Twinning–detwinning behavior during the strain-controlled low-cycle fatigue testing of a wrought magnesium alloy, ZK60A

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Abstract

The twinning and detwinning behavior in a strongly textured magnesium alloy was investigated using in situ neutron diffraction during the cyclic deformation along the prior extrusion direction at the fully reversed total constant strain amplitude of 1.2% at room temperature. The initial preferred orientation places the $c$-axis in most grains perpendicular to the loading axis, and this favors extensive $\{10\overline{1}2\}$ $\{10\overline{1}1\}$ twinning under compressive loading. In contrast, the grains are not favorably oriented to undergo such twinning during monotonic tensile loading along the prior extrusion axis. This is the reason for the well-known tension–compression strength asymmetry of wrought magnesium alloys. The strength in compression is controlled by the stress required to activate twinning, while the strength in tension is controlled by the harder non-basal slip mechanisms. The unique orientation relationship between the parent grains and the twin grains favors detwinning during the subsequent loading reversal. In situ neutron-diffraction results indicate that such twinning and detwinning alternates with the cyclic loading, i.e. most of the twins formed during compression are removed when the load is reversed. However, a small volume fraction of residual twins gradually increases with increasing cycles, which may be an important factor in dictating the low-cycle fatigue behavior of the magnesium alloy.

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1. Introduction

The magnesium alloys have recently attracted a great deal of research interest because of their potential applications for lightweight structural components [1–5]. However, the number of applications for which magnesium alloys can be used is restricted because they always exhibit a high directional anisotropy and are hard to deform at room temperature, owing to their hexagonal crystallographic structure and limited deformation modes [1]. In this regard, mechanical twinning plays a significant role in the magnesium deformation. At room temperature, twinning may help this material to satisfy the von Mises criterion, which requires five independent deformation systems for an arbitrary homogeneous straining. Aside from the slip of dislocations with $(c+a)$ Burgers vectors, which is recognizably a very hard deformation mechanism, twinning is the only active deformation mode that can provide straining along the $c$-axis at room temperature [6].

Most common wrought magnesium alloys usually display a strong in-plane tension–compression asymmetry [7]. This phenomenon has been attributed to the mechanical twinning on the $\{10\overline{1}2\}$ planes along the $(10\overline{1}1)$ directions during compression along the prior working direction (rolling or extrusion) but not during tension along the same direction [1]. This is primarily the result of strong textures, where a significant fraction of grains orientated with their...
c-axis nearly perpendicular to the prior working direction [8], and the polar nature of twinning [9]. The monotonic compressive curve with the activation of twinning typically exhibits a sigmoidal (S-shaped) shape, where a low stress plateau is initially observed, followed by a higher hardening-evolution rate [10].

Because it is such an important deformation mode of Mg alloys, mechanical twinning has been the focus of a number of recent studies [10-18]. For magnesium alloys, with \(\text{c/a} \sim 1.624 < \sqrt{3}\), the \(\{10\overline{1}2\} \langle10\overline{1}1\rangle\) tensile twinning mode can accommodate extensions along the c-axis of the hexagonal lattice but not contractions along the same direction. This tensile twinning results in a nearly 90° (~86.3°) reorientation of the basal pole, as shown in Fig. 1a [1]. Thus, detwining may occur in the twinned areas at subsequent reversed loading with the tensile stress applied along the c-axis of the twinned materials (Fig. 1b) [12,13,19]. In other words, twins can disappear or become narrower under reversed loading or unloading, and can reappear under reloading. Brown et al. [14,15], Gharghouri et al. [11] and Oliver et al. [16] found that twinning and detwinning appear alternately in cyclic loading using in situ neutron scattering. Lou et al. [17] revealed twinning during in-plane compression and detwining upon the subsequent tension of an Mg alloy, AZ31B, sheet using metallography, acoustic emission and X-ray texture measurements. This twinning-detwinning behavior produces unusual hysteresis loops during cyclic deformation [15-17].

However, the cyclic twinning-detwinning behavior of magnesium alloys is far from fully understood. In the current research, the twinning-detwinning behavior of wrought magnesium alloy, ZK60A, was investigated at the Los Alamos National Scattering Center (LANSCE) using in situ neutron diffraction. This is the first detailed report of twinning-detwinning behavior of the alloy ZK60A, and the results serve to highlight the similarities and distinctions between two of the most common wrought magnesium alloys, AZ31B and ZK60A. Implications for the low-cycle fatigue behavior of wrought magnesium alloys in general are discussed.

2. Experimental details

The magnesium sample used in this study was machined from a commercial extruded magnesium plate with T5 temper (solution-treated at 535 °C for 2 h, quenched in hot water and aged at 185 °C for 24 h), which has a nominal composition of 6.0% Zn, 0.5% Zr (wt.%) and Mg as balance. The initial texture exhibits the combined features of the typical rolling and extrusion textures (Fig. 2). There are two major texture components, one with the basal poles perpendicular to the plate normal (ND) and the other with the basal poles parallel to the transverse direction. There is a greater angular spread in the pole density from the ND toward the extrusion direction (ED) than toward the transverse direction (TD). In this study, those grains with their initial c-axis parallel to the plate normal (Fig. 2) were extensively twinned under the compressive loadings and detwinned during the subsequent reversed loadings, and their behavior was readily captured using in situ neutron diffraction.

In order to analyze the microstructures by optical microscopy, the cylindrical specimens were cut in the ED-ND plane. Specimens were prepared using standard metallographic techniques, finishing with 1 \(\mu\)m diamond paste in methanol. Acetic picral solution (5 g picric acid, 10 ml water, 5 ml acetic acid and 90 ml ethanol) was employed to etch the specimens for 5–10 s, which revealed the grains and/or twins.

In situ neutron-diffraction measurements were conducted on the Spectrometer for MMaterials Research at Temperature and Stress (SMARTS) (Fig. 3) at the Manuel Lujan Jr. Neutron Scattering Center, LANSCE. Details of the SMARTS have been published elsewhere [10,14] and only a short description is presented here. The threaded-end sample, having a 19.05 mm gage section with a 6.35 mm diameter, was mounted in the horizontal load frame, and the prior plate normal was carefully aligned in the horizontal direction so that a large volume fraction of grains have their basal poles in the diffraction plane (horizontal), but transverse to the loading direction. The loading axis is oriented at 45° relative to the incident beam (Fig. 3) and the two detector banks situated at ±90° simultaneously record two complete diffraction patterns with diffraction vectors parallel (\(Q_l\)) and perpendicular (\(Q_\perp\)) to the applied load. The cyclic testing was performed using the Instron-customized load frame under fully reversed cyclic loading conditions at a constant total strain amplitude of 1.2% with a triangular waveform at room temperature. The macroscopic strain was measured using an extensometer attached to the sample, and the imposed cyclic frequency was 0.5 Hz. The initial loading was compressive, which results in significant extension twinning during the first quarter cycle.

\[\text{Fig. 1. Schematic of the } \{10\overline{1}2\} \langle10\overline{1}1\rangle \text{ tensile twin system in magnesium: (a) 86.3° reorientation of the twin grain relative to the parent grain [1]; (b) applied loading directions with respect to the c-axis [19]. The solid arrows indicate the applied loading directions favorable for the tensile twinning, and the open arrows indicate the applied loading directions unfavorable for the tensile twinning.}\]
Owing to the high penetration of neutrons into the bulk materials, the entire volume of the specimen can be probed by the neutron beam regardless of the specimen orientation during the measurements. Twinning takes place throughout the volume of the sample; the full penetration of the samples by neutrons provides reliable volumetric information. It is thus quite easy to detect twinning by neutron diffraction, which results in a change in the peak intensity. The transfer of the f0002g diffraction intensity between the two detector banks is a direct measure of the twinned volume fraction and allows us to monitor the evolution of the twinned microstructures. In particular, the unique orientation relationship between the parent grains and the twin grains coupled with the SMARTS configuration facilitates the study of the cyclic twinning–detwinning behavior.

3. Results and discussion

Fig. 4 clearly shows that the hysteresis loops loaded along the extrusion direction (Fig. 4a) are distinct from those loaded along the transverse direction (Fig. 4b). In Fig. 4b, the hysteresis loops are symmetric between the compression and tension cycle, which are usually the result of the dislocation slip-dominated deformation in most materials [20], while in the current Mg alloy, the cyclic deformation behavior along the transverse direction with the normal-shaped loops (Fig. 4b) are to be studied using in-situ neutron and synchrotron diffractions, and the results will be reported shortly. In contrast, the hysteresis loops in Fig. 4a are asymmetric, with a sigmoidal shape characteristic of mechanical twinning [15]. Upon the initial compressive yielding at ~150 MPa, the sample showed little hardening and the maximum compressive stress at 1.2% was ~170 MPa. This type of strain hardening plateau is typical of materials which deform by twinning, including hexagonal close-packed Mg alloys [10,13,21] and Zr alloys [22,23], as well as low-symmetry martensite shape memory alloys such as NiTi [24]. Upon reversal, a significant Bauschinger effect was observed, such that the material yielded even before the stress became positive. The reverse yielding is much more gradual than the abrupt elasto-plastic transition observed during the initial compressive loading. During the reversal, at ~0% strain the loop shows an inflection, and beyond this point, the hardening rate rapidly increases. It will be shown below that this increase in the hardening rate correlates with the exhaustion of the detwinning mechanism. Once the grains are completely detwinned, the resulting orientation is hard with respect to tensile deformation by basal slip. This demands activation of the harder non-basal dislocation slip [6] or {10T1} ⟨10T2⟩ compression twinning mechanisms [21,25]. Thus, a high maximum tensile stress of ~310 MPa was reached.

On the unloading from +1.2% strain, the deviation from linear elastic unloading occurs roughly at a stress of ~100 MPa and a strain of +0.2%. Because the yielding during the second compression cycle started early, more compressive plastic strain was induced prior to reversing a second time. Interestingly, the maximum compressive stress during the second compressive cycle was similar to the first, and this is an additional testimony to the very low hardening rate which accompanies twinning dominated deformation in Mg alloys. Furthermore, it suggests that more twinning occurs during the second cycle than the first, and the in situ neutron diffraction data discussed below corroborate this conclusion. The maximum tensile
stress during the second cycle was comparable to that of the first as well; however, the inflection of the tensile curve appeared later, at a strain of \(\sim 0.5\%\). Again, it is understood that more twinning took place during the second compressive cycle, and more strain is required to detwin the material during the second tensile cycle than during the first. The in situ diffraction data enable us to make firm conclusions regarding these points. The subsequent hysteresis loops closely follow the shape of the second cycle. Nevertheless, in the compressive quarter cycles, the material showed an ever increasing hardening rate during the later cycles (see, for example, the hysteresis loop of the cycle 200 in Fig. 4a); in addition, the maximum compressive and tensile stresses developed distinctly with increasing cycles (Fig. 5).

The variation of the stress response with the number of fatigue cycles is an important behavior of the low-cycle fatigue process. Such a cyclic-stress response depends mainly on the mechanical and/or cyclic stability of the intrinsic microstructural features, particularly as a result of the competitive processes between the hardening from the multiplication of dislocations [26] and the twin boundaries as barriers to dislocation slip [27], and the softening due to the annihilation and rearrangement of dislocations [26,28,29]. The peak compressive stress exhibits continuous cyclic hardening until the final fracture. The neutron diffraction data suggest that this is due to an increasing volume fraction of twins which form during the cycling. On the other hand, the material exhibits a tensile hardening during the initial 10 cycles and then the cyclic tensile softening dominates until a final abrupt stress drop. The overall shape of the hysteresis loops and the neutron diffraction data again provide an explanation for this more complicated observation.

As stated in the experimental section of this paper, complete diffraction spectra were collected using the time-of-flight technique in detector banks located at \(\pm 90\%\) and \(90\%\) from the incident beam. Due to the \(45\%\) orientation of the sample stress axis, these detector banks collect information from grains with their diffracting plane normal vectors oriented parallel and transverse to the stress axis, respectively. The diffraction data reveal a number of different aspects related to the structure and stress state of the material. For example, the lattice spacings derived from Bragg's law can be used to determine the state of internal lattice strain (and stress via Hooke's law) [10]. The appearance or disappearance of peaks can signify a phase transformation or, in the present case, mechanical twinning. Due to the \(\sim 90\%\) reorientation of the basal poles which occurs with \(\{10T2\}\) \((10T1)\) twinning and the initial texture, the changes in the basal \(\{0002\}\) peak intensities in the parallel and transverse detector banks can be directly related to the
degree of twinning that has occurred. This fact was exploited to advantage in previous in situ studies of twinning in Mg [11,15].

In short, the initially high basal \{0002\} peak intensity in the transverse bank (Bank 1) is indicative of the large volume fraction of “parent” grains which are favorably oriented for twinning during the compression cycle, and can be correlated with the high intensity in the basal \{0002\} pole figure parallel to the ND of the initial texture (Fig. 2). The initially low intensity in the parallel bank (Bank 2) correlates with the initially low intensity in the \{0002\} basal pole figure along the ED (Fig. 2).

Fig. 6 shows the normalized-diffraction intensity evolutions of the basal \{0002\} peak during the cyclic deformation along the extrusion direction corresponding to the hysteresis loops presented in Fig. 4a. The intensity data is plotted as a function of the “run number” (which was used for the experimental bookkeeping to identify the diffraction data), and the actual cycle number is also indicated in the plots. An increase in the \{0002\} peak intensity within the parallel bank indicates an increase in the volume fraction of twins and may be correlated with a concurrent decrease in the corresponding intensity diffracting from the parent grains in the transverse bank. A decrease in the parallel bank intensity is, in the present set of experiments, correlated with detwinning events (or the shrinking of twins and reversion to the parent orientation).

A detailed view of the intensity evolution is provided for the first few cycles in Fig. 7. At the initial compression, the intensity of the basal \{0002\} poles remained unchanged because the deformation is elastic and the initial microstructure of ZK60A with T5 temper is free of twins (Fig. 8a). Upon yielding, at a strain of \(\sim 0.2\%\) (Fig. 4a), the intensity begins to gradually increase, which indicates that the twinning was activated, and a lot of needle-like twin bands were observed in some favorably oriented grains at a strain of 1.2% (Fig. 8b). Previous in situ diffraction experiments focused on the monotonic deformation have shown that as much as 80% of the volume fraction of favorably oriented grains can undergo twinning during the first 8% strain [10,30]. There is a direct relationship between the volume fraction of twins, \(f_t\), the characteristic twinning shear, \(\gamma_t\) (for tensile twins in Mg alloys, \(\gamma_t = 0.130\) [31]), the Schmid factor of the twin variant and grain orientation, \(m^{B,\gamma}\), and the macroscopic strain, \(\varepsilon\), accommodated by twinning:

\[
\varepsilon = f_t m^{B,\gamma} \gamma_t
\]

In the present case, the Schmid factor for twinning in the parent grains with basal poles parallel to ND or TD can be approximated as \(\sim 0.5\). Determining the volume fraction of twins from the increase in the \{0002\} intensity within the parallel bank confirms that the plastic deformation during the compression stroke is dominated by twinning. Because twinning is dominating the strain accommodation, and the overall strain hardening rate is low, it can be inferred that massive twinning leads to a low strain-hardening rate (Fig. 4a) [19].

The stress–strain response departs from linear elastic compressive unloading almost immediately (Fig. 4a), indicating the activity of some plastic dissipation. Correspondingly, as soon as the straining is reversed, the \{0002\} intensity began decreasing. This indicates that detwinning requires much lower absolute stresses to occur in alloy ZK60A relative to the stress required to activate the twins themselves. This places the present results in agreement with previous twinning–detwinning observations of alloy AZ31B [15,17]. In fact, the detwinning begins so quickly that it is suggested that the twins and parents must contain significant internal stresses that drive the detwinning event. This hypothesis is presently being explored in detail using the collected in situ lattice strain data, and will be reported shortly.

The \{0002\} intensity in the parallel bank, indicative of the twin volume fraction, continued to decrease all the way back to the background intensity until the plastic compres-
Sive strain was recovered at 0%. As discussed and suggested in the discussion of the hysteresis loops above, this result confirmed that the twin grains formed during the compressive deformation were entirely removed by detwinning, which was corroborated by the observed disappearance of twins at the maximum tensile strain of +1.2% in Fig. 8c. Again, this trend explains why the stress–strain curve becomes concave up at 0% (Fig. 4a), and beyond this point, the deformation was accommodated by the harder deformation mechanisms within the parent grains, such as prismatic \(<a\> and pyramidal \((c+a)\) dislocation slip [32] along with possible \(\{10\bar{1}1\}\) \(\{10\bar{1}2\}\) compression twinning [21,25]. These mechanisms result in a higher strain hardening rate up to the maximum tensile stress (Fig. 4a), but they do not result in a rapid texture evolution, thus the \{0002\} peak intensities remain unchanged during this portion of the hysteresis loop (compare Figs 4a and 7).

During the unloading from the maximum tensile strain, the \{0002\} pole intensity was equal to the background intensity through the zero stress until the plastic deformation begins again during the second compression stroke at a strain of +0.5%, at which point the intensity curve increased once again (Fig. 7). Actually, the plastic straining began at +0.2% (Fig. 4a); however, we did not collect in situ diffraction data at this point. Because the second compressive straining induces more plastic strains than that introduced in the first cycle (Fig. 4a), a large volume fraction of twins were formed and, consequently, the maximum intensity is higher at the end of the second compressive cycle than that of the first cycle.

During the second tensile reversal, the inflection point in the hysteresis loop appeared at \(~+0.5\%\), which is considerably larger than the strain required to reach the inflection point during the first tensile reversal, but is comparable to the starting point of the second compression (Fig. 4a) because a larger volume fraction of twins is available to be detwinned and, concurrently, more tensile strain can be accommodated by detwinning. Notably, the strain accommodated by detwinning is the same as that of twinning, only opposite in sign (see Eq. (1)). This hypothesis is confirmed by the fact that the \{0002\} peak intensity in the parallel bank does not return close to the background level at 0% strain as in the first cycle.
Unfortunately, diffraction data were not collected at levels intermediate to $+0$ and $+1.2\%$ to confirm the exact strain level at which the detwinning mechanism is exhausted; however, it can be inferred to be $\sim0.5\%$ based on the hysteresis-loop shapes of the first and second cycles (Fig. 4a).

The cyclic hardening and softening behavior can also be explained by the variations in the loop shapes, when considered in light of the corresponding in situ neutron diffraction data. The continuously increasing basal $\{0002\}$ peak intensity in the parallel bank and the corresponding decreasing of the $\{0002\}$ peak intensity in the transverse detector bank at the point of maximum compressive strain indicate the presence of an ever increasing volume fraction of twins (Fig. 6). This coincides with the compressive hardening of the material with the increasing number of cycles (Fig. 5). Similarly, the inflection point on the tensile side of the hysteresis loop is delayed to an increasing strain level (Fig. 4a), and thus the exhaustion of detwinning is continually delayed to higher strain levels. The strain at which the hysteresis loop inflects is important because it is only beyond this point that the strain hardening rapidly increases, and the cyclic softening observed on the tensile side of the hysteresis loops at the later cycles (Fig 5) is most probably related to the decreasing amount of the post-detwinning dislocation-based flow in the parent grains. From approximately 25 cycles onwards, the basal $\{0002\}$ peak intensity in the parallel bank never returned to the background level (Fig. 6), suggesting that there is a residual twin content that remained throughout the entire cycle. With the increasing number of cycles, the volume fraction of residual twins increases (Fig. 6), resulting in the ever-increasing hardening rate observed in the compressive quarter cycles of the hysteresis loops (Fig. 4a). It is suggested that an increasing amount of dislocation debris within the material makes twinning–detwinning more difficult with cycling, which could simultaneously explain both the cyclic hardening on the compression side and the cyclic softening on the tensile side of the hysteresis loops.

Fig. 9 shows the typical fractography of the magnesium sample fatigued at a total constant strain amplitude of $1.2\%$ at room temperature. There exist a great number of parallel traces, and secondary cracks propagated in a transgranular mode along specific directions, which are presumably related to twin boundaries. The connection between fatigue cracking and deformation twinning will be further investigated by sectioning through the fracture surface and performing electron backscattered diffraction, in conjunction with fractographic stereology, as has recently been applied to aluminum alloys [33]. During the cyclic deformation, there are presumably strong interactions between the slip and twinning [9,22,27]. This is particularly important because the newly formed twins are in a plastically hard orientation with their basal poles parallel to the compressive straining direction [15]. Furthermore, the increasing volume fraction of residual twins with cycles may serve as barriers to the dislocation motion and vice versa [20,21]. Such complicated deformation interactions may be important damage accumulation mechanisms that ultimately lead to the massive shear-band formation and crack initiation, once slip and twinning cannot accommodate further plastic straining [9,19].

In summary, the intensity evolutions of $\{0002\}$ basal poles observed via in situ neutron diffraction measurements (see Figs. 6 and 7) can be correlated with the activation of the $\{10\overline{1}2\}$ $\{10\overline{1}1\}$ twinning in this material (see Figs. 1–3), and these results may be used to explain the strangely shaped hysteresis loops (Fig. 4a).

4. Summary and conclusions

The cyclic deformation of the magnesium alloy, ZK60A, was investigated using in situ neutron scattering to examine the twinning–detwinning behavior. The major conclusions are summarized as follows:

(i) the unique orientation relationship between the parent and the twin grains in the magnesium alloys facilitates the investigation of the twinning–detwinning behavior using the two-detector in situ neutron diffraction system of the SMARTS spectrometer;

(ii) the intensity transfer of $\{0002\}$ basal poles in the two detector banks can be reasonably related to the activation of $\{10\overline{1}2\}$ $\{10\overline{1}1\}$ twinning in this material, which are characterized by the strangely shaped hysteresis loops;

(iii) the fact that twinning and detwinning alternates with the cyclic loading was confirmed using in situ neutron scattering, i.e. most twins formed during compression are removed via detwinning when the load is reversed;

(iv) a small volume fraction of residual twins was detected, which gradually increases with increasing cycles, and may be an important factor in understanding the low-cycle fatigue behavior of the magnesium alloy;
(v) overall, the observed cyclic twinning–detwinning behavior in ZK60A alloy was similar to the phenomenon reported for AZ31B alloy, although their initial textures are significantly different.

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