Acta Materialia 135 (2017) 14–24

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

Full length article

Individual effect of recrystallisation nucleation sites on texture weakening in a magnesium alloy: Part 1- double twins

Dikai Guan, W. Mark Rainforth*, Junheng Gao, Joanne Sharp, Brad Wynne, Le Ma

Department of Materials Science and Engineering, University of Sheffield, Sheffield, S1 3JD, UK

ARTICLE INFO

Article history:
Received 11 March 2017
Received in revised form
1 June 2017
Accepted 7 June 2017
Available online 8 June 2017

Keywords:
Magnesium alloy
Recrystallisation
Deformation twin
Texture evolution
Precipitation

ABSTRACT

Recrystallised grain nucleation, grain growth and corresponding texture evolution in a cold-rolled rare earth containing WE43 Mg alloy during annealing at 490 °C was fully tracked using a *quasi-in-situ* electron backscatter diffraction method. The results show nucleation sites, such as double twins, can weaken the deformed texture and for the first time provide direct evidence that recrystallised grains originating from double twins can form the rare earth texture during annealing. Precipitation and recrystallisation occurred concurrently during most of the annealing period, with precipitates forming preferentially along prior grain and twin boundaries. These precipitates effectively retard the recrystallisation due to particle pinning leading to an excessively long time for the completion of recrystallisation. A large portion of recrystallised grains were observed to have ⟨0001⟩ poles tilted 20–45° away from the normal direction. The RE texture emerges during the nucleation of recrystallised grains and is maintained during subsequent uniform grain growth, which results in a stable RE texture being developed as recrystallisation progresses. The uniform grain growth could be attributed to solute drag suppressing the grain boundary mobility of those grains that had recrystallised with a basal texture and precipitate pinning restricting potential orientated grain growth.

1. Introduction

Pure Mg and Mg alloys commonly have a sharp basal crystallographic texture after cold or thermomechanical processing [1–5]. Subsequent annealing involving grain growth usually strengthens the texture intensity [6,7]. Various thermomechanical processing and alloying approaches have been employed to weaken the strong deformed texture [8–11]. The most encouraging discovery was that the addition of rare earth (RE) elements into pure Mg or conventional Mg alloy systems can effectively weaken the texture due to the appearance of a new texture component during thermomechanical processing and/or subsequent annealing [1,9,10,12–15]. Because this new texture component can be only found in Mg-RE alloys in most cases, it has been defined as “RE texture” [13]. Frequently reported RE textures are the $\langle 11\bar{2}1\rangle$//ED (extrusion direction) produced after extrusion, and peak texture intensity reduced and tilted towards the transverse direction (TD) after rolling and/or subsequent annealing [2,16,17].

RE textures normally reduce the basal texture intensity, anisotropy and asymmetry of Mg alloys. Consequently, these alloys can be deformed more homogeneously with improved formability [13,18]. However, the exact origin of RE texture is still a matter of debate. So far, numerous potential proposed mechanisms of RE texture formation have been reported. The factors in these hypotheses include the choice of preferential nucleation sites [2,4,9,12,13,19–21], increased activity of pyramidal <c+a> slip [5], oriented grain growth and solute drag or particle pinning along some specific grain boundaries (GBs) [22–25]. It is impossible to elucidate and validate all the potential mechanisms in one piece of experimental work. Therefore in this work, we only address the issue of which nucleation site(s) and/or subsequent growth of corresponding recrystallised grains is(are) directly related to the RE texture formation.

Deformed GBs, shear bands, deformation twins and second phase particles are the four proposed recrystallisation nucleation sites [10,20,22,26–28]. There is a consensus that the latter three sites can produce recrystallised grains with a wide range of orientations and randomise the basal texture [1,10,22,25]. However, there is no agreement which nucleation site is the main source that produces grains with RE texture orientations. Moreover, the growth of recrystallised grains at individual nucleation sites remains a

* Corresponding author.
E-mail address: m.rainforth@sheffield.ac.uk (W.M. Rainforth).

http://dx.doi.org/10.1016/j.actamat.2017.06.015
1359-6454/

challenging puzzle, and therefore the contribution that each makes to the full recrystallised texture is still a wide open question [1,10]. A variety of nucleation sites commonly coexist in Mg alloys after deformation, which makes it difficult to identify how recrystallised grains evolve from an individual nucleation site. Although particle stimulated nucleation (PSN) can be ignored in dilute Mg alloys or solid solution treated Mg alloys, deformation twins usually accompany shear bands in Mg-RE alloys where RE texture is also observed [18,21,25]. It is not clearly known whether recrystallisation in shear bands or twins is mainly responsible for RE texture formation. The unavoidable coexistence of twins and shear bands in most of the experimental cases makes it difficult to address this question. Therefore, to clarify the exact origin of RE texture, it is logical to design experiments to individually investigate the effect of shear bands and twins.

In our previous study [12] we showed that double twins (DTWs) can act as preferential nucleation sites for recrystallisation and the recrystallised grains originating from DTW-DTW and DTW-GB intersections can grow beyond the twin boundaries and expanded into deformed parent grains. Therefore, DTWs made the main contribution to the recrystallised texture, which contrasts the opposite view that has dominated in the past decades that recrystallisation in twins makes only a limited contribution [10,22,29–31]. Nevertheless, because the grain size was very large and therefore the examined area had only limited grains, the recrystallised texture may not have fully represented the bulk sample. Furthermore, some research reported the recrystallised texture also depends on subsequent growth of recrystallised grains [6,7,20,27,32], and so the debate remains: when does the RE texture orientation selection process occur [1,10]? During nucleation or the following grain growth stage?

To address these fundamental questions, we investigated the entire recrystallisation process from the as-cold rolled state to full recrystallisation condition of a Mg-RE alloy. The prior solid solution treatment and cold rolling process were intentionally adjusted to ensure that double twins were the dominant deformation feature in this work. We successfully employed a *quasi-in-situ* EBSD method to allow a site-specific method to track the recrystallisation process at each point in the microstructure and therefore to follow the nucleation at DTWs, subsequent recrystallised grain growth and texture evolution. To the best of our knowledge, this work for the first time provided direct evidence that recrystallised grains originating from double twins can form RE texture during annealing. The RE texture orientation selection took place during the nucleation process and grew uniformly during the subsequent grain growth stage. The individual role of shear bands on the texture weakening was excluded in the current work, but will be reported in a separate paper. These findings have filled some fundamental gaps mentioned in two recent review papers discussing the origin of RE textures [1,10] and could shed light on designing new wrought Mg alloys and optimising the thermomechanical processes to improve formability of commercial Mg alloys.

2. Experimental procedure

The alloy examined is the same as used in Ref. [12]. A rectangular plate 50 (ED) × 25 × 6 mm^3 was cut from the extruded bar for heat treatment and cold rolling. Solid solution treatment was carried out in a tube furnace with a continuous argon flow at 525 °C for 1 h (SST1H), followed by cold water quenching. The SST1H sample was then cold rolled with a reduction of 20% in one pass. The EBSD sample procedure can be found in Ref. [12].

EBSD was performed using a FEI Nano Nova 450 field emission gun SEM fitted with Oxford Instruments HKL NordlysMax^3 EBSD detector. The EBSD data were analysed via using HKL CHANNEL5 software. Fiducial marks were made on the surface of the cold-rolled sample after OPS polishing. Thus the area scanned for EBSD was relocated after further annealing to allow re-scanning of the same area. The *quasi-in-situ* EBSD collected data from a large area of about 1.4 mm^2 from the middle part of RD-ND plane. The EBSD scans were taken after cold rolling and after 5, 12, 21, 39, 90, 114, 163, 242, 341, 520, 920, and 1520 min annealing at 490 °C. The details of *quasi-in-situ* EBSD procedure were listed in Ref. [12].

3. Results

3.1. Microstructure after solid solution treatment and cold rolling

The as-received extruded sample microstructure was reported in Ref. [12]. Fig. 1 presents a large scale EBSD Inverse Pole Figure (IPF) map of the SST1H sample. The average grain size was found to be 82.3 μm. The inset (0002) pole figure (PF) indicated a majority of grains in the SST1H sample had their basal (0001) plane parallel to the ED. However, the intensity of 6.5 multiples of uniform density (mud) was lower than that in conventional non-RE Mg alloys [6,31,33].

Fig. 2(a) shows a backscattered SEM image of the SST1H sample after cold rolling for *quasi-in-situ* EBSD experiment subjected to 20% thickness reduction in one pass. Although the solution treatment time was only 1 h, nearly all the initial intermetallic compounds dissolved into the matrix except some sparsely distributed RE enriched particles, which were also shown in samples after 24 h solid solution treatment [12]. Therefore, the effect of second phase particles on the recrystallised texture would have been negligible. Moreover, Robson et al. [19] investigated the texture of the recrystallised grains originating from second phase particles via particle stimulated nucleation (PSN) appears to be random. Imandoust et al. [1] also stated the PSN recrystallisation mechanism could not be treated as a main source of RE texture, since PSN normally produces random texture.

Fig. 2(b) gives a corresponding band contrast (BC) image and shows that deformation twins can be clearly found in nearly all deformed grains. To identify twin types, the special boundary component has been superimposed. Fig. 2(c) presents the distribution of misorientation angles between neighbouring points in this map. In addition to the low misorientations associated with low angle GBs, there were two peaks around 38° and 86°

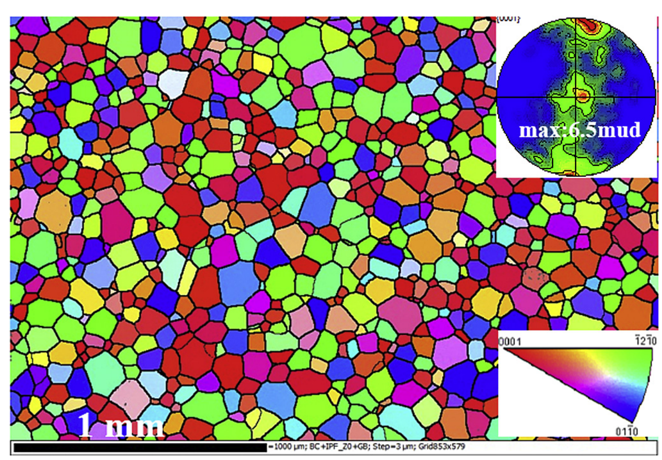

Fig. 1. EBSD IPF image of sample SST1H. The insets are corresponding (0002) pole figure and legend of IPF map. The extrusion direction (ED) is horizontal and observation along radial direction was applied to IPF triangle. (0002) pole figure is shown in the same orientation as the corresponding map.