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The role of atomic scale segregation in designing highly ductile magnesium alloys

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ABSTRACT

Two solid solution binary magnesium-rare earth (RE) alloys, Mg-1wt.% Gd and Mg-1wt.% Dy were subjected to large strain hot rolling followed by recrystallization annealing at different temperatures for 60 min. Recrystallization and grain growth in Mg-1Gd led to stepwise change in the deformation texture, highlighted by a complete disappearance of the basal texture component developed during rolling. In case of Mg-1Dy, texture changes upon annealing were less accentuated, characterized by gradual softening of the deformation basal texture with increasing annealing temperatures. The development of favorable RE-textures during annealing is believed to be a result of solute segregation to planar defects, such as grain boundaries. It was observed using atom probe tomography that Gd atoms show a much higher grain boundary segregation tendency than Dy atoms. The enhanced segregation behavior of Gd manifests during annealing into a strong growth advantage of recrystallized off-basal orientations over basal orientations originating from the deformation microstructure, continuing as discontinuous grain growth at higher annealing temperatures and longer durations. No distinctive growth advantage was witnessed during recrystallization and grain growth of Mg-1Dy. Different segregation effects between the two RE elements and the resulting annealing texture evolution strongly impacted the room temperature tensile ductilities of both alloys. Mg-1Gd seemed to exhibit concomitant precipitation hardening effects, thereby displaying higher tensile strength along with enhanced ductilities. This work demonstrates the exciting possibility of being able to selectively exploit grain boundary characteristics using RE elements as an effective mechanism to tailor the microstructure during thermomechanical processing, thereby improve the mechanical performance of Mg alloys.

1. Introduction

Conventional wrought Mg alloys (e.g. AZ31) are typically characterized by sharp deformation textures associated with strong mechanical anisotropy. By contrast rare-earth (RE) containing Mg alloys develop modified textures with weaker intensities that contribute to the enhancement of formability at ambient temperatures and the reduction of mechanical anisotropy [1–3]. Such texture transition, as a result of RE alloying to Mg has been primarily attributed to recrystallization and grain growth phenomena, wherein evolution of new, softer orientations (i.e. more favorably oriented for basal slip) has been observed [1,4]. Such characteristics of Mg-RE alloys during recrystallization are quite interesting, since previous studies on conventional alloys have established that recrystallization is not effective in bringing forth qualitative changes in the deformation texture [5–7]. Although there have been quite a few theories on how RE elements might affect the development of recrystallization and grain growth textures in Mg-RE alloys [2,4,8–10], the principal mechanisms on how RE orientations nucleate in the deformation microstructure and subsequently grow during recrystallization are still not fully understood and remain subject to greater scrutiny.

Previous investigations on dilute binary Mg-RE alloys [11] suggest that the degree of observed texture modification depends strongly upon the choice of RE addition, as well as the material processing technique. It was found that recrystallization in shear bands [2,11–13], deformation twins [14–16] and in the vicinity of particles [10,12,14,17] are typically the most potent means of modifying the strong deformation texture in magnesium. For instance, shear band recrystallization and grain growth in solute

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based Mg-1wt.%Gd were found to result in significant texture changes unlike the precipitate-based Mg-1wt.% Ce alloy that revealed none or negligible deviation from the rolling deformation texture [11]. The observed texture changes in the presence of Gd were attributed to favorable growth of non-basal orientations having higher local stored energies and greater mobility, as a result of the higher angle boundaries between them and the deformed parent grains. By contrast, the lack of RE-induced texture modification in the Mg-1Ce alloy was primarily attributed to the detrimental role of secondary $Mg_{12}Ce$ phases, which were fine enough to pin the grain boundaries, and thereby, restrict any selective growth-related evolution of soft, non-basal orientations.

Numerous studies [9,12,18–24] attribute such drastic solute RE-related texture modification during recrystallization and grain growth to arise from the tendency of RE-segregation to stacking faults and grain boundaries. It is suggested that the latter retards dynamic recrystallization (DRX) by suppression of grain boundary migration, which can be considered a necessary condition to produce the RE texture [25]. DRX in extruded Mg-RE alloys have been known to result in strong texture modification and randomization, unlike in conventional extrusion Mg alloys, where the DRX texture often closely follows the orientation of the deformed grains that have their basal planes primarily aligned with the extrusion direction [2,25]. While RE-segregation to stacking faults can alter the stacking fault energies, thus influencing recovery and recrystallization [12,14,24], the importance of the segregation of RE atoms to grain boundaries causing solute drag lies in its role in retarding some recrystallization mechanisms more than others to allow oriented growth of RE nuclei within deformation heterogeneities, such as shear bands or compression twins [4,18].

The present work is an attempt to develop a mechanistic understanding of the RE effect activated at very low levels of solute addition. We consider two RE elements; Gd and Dy, with similar atomic size (Dy ~179 p.m. vis-à-vis Gd ~180 p.m.) and atomic weight (Dy ~163 u vis-à-vis Gd~157 u). As a result of their large atomic sizes, both elements would cause misfit strains in the matrix, which are relaxed as the atoms diffuse to grain boundaries. Despite the strong segregation potential of both RE elements, we also bear in mind that differences in the solubility in magnesium could limit the maximum grain boundary concentration of RE, and hence, influence the mechanisms of texture evolution during recrystallization and grain growth. This is shown in the results by comparing the textures and microstructures of both alloys at various annealing conditions. Finally, it will be shown that the effects investigated here are critical in steering alloy design towards a new class of highly ductile magnesium alloys.

2. Experimental procedure

2.1. Thermomechanical treatment

Binary Mg-1wt.% Gd and Mg-1wt.% Dy alloys were cast and homogenized as per Ref. [11]. Table 1 shows the respective chemical compositions obtained by wet chemical analysis. Rolling blocks of dimensions; 60 mm × 40 mm x 4 mm were hot rolled at 450 °C (nominal furnace temperature) to 80% thickness reduction in a single-rolling pass and rapidly water quenched. With a true strain of 1.6 ($\phi_h = \ln(h_1/h_0)$; h_1 and h_0 final and initial thicknesses), the single-pass approach enforced a severe plastic deformation condition that gave rise to significant shear banding. Subsequently, the rolled specimens were subjected to recrystallization annealing treatments at four different temperatures, viz. 300 °C, 350 °C, 400 °C and 450 °C, for duration of 60 min followed by water quenching. The annealing trials were conducted in a sand bath furnace with an average heating rate of ~70–80 °C/s.

Table 1
Chemical composition of the investigated alloys determined by wet chemistry techniques.

Alloy	Gd (wt.%/at.%)	Dy (wt.%/at.%)	Mg (wt.%/at.%)
Mg-1Gd	1.1/0.19	–	balance
Mg-1Dy	–	1.1/0.22	balance

2.2. Recrystallization and grain growth measurements

Recrystallization kinetics were studied by means of Vickers micro-hardness measurements with an applied load of 1N that were performed on rolled and annealed specimens at 325 °C for various holding times ranging from 5 s to 90 min. At least 5 hardness measurements were acquired for each annealing time (t). The corresponding recrystallized volume fraction (X_V) was subsequently calculated by the following expression,

$$X_V \frac{HV_0 - HV(t)}{HV_0 - HV_{final}} \tag{2.1}$$

where, HV_0 and HV_{final} are the initial and the final hardness values after annealing at 325 °C for a maximum duration of 90 min, $HV(t)$ is the hardness value measured for an intermediate annealing time ‘t’. In order to investigate anomalous grain growth behavior observed in the Mg-1Gd alloy, grain growth kinetics experiments were performed at 450 °C for varying annealing durations ranging from 4 to 1230 min.

2.3. Texture and microstructure characterization

Optical microscopy, X-ray diffraction (XRD) and electron backscatter diffraction (EBSD) measurements were utilized for texture and microstructure 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 grains and grain boundaries. 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 [26] 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 analysis were subjected to electro-polishing in Struers AC-2 reagent using Lectro-Pol 5. For Mg-1Dy, a voltage of 34 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 was subsequently analyzed using conventional EBSD analysis software and MTEX. Grain size measurements were performed using the linear intercept method and the deviation in mean values was reported in terms of standard error (S.E.). Phase identification was performed using energy dispersive X-ray spectroscopy (EDX).