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The influences of alloying additions and processing parameters on the rolling microstructures and textures of magnesium alloys

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Abstract

The combined effects of lithium additions (1–3 wt%) and processing parameters (rolling temperature, annealing) on the microstructural and texture evolution of pure Mg and Mg–3 wt% Al–1 wt% Zn alloy have been studied. Following rolling the basal planes were aligned with the sheet surface, although the basal poles were split and rotated towards the rolling direction. Lithium additions increased the rotation of basal poles in the rolling and transverse directions; an increase in the rolling temperature was associated with decreased rotation in the rolling direction and some broadening of texture in the transverse direction. Recrystallization during rolling varied between alloys, but had little influence on the texture. Recrystallization, and particularly grain growth, during annealing resulted in a single peak in the basal poles replacing the split observed following rolling. Texture is interpreted in terms of deformation, recrystallization and grain growth. Microstructural and texture evolution during industrial forming processes are also discussed.

Keywords: Magnesium; AZ31; Rolling; Annealing; Texture

1. Introduction

The dominant slip mode in hexagonal close packed (hcp) metals has the Burgers vector $1/3\langle 1\,1\,\bar{2}\,0\rangle$ (or $\langle a\rangle$), and in magnesium the primary slip plane is the basal $(0\,0\,0\,1)$ [1]. Mechanical twinning, which tends to occur on the $\{1\,0\,\bar{1}\,2\}$ planes in the $\langle 1\,0\,\bar{1}\,1\rangle$ direction, only occurs during c-axis tension [2]. The glide of dislocations on non-basal prismatic and pyramidal planes is thermally activated, and contributes to the increase in formability associated with magnesium above ~225 °C [1,3]. The formability of magnesium is limited because the glide of $\langle a\rangle$ dislocations does not provide sufficient independent slip systems to meet the von Mises criteria for homogeneous plastic deformation.

Lithium alloying additions improve the room temperature formability of magnesium [1]. This improvement is associated with non-basal slip, and the decrease in the axial ratio (c/a) of magnesium as a function of lithium alloying additions has been cited as the reason for enhanced glide of dislocations on prismatic planes [4]. As lithium is added to magnesium the c value of the hcp crystal falls faster than the a value, which leads to a decrease in the c/a ratio [5]. The c/a decrease results in the Peierls stress for basal slip increasing relative to that for prismatic slip, and hence increases activation of non-basal prismatic planes. Texture modeling and TEM studies indicate that lithium additions also increase the glide of $\langle c+a\rangle$ dislocations on $\{1\,1\,\bar{2}\,2\}$ pyramidal planes [6,7]. There is evidence that $\langle c+a\rangle$ dislocations dissociate into partial dislocations [8], and lithium may lower the energy of the stacking fault, and thereby increase the stability of the glissile dislocation configuration [7]. The $\{1\,1\,\bar{2}\,2\}\langle 1\,1\,\bar{2}\,\bar{3}\rangle$ system provides additional slip modes and satisfies the von Mises criteria, which explains the increase in formability associated with lithium alloying additions.

During rolling and extrusion of hcp materials a texture develops consisting of basal planes aligned parallel to the rolling direction (RD) or extrusion direction (ED) [9]. In magnesium there is often spread of basal poles in the RD, and in some cases a split in the basal poles is observed. The texture depends on factors including rolling reduction [10], rolling temperature [11,12] and initial texture [13]. Through-thickness texture variations have also been reported [14]. Alloying additions [15], including lithium [6], have been observed to alter the crystallographic texture. This is because alloying additions can alter the

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balance of deformation mechanisms, which in turn influences the texture. The texture may also evolve during recrystallization and grain growth [16–19].

Magnesium sheet does not currently exhibit sufficient formability, which means that either existing alloys must be modified to enhance sheet ductility, or else new alloys must be developed. In addition to developing novel processing techniques and modifying the microstructure there is potential to improve the properties of magnesium wrought products by modifying the *c/a* ratio of its hexagonal close packed (hcp) lattice [1,4] and the crystallographic texture [18,20]. This study investigates the effects of alloying elements (lithium), processing parameters (rolling temperature and annealing) and microstructural evolution (recrystallization and grain growth) on the texture of pure magnesium and the commercial AZ31 alloy.

2. Experimental procedure

Mg–1 wt% Li, Mg–3 wt% Li, AZ31 and AZ31–3 wt% Li were prepared in a Lindberg electrical resistance furnace under protective gas cover. The alloy compositions are listed in Table 1. The alloys were melted in mild steel crucibles at ~680 °C, and 42 mm diameter coupons were cast in copper moulds with heated risers. In order to complete additional rolling trials a 6 mm × 100 mm × 125 mm plate of AZ31 was cast into a tool steel mould preheated at 400 °C. All material was annealed in a SPX Blue M electric furnace for 8 h at 380 °C with an argon atmosphere. 6 mm × 15 mm × 30 mm test specimens were machined from the annealed material.

Specimens cut from coupons of all four alloys were rolled in seven passes to a true strain of 0.5 at temperatures of 150 and 350 °C using a laboratory-scale Stanat rolling mill. Specimens cut from the AZ31 plate were rolled in two passes to a true strain of 0.12 at 150 °C in order to observe microstructural evolution at lower strains and temperatures. All specimens were placed in a furnace for 15 min prior to rolling to allow them to reach temperature; between rolling passes specimens were replaced in the furnace for 3 min in order to regain temperature. Following the final rolling pass the specimens were immediately quenched in water. A section of the material rolled at 350 °C was also annealed for 10 min at 400 °C in an argon atmosphere and then quenched in water.

Microstructural characterization was performed on material following annealing for 8 h at 380 °C, and on specimens in the as-rolled, and in the rolled and annealed conditions. For optical microscopy specimens were cut using a diamond blade and mounted in resin. Mounted specimens were ground with 4000 grit SiC paper and mechanically polished using 6, 3 and 1 μm diamond paste. Final polishing was performed using 0.3 μm alumina solution. Specimens were etched for 1 s in acetic picral (4.2 g picric acid, 70 ml ethanol, 10 ml water and 10 ml acetic acid). Grain sizes were calculated using a mean linear intercept (MLI) average of 250 grains. Optical micrographs of the rolled material are viewed in the plane containing the RD and normal direction (ND); the RD is horizontal.

Electron backscattered diffraction (EBSD) presented in this study was carried out on the AZ31 plate specimens rolled to a true strain of 0.12 at a temperature of 150 °C. In specimens rolled to the higher strain of 0.5 the indexing levels were insufficiently high to generate meaningful orientation data. Following mechanical polishing, specimens were prepared for EBSD by electropolishing at 12 V for ~30 s in a solution of 30 ml nitric acid in 70 ml ethanol at −30 °C in order to remove surface strain. EBSD data was acquired using a Hitachi S-3000 variable pressure scanning electron microscope (VPSEM) and HKL Channel 5 EBSD software (HKL Technology, Denmark). The VPSEM was operated at 20 kV in high vacuum mode and the specimen was tilted at 70°. For orientation mapping the scan step size was set at 0.5 μm. EBSD orientation micrographs of the rolled material are viewed in the plane containing the ND and the transverse direction (TD); the TD is horizontal.

The crystallographic texture was measured in specimens rolled to a true strain of 0.5. For measurement of texture the specimen surface was ground until 40% of the sheet thickness was removed. The ground surface was polished with 6, 3 and 1 μm diamond paste, and then etched for 60 s in 10% nital in order to remove surface strain. X-ray texture analysis was performed using a Siemens D-500 diffractometer. Incomplete pole figures of (0 0 0 2), $\{1\,0\,\bar{1}\,0\}$ and $\{1\,0\,\bar{1}\,1\}$ were recorded, and the orientation distribution function (ODF) was constructed using TexTools, a texture analysis software. Recalculated pole figures were derived from the ODFs.

3. Results and discussion

3.1. Alloys and microstructures

The microstructures of Mg–1Li, Mg–3Li, AZ31 and AZ31–3Li coupons following annealing for 8 h at 380 °C are shown in the optical micrographs presented in Fig. 1a–d. As would be expected from the Mg–Li binary phase diagram [21], Mg–1Li and Mg–3Li are solid solution alloys. AZ31 and AZ31–3Li both contain precipitates; lithium additions alter the precipitate sizes and distributions in AZ31. MLI grain sizes of the homogenized material are shown in Table 2. Addition

Table 1
The compositions of Mg–1Li, Mg–3Li, AZ31 and AZ31–3Li (wt%)

Alloy	Mg	Al	Zn	Li	Mn	Fe	Si
Mg–1Li	Bal.	0.003	0.004	1.088	0.001	0.005	0.002
Mg–3Li	Bal.	0.003	0.004	3.034	0.001	0.005	0.002
AZ31	Bal.	3.098	0.842	<0.001	0.338	<0.001	0.016
AZ31–3Li	Bal.	3.052	0.858	3.081	0.304	<0.001	0.016

Table 2
MLI grain sizes following annealing for 8 h at 380 °C

Alloy	Grain diameter (μm)	S.D.
Mg–1Li	613	31
Mg–3Li	219	24
AZ31 (coupon)	73	11
AZ31 (plate)	150	19
AZ31–3Li	109	13