EVOLUTION OF HARDENING IN AZ31B MAGNESIUM SHEET

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The monotonic and cyclic mechanical behavior of O-temper AZ31B Mg sheet was measured in large-strain tension/compression and simple shear. Metallography, acoustic emission (AE), and texture measurements revealed twinning during in-plane compression and untwining upon subsequent tension, producing asymmetric yield and hardening evolution. A working model consistent with the micro- and macro-experimental data was constructed. Plastic straining occurs preferentially by basal slip, which provides two independent slip systems with low critical resolved shear stress. The macroscopic flow stress is determined by the required activation of higher-stress mechanisms to accommodate arbitrary deformation: predominantly non-basal slip for initial tension, twinning for initial compression, and untwining for tension following compression. The nucleation stress for twinning is larger than that for untwinning. Increased accumulated hardening increases the twin nucleation stress, but has little effect on untwinning. Multiple-cycle deformation tends to saturate after a few cycles of +/- 0.02 strain, but does not saturate for +/-0.04 strain cycles by the time fracture intervenes at 10 cycles.
Dedicated to my family
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CHAPTER 1

INTRODUCTION

With increasing demand for the application of light materials in the transportation industry, the plastic deformation behavior of magnesium alloys has been of recent interest. Compared with casting counterparts, wrought magnesium alloys have better mechanical properties, including tensile properties [1, 2] and fatigue resistance [3]. However, because of poor formability at room temperature, the large-scale utilization of sheet-formed magnesium alloys, for example AZ31B magnesium alloy sheet, has not developed.

The low formability at room temperature mainly arises from the limited number of slip systems in the hexagonal close packed (HCP) Mg alloys. The dominant slip system of magnesium AZ31B alloy at room temperature is slip in the close packed direction ($<11\bar{2}0>$ or $<a>$) on the basal (0001) plane [1]. The critical resolved shear stress (CRSS) of basal slip in pure magnesium is about 0.5MPa [4-6]. Other slip systems, such as non-basal slip of $<a>$ on prismatic $\{10\bar{1}0\}$ planes [7], $<a>$ on pyramidal $\{10\bar{1}1\}$ planes and $<c+a>$ on pyramidal $\{11\bar{2}2\}$ planes [8, 9] were also observed in magnesium, although their critical resolved shear stresses are two orders higher than basal slip [4, 6]. According to the Von Mises criterion [10, 11], five independent slip systems are needed to accommodate the homogeneous deformation of polycrystalline materials. Basal $<a>$
slip, prismatic $<a>$ slip and pyramidal $<a>$ slip provide only four independent slip systems. Pyramidal $<c+a>$ slip, which in principle provides the additional independent slip systems, is unavailable at room temperature because of its high CRSS [9, 12]. At elevated temperature, the activation of pyramidal $<c+a>$ slip and other non-basal slip occurs at lower CRSS, reducing flow stress and increasing formability [13, 14]. At room temperature, twinning can provide an independent deformation mechanism (in addition to basal and non-basal $<a>$ slip systems) to satisfy the Von Mises criterion [15].

In HCP crystals, the twinning systems are strongly correlated with $c/a$ ratio [16, 17]. The $c/a$ ratio for pure magnesium is 1.624 [1], which is close to, but less than, the ideal hard-sphere value of 1.633. Two twin modes, $\{10\bar{1}2\}<10\bar{1}1>$ and $\{10\bar{1}1\}<10\bar{1}2>$, have been reported in magnesium [17], with $\{10\bar{1}2\}<10\bar{1}1>$ being the most common and easily-activated twin in magnesium and many other HCP metals [1, 4, 16]. A CRSS for twinning, while low [18], is not well established because twin nucleation is inhomogeneous and depends on microstructure features [16, 19]. A fresh twin has a higher nucleation stress than the stress to propagate an existing twin [19, 20].

Because of crystal symmetry, the shear direction for $\{10\bar{1}2\}$ twin reverses at $c/a = \sqrt{3}$. Since the $c/a$ ratio of magnesium is smaller than $\sqrt{3}$, the $\{10\bar{1}2\}$ twin is a 'tension' twin, that is, its activation is associated with extension parallel to the c-axis in the HCP crystal structure [17], and with contraction in a direction lying normal to $<c>$, i.e. $<a_1>$, $<a_2>$, or $<a_3>$. Because of the polar nature of twinning, the shear can occur only in one direction rather than opposite directions [14, 15]. Therefore, a contraction along the c-axis cannot be accommodated by a $\{10\bar{1}2\}$ twin. In magnesium, a theoretical maximum
extension of 6.4% along c-axis can be accommodated by complete reorientation of \(\{10\bar{1}2\}<10\bar{1}\bar{1}>\) twins [15]. After twinning, the c-axis will reorient to lie approximately in the original basal plane [21].

Rolled AZ31B magnesium sheet alloy usually has very strong basal texture generated by rolling [1, 9, 22, 23], where the c-axis of HCP lattice is predominantly aligned parallel to the sheet normal [23, 24]. A state of stress which causes an extension in the sheet normal direction will activate twinning at low stress, while a state of stress that causes contraction normal to the sheet plane does not activate twinning [9, 15, 22, 25-28]. Conversely, an in-plane compression activates twinning but in-plane extension does not [19]. Of course, local inhomogeneities from grain-to-grain interactions can activate limited twinning, particularly in view of the limited number of independent slip systems, as can orientations of some grains that don’t lie in the predominant basal texture. Sheet materials are typically formed by combinations of bending and stretching, both of which are dominated by in-plane loading, with through-thickness stresses much smaller. Therefore, the in-plane plastic deformation properties are of interest for sheet forming application. The yield stress for in-plane compression of Mg sheet is typically one half of that for in-plane tension [27, 29]. After yield, the compressive hardening curve exhibits an inflected stress-strain curve with initially low strain hardening rate, distinct from the tensile behavior of AZ31B Mg alloy [21, 23, 27, 30, 31]. At larger compressive strain, when twinning is exhausted or nearly so and slip dominates, the compressive hardening curve takes on the appearance of the tensile one [23]. At high temperatures, as twinning is suppressed by lower slip system CRSS’s, the inflection hardening curve disappears [23].
In addition to dislocation slip and twinning, untwinning (or detwinning) may occur in a twinned material. It is responsible for the shape memory effect in shape memory alloys [32-34], where untwinning is the growth of one variant in martensite at the expense of another [32, 34]. Microscopically, untwinning can be characterized by the disappearance of existing twin bands. Deformed magnesium alloy, which has a high density of twins, can undergo untwinning [35, 36]. Twins can disappear or become narrower under reverse loading or unloading, and can reappear under reloading. The crystal deformation process of untwinning is similar to twinning, although nucleation is not required or occurs more readily. Therefore, untwinning can also result in an inflected and concave strain hardening behavior [6]. The strain caused by twinning in compression can be reversed by untwinning in subsequent tension. Untwinning is a contraction of twinned regions, a process that does not require nucleation [20]. The stress required for untwinning is less than that for twinning nucleation, but greater than that for twinning growth [20]. In cyclic loading, twinning and untwinning appear alternately [28].

Understanding the large plastic behavior of sheet alloys along non-proportional strain paths is an important requirement for sheet metal forming application. The Bauschinger Effect [37], which refers to a lower yield stress developed with strain upon reverse loading following an initial strain/stress path, has been related to various mechanisms: residual stresses generated in forward deformation [38], Orowan loops around strong precipitates [39, 40], internal stress from dislocation interactions [41], and dislocation pileups at grain boundaries [42]. Regardless of mechanism, the macroscopic interpretation involves development of a “back stress” during loading that assists reverse loading.
The foregoing interpretations of the Bauschinger Effect rely on material hardening characteristics of dislocation slip. In materials that twin significantly, twinning and its interaction with slip can provide alternate Bauschinger Effect mechanisms. The twin boundaries operate as hard but deformable obstacles, with dislocation pileups developing at the twin boundaries which generate a long-range back stress field and large Bauschinger Effect [43]. In magnesium alloys at room temperature, where deformation in some stress states depends intimately on twinning, large asymmetry of cyclic deformation has been noted [44], but not studied in detail, particularly at large strain. Noster et al. [45] reported a pronounced Bauschinger Effect for small strain reversal. Consistent with the role of twins as obstacles for slip, pre-compression followed by tension was reported to produce a larger Bauschinger Effect than the opposite path, pre-tension followed by compression.

In contrast to extensive studies of magnesium bulk alloys, there is little data for large-strain in-plane compression of magnesium sheet alloys. Such measurements are limited by buckling. The large-strain cyclic deformation behavior of magnesium alloy, important to sheet metal forming behavior and simulation, has not been reported. Knowledge of the Bauschinger effect, and more generally the evolution of yield and hardening under non-propotional loading paths is required to enable development of novel forming methods to take advantage of the unusual plastic properties of wrought Mg sheet.

In the current work, the constitutive behavior of O-tempered AZ31B Mg sheet alloy was investigated at room temperature. Monotonic and continuous reverse-path tests were conducted in uniaxial tension/compression and simple shear. Novel test designs
were utilized to obtain large-strain deformation [46-51]. In order to understand the origins of the mechanical response, optical metallography, texture analysis and acoustic emission measurements were conducted in parallel with the mechanical tests.
CHAPTER 2

EXPERIMENTAL PROCEDURES

O-temper Mg AZ31B sheet alloy was mechanically tested at room temperature using novel tension/compression [46, 47] and simple shear [48-51] tests. The deformation mechanism of the mechanical response was revealed by standard optical metallography, texture measurement, and acoustic emission. The material and tests are introduced briefly, with reference to more complete information appearing in the literature.

2.1 Materials

AZ31B magnesium alloy sheet, which is a commercial alloy produced by Mark Metal Inc. [52], has chemical compositions and elastic properties as listed in Table 1. The additional aluminum and zinc act as solute atoms to strengthen magnesium alloy [53]

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<th>Chemical Composition (wt%) [52]</th>
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<td>Mg</td>
<td>Al</td>
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<tr>
<td>95.8</td>
<td>3.0</td>
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Table 1. Chemical compositions and general mechanical properties of AZ31B Mg alloy
Three thicknesses and processing paths were tested. Sheet with 6.4 mm thickness was received in the O-condition. Sheet with 3.2 mm thickness was received in the H24 condition. Sheet with 1.0 mm thickness was processed by a high density infrared [54] heating and rolling technique starting from O-temper 6.4 mm sheet at Oak Ridge National Laboratory. To eliminate the residual stresses from cutting and processing and to standardize the microstructures, all the materials were annealed at 345 °C for 2 hours to obtain the O-temper [53, 55].

2.2 Uniaxial Tensile Tests

Standard ASTM tensile specimens [56] were machined parallel to the rolling direction (RD), transverse direction (TD), and 45° to the rolling directions (45°), with a rectangular cross-section of 13mm width by 1, 3.2, or 6.4mm thickness and a gage length of 50 mm. Testing was carried out using an Instron 1322 universal testing machine with load cell and laser extensometer [46].

Figure 1 shows typical uniaxial tensile test results. As shown in Figure 1a, in the normal tensile testing strain rate range of $10^{-4}$/sec to $10^{-3}$/sec, there is little effect of rate. The remainder of tests in the current work was carried out at a nominal initial strain rate of $1\times10^{-3}$/sec at a constant crosshead speed. Figure 1b shows that the sheets of three thicknesses have nearly identical mechanical properties. Note the abrupt fracture behavior, quite different from ductile failure by plastic strain localization for low-strength sheet alloys with cubic crystal structures.
Figure 1. Baseline tensile hardening

a) Effect of strain rate; b) Comparison of three material thicknesses
The Lankford coefficient (r-value) was measured by interrupting tensile tests at known axial extension and measuring the corresponding specimen width. Because the thickness change was small and thus involved large relative measurement uncertainty, the axial strain and width strain were used with assumed volume constancy to infer the thickness strain, and thus the r-value:

\[ r = \frac{\varepsilon_w^p}{\varepsilon_l^p} = -\frac{\varepsilon_w^p}{\varepsilon_w^p + \varepsilon_l^p} \]  \hspace{1cm} (1)

where \( p \) refers to plastic strain, and \( l, w \) and \( t \) refers to length, width and thickness, respectively. The plastic strains were obtained from the measured strains by subtracting elastic strains, as follows:

\[ \varepsilon_l^p = \varepsilon_l - \frac{\sigma_l}{E} \]  \hspace{1cm} (2)

\[ \varepsilon_w^p = \varepsilon_w + \mu \frac{\sigma_l}{E} \]  \hspace{1cm} (3)

where \( E \) is Young's modulus (Table 1) and \( \mu \) is the shear modulus (Table 1), and \( \sigma_l \) is the longitudinal stress.

2.3 Compression and Cyclic Tests

Large compressive strains, which are critical for many sheet metal forming operations, cannot be obtained easily because of the limitation of buckling. A test method developed by Boger et al. [46], which relies on through-thickness sheet stabilization to avoid buckling, was used to extend the attainable strain range in compression to approximately -0.08. A schematic of the novel tension/compression test [46] and the sample dimensions are shown in Figure 2. Two flat steel plates and a hydraulic cylinder
system were used to provide side force to support the exaggerated dogbone specimen. Side forces of 12 kN were used to stabilize the sheet sample. A laser extensometer [46] was used to measure specimen extension directly.

The cyclic loading test was carried out using 3.2 mm and 6.4 mm thick samples. All mechanical tests were carried out under displacement control, except the multiple-cycle tests which were performed under strain control to improve the precision of the strain limits for each cycle. The experiments were repeated to establish reproducibility; the standard deviation for compression was found to be ±6 MPa.

\[
G = 36.8 \text{ mm}, \quad W = 15.2 \text{ mm} \\
B = 50.8 \text{ mm}, \quad L > 3 \text{ mm}
\]

Figure 2. Geometry of T/C test and sample dimensions [46]
The stabilizing side force requires correction for two effects in order to obtain uniaxial stress-strain curves comparable to standard tensile testing: 1) friction between the sample surface and supporting plates, which reduces the effective axial loading force, and 2) biaxial stress state. Analytical schemes for making corrections for each of these have been presented by Balakrishnan [47] and Boger et al. [46], as discussed below.

Friction is minimized mechanically by sandwiching 0.35mm Teflon® sheet between the specimen and side plates, and by applying the side force through freely turning rollers. The remaining friction force, \( F_f \), can be estimated using a Coulomb friction law [57]:

\[
F_f = 2 \mu F_{side}
\]  

(4)

where \( F_{side} \) is the side supporting force and \( \mu \) is the up-to-now unknown friction coefficient. The friction force must be subtracted from the measured force to obtain the effective force.

The correction for biaxiality is simpler and much smaller than that for friction, Figure 3. The side force is converted to a stress using the area of the specimen in contact with the side plates, and a Von Mises yield criterion (or other choice) is used to find the equivalent tensile stress for the biaxial state. Previous work showed that the choice of yield function has a minimal effect on this correction [46].

The unknown friction coefficient, \( \mu \), Equation 4, is fit for each test utilizing side support as follows. Two identical test specimens are tested at the same rate using a) standard tensile testing techniques (i.e. with side support), and b) tensile testing with side support at given side loading. The side-loaded tensile curve is first corrected for biaxial tension, then is compared with the standard tensile curve. These two curves are brought
into registry by adjusting \( \mu \) until they agree. This value of \( \mu \) is then used to correct compression or cyclic tests of similar specimens tested at the same rate using the same side force. Details of the correction procedures have been presented elsewhere [46].

![Graph showing stress-strain relationship](image)

Figure 3. Raw and corrected tensile hardening from uniaxial tensile test and test with stabilizing side plate force

2.4 Simple Shear Test

A recently-devised simple shear test [49-51], shown schematically in Figure 4, provides an alternate stress state for testing and avoids the strain limitation caused by tensile necking. A set of specially-designed fixtures assures that there is little axial extension of the specimen and that the clamped edges remain parallel and at nominally fixed distance apart, thus producing simple shear. Strain is computed from the output of a
A special mechanical extensometer that records the relative axial displacement of the edges of the deformed region on the specimen. The shear stress $\sigma_{12}$ and engineering (not tensor) shear strain $\gamma_{12}$ are calculated as following:

$$\sigma_{12} = \frac{F}{L \cdot t} \quad \gamma_{12} = \frac{u}{b}$$  \hspace{1cm} (5)

![Figure 4. Geometry and dimensions of the simple shear test](image)

2.5 Metallography and Texture Measurement

Samples used for optical metallography were cut from the gauge regions where the material was deformed uniformly. Specimens were mounted and successively ground and polished, finishing with $1\mu m$ diamond paste in ethanol. Acetic picral solution (4.2 g picric acid, 10 ml acetic acid, 70 ml ethanol and 10 ml water) [14] was then used to etch
for 5-10s. Care was taken to etch and examine specimens immediately after polishing to avoid oxidation effects. Polishing in some case produced surface porosity, consistent with reports in the literature [58].

Crystallographic texture measurement was performed by X-ray reflection. The surface the samples under various deformation conditions were examined using PAD-V diffractometer. Pole figures for (0002), (10\bar{1}0) and (10\bar{1}1) were calculated and generated using popLA software [59].

2.6 Acoustic Emission

Acoustic emission (AE) technology is a non-destructive testing method used widely in industry for detecting leakage in vessel and pipe systems. The internal structure evolution, such as crack growth, dislocation slip and twinning, can also be monitored during plastic deformation [60]. In recent years, deformation of magnesium alloys has been studied using AE [61-65]. Two types of AE signal are shown in Figure 5, burst AE and continuous AE. The start and end of a burst signal can be distinguished from background noise, in contrast to a continuous signal [66]. A burst-type AE signal with variable [67] amplitude is identified with twinning [68, 69], in contrast to the continuous [69], much less intense [70] AE signal corresponding to dislocation slip. The AE signal is dominated by the formation of twins [71], i.e. the nucleation of twins rather than their growth [67].
In-situ AE measurement was performed during selected mechanical tests using a Vallen-Systeme AMSY4 AE workstation [72] and a Deci SE150-M acoustic sensor with a Vallen-Systeme AEP3 preamplifier [72] mounted outside of the gage length of each specimen. The acoustic threshold was set immediately prior to each test at a level just above ambient noise. Other system parameters include: bandwidth of 100-450 kHz, 40 db pre-amplifier gain, rearm time of 4 ms and duration discrimination time of 400 μs.

AE signals may be analyzed in a variety of ways. Cumulative AE count [67], AE time count rate [68, 69] and strain count rate were used to analyze the mechanism of the mechanical response. Cumulative AE count is the sum of the counts of all AE events. It is associated with the extent of twin formation in the material [68]. AE time count rate or strain count rate are the time or strain derivative of the AE cumulative count, and is similarly associated with the rate of twin nucleation.
CHAPTER 3

RESULTS

The deformation mechanisms of Mg have been reported as outlined in the Introduction, as has the mechanical response of textured Mg sheet under either monotonic or small-strain cyclic deformation. The testing reported here seeks to clarify the evolution of hardening under large-strain non-proportional deformation paths.

3.1. Monotonic Tension and Compression Tests

Large asymmetry of yield and hardening evolution, Figure 6, was observed in monotonic tensile and compressive tests, consistent with reports in the literature [23, 27, 29, 31, 45]. With the exception of the larger compressive strains attainable for the thicker material, there is little difference in the response of material of the two thicknesses. While the tensile curves exhibit the standard concave-down appearance (i.e. steadily decreasing hardening rate), the compressive curves exhibit an unusual concave-up aspect. An inflection is just visible at the limit of attainable strain for the 6.4 mm thick specimens, at approximately 0.08 strain. The 0.2% offset tensile yield stresses for RD, 45° and TD are 164 MPa, 180 MPa and 192 MPa, respectively. For comparison with data to be presented later, the 0.4% offset tensile yield stresses are also of interest: 167MPa (RD), 182MPa (45°), 191MPa (TD), well within experimental scatter (± 6MPa) of the 0.2% offset ones.
The unusual compressive behavior is related to twinning [23, 27, 29, 31], as will be shown later. The 0.2% offset compressive yield stresses in compression (3.2mm material) are closely grouped: 104 MPa, 105 MPa and 110 MPa for RD, 45° and TD, respectively, with corresponding 0.4% offset compressive yield stresses of 106 MPa, 107 MPa and 112 MPa for RD, 45° and TD, respectively. Thus, tensile yielding exhibits significant anisotropy while compressive yielding does not. Comparison of Figures 5a with 5b reveals only insignificant differences between the mechanical response of the AZ31B material with different thicknesses. In the remainder of this paper, results will be shown for only one thickness unless a significant difference was noted.
Figure 6. Uniaxial hardening in tension and compression in three directions:

a) 3.2 mm thick material, b) 6.4 mm thick material
In-plane strain anisotropy and its evolution are also apparent, Figure 7. The r-values increase with plastic strain in three directions, with decreasing rates, throughout the attainable strain range.

Figure 7. Evolution of r-value with tensile strain
3.2. Cyclic Tension/Compression Tests

Large-strain compression-tension-compression (C-T-C), Figure 8, and tension-compression-tension (T-C-T) tests, Figure 9, were conducted for 3.2 mm thickness sheet in three directions. Figures 6, 8 and 9 illustrate the similarity of stress evolution among the three tested directions, although there are differences in yield stress related to textural anisotropy as discussed later. Subsequent plots will focus on a single direction unless significant difference was noted.

Figures 8b and 9b are replotted versions of Figure 8a and 9a, useful for revealing the evolution of hardening. In these alternate plots, the absolute stress is shown, that is, compressive stresses are shown with reversed sign. Instead of standard true geometrical strain, the absolute value of each strain increment is summed to produce an accumulated absolute strain, $\varepsilon_{abs}$:

$$\varepsilon_{abs} = \sum_{i=1}^{n} |\varepsilon_i|$$ \hspace{1cm} (6)

where $\varepsilon_i$ refers to the strain accumulated in each of the n proportional-path segment of the test. Plots in the form of Figures 8b and 9b may readily be compared to those expected for those obeying standard material models, i.e. those with isotropic hardening and symmetric yield (tension identical to compression). Under those conditions, plots such as Figure 8b and 9b collapse to a single curve, like that shown as “monotonic tension”, with elastic unloading and loading excursions whenever the stress state is reversed. The very dramatic departure of AZ31B from this “standard”, or idealized, behavior is apparent.
The C-T-C tests reveal an inflected tension portion following the compressive prestrain, an effect notably absent in the monotonic tensile tests. Similarly, the T-C-T tests show no inflection upon initial tensile deformation, but the inflection appears during compression and subsequent tension. In all cases, the inflected hardening curve is accompanied by yield stresses considerably less than those for the "normal" hardening behavior.
Figure 8. Single-cycle strain hardening for C-T-C

a) True stress vs true strain

b) Absolute true stress vs accumulated absolute strain
Figure 9. Single-cycle strain hardening for T-C-T

a) True stress vs true strain

b) Absolute true stress vs accumulated absolute strain
Multiple-cycle uniaxial tests were conducted using strain control for better cycle-to-cycle reproducibility. Strain ranges of 0.04 and 0.07 were set. All tests started with tension, and cycles were numbered starting from the first point of maximum compressive strain. (The initial tension and compression are labeled Cycle 0).

Figure 10 shows selected cycles from multiple-cycle tests. Both small and large strain amplitudes show a tendency to approach a saturated, static hysteresis loop, although in the case of large strain amplitude, fracture initiated after 10 cycles before complete saturation was obtained. No fracture occurs even after 20 cycles at the small strain amplitude.
Figure 10. Multiple cycle tests

a) Small strain, 0.04; b) Large strain, 0.07
The saturation behavior of the cyclic tests may be quantified by the evolution of the maximum stress \( (\sigma_{\text{max}}, \text{positive in tension}) \), the minimum stress \( (\sigma_{\text{min}}, \text{negative in compression}) \), and the stress amplitude \( ((\sigma_{\text{max}}-\sigma_{\text{min}})/2) \), Figure 11. The stress amplitude for both strain ranges saturates within 3 to 5 cycles. For the small-strain-range test, the maximum and minimum stresses saturate in a similar period. For the large-strain-range test, the minimum stress continuously increases while the maximum stress first increases, then decreases such that the average stress decreases continuously at large strains. This behavior may be related to damage evolution which reduces the tensile strength while having less effect on the compressive one. The fact that the large-strain amplitude specimens fracture in tension at the 10th cycle tends to agree with this interpretation.
Figure 11. Relationship between the stress and cycle number

a) Small strain, 0.04; b) Large strain, 0.07
3.3 Simple Shear Test

In-plane simple shear is of interest for testing of basal-textured AZ31B Mg sheet because the in-plane principal strains nominally are opposite and equal, thus producing small (ideally zero) thickness strains. However, both measurement and simulation in Mg sheet show that small through-thickness extensile strains, 0.02, occur during the initial deformation in simple shear. Figure 12 shows a typical result for simple shear in the RD. TD and 45° have similar flow curves as RD. Yield occurs at an equivalent tensile stress of $\bar{\sigma} = \sqrt{3} \sigma_{12} = 175$ MPa (0.2% offset). An equivalent extensional strain, $\bar{e} = \frac{\gamma_{12}}{\sqrt{3}} = 0.13$, less than the tension limit, is attained before fracture. Up to a strain of approximately 0.05, strain hardening is minimal, with more rapid hardening thereafter. At a monotonic equivalent tensile strain of $\gamma_{12} = 0.13$ the equivalent flow stress is 302 MPa. Upon reversal of shear, a significant Bauschinger Effect appears.
Figure 12. Cyclic simple shear test results

a) True stress-strain; b) Effective stress-strain
3.4 Microstructure Evolution

Figure 13 summarizes the microstructure evolution for several strain states. The initial microstructure of AZ31B Mg alloy after annealing is free of twins [14]. At a uniaxial tensile strain of 0.075, a small number of twins are arranged in needle-like narrow bands in some grains, most of the grains being twin free. The area fraction of twins is estimated from micrographs, and the uncertainty of area measurement is estimated to be ±5% at larger twin fractions. The uncertainty arises when twins intersect and it becomes difficult to distinguish twinned from untwined regions. At a tensile strain of 0.075, an area fraction of 4% of the material is twinned, which is consistent with recent report [18]. When the material is under compressive stress, more twins in wider twin bands are observed. The area twin fraction increases from 21% (ε = -0.023), to 34% (ε = -0.046), to 43% (ε = -0.072). Upon subsequent reverse tension to 0.04 strain, the wide twin bands formed during compression disappear. Needle-shaped twins, similar to those observed in monotonic uniaxial tension, appear in the material. The area fraction of twins drops to about 6%, slightly larger than that of monotonic tension. As shown in Figure 13, twinning also occurs in monotonic simple shear, although at a slower rate than compression. Wide-band twins, similar in morphology to the compressive twins, can be readily identified.
Figure 13. Evolution of microstructure under various loading paths
3.5 Texture Evolution

Figure 14 illustrates the evolution of crystallographical texture during a C-T-C test. The scale indicates the diffraction intensity normalized to the randomly distributed intensity, i.e. a value of 1.0 indicates the average intensity. The initial texture of the annealed magnesium AZ31B sheet alloy is shown in Figure 14a. The (0002) basal pole figure exhibits a strong basal texture, with the majority of c-axes aligned in the sheet normal direction. There is some spreading of the basal pole along the rolling direction. As reported in the literature, tensile straining does not alter the basal texture significantly [13, 14]. After in-plane compression along rolling direction, Figure 14b, the basal pole moves to the rolling direction (i.e. the compression axis). Figure 14c is the texture after the subsequent reverse tension along the rolling direction; it has a strong basal texture similar to the initial, annealed material. There is no preferred orientation for the prismatic plane \{10\bar{1}0\} or pyramidal plane \{10\bar{1}1\} throughout the deformation.
Figure 14. Crystallographical texture in various deformation stages. The scale at right indicates the relative diffraction intensity (1.0 = random)
3.6 In-situ Acoustic Emission

Figures 15a and 15b show the cumulative acoustic emission count and time count rate during a T-C test. During the initial tension, the count rate is small, less than $10^4$/s. During subsequent compressive yield, the count rate increases dramatically, and two strain ranges with intense count rate are exhibited. The AE results are consistent with the metallographic sections, pole figures and literature. Very limited twinning takes place during in-plane tension starting from the initial texture. Upon in-plane compression, however, a rapid burst of twin creation occurs over a strain range of 0 to -0.02, followed by lower rate from -0.02 to -0.05, followed by another burst from -0.05 to -0.07 (the largest compressive strain attained in this test). It appears that there are two activation stresses for twinning in this alloy, either related to two twin mechanisms or two sets of twin orientations. Rapid strain hardening accompanies the accumulated twin production, consistent with the role of twins as slip obstacles [17].

Figures 16a and 16b present AE and stress-strain results for a C-T test. Similar to the foregoing T-C test, initial compression induces two bursts of high twin rate separated by a region of low twin rate. Again, the accumulated twin production is accompanied by high strain hardening. The reverse tension hardening occurs at a much lower stress than initial compression curve. This is consistent with the metallographic results showing the removal of twins throughout this region, or “untwining” presumably without the large nucleation stress needed for twinning. However, the AE data shows only minimal activity in this region, similar to that for initial tension. Apparently the untwinning operation produces much lower acoustic signals than the twinning operation, which would be consistent with a lower activation stress.
Figure 15. Acoustic emission during T-C test

(a) Cumulative AE count; (b) Time AE count rate
Figure 16. Acoustic emission during C-T test

(a) Cumulative AE count; (b) Time AE count rate

RD, t = 3.2 mm
CHAPTER 4

DISCUSSION

As shown by the foregoing experiments, significant asymmetric yield and hardening behavior is exhibited in magnesium AZ31B sheet alloy at room temperature. An inflected and concave strain hardening flow curve was observed in both compression and reverse tension. A large Bauschinger effect occurs during cyclic testing or simple shear testing. As has been widely reported in the literature, the origin of these effects lies in the activation of twinning.

As discussed in the Introduction section, the easily-activated \{10\bar{1}2\} twin found in Mg alloys is a ‘tension’ twin, i.e. one which can be activated by extension parallel to the c-axis in a HCP crystal structure \[1, 4, 16, 17\], or equivalently, by in-plane compression \[19\]. The initial crystallographical texture of AZ31B magnesium sheet alloy exhibits a strong basal texture, where the majority of c-axes are aligned in the sheet normal direction with some spreading in the rolling direction, the prismatic planes \{10\bar{1}0\} and pyramidal planes \{10\bar{1}1\} are randomly distribute in the sheet plane initially. An in-plane compressive stress results in twinning, while in-plane tension with a contraction along c-axes does not. Therefore, optical microscopy shows only a small number of local, narrow band twins after in-plane uniaxial tension. These twins are produced under tension stress state because the c-axis orientation of some grains maybe aligned close to

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the loading direction, or because of stress state inhomogeneities introduced by inter-grain interactions [18]. It should be noted that a spreading of basal pole along the rolling direction allows easier activation of basal slip by RD tension. The strong basal texture in TD requires activation of non-basal slip, which increases the yield stress. The higher incidence of non-basal $<a>$ slip relative to basal $<a>$ slip during TD tension is responsible for the higher r-value of TD than that of RD and 45° [13]. Under tension, there is little thickness strain, but the activity of non-basal $<a>$ slip produces large width strain, thus large r-value [18]. During tension, non-basal slip is progressively activated as stress increases, thus, r-value increases with tensile strain.

After in-plane compression, significant wide-band twins appear and the basal pole of the texture shifts rapidly to the rolling direction. This is characteristic of twinning, which rotates the c-axes toward the compressive loading direction. An area fraction of 43% twinned area is observed at a compressive strain of 0.072.

Subsequent in-plane tension produces an extension along the c-axes of these newly-aligned crystals, activating an untwining process. Untwining rotates the c-axes back to the sheet normal direction, thus the strong basal texture again appears in the texture. The microstructure shows that the originally formed wide-band twins disappear, instead, only a few narrow-band twins remain, similar to the uniaxial tensile condition. Untwining removes the original twins and produces the reverse of the previous strain caused by twinning. The untwining flow curve is very similar in appearance to the original compression curve, i.e. with a sigmoidal aspect having an inflection.

A working model of the deformation mechanisms under reverse loading may be constructed consistent with the experimental results presented here and with those
appearing in the literature [23, 26]. The conceptual result is shown schematically in Figure 17 for three strain paths: monotonic tension, monotonic compression, and reverse tension following compression. The first and last of these are experimental ones, with the monotonic compression curve being a composite curve, derived as outlined below.

As shown on Figure 17, the initial plastic modulus at yield in tension (after a brief yield point phenomenon presumably related to twinning) is approximately 2000MPa. This slope may be conveniently used to identify two other characteristic points on the stress-strain curves. When associated with concave-up curvature, it represents a midpoint between twin-dominated and slip-dominated flows. When associated with concave-down curvature, it signals the end of the twin-facilitated flow and may be used to characterize slip resistance, or “slip-dominated flow stress”.

The composite monotonic compression curve consists of a first experimental portion and second portion translated from an experimental reverse tension curve (which extends the attainable strain range). The second curve is translated in stress and strain until the points on the two curves having 2000MPa slopes (and concave-up curvature) coincide.
Figure 17. Schematic of strain hardening. Note that the monotonic compression curve is a composite one, constructed as described in the text.

The working model may be described as follows. Monotonic tension exhibits the normal strain-hardening aspect encountered in non-twinning materials, although some small amount of twinning is present, as shown by metallography and AE, presumably as required for arbitrary deformation with activation of only 4 independent slip systems. Because twinning is difficult during in-plane tension, the constraints of the limited number of independent slip systems cause a high yield and flow stress, with hardening consistent with a predominant slip deformation.

Monotonic compression exhibits a lower yield stress and initial hardening rate than tension because twinning is facilitated and the Von Mises – required five
independent slip/twinning systems are readily activated. However, after a strain of approximately 0.05, the hardening rate increases rapidly as the capacity for twinning is exhausted locally within the polycrystal. The reverse tension flow curve exhibits lower yield stress than monotonic compression, consistent with reported low twin propagation stresses relative to twin nucleation stresses [19, 20]. Untwining does not involve nucleation of twins, thus, the stress of untwining is less than that of twinning [20].

The simple shear test exhibits a similar, but less dramatic, flow curve. Initial yield is followed by a low hardening rate up to a strain of 0.05, at which time the hardening rate increases, giving a concave-up aspect. This appearance is consistent with twinning and concurrent slip. However, twinning cannot occur without an accompanying through-thickness extensile strain in the initially basal-texture sheet. After a simple shear strain of 0.1, the thickness strain was carefully measured and found to be 0.02 with a measurement uncertainty of ±0.007, consistent with the presence of twinning. The sheet area of the shear specimen is not constant during the deformation, as would be expected for an isotropic material or for a rigorously controlled shear test. It appears that the compliance of the fixture or laterally slipping in the grips allows some material contraction normal to the applied displacement. The in-place compression under a principal compressive stress is less than the more difficult in-plane extension under the principal tensile stress, thus favoring a net contraction in the plane and net extension through the thickness.

As discussed in the Introduction and Experimental Procedure sections, burst-type AE counts are associated closely with twinning events. Figure 18 shows the experimentally measured AE cumulative counts and strain count rate from a compression test and the corresponding areal twinning fraction obtained by metallographical
examination. After a compressive strain of 0.05, a second stage of high count rate occurs. However, as shown in Figure 18b, the areal fraction of twinning increases at a steadily decreasing rate, seemingly in disagreement with the AE data. In fact, this discrepancy confirms reports in the literature [67-69, 71, 73] that the AE counts are related to twin nucleation and initiation, rather than to growth and thickening. Growth of twins has been reported to contribute to AE signals one-to-two orders of magnitude less than nucleation [67]. It is also for this reason that the AE signal for untwining, which does not require nucleation, is quite small, Figure 16.

With this interpretation of AE signals in mind, the strain-count-rate plot of Figure 18a can be understood. The first peak is associated with nucleation of twins of the principal type in Mg, \{10\bar{1}2\} twin. As straining proceeds, fewer new twins are nucleated because of unfavorable grain orientations and the already activated ones continue to grow without an accompanying large AE signal. As these favorable twins become exhausted, the stress rises and either a new type of twin begins to be nucleated (for example, \{10\bar{1}1\} and \{30\bar{3}2\} twins have been observed in large tensile strains in Mg [18]), or possibly unfavorably-oriented grains can accommodate twinning at the high stress.

The basis for the rate fraction attributable to twinning plotted in Figure 18b requires explanation. It is computed from the fit cumulative twin fraction in Figure 18b by noting that a fully twinned Mg single crystal produces an extensile strain of 6.4% [15]. However, for a basal textured sheet with random in-plane orientation of the <a> axes, the equivalent extensile strain is 5.9%. Assuming that the areal twin fraction is proportional to the strain from twinning, the twinning strain can be computed:

$$\varepsilon_{tw} = F_{\text{twin}} \cdot 0.059$$

(7)
Similarly, the slope of the twin fraction curve gives the twin fraction rate, i.e. the increase of twin fraction per unit strain, $\dot{F}_{\text{twin}}$:

$$
\dot{F}_{\text{twin}} = \frac{dF_{\text{twin}}}{d\varepsilon_{\text{total}}}
$$

(8)

and the fraction strain rate attributable to twinning is:

$$
\dot{\varepsilon}_{\text{twin}} = \frac{d\varepsilon_{\text{twin}}}{d\varepsilon_{\text{total}}} = \dot{F}_{\text{twin}} \cdot 0.059
$$

(9)

Since the total strain rate is contributed by slip strain rate and twinning strain rate, thus

$$
\dot{\varepsilon}_{\text{total}} = \dot{\varepsilon}_{\text{twin}} + \dot{\varepsilon}_{\text{slip}}
$$

(10)
Figure 18. Evolution of AE count and areal twin fraction with compressive strain

a) Cumulative count and strain count rate

b) Fraction strain rate attributed to twinning
Calculations such as these, which are clearly approximate, complete the basic working model. During compression, the strain mechanisms are dominated by slip while the flow stress is dominated by the stress needed to activate twinning in order to complete the Von Mises required 5 independent deformation mechanisms. The initial twinning activation stress is high, with twinning growth occurring more readily (and with little AE signal). However, twinning of any type is both polar and of limited extent. In addition, twins function as obstacles to dislocation motion. For both these reasons, the flow stress inevitable increases as twinning proceeds, at first slowly during the growth stage, before exhaustion is approached, and before a dense set of twin obstacles is established. The stress then rises rapidly as exhaustion approaches and density increases, thus allowing activation of non-preferred twin types and orientations. Eventually, the flow stress becomes so high that local cracks are created and propagated by incompatibility caused by removing twinning from the mix of independent deformation mechanisms.

The cumulative count results in T-C and C-T tests were smoothed by polynomial fitting and differentiated in order to obtain strain count rate curves, Figure 19. The count rates in compression, Figure 19a, are much larger than those in tension, Figure 19b, which indicates the nucleation of twins contributes most to the AE signal, compared with twin growth and dislocation slip. Two peaks, which are related two different twinning modes or twinning orientations as discussed before, can be detected in the compression parts of either T-C or C-T. However, small AE signals in the tension part of T-C and C-T, Figure 19b, are created by the combined effect from dislocation slip, and some limited twinning or untwinning.
Figure 19. Smoothed AE Strain count rates in different deformation stage

a) Compression in C-T and T-C

b) Tension in C-T and T-C
A working model has been established which accounts for the principal characteristics of monotonic and reverse deformation paths in terms of slip, twinning and untwinning. Continuous reverse tests can also reveal secondary characteristics of interest for formulating an accurate constitutive model.

T-C-T tests were carried out with initial tensile prestrains of various values, followed by fixed compressive and tensile strain ranges of \( \sim 0.07 \), Figure 20. Raw plots like Figure 20a are difficult to interpret directly, but may be summarized in terms of yield stresses following the transitions, Figure 20b and Figure 20c. While it is traditional to use a yield stress defined by a 0.2% offset, this value is insufficient to capture the essence of reverse yielding. Presumably because of large micro-plastic backflow of dislocations piled up at twin obstacle [17, 43], low effective unloading moduli and gradually curve elastic-plastic transitions are observed upon the stress transitions. As shown in Figure 20b, the choice among slightly larger offset yield stresses, 0.4%-1.0% makes little difference, but the 0.2% offset yield stress still lies in the rapidly-changing portion of the stress-strain curve.

The behavior illustrated in Figure 20a and 20b is typical of all of the transitions observed. Therefore, 0.4% offset yield stresses are used for comparison hereafter. In particular, Figure 20 shows that tensile prestrain more than 0.03 increases the subsequent compressive yield stress. The tentative conclusion is that the multiplication of dislocations (and creation of a small number of local twins) in tension creates obstacles that remain effective upon compressive loading, presumably by raising the activation stress for twinning, which according the working model, determines the flow stress.
Figure 20c shows the subsequent compressive yield stress and the 2nd tensile yield stress (i.e. after variable tensile prestrain, the -0.07 compressive strain) as a function of the flow stress at the end of the first tension path. While the compressive yield stress (dominated by twinning) increases markedly (30 MPa) corresponding to the pre-stress increase of 80 MPa, the second reverse tension stress (dominated by untwinnning) occurs at less than half the compression value, and does not increase with pre-stress. These results indicate that dislocation multiplication affects the twin nucleation stress, but has little or no effect on the untwinnning stress, which does not require nucleation. The untwinnning stress is approximately half of the twinning stress.
Figure 20. Tension-Compression-Tension (T-C-T) results for various initial tensile prestrain: a) Raw stress-strain data; b) Variation of reverse compressive yield stress based on several offset yield criteria; c) Variation of reverse compressive yield stress (0.4% offset) and second tensile yield stress (0.4% offset) with the final flow stress at the end of the tensile prestrain.
Figure 21 analyzes C-T-C tests with various levels of initial compressive strain followed by fixed tensile strain and final compressive strain. Figure 21a is the raw data and Figure 21b relates reverse hardening parameters to initial compressive prestress (similar to Figure 20c for T-C-T tests). Consistent with the previous T-C-T results, the tension yield (untwining controlled after the initial compression-produced twins) is unaffected by prestrain greater than 0.03 and occurs at a value of approximately 30-50 MPa. The slip-dominated flow point (where $E_{\text{plastic}} = 2000$MPa) in tension increases dramatically (120MPa) with increasing compressive prestress (100 MPa), thus implying that even twinning followed by untwining (and of course the even large accumulated slip) leaves behind a high density accumulated slip obstacles. The final compressive yield, in spite of the wide variation of flow stress immediately prior to the reversal, is almost unaffected by the preceding cycle parameters, as is the subsequent hardening. It should be noted that the estimated slip activity preceding the final compression is nearly the same in all cases, consistent with the constant final compression yield stress observed.
Figure 21. Compression-Tension-Compression (C-T-C) results for various initial compressive prestrains: a) Raw stress-strain data; b) Variation of characteristic yield stresses (0.4% offset) and slip dominated flow points ($E_{\text{plastic}} = 2000\text{MPa}$) with the final flow stress at the end of the compressive prestrain.
In order to isolate the effect of untwining better, C-T-C tests were conducted with similar compressive prestrains of 0.07, followed by tension to various strains, and then final compressive loading, Figure 22a. The tensile part of the cycle subjects the material to more untwining and slip. As shown in Figure 22b, the second compressive yield stress increases (90 MPa) with increasing tensile flow stress just prior to the reversal (180 MPa). But between the reverse tensile flow stress of 160 MPa and 150 MPa, the second compressive yield stress remains constant. Comparison with Figure 20c shows that the compressive yield following tension increases with pre-tensile stress. In one case the prestrain involves very limited twinning (~ 4%) whereas in the other there is nearly complete twinning and variable untwining. The similarity of the results suggests that the compressive yield stress, determined mainly by twinning, is related to the accumulated slip activity, in the form of dislocation density.
Figure 22. Compression-Tension-Compression (C-T-C) results for various reverse tensile strains: a) Raw stress-strain data; b) Variation of 2nd compressive yield stress (0.4% offset) with the final flow stress at the end of the tensile prestrain
CHAPTER 5

CONCLUSIONS

Several mechanical and microstructural techniques have been used to reveal the nature and origin of continuous and reverse strain hardening of AZ31B magnesium sheet alloy. The techniques include a large-strain, continuous tension/compression test, a simple shear test, acoustic emission technology, optical metallography, and X-ray texture measurement.

The following conclusions were reached:

1. Twinning and untwining respectively produce remarkably similar compressive and tension after compression hardening curves, although untwining usually occurs at lower stress.

2. A sigmoidal, inflected flow curve is associated with twin-dominated deformation. The initial low hardening rate is associated with the easy growth of twins and the rapid hardening portion is associated with exhaustion of twinning and twins operation as obstacles.
3. The plastic strain in all states is dominated by slip, although the flow stress is related to mechanisms specific to each case: twinning in initial compression, non-basal slip in initial tension, and untwining in tension after compression.

4. The nucleation and propagation stress for twinning increases with accumulated slip activity.

5. AE signals are associated with twin nucleation events, not propagation. Therefore, they cannot be related quantitatively to strains produced by twinning or twin fractions.

6. Untwining requires little or no nucleation stress, and, correspondingly, generates minimal AE signals.

7. AE shows two bursts of twin nucleation during straining. The second appears to be associated with secondary twins identified in the literature as \{10\bar{1}1\} and \{30\bar{3}2\} twins [18].

8. Under tensile test, TD has the largest r-value, while RD has the smallest r-value, consistent with the texture-predicted ease of non-basal slip [13, 14]. R-value increases with tensile straining as non-basal slip is progressively activated at higher stress.
9. Contrary to the behavior of ductile alloys with cubic crystal structures, fracture occurs in simple shear at extensile strains less than those attainable in uniaxial tension. This appears to be related to higher strain constraint of simple shear and the difficulty of HCP alloys to meet the Von Mises condition.
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