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The effect of high yttrium solute concentration on the twinning behaviour of magnesium alloys

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Abstract—The deformation behaviour of two single phase binary alloys, Mg–5Y and Mg–10Y, have been examined. In compression, two twin types were observed, the common $\{10\bar{1}2\}$ twin as well as the less common $\{11\bar{2}1\}$ extension twin. It is shown that the $\{11\bar{2}1\}$ twin is much less sensitive to solute concentration than the $\{10\bar{1}2\}$ twin, and it is suggested that the simple atomic shuffle of the $\{11\bar{2}1\}$ twin reduces the solute strengthening imparted by Y additions. The common $\{10\bar{1}2\}$ twin showed significant hardening as a result of alloying with Y. An analysis of solute behaviour has indicated that of the four chemical parameters investigated, i.e. atomic size, shear modulus, electronegativity and solute distribution, it appears to be the larger atomic radius of Y compared to Mg that increases the stress required to activate the $\{10\bar{1}2\}$ twin. It is suggested that the large atomic radius inhibits the atomic shuffling process which accompanies the twinning shear in this twin type.

Keywords: Magnesium; Twinning; Deformation; Solute strengthening

1. Introduction

The room-temperature deformation behaviour of magnesium alloys is fairly well described [1–5]. Experiments on single crystals [6–10], polycrystals [5,11–13] and the use of various modelling techniques have revealed that the softest and most commonly activated deformation mode is basal slip. In addition to slip on the basal plane, slip is also observed on the prismatic and second-order pyramidal planes, but these require much higher stress states to activate than basal slip. Magnesium also deforms by twinning. The activation of the $\{10\bar{1}2\}$ (*c*-axis extension) twin is prolific at room temperature, and the stress required to activate this mode lies somewhere between the critical resolved shear stress (CRSS) for basal slip and prismatic slip [13]. Being one of the softer modes, $\{10\bar{1}2\}$ twins often accommodate a significant portion of the applied strain during the early stages of deformation [14], depending on the texture and strain path.

It has been shown [15] that the stress required to activate a twin is analogous to that for slip, i.e. there is a minimum shear stress that is required to be applied in the twinning shear direction to propagate the twin and accommodate the applied strain. For the case of two-phase alloys, it has been shown that this stress can be significantly increased by the precipitation of second-phase particles [16,17]. However, the effect of solutes on the stress required to activate twins is not very well established. On the one hand, there has been experimental work indicating that zinc in solid solution in magnesium has no effect on the stress required to propagate a twin [18]. On the other hand, there are indications of significant twin hardening in alloys containing high solute concentrations of rare earth (RE) elements [19]. There have been other anomalous observations in regard to twinning of magnesium alloys in the presence of high RE solute concentrations. Two separate studies report the complete suppression of twins in alloys with >8wt.% Y [20,21]. It therefore appears that RE elements may have a significant impact on the twinning behaviour, but it is not yet clear why. Since twinning is such an important deformation mode in magnesium alloys, we have chosen to investigate this issue in more detail. To examine the effects more closely we describe here the twinning behaviour of two Mg alloys, containing 5 and 10 wt.% Y heat treated to be single phase. These compositions were chosen based on previous reports [20,21] that show the higher concentration alloy (Mg–10Y wt.%) may exhibit twinning suppression.

2. Experimental methods

The two alloys, Mg–5Y and Mg–10Y, were made by melting pure magnesium with a master alloy of Mg–27Y in the appropriate proportions; the compositions are given in Table 1. Melting was carried out in a resistance furnace. The 2 kg melts were held in a steel crucible under the shielding gas commercially sold under the name of AMGAS.

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Table 1. Composition of the two alloys (wt.%). Measurements made using inductively coupled plasma atomic emission spectroscopy.

	Mg	Y	Al	Si
Mg–5Y	Bal	5.12	0.02	0.03
Mg–10Y	Bal	10.7	0.01	0.03

After melting, the alloys were poured into a steel chill mould to ensure relatively rapid solidification. The as-cast ingots were sectioned into specimens with an approximate size of 40 mm × 80 mm, and a thickness of ~25 mm. The sections were solution treated in a tube furnace under a flowing argon atmosphere for 5 h at 520 °C followed by an immediate cold water quench. The solution treated alloys were hot-rolled at 500 °C to a strain of 50% in three passes. After hot rolling, a set of annealing trials were carried out to examine the recrystallization behaviour. From these trials it was decided that the specimens would be annealed at 500 °C for 1 h, producing average grain sizes of 75 μm in the 5Y alloy and 65 μm in the 10Y alloy. Although not exactly the same, the average grain sizes of the two alloys are quite similar, 70 μm (±5 μm). Note that at 500 °C both of the alloys are single phase at equilibrium.

Cylindrical samples with a diameter of 8 mm and a height of 12 mm were wire cut from the annealed plate. These cylindrical samples were compression tested at room temperature in an Instron load frame at an initial strain rate of 0.001 s^{-1}. Initially, samples were deformed to failure. Subsequently additional tests were carried out to total strains of approximately 0.01, 0.02, 0.05 and 0.1. The strain was measured using a non-contact video extensometer which reads displacement from markers placed on the sample surface.

After compression testing the samples were metallographically prepared by standard techniques followed by a final polish with colloidal silica. For optical microscopy, two techniques were used to reveal the microstructure. The first method was etching with the commonly used acetic picral etchant. This reveals the microstructure through orientation etching and grain boundary enhancement. The second method used was a short etch of ~3 s in a solution of 2% nitric acid in ethanol. This revealed the microstructure of these particular alloys quite well, and the fine twins were more easily seen with this etching technique using differential interference contrast (or Nomarski contrast [22]) in the optical microscope. This was also the method used to prepare samples for electron microscopy. The average grain size was determined using the linear intercept method on samples etched in acetic picral. The volume fraction of twins was measured from optical micrographs taken with Nomarski contrast using the point counting method. Additional twin volume measurements were also taken using the electron backscattered diffraction (EBSD) data, see below.

The deformed microstructure of selected specimens was examined using EBSD, which was carried out in an FEI Quanta field-emission gun scanning electron microscope equipped with a TSL EBSD system. Most of the post-processing of the EBSD data was done on the TSL orientation imaging (OIM) software. However, some inadequacies in the texture contouring module of this system necessitated the use of HKL software, known as Channel 5 in some instances. Note that for hexagonal crystal structures, the data from TSL requires a conversion into a different reference frame in order for the crystallographic data to plot correctly in the HKL software platform.

X-ray diffraction (XRD) was used to measure the texture of the starting materials. Texture measurements were made using a Panalytical MRD with point focus optics for the {0002}, {10$\bar{1}$0} and {10$\bar{1}$1} planes in 5° increments. The background correction, defocusing correction and pole figure plotting were carried out using LaboTex 3 software.

3. Results

The starting alloys had a similar grain size of 70 μm (±5 μm), and a fairly weak texture (Fig. 1). Samples of each alloy were compressed in the rolling direction to failure, and the flow curves are shown in Fig. 2. To help benchmark these results, a flow curve from the literature [23] is also given, showing a compression curve for pure magnesium with comparable grain size and texture. It can be seen that the Mg–5Y alloy showed an exceptionally large strain to failure of >0.4, while the Mg–10Y alloy showed a more modest strain to failure of ~0.24. The microstructures observed after failure are given in Fig. 3a and b. It can be seen that both samples show prolific deformation twinning. The Mg–5Y alloy shows more twinning than the more concentrated alloy, but this sample had also achieved a larger strain before failure. To clarify this issue, samples were compressed to different strain levels between 0.01 and 0.1. Typical examples of the resultant microstructures are shown in Fig. 3c and d. It is clear that the Mg–5Y alloy shows significantly more twins than the Mg–10Y alloy for the same strain level. The volume fraction of twins was measured optically and the results of this analysis are summarized in Fig. 4. It can be seen that at all levels of strain examined, the Mg–5Y alloy showed a significantly higher volume fraction of twins compared to the Mg–10Y alloy. Note that the data in Fig 4 is shown in terms of plastic strain, not total strain. It can also be seen that for a given volume fraction of twins, the flow stress is significantly higher in the 10Y alloy, compared to the lower concentration alloy.

The twinning behaviour was further examined with EBSD, and prolific activation of {10$\bar{1}$2} twins in the

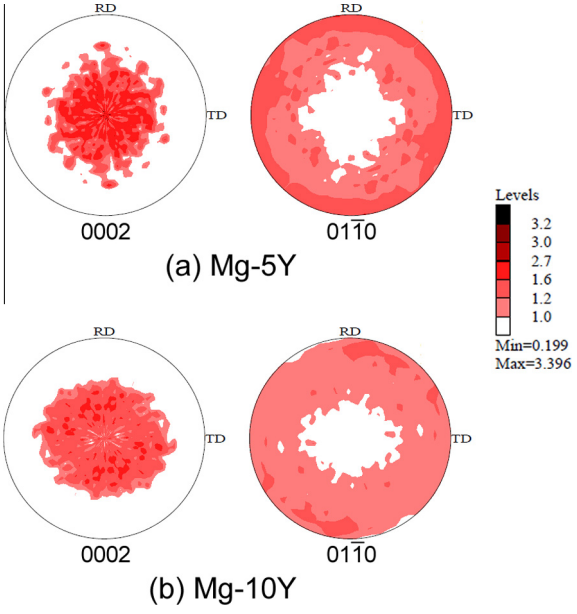

Fig. 1. Textures of the two alloys measured using X-ray diffraction.