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Texture evolution during static recrystallization of cold-rolled magnesium alloys

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ABSTRACT

Texture evolution in cold-rolled Mg-0.3Zn-0.1Ca, Mg-0.4Zn and Mg-0.1Ca (at.%) alloys during static recrystallization is monitored using a *quasi-in-situ* electron backscatter diffraction (EBSD) method. The *quasi-in-situ* EBSD results show that most of recrystallized grains formed in the early stage of recrystallization have randomised orientations in the ternary alloy and they grow uniformly during the recrystallization process. The formation and uniform growth of these recrystallized grains with randomised orientations give rise to a weak texture in fully recrystallized samples of the ternary alloy. A weak recrystallization texture also forms in the early stage of recrystallization in the two binary alloys, but it is gradually replaced by a strong basal texture via the preferential growth of recrystallized grains with specific orientations. The grain size in the ternary alloy is smaller than those in the two binary alloys at each stage of recrystallization, and the grain size distribution in the ternary alloy is significantly narrower than those in the two binary alloys after full recrystallization. Solute segregation to grain boundaries is observed in all three alloys in the fully recrystallized state. It is hypothesised that Zn and Ca atoms in the ternary alloy segregate strongly to high-energy boundaries of the recrystallized grains that would otherwise grow preferentially in the counterpart binary alloys, and that this co-segregation would significantly reduce the boundary mobility, by reducing grain boundary energy and enhancing solute dragging effect, and therefore lead to a more uniform growth of recrystallized grains with randomised orientations.

1. Introduction

Thermomechanically processed sheets of conventional magnesium alloys, such as AZ31 (Mg-3Al-1Zn-0.3Mn, wt.%), generally have a strong basal texture. Such a texture corresponds to the [0001] axis (*c*-axis) of most grains being nearly parallel to the normal direction (ND) of the sheet. Strong basal texture generally leads to unsatisfactory formability at near room temperatures, and therefore weakening the texture of Mg sheet alloys is a major area of research [1–3]. The addition of rare-earth (RE) elements such as Ce, Nd, Gd and Y, either individually or in combination, to Mg sheet alloys significantly weakens the recrystallization texture [4–12], and the combined addition of RE and non-RE elements, such as zinc, can generate an even weaker recrystallization texture than the single addition of RE [13–17]. Ca has been reported as a cheaper alternative to the more expensive RE, with its addition also resulting in a weakened texture after recrystallization and improved ductility and formability in Mg–Zn based sheet alloys [18–24]. When Ca (or RE) and Zn are added together to Mg sheet alloys, the most intensive texture poles are split and tilted towards the transverse direction (TD).

The weakened recrystallization texture has been proposed to be related to particle stimulated nucleation (PSN), shear band induced nucleation (SBIN), and deformation twin induced nucleation (DTIN). In studies of recrystallization behaviours of aluminium alloys [25,26] and steels [27], second-phase particles have been observed, and PSN was subsequently postulated to be the cause of weakened texture in these alloys [25]. In Mg alloys, particles have been reported to provide heterogeneous nucleation sites for recrystallized grains with randomised orientations, which results in a weakened texture after recrystallization [28,29]. Subsequent studies [30,31], however, have suggested that the texture

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weakening is not caused by second-phase particles, as texture weakening is also observed in dilute Mg sheet alloys that contain essentially no second-phase particles. Therefore, PSN is not likely to be the cause of texture weakening, particularly in solid solution alloys.

In plastically deformed metals and alloys, shear bands are narrow regions of intensive plastic deformation [32]. The recrystallization inside shear bands has been reported to occur in copper [33], aluminium [34] and steels [35]. In these metals, the orientations of recrystallized grains formed inside shear bands are case dependent—grains formed in shear bands in brass have randomised orientations [36], but grains formed in shear bands in aluminium have a strong texture [34]. In Mg and its alloys, the recrystallized grains formed inside shear bands have relatively randomised orientations [15,37–39], and the formation and growth of these grains lead to a weakened basal texture. Because a large fraction of shear bands is observed after plastic deformation in RE-containing magnesium alloys [5,40–42], the SBIN has been considered to be critical for the occurrence of the weakened recrystallization texture in RE-containing alloys. However, shear bands are also observed in RE-free magnesium alloys, such as AZ31 [42] and Mg-1wt.%Zn alloy [8], after plastic deformation, but these alloys have a strong basal texture after recrystallization. Therefore, the effectiveness of SBIN on texture weakening in bulk samples is still debatable.

In magnesium and its alloys, deformation twins have been reported to act as nucleation sites for recrystallized grains [43–46]. DTIN is based on the assumption that twins could provide nucleation sites for recrystallized grains with randomised orientations [47]. It has been reported that the RE addition resulted in the formation of more contraction and secondary twins, which provide more nucleation sites for recrystallized grains with randomised orientations [5,18,48,49]. Therefore, these studies suggest that DTIN is important to the development of weakened recrystallization texture in the RE-containing alloys. However, the recrystallised grains that nucleate inside twins do not grow beyond the twin size [50,51], therefore the effectiveness of DTIN on texture weakening in bulk samples is likely to be limited.

In addition, it has been proposed that the recrystallization texture is determined not merely by the orientations of recrystallized grains during nucleation, but also by the preferential growth of recrystallized grains of specific orientations [32,52]. The preferential growth of recrystallized grains with their *c*-axis nearly parallel to the ND has been reported in pure Mg [53] and AZ31 sheet [54,55], which were considered to contribute to the formation of a strong recrystallization texture. However, it is still unclear whether RE or Ca additions can suppress such preferential grain grow during static or dynamic recrystallization.

In order to address these issues, a *quasi-in-situ* electron backscatter diffraction (EBSD) method is used in the present study to monitor the texture evolution in the same area of cold-rolled samples of Mg-0.3Zn-0.1Ca, Mg-0.4Zn, and Mg-0.1Ca (at.%) alloys from the early stage (within a few seconds of annealing) to the full stage of static recrystallization. The Mg-0.3Zn-0.1Ca alloy is selected because the dilute Mg–Zn–Ca alloy sheet has been reported to have weakened recrystallization texture [18–24]. In addition, compared to the texture weakening in RE-containing Mg alloy sheets, the texture weakening in Ca-containing alloy sheets is less studied. For comparison with the texture evolution in the Mg-0.3Zn-0.1Ca alloy, the texture evolutions in Mg-0.4Zn and Mg-0.1Ca binary alloys are also examined, so that the influence of individual addition of Zn or Ca element on texture evolution can be revealed. The *quasi-in-situ* EBSD method is used for the first time to examine the texture evolution during static recrystallization in the same area of cold-rolled Mg alloys. In previous studies, the *quasi-in-situ* EBSD method has been used to examine the microstructural evolutions of Mg alloys during plastic deformation [56–58]. The examination of microstructure evolution in the same area of specimen during recrystallization were generally made using *in-situ* EBSD method [59–62], which has much more complex experimental setup and limitation on sample size than the *quasi-in-situ* EBSD method.

The primary purpose of this paper is to provide the results of systematic observation of texture evolution of Mg-0.3Zn-0.1Ca, Mg-0.4Zn and Mg-0.1Ca (at.%) sheets during static recrystallization, and on the basis of *quasi-in-situ* EBSD observations and analysis, an alternative interpretation is provided to explain the cause of texture weakening during annealing; allowing the relationship between solute drag of grain boundary and the weakened recrystallization texture to be discussed.

2. Experimental procedure

Three alloys, with nominal compositions of Mg-0.3Zn-0.1Ca, Mg-0.4Zn, and Mg-0.1Ca (all in at.%) were cast at 760 °C under argon atmosphere, followed by 400 °C homogenisation for 24 h. Homogenised samples for cold rolling were 3.25 mm in thickness. The thickness reduction per pass was about 0.1–0.15 mm. In total, six samples were prepared from the three alloys. One sample from each binary alloy was cold rolled to 23% thickness reduction. Four samples were sectioned from the ternary alloy. Three of them were cold rolled to 23% thickness reduction and the other was reduced by 40% thickness reduction. The as-cold-rolled samples were metallographically prepared using SiC paper, 50 nm diameter silica suspension, and subsequently argon ion beam polished using a Gatan precision etching and coating system (PECS).

EBSD was performed using a FEI Quanta 3D-FEG scanning electron microscope (SEM) equipped with a Pegasus Hikari EBSD detector. Fiducial marks were made on the specimen rolled surfaces, such that the area of the EBSD scan could be accurately re-analysed following each annealing step. The *quasi-in-situ* EBSD captured about 1.2 mm^2 of the rolled surface in the as-cold-rolled state, and of those after 5, 20, 40, 100, 300, and 900 s annealing at 350 °C. This annealing process was applied to the binary alloy bulk samples, and to the samples of ternary alloy rolled to thickness reductions of 23% and 40%. A sample of ternary alloy rolled to 23% thickness reduction was also separately annealed at 290 °C. Because recrystallization kinetics was expected to be slower at a lower annealing temperature, the EBSD scans were then taken after 5 min, 15 min, 1 h, 4 h, 20 h, and 68 h annealing. To investigate the static recrystallization in a shear band, EBSD scans were then taken after 14, 20, 28, 38, 50, 65, and 115 s annealing at 350 °C of a sample of ternary alloy rolled to 23% thickness reduction. After each EBSD scan, the samples were sealed in aluminium foil and annealed in a salt bath. After annealing, the samples were quenched in cold water and slightly polished using silica suspension, to remove any oxidised layer or tarnish in order to permit the next EBSD scan. The thickness reduction of samples, as a result of polishing, was less than 1 μm, measured by micrometre and/or SEM. The step size for large area EBSD scans was 1 μm, whilst a step size of 50 nm was used to investigate static recrystallization in shear bands. The results of EBSD scans were processed using TSL OIM 6 software, which was capable of showing grain orientations and grain sizes. The size of a grain was represented by the diameter, which was calculated based on the measurement of the area of this grain, $2\sqrt{\frac{Grain\ area}{\pi}}$. The average size of recrystallized grains in each annealed state was calculated by counting all recrystallized grains within the 1.2 mm^2 EBSD scan area.

In the *quasi-in-situ* EBSD method, the formation and growth of recrystallized grains in what are three dimensional (3D) bulk samples are represented by observations in 2D sectioned planes.