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ON THE IMPACT OF SECOND PHASE PARTICLES ON TWINNING IN MAGNESIUM ALLOYS

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Abstract

Deformation twinning is an important deformation mode in magnesium alloys. Despite this, little is known on the extent to which the stress for twinning can be altered by a dispersion of second phase particles. The current paper presents a series of findings on the role of differently shaped particles on the characteristics of the twins that form. It is shown that coherent rod shaped particles in Mg-Zn alloys have little obvious effect locally on the twin boundaries but that the twin number density is increased by their presence. Plate particles in a Mg-Al-Zn alloy cause obvious perturbations to the twin interface. Loops of twinning dislocations left around the particles eventually collapse into the particle interface, a phenomenon that is evidently facilitated by stress concentration on leading twin dislocation and stress relaxation in the adjacent material.

Introduction

The $\{10\bar{1}2\}$ twin plays an important role in the mechanical response of magnesium and its alloys. In some cases it dominates it. Two important examples are the compression of wrought plate in its plane and the compression of extruded material along its extrusion direction. In these cases the first few percent of strain can sometimes be attributed entirely to the $\{10\bar{1}2\}$ twin [1]. The practical consequence of these facts is a stress-strain curve in compression that differs in both the yield strength and strain hardening response to that seen in tension. It is not uncommon for the yield stress of an extrusion tested in compression to fall below that found in a tension test by a factor of 2.

When twinning dominates the plastic response, the sensitivity to other processing or "service" variables differs to that seen when slip dominates. For example, it is known that the stresses required for twinning are less sensitive to temperature and strain rate than those seen for slip. Consequently, the yielding stresses in extrusions tested in compression along the extrusion direction show relatively little change with temperature and strain rate [2-4].

The grain size also has a different impact upon yielding when twinning is prevalent. In their review of the literature, Meyers et al. [5] found that the Hall-Petch slope – i.e. the sensitivity of yield stress to grain size – is invariably higher when twinning dominates. In magnesium alloys, it has been found that the Hall-Petch slope for extrusions tested in compression falls approximately twice that seen in tension (i.e. ~10 MPa.mm$^{1/2}$ compared to ~5 MPa.mm$^{1/2}$) [6]. This obviously has important consequences for the strength of compression members and for the tension-compression yield asymmetry.

We turn now to the strengthening effect of fine dispersed precipitates, the subject of this short communication. For slip the basic principles are well known (e.g. [7]). At low particle volume fractions, small inter-particle distances, which force passing dislocations to bow to smaller radii, give higher strengths. Non-shearable particles facilitate higher work hardening rates – due primarily to geometrically necessary Orowan loops left in the vicinity of particles. At higher volume fractions, the back-stress generated by non-relaxed elastic stresses becomes important and kinematic work hardening is enhanced. For twinning, the effects are less well understood.

First, one must determine how the separate processes of twin nucleation (formation) and growth respond to the presence of particles. Here, we restrict our interest to growth. Richman [8], in his survey of twinning phenomena in bcc metals, examined the interaction between twins and FeBe$_2$ particles in an alloy of Iron and 25 at% Be. Upon encountering a particle, the twins "pinched off". The scenario painted by Richman following his microscopy work is sketched in Figure 1, in which his original diagram is closely followed. Twinning dislocations loop around the particle in a manner similar that followed by slip dislocations. Only here an inclined twin interface is built up around that particle. The particle and the material around it are not sheared in this case, though they are highly stressed. It is imagined that Orowan type precipitation hardening occurs in such an instance.

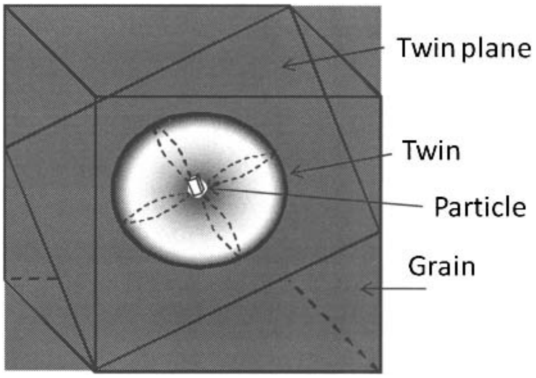

Figure 1. A twin centred on a particle producing a "donut" effect where loops of twinning dislocations build up around the particle. Diagram sketched from a schematic provided by Richman [8].

In his transmission electron microscope study on a magnesium-5% zinc alloy, Clark [9] observed the interaction between dislocations, twins and MgZn′ particles. The particle phase is a transition structure with a high coherency with the magnesium lattice. Basal dislocations were not able to shear the precipitates, but twins did. Sheared precipitates were seen to be "engulfed" by the twin. A schematic based on Figure 11 of Clark's paper is provided in Figure 2. This shearing is in marked contrast to the non-shearing particles observed by Richman. It is interesting that the same obstacle was not sheared by slip dislocations. Clark ascribed this to two effects: i) the relatively small shear associated with the twinning dislocations (Burgers vector $b_T=h\gamma$ where h is the step height, $2d$=0.38 nm in the current twin, and γ=0.13 is the twinning shear, thus b_T~0.05 nm) and ii) the supposed difficulty for a twin dislocation to bow around a particle due to its confinement in the twin interface.

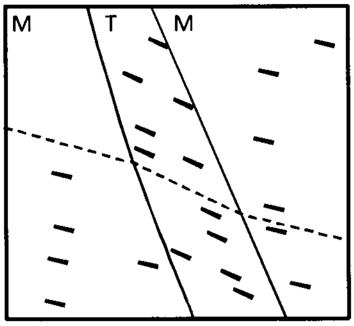

Figure 2. Particles of MgZn' sheared and thereby "engulfed" by the progress of a twin through a grain in Mg-5Zn [9]. Schematic based on Figure 11 of the paper.

Gharghouri et al. [10] studied the interaction between twins and precipitates in Mg-7.7Al, which forms plate shaped particles upon aging. In their study, twins were observed to be held up by particles, to engulf particles and in some instances to bypass particles. This last situation is shown schematically in Figure 3, which follows a published transmission electron microscope image. Important for the present consideration is the fact that Ghargouri et al. maintain that although the precipitates were engulfed by their twins they were *not* sheared. This conclusion appears to be based largely on their determination of the yield stress of the precipitate, which was found to be in the order of 1 GPa, approximately an order of magnitude higher than the yield stress of the matrix. The engulfed precipitates were seen to be inclined ~4° to their initial orientation in the direction of the twinning shear.

Two of the present authors have examined an alloy similar to that employed by Clark – Mg-5%Zn [9]. In that work, we concluded that the precipitates did not show any rotation, nor were they sheared by either twins or dislocations. However, precise measurements of rotation were hindered by foil warping and difficulties in obtaining sufficient contrast. Basal faults in the twin were used as fiducial markers but we acknowledge that this approach assumes a perfectly aligned c-axis rod in the matrix prior to twinning and this may not always be the case. Nevertheless, the study showed that twin nucleation was enhanced by precipitation but that twin growth was suppressed by it.

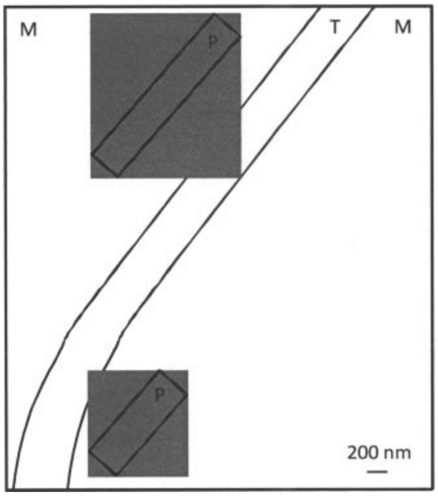

Figure 3. Sketch of a twin bypassing two precipitate particles in aged Mg-7.7Al as seen by Ghargouri et al. [10].

In the present study, we employ some additional microscopy to determine how twins interact with precipitates in magnesium alloys strengthened by MgZn' rods (Mg-5Zn) and $Mg_{17}Al_{12}$ plates (Mg-9Al-1Zn).

Methodology

Extruded magnesium round bar containing 5 wt% Zn (Z5) was employed in the study. A two step solution treatment was performed following extrusion, 2 hr at 330°C followed by 6 hr at 400°C. Aging was performed at 250°C, 200°C, 150°C and 110°C for 10 hr, 18 hr, 8 days and 32 days. After Clark [9] and Gao and Nie [11], the rod precipitates thus produced are a coherent transition phase referred to as MgZn′ or β_1' with their long axis parallel to the c-axis of the magnesium matrix. Compression specimens 8 mm in height and 5.4 mm in diameter were examined using Electron Backscatter Diffraction and transmission electron microscopy after deformation to a strain of 5%. The grain size was 30 μm and the texture was typical for a magnesium extrusion. More information is presented in reference [12].

Rolled magnesium alloy with 8.6 wt% Al and 0.8 wt% Zn with 0.24% Mn (AZ91) was also examined. Solution treatment was performed at 420°C for 24 hours. Aging was performed at 200°C for 2 hr, 30 hr and 20 days. The precipitates produced are plates of $Mg_{17}Al_{12}$ that lie on the basal plane. The grain size was 70 μm and the texture was typical for a magnesium plate although there were a few grains that had their c-axis tilted towards the transverse direction. Compression specimens were cut to enable compression to be performed along the rolling direction. Transmission electron microscopy was carried out after deformation to different strains. More information will appear in a future publication.

Specimens for EBSD were prepared using electropolishing in 5% nitric acid in ethanol at 20V for 30-45 s. The same solution was used for chemical cleaning for 5-10 s. For TEM sample preparation, a Gatan plasma ion polishing system was employed.

Results and Discussion

To gain insight into how twins interacted with precipitates at their encounter, precipitates in the vicinity of $\{10\bar{1}2\}$ twin interfaces were inspected using transmission electron microscopy. In all cases images were produced on the $<11\bar{2}0>$ zone axis. This is the "edge on" condition where the twin plane normal lies in the plane of the image, as does the twin shear direction $<\bar{1}011>$.

In Figure 4 two images taken near to a twin boundary in the Z5 alloy aged at 150°C are shown. In each case instances can be seen where the inclination of the precipitate long axis differs on either side of the twin boundary. As mentioned above, this was not always detected in this alloy. The effect appears similar to that seen by Clark [9] but due to the scarcity of the observations, further work is required to be certain. The sense of the shear provided by the twin, determined from inspection of the inclination of the twin boundaries to the matrix c-axis and noting the polarity of the twinning is such as to give extension along c. The slight rotation of the precipitates that can be seen near these twin boundaries is the right sense according to the twinning shear. Of considerable interest in Figure 4 is that there is no obvious sign of any "pinching off" or dislocation looping where the twins meet the precipitates. The twin boundaries show quite strong contrast in these images such that it is possible to detect ledges in the twin interface. These seem to be separated by longer regions of coherent twin interface on the lower boundary in Figure 4a compared to the upper boundary. At this lower interface some overlap of the flat portions of twin interface can be seen and this reflects the bowing of a twinning dislocation between the upper and lower surfaces of the foil. No such effects are seen to be concentrated at the particles. The precipitates appear to have been sheared with minor disruption to the twin interface.

As an aside, the height of the ledges in the twin boundary in Figure 4a is in the order of 2 nm. This equates to a segment of incoherent twin boundary comprised of 5 twinning dislocations.

In the case in Figure 4, the precipitates are rod shaped and are located well within the foil, whereas the twin boundary traverses the foil width. This may obscure features of the interaction. However, in the case of the plate shaped precipitates seen in AZ91, the plates are sufficiently large (295-825 nm) to traverse the whole foil thickness. An example of the twin-particle interaction seen in alloy AZ91 is shown in Figure 5. In this instance, the twin appears to have been held up at the particle in the lower right corner. It is also clear that twinning dislocations are looped around the precipitate to the right of centre in this image. Here the precipitate remains unsheared.

In Figure 5, the twin is thin. The thickening of the twins is not prevented in this material, however, and an image of a thicker twin is presented in Figure 6. In this case there is no obvious looping of twinning dislocations around the precipitates. However, some larger scale curvature concave to the twin can be seen on the right of the image. This requires the existence of loops of twinning dislocations. The twin interface is also considerably perturbed in the top left of the image.

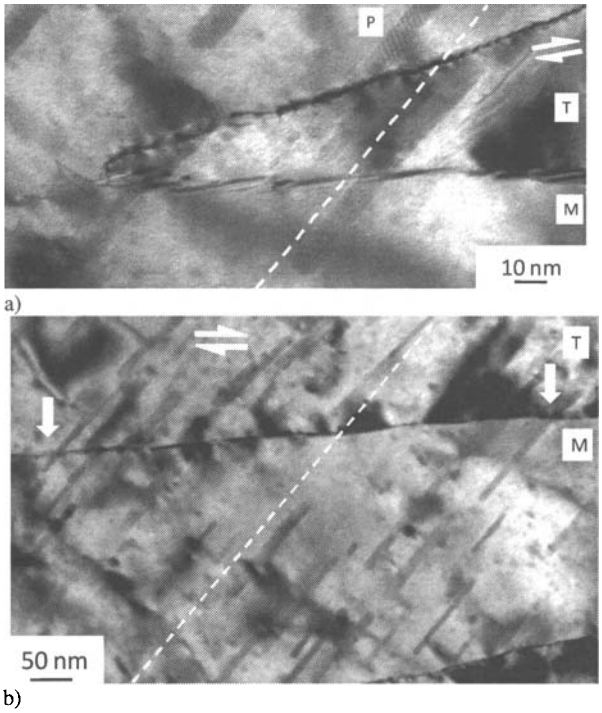

Figure 4. TEM images of $\{10\bar{1}2\}$ twin interfaces seen in alloy Z5 aged at 150°C and compressed along the extrusion direction to 5% strain. The twins shown share the $<11\bar{2}0>$ zone axis.

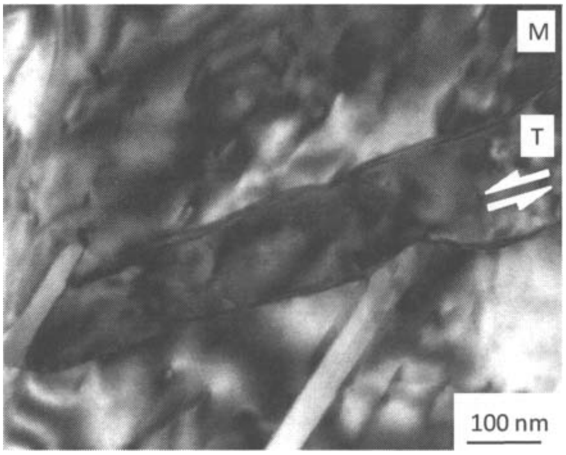

Figure 5. TEM image of a $\{10\bar{1}2\}$ twin seen in alloy AZ91 peak aged at 200°C and compressed along the extrusion direction to 8.5% strain.

Importantly, Figure 6 shows that the plate particles are strained considerably. Whether elastic or plastic, such a large strain requires considerable stresses. This consequently entails i) stress concentration, most likely in the form of pile-ups of twinning dislocations (seen as regions of twin boundary with an "elliptical" curvature) and ii) stress relaxation, most likely in the form of basal slip at the twin tip (possibly along with other emissary dislocations [13]) and basal slip inside the twin. Indeed, we have noted previously [12] that the hardening of accommodation slip is likely to comprise an important contribution to the hardening of twinning by particle addition.

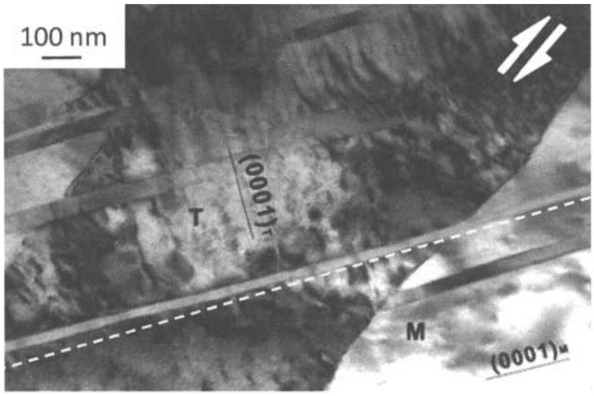

Figure 6. TEM image of a $\{10\bar{1}2\}$ twin seen in alloy AZ91 peak aged at 200°C and compressed along the extrusion direction to 8.5% strain.

A difficult observation to reconcile is that in Figure 6, the twinning dislocations appear to have collapsed into the matrix-precipitate interface. For this to occur at the interface of a strong (σ_y~1 GPa) rigid (E~80 GPa) particle is not easy to understand. Shear along the particle interface will go some way towards accommodating the incompatability strains. Such slip is shown in Figure 7. Considerable dislocation activity can be seen emanating from the tip of the particle and extending into the twin interior. Indeed, Gharghouri et al. [10] noted high "accommodating" slip activity on the perimeter of equivalent particles in their alloy.

Figure 6 also shows the process of collapse of loops of twinning dislocations into the particle interface. On the right of the particle, the "collapse" has occurred to a further extent than on the left. In the case of the latter, the twinning dislocations have to glide "past" each other and through the stable vertical array configuration so as to meet the particle interface. These dislocations are not subject to the same pile-up stress magnification that drives the dislocations into the other face of the particle. A similar asymmetry is seen in the loops in Figure 5.

It now remains to observe the interaction between twins and precipitates at a lower magnification. EBSD analysis (see [14]) was employed to observe twin growth and formation in alloy Z5 as a function of aging condition. The results are plotted in Figure 8 as a function of the yield stress. As hardening due to aging progresses, the twin number density rises. This can be attributed to increased resistance to twin growth over nucleation. The higher stresses required for twin propagation enable more twins to form. At the same time the twin volume fraction drops. With increasing aging up to peak hardness, the volume fraction continues to drop. So too does the number density. However, the number density does not drop below that seen in the unaged condition, even during overaging. Although overaging does not alter the twin number density much, it lifts the volume fraction considerably. The effect of this exceeds what one would expect from a stress based argument alone; that is, the overaged datum falls outside of the relationship between stress and volume fraction seen during aging. This may be due to simultaneous effects of particles on slip. The extent of twinning depends on its competition with slip.

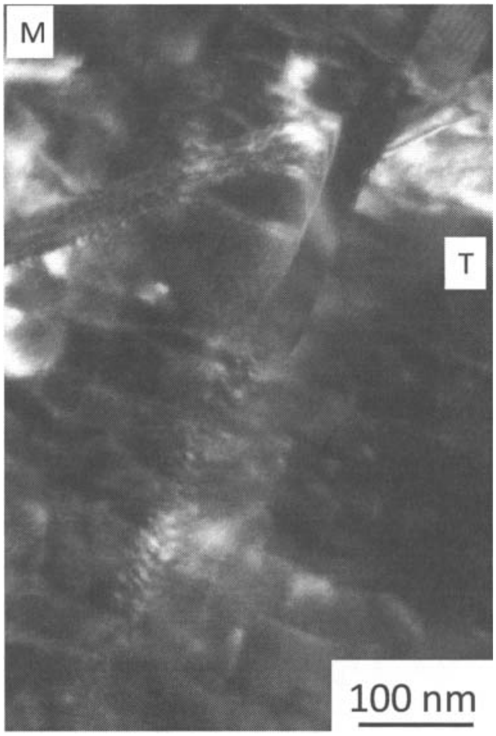

Figure 7. TEM image of a $\{10\bar{1}2\}$ twin seen in alloy AZ91 peak aged at 200°C and compressed along the extrusion direction to 8.5% strain.

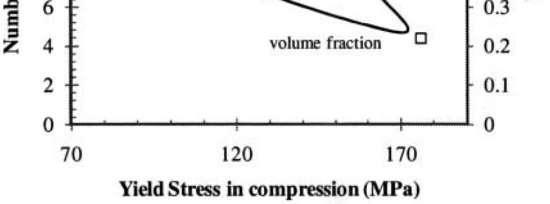

Figure 8. Twin volume fraction and number density determined using EBSD for magnesium alloy Z5 extruded and tested in compression to a strain of 5% after aging to different levels of yield stress.

Conclusions

1. Rod shaped precipitates in magnesium alloy Z5 seem to shear during twinning although more accurate measurements of the effect are required.
2. No obvious Orowan type looping is seen where the rods intersect a twin interface.
3. Orowan type looping is seen where twins meet plate shaped precipitates in alloy AZ91.
4. These loops collapse into the particle interface, driven by pile-up stress magnification and eased by slip relaxation into the twin.
5. In Z5 aging initially promotes twin nucleation but consistently depresses the twin volume fraction.

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