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Reducing the tension–compression yield asymmetry in a Mg–8Al–0.5Zn alloy via precipitation

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Tension–compression asymmetry in Mg–8Al–0.5Zn alloys has been studied as a function of precipitation state. It has been shown that the presence of precipitates significantly reduces yield asymmetry compared with solution treated material. This reduction in asymmetry was attributed to reduced rates of twinning in the presence of $Mg_{17}Al_{12}$ precipitates. This has been confirmed by texture and microstructure analyses, which show a reduction in the scale and volume fraction of twins in aged and solution treated samples examined at equivalent levels of strain.

Keywords: Yield asymmetry; Magnesium alloys; Precipitates; Neutron diffraction; Twinning

In contrast to cubic metals, the yield strength of most magnesium alloys differs significantly between tension and compression [1,2]. This effect complicates the deformation and forming of magnesium alloys and the ability to develop mathematical models to describe this behavior. The yield asymmetry has been attributed to the polar nature of tensile twinning on the $\{10\bar{1}2\}$ plane. For HCP magnesium with a c/a ratio of approximately 1.622 tensile twinning is activated by a compressive stress parallel to the basal plane or a tensile stress perpendicular to the basal plane [3]. In polycrystalline samples the orientation of the applied stress relative to the basal plane is dependent on the crystallographic texture of the material and, as a result, the yield asymmetry is sensitive to texture. For example, the yield asymmetry is lower in cast alloys [4], which tend to have weak textures compared with wrought alloys [5].

In addition to texture, microstructure can also influence twinning behavior. It has been shown [6] that grain size refinement can reduce twinning and, therefore, yield asymmetry. It has also been reported that the presence of precipitates modifies the process of twinning in aged magnesium alloys [7–9]. The interaction between twinning and precipitates has been proposed to primarily arise from the difficulty experienced by migrating twin boundaries in propagating through a high density of precipitates [8]. Most recently, Stanford and Barnett [9] have reported reductions in twin size and total twin volume fraction in an aged binary Mg–5% Zn alloy.

Commercial Mg–Al–Zn (AZ series) magnesium alloys present an excellent opportunity to study this effect since a relatively large density of precipitates can be produced by aging. Moreover, there exist previous detailed microstructural studies that have shown a strong interaction of precipitates with migrating twin boundaries [8]. In an attempt to explore the role that $Mg_{17}Al_{12}$ precipitates play in modifying the tension–compression asymmetry in an AZ80 alloy tension (6.7 mm wide, 1 mm thick and 40 mm long) and compression ($10 \times 9.45 \times 13$ mm) samples were machined from a direct chill cast AZ80 (Mg–8 wt.% Al–0.5 wt.% Zn) ingot. These samples were solution treated for 24 h at 415 °C, resulting in an equiaxed microstructure (Fig. 1a) with an average equivalent area diameter [10] grain size of 32 μm. The tension and compression samples were then aged at 200 °C for 72 h to obtain a uniform distribution of mainly continuous precipitates (Fig. 1b). Sample preparation for optical and scanning electron microscopy (SEM) has been described elsewhere [11].

Tensile tests were conducted on a servo-hydraulic Instron load frame at a strain rate of $1 \times 10^{-3}\ s^{-1}$. Compression tests were carried out on a screw driven Instron test machine. The uniaxial stress was applied normal to the casting direction (i.e. the centre of the as cast pole figure in Figure 3a). During compression tests molybdenum disulphide-coated with Teflon was

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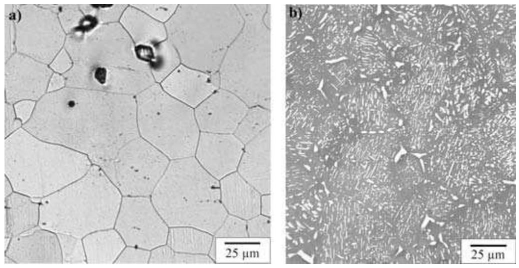

Figure 1. (a) Optical micrograph showing the grain structure of a solution treated AZ80 sample. (b) Scanning electron micrograph of the same material after aging.

used as a lubricant and a strain rate of $1 \times 10^{-3}\ s^{-1}$ was applied. Negligible barrelling was observed after testing, confirming the uniformity of deformation in compression. The load–displacement data from the load frame were corrected for machine compliance and then used to calculate true stress and true strain. Yield strength values reported here correspond to a 0.2% offset.Texture measurements were made on the E3 spectrometer at the Canadian Neutron Beam Centre, Chalk River Laboratories, Canada. The neutrons had a nominal wavelength of 2.2 Å and the cross section of the beam was 25.4 × 25.4 mm. Four pole figures, $\{0001\}_{Mg}$, $\{10\bar{1}0\}_{Mg}$, $\{10\bar{1}1\}_{Mg}$ and $\{10\bar{1}2\}_{Mg}$, were measured for each sample. The volume fraction of $\{10\bar{1}2\}$ tensile twinned material was estimated from the $\{0001\}$ diffraction data (for details see below). Electron backscatter diffraction (EBSD) was performed using a JEOL 6500F field emission gun scanning electron microscope with HKL Channel 6.0 software. Sample preparation for EBSD has been described elsewhere [11].

The tension–compression asymmetry described above is clearly illustrated in the compressive and tensile true stress–true strain curves for the solution treated alloy (Fig. 2). Both the yield strength and the work hardening rate showed a marked asymmetry. The ratio of the yield strength in compression to the yield strength in tension (denoted the asymmetry ratio here) was 1.15. The work hardening rate in tension was also significantly lower than that in compression throughout the entire stress–strain curve.

It is well known that pure magnesium exhibits a lower yield strength and rate of work hardening (until twinning is exhausted) when plastic deformation is dominated by twinning rather than slip [12]. The behavior observed here can thus be interpreted as a manifestation of this tendency, attenuated by the relatively weak texture. For a random texture there will be more twinning in compression than in tension, however, the initial texture of the material used in the current study was not random (see Fig. 3a). Indeed, the texture prior to compression was such that it exhibited a preferred alignment of basal poles normal to the rolling direction. The lower yield strength and rate of work hardening exhibited by the solution treated material in tension relative to compression were consistent with the larger fraction of grains oriented favorably for $\{10\bar{1}2\}$ twinning in tension compared with compression.

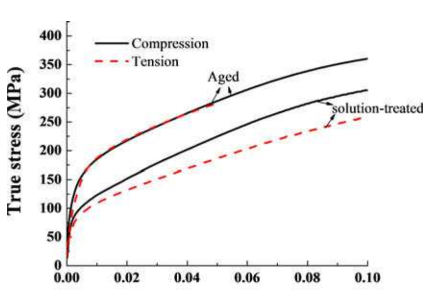

Figure 2. The true stress–true strain response of solution treated and aged AZ80 alloy tested in compression (solid lines) and tension (dashed lines).

Also shown in Figure 2 are the tensile and compressive stress–strain curves for the aged alloy. In this state the stress–strain curves showed identical yield and work hardening responses up until fracture (giving an asymmetry ratio of 1.0). The only significant difference in the tensile and compressive stress strain curves was the ductility, which is lower in tension than in compression.

In order to evaluate whether the differences between the solution treated and aged samples could be attributed to differences in twinning behavior the crystallographic texture was measured as a function of strain. While both slip and twinning contribute to texture evolution, the large reorientation associated with tensile twinning (86° rotation of the <c> axis) results in very significant and rapid texture evolution. The textures of the aged and solution treated samples were measured for several levels of equivalent plastic strain. For example, Figure 3 shows the result obtained at 2% strain for compression in aged and solution treated samples. In the solution treated sample (Fig. 3b) reorientation due to tensile twinning causes the basal poles originally close to the transverse direction to switch to new orientations close to the compression direction (the centre of the pole figure). Conversely, the texture in the aged and deformed material (Fig. 3c) was similar to the initial undeformed texture (Fig. 3a) after 2% strain.

These results can be interpreted in a more quantitative manner by determining the volume fraction of $\{10\bar{1}2\}$ tensile twins from the experimentally measured $\{0001\}$ pole figures. Texture analysis on undeformed material and on samples deformed in compression suggests that the grains present within the outer rim of the $\{0001\}$ pole figure, corresponding to tilt angles between 50° and 90°, should undergo $\{10\bar{1}2\}$ tensile twinning. The volume fraction of grains where twinning is expected can thus be calculated by integrating the normalized intensity from the outer 40° rim of the $\{0001\}$ pole figure. As deformation proceeded it was observed that the volume fraction of grains in this rim decreased as the grains reoriented due to twinning. Therefore, the decrease in volume fraction of grains in this rim was used as a measure of the twinned volume fraction. A similar approach has recently been applied by Proust et al. [13] to determine the twin volume fractions in an AZ31 magnesium alloy.

Figure 3d shows the $\{10\bar{1}2\}$ tensile twin volume fractions, calculated using this procedure, for solution treated (dashed lines with closed circles) and aged (solid lines with open circles) samples at 2% and 5% strain.

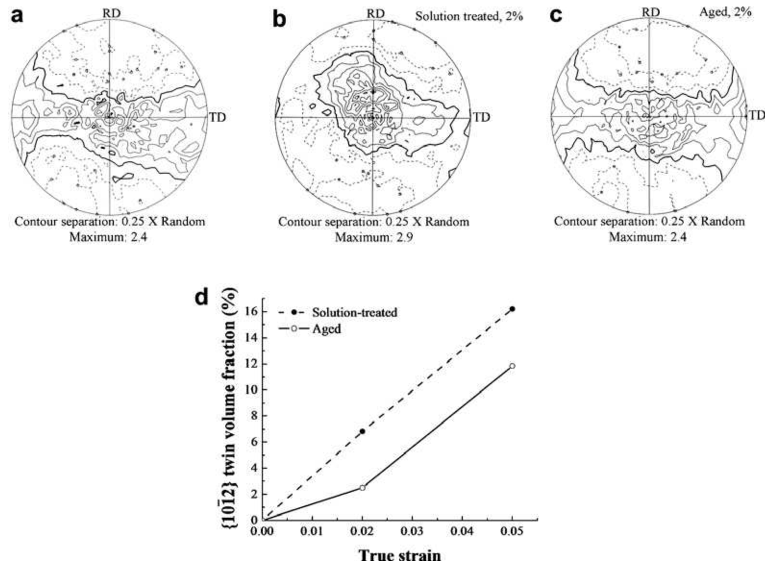

Figure 3. Stereographic basal pole figures for (a) solution treated AZ80 sample, (b) sample solution treated and compressed to 2% strain, (c) sample aged and compressed to a strain of 2% and (d) $\{10\bar{1}2\}$ twin volume fractions (%) as a function of true strain for solution treated (dashed lines with closed circles) and aged samples (solid lines with open circles). Note: the compression direction is at the centre of the pole figure. The pole figures are contoured in multiples of random distribution (m.r.d) with the thick solid black line corresponds to 1 m.r.d. The contour levels above and below 1 m.r.d are given by solid and dotted lines, respectively, in 0.25 m.r.d steps.

The solution treated samples had a higher volume fraction of $\{10\bar{1}2\}$ tensile twins at 2% and 5% compared with the aged samples. At 2% strain the ratio of the twinned volume fraction in the solution treated sample to that in the aged sample was approximately 3, i.e. volume fractions of 6.9% and 2.5%, respectively. At 5% strain the ratio has decreased to approximately 1.5.

Further evidence of lower tensile twinning activity in the presence of precipitates can be obtained from microstructural observations. Figure 4a and b shows typical EBSD maps (grain boundary maps) of solution treated and aged samples after 2% compression. The highlighted tensile twinned regions in these maps were identified as being surrounded by boundaries with an 86° (±5°) $< 11\bar{2}0 >$ boundary. Qualitatively, it was observed that the fraction of tensile twins in the aged material was lower than in the solution treated case, in agreement with the analysis of the bulk texture measurements.

Grain boundaries are generally considered to be the most common sites for twin nucleation in these alloys [14–16] and thus anything that acts to modify the structure or the chemistry of these boundaries could also influence twin nucleation. In aged materials discontinuous and continuous precipitates at grain boundaries could modify potential twin nucleation sites. Moreover, precipitation will alter the local boundary chemistry and could, therefore, affect the rate of twin formation. Once nucleated, the lengthening and widening of twins has been described in terms of various dislocation glide-based mechanisms [14,17–19]. The presence of obstacles in the path of a migrating twin boundary can markedly influence the twinning behavior of an alloy. For example, the early work of Partridge [20] on pure magnesium illustrated the pinning of twin boundaries due to oxide particles. More recently, Serra et al. [21] studied the interaction of a moving $\{10\bar{1}2\}$ twin boundary with clusters of self-interstitial atoms and vacancies in zirconium using atomistic simulations. These results indicate that obstacles such as these cause strengthening by pinning the twinning dislocations and restricting boundary motion. Finally, the detailed work of Gharghouri et al. [8] on a binary Mg–Al alloy containing β-$Mg_{17}Al_{12}$ precipitates showed many examples of twin boundary–precipitate interactions, including cases of twins bowing around and bypassing precipitates.

While more detailed examinations are required to unambiguously identify whether twin nucleation, twin growth or both processes are strongly modified in the aged samples compared with the solution treated samples, Figure 4 provides evidence which can be considered in the context of the work cited above. It should be noted that in these EBSD maps the twins are significantly finer in the aged sample compared with the solution treated sample. Preliminary observations on samples deformed to larger strains showed that the twin boundaries became curved in the presence of precipitates, similar to the observations of Gharghouri et al. [8]. It is clear that the interaction between migrating

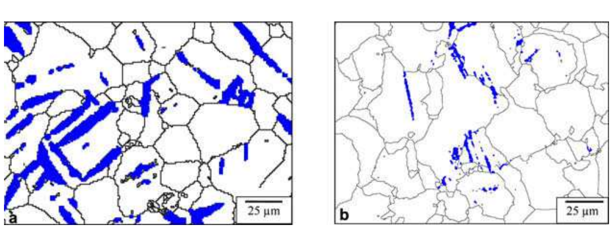

Fig. 4. EBSD grain boundary maps (black boundaries are 15° and higher misorientation boundaries) with tensile twins highlighted for (a) solution treated and (b) aged samples each after having been strained 2% in compression. The twinned area was identified as those areas surrounded by boundaries having a 86° (±5°) $< 11\bar{2}0 >$ boundary disorientation. The compression direction is oriented horizontally in these maps.

twin boundaries and precipitates hinders the rate of twin propagation, but it is unclear how strong the effect of the precipitates is on the nucleation of twinning. Determining whether the number density of twins (i.e. the number of twins nucleated) is significantly different for the solution treated and aged samples is much more difficult, given that the thickness of the twins observed in the aged material was at the limit of resolution of the EBSD technique.

As a final note, the level of tension/compression asymmetry in the solution treated material can be much larger for wrought samples with a strong basal texture (maximum intensity of 7 m.r.d). Here one expects significant $\{10\bar{1}2\}$ twinning in compression and little $\{10\bar{1}2\}$ twinning in tension. Preliminary results showed a pronounced yield asymmetry (asymmetry ratio of 0.59) in the solution treated sample. However, aging reduced the asymmetry drastically to an asymmetry ratio of 0.87, i.e. the precipitates were extremely effective at reducing the tension/compression asymmetry in this case. The current results are qualitatively similar to those previously reported by Kleiner and Uggowitzer [22] in an extruded AZ80 alloy (0.52 in solution treated to 0.76 in aged). The small differences between their work and the current study was presumably related to the less intense initial texture (maximum intensity of 4 m.r.d) and different heat treatment conditions, which produced a different state of precipitation.

The current work illustrates that the difference in the tensile and compressive yield strengths of a solution treated AZ80 magnesium alloy can be reduced and in some cases eliminated when the sample is aged to contain a high density of $Mg_{17}Al_{12}$ precipitates. While the work shown here concerns material having a relatively weak starting texture (arising from casting), the effect of precipitates on the tension/compression asymmetry is even more striking in a highly textured material. The results presented here suggest that $Mg_{17}Al_{12}$ precipitates affect twin growth, however, their influence on twin nucleation is unclear. Nonetheless, it is concluded that precipitation of this phase is an effective approach to reducing the tension/compression asymmetry in magnesium–aluminum alloys.

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