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Twin recrystallization mechanisms in magnesium-rare earth alloys

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ABSTRACT

Binary Mg-1 wt.% Gd and Mg-1 wt.% Ce wrought alloys, with basal type starting texture, are subjected to plane strain compression. Specially oriented specimens invoking *c*-axis extension (In Plane Compression) and *c*-axis compression (through thickness compression) were deformed to 6% and 13% respectively. Deformation in Mg-1Gd under in plane compression (IPC) led to evolution of sharp 'prismatic fiber' texture, whereas a weak basal texture was witnessed for Mg-1Ce. In case of through thickness compression (TTC), Mg-1Gd gave rise to a split basal component, whereas Mg-1Ce resulted in a weak basal component with large off-basal spread. Microstructurally, Mg-1Gd displayed unequivocal activation of both $\{10\bar{1}2\}$ tension twinning and $\{10\bar{1}1\}$ compression twinning during IPC, while profuse $\{10\bar{1}1\}$ compression twinning for the TTC mode. Majority of the compression twins underwent second generation $\{10\bar{1}2\}$ tension twinning to give rise to $\{10\bar{1}1\}$–$\{10\bar{1}2\}$ double twins. Compression and double twinned regions showed strong slip activity leading to the formation of fine recovered sub-structure. On the contrary, Mg-1Ce exhibited a conventional deformation response with twinning being predominantly of $\{10\bar{1}2\}$ tension type. Annealing led to preservation of the deformation texture components along with strong texture weakening. Recrystallization in Mg-1Gd commenced discontinuously in compression and double twins, which at higher temperatures showed favorable growth behavior consuming the still deformed matrix and tension twins. Mg-1Ce displayed continuous recrystallization marked by recovery and growth of orientationally soft tension twins along with nucleation in the vicinity of particles.

1. Introduction

In recent years, texture control in magnesium alloys via rare earth (RE) addition has gained considerable interest. The advantage of dilute addition of rare earths to obtain weaker and deformable textures in wrought magnesium alloys has been reported by many researchers [1–7]. Rare-earth related texture modification has been mostly associated with recrystallization and grain growth [2,7]. However, only certain recrystallization mechanisms have been known to display the desired departure from conventional sheet textures viz. particle stimulated nucleation [8], shear band nucleation [4,9] and deformation twin nucleation [6].

In a previous investigation [4], wrought Mg-RE alloys were subjected to severe shear banding during large strain rolling, and subsequently annealed at selected temperatures. Recrystallization resulted in nucleation of both basal and off-basal orientations. Subsequent nuclei and grain growth led to evolution of off-basal texture components at the expense of the as-deformed basal texture. The following key conclusions were drawn from the results:

(1) Texture modification was primarily triggered during recrystallization and grain growth. Off-basal grains indicated favorable growth kinetics over the basal-oriented grains.
(2) The choice of RE element is instrumental in determining the degree and extent of texture modification. In this respect, the binary Mg-1Gd alloy showed a strong texture medication potential as compared to the Mg-1Ce alloy.

In order to obtain a complete mechanistic understanding of deformation and recrystallization related texture evolution in Mg-REs, it is necessary to also understand the influence of RE alloying on the formation of twinning and the related recrystallization behavior.

Deformation twinning is a quintessential component during deformation of hexagonal close packed (HCP) materials with low crystal symmetry. Magnesium, being an HCP metal, is unable to provide sufficient slip modes to ensure homogeneous deformation at room temperatures [10,11]. In such cases, the mechanical response strongly relies on the role of twinning that compensates for strain imposed along the *c*-axis. However, due to its characteristic polar nature, twinning influences deformation in primarily two pathways: firstly, the twinned volume reorients the crystal,

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such that a mirror symmetry relative to the parent grain is created. Such reorientation in turn drastically modifies the crystallographic texture. Thus, the role of twinning in understanding the evolution of deformation texture in magnesium and its alloys is indispensable. Secondly, the rapid reorientation of the twinned region leads to the twin boundaries acting as strong barriers for further dislocation slip, thereby promoting work hardening [12]. However, in the presence of anisotropic slip behavior in conventional Mg alloys, such twinning induced hardening also acts as one of the primary mechanisms promoting failure. Fracture by means of tension twinning initiates as a result of the re-orientation of the *c*-axis toward the compression direction, whereby a hard non-deformable orientation (crystallographic hardening) is produced leading to subsequent void nucleation [7]. In case of compression and double twins, twinning aligns the basal planes favorably for basal slip, whereby the twinned regions undergo much more plastic deformation in comparison with the neighboring matrix. An energetic resistance of compression twin boundaries against growth and large difference in degree of strain accommodation between twin interior and matrix trigger high local stresses at the twin boundaries, leading to subsequent shear failure [10].

With regard to twinning induced recrystallization, potency of nucleation and related texture contribution is primarily determined by the twin type (tension or compression) and twin generation (primary or secondary) [13,14]. Studies on pure Mg single crystals tested under *c*-axis compression to trigger compression twinning revealed that recrystallization was of continuous nature based on extensive recovery, and that recrystallized grains had inherited the orientation of their twin host, which was obviously different from the initial matrix orientation [15]. Recent observations additionally suggested that recovery in twins occurs dynamically by simultaneous prismatic slip that rotates the new grains in the twin about their *c*-axes leading to subsequent randomization of the texture [16].

In the case of Mg-RE alloys, previous investigations [2,9,17,18] did not show perceptible distinctions in the twinning behavior over conventional Mg alloys, i.e. compression twinning was still less favorable than tension twinning when loaded accordingly. This aspect, however, remains a subject of debate in the Mg literature and it is yet to be determined whether twin recrystallization contributes to the RE-texture modification phenomenon. The current study investigates deformation twinning and subsequent recrystallization in binary Mg-1 wt.% Gd and Mg-1 wt.% Ce alloys. The results indicate that deformation twinning behavior in Mg-REs can indeed be modified in comparison with conventional cases, with competitive nucleation of both tension type and compression type twinning. However, such desired behavior is strongly based on the choice of RE-alloying element. Static recrystallization and grain growth preserves the deformation texture components and additionally promote strong texture weakening. The underlying mechanisms active during deformation, recrystallization and grain growth have been correlated with alloying chemistry and discussed within the purview of magnesium-rare earths.

2. Experimental procedure

Binary Mg-1Ce and Mg-1Gd (wt.%) alloys were produced as per Ref. [4]. Rolling blocks of dimensions 80 mm × 40 mm × 40 mm were machined from the cast materials and hot rolled at 450 °C (nominal furnace temperature) to a final thickness of 12 mm, imposing a total thickness reduction of 70% in seven passes. The rolling treatment was performed in order to achieve a suitable grain refinement and a well-defined sheet texture that was utilized later for obtaining specially oriented specimens for the deformation experiments. A following recrystallization treatment was conducted at specific annealing parameters, so that texture changes were insignificant, yet the microstructure was fully recrystallized and stress relieved. For each alloy different annealing conditions were used to achieve the aforementioned target, since Ce and Gd have a different potential for texture modification. Thus, Mg-1Ce was annealed at 400° C for 1 h and Mg-1Gd was annealed at 350 °C for 5 min, both followed by rapid quenching in water.

Deformation experiments were carried out in plane strain compression (PSC) at ambient temperature and a constant strain rate of $10^{-2}\ s^{-1}$. PSC was performed in a channel-die using a conventional screw-driven ZWICK testing machine. Hexagonal boron nitride powder was used as a lubricant to minimize friction between the die surfaces and the specimen. To evaluate the influence of texture on recrystallization and microstructure development during subsequent annealing, channel-die specimens with dimensions of 12 mm (longitudinal direction; LD) × 10 mm (transverse direction; TD) × 4 mm (compression direction; CD) were machined from the hot rolled alloys in two different orientations with respect to the PSC loading axis (c.f. Fig. 1d). TTC Specimens were compressed along the sheet normal direction 'ND' (through thickness compression), while IPC specimens were compressed along the sheet rolling direction 'RD' (in-plane compression). Deformation was conducted up to ~8% and 13% strain for IPC and TTC samples, respectively. The strains were chosen such that the deformed specimens exhibited strongly twinned microstructures. The stress–strain curves were obtained by means of automated measurement of the load and displacement with an average data acquisition frequency of 50 Hz. At least three tests were conducted for each loading orientation and alloy to ensure reproducibility of the stress–strain data. The results presented here show the average flow curves. For the recrystallization study the deformed specimens were annealed at four different temperatures ranging from 300 °C to 450 °C for one hour followed by water quenching.

Optical microscopy, X-ray diffraction (XRD) and electron backscatter diffraction (EBSD) measurements were utilized for texture and microstructural characterization. Specimens for optical microscopy were prepared by conventional grinding and diamond polishing (down to 0.25 μm), followed by electro-polishing in a 5:3 solution of ethanol and H_3PO_4 for 30 min at 1.93 V. Acetic picral and 5%-Nital were used as etchants to reveal the microstructure. XRD pole figure measurements were conducted using a Bruker D8 Advance diffractometer, equipped with a high resolution area detector, operating at 30 kV and 25 mA, using filtered iron radiation and polycapillary focusing optics. The measurements were performed on the specimen mid-plane to obtain the bulk texture and avoid any surface effects. The quantitative texture analysis toolbox MTEX [19] was employed to calculate the orientation distribution function (ODF) and full pole figures from the incomplete pole figure measurements. EBSD measurements were performed with a LEO-1530 scanning electron microscope (SEM) equipped with a field emission gun (operated at 20 kV) and an HKL-Nordlys II EBSD detector. Specimens for EBSD were subjected to electro-polishing in Struers AC-2 reagent using Lectro-Pol 5. For Mg-1Ce, a voltage of 26 V was applied for 25 s and for Mg-1Gd 41 V for 30 s. All specimen preparation techniques were conducted under ambient temperature conditions. The acquired raw EBSD data were subsequently analyzed using conventional EBSD analysis software and MTEX. The particle sizes of secondary phases were measured using a lineal intercept method from multiple SEM images. The area fraction was obtained using an image analysis software.