramidal $\langle c+a \rangle$ type II	6	2

Example EBSD inverse pole figure, image quality map and dislocation density maps for AZ31 showing newly formed twins for three sample regions