Journal of Alloys and Compounds 638 (2015) 267–276

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

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

Microstructure, mechanical behavior and low temperature superplasticity of ECAP processed ZM21 Mg alloy

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ARTICLE INFO

Article history:
Received 28 January 2015
Received in revised form 26 February 2015
Accepted 3 March 2015
Available online 10 March 2015

Keywords:
ZM21 alloy
Equal channel angular pressing (ECAP)
Ultra-fine grained (UFG)
Mechanical asymmetry
Superplasticity

ABSTRACT

In this study, ultra-fine grained ZM21 Mg alloy was obtained through two-stage equal channel angular pressing process (ECAP) at temperatures of 200 and 150 °C. For each stage four passes were used. Plastic behavior, mechanical asymmetry and low temperature superplasticity of ultra-fine grained ZM21 alloy were investigated as a function of processing condition with particular attention to microstructural and texture evolution. Microstructural observations showed that after the first stage of ECAP an equiaxed ultra-fine grain (UFG) structure with average size of 700 nm was obtained. Additional stage did not cause any further grain refinement. However, Electron Backscattered Diffraction analysis showed that the original extrusion fiber texture evolved into a new one featuring a favorable alignment of the basal planes along ECAP shear planes. Such a preferential alignment provided a considerably higher Schmid factor value of 0.32, resulting in a remarkable loss in tensile yield stress, from 212 to 110 MPa and an improvement of the tensile fracture elongation, from 24% to 40%. Tensile and compression tests at room temperature revealed that yielding asymmetry could be alleviated by either weakening of basal plane fiber texture or by grain refinement. Tensile tests at 150 °C showed that texture supplies a significant contribution to plastic flow and elongation, making dislocation slip the dominant mechanism for deformation, while grain boundary sliding was not actively operated at this temperature. However, at 200 °C the effect of texture on fracture elongation of UFG alloys was subtle and the impact of grain size became more important. Hence, UFG samples exhibited maximum elongation values exceeding 370% at a strain rate of $5.0 \times 10^{-4}\ s^{-1}$, confirming that the flow stress has notable texture dependence, while superplastic ductility was strongly influenced by grain size, being detectable only in UFG samples.

1. Introduction

Being the lightest commercial structural metal, with desirable damping capacity, good castability and recyclability, magnesium has been attracting much attention as a potential metallic material in a wide range of automotive, military, electronics and aerospace applications. However, owing to its hexagonal closed-packed (HCP) crystal structure providing limited number of active slip systems, magnesium exhibits poor formability at room temperature [1–3]. Generally, deformation of Mg at room temperature is mainly governed by basal slip $\{0001\}\ \langle 11\bar{2}0\rangle$ since critical resolved shear stress (CRSS) for the basal slip at room temperature is much lower than those for non-basal slip systems. Basal planes alone provide only two independent slip systems which are much less than the required five independent slip systems to satisfy the Taylor criterion [4], leading to poor ductility of Mg at room temperature. Previous investigations showed that with increasing of temperature, the CRSS of non-basal slip systems decrease gradually while that of basal slip remains constant. Therefore, improved formability of Mg at elevated temperatures (>180 °C) is associated with the activation of non-basal slip systems [5,6]. Moreover, limited active slip systems in Mg results in the formation of a strong crystallographic texture upon conventional thermo-mechanical processing such as rolling and extrusion. Therefore, mechanical properties of wrought Mg alloys are strongly influenced by texture orientation induced by previous forming processes [7–9].

In addition to dislocation slip, magnesium exhibits a strong tendency for twining, especially for $\{10\bar{1}2\}\ \langle 10\bar{1}1\rangle$ extension twin accommodating extension along the *c*-axis. Accordingly, twinning

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http://dx.doi.org/10.1016/j.jallcom.2015.03.029
0925-8388/

in Mg plays a key role in plastic deformation upon contribution to meet the Taylor criterion. Compared to metals with face-centered and body-centered cubic structures, Mg is a low-symmetry material having axial ratio (c/a) of around 1.633 in its crystal lattice [10,11]. As the axial ratio in Mg is less than $\sqrt{3}$, the activation of extension twin is achieved when tensile and compression stresses are perpendicular and parallel to the basal planes, respectively, producing marked tension–compression yield asymmetry [8,12–14]. Tension–compression yield asymmetry restricts Mg alloys from structural applications in which parts are simultaneously subjected to tension and compression stresses (e.g. in bending or in axial tension–compression cycles). Previous studies showed that the mechanical asymmetry in Mg alloys could be alleviated by several ways such as texture weakening [15], alloying elements [16], heat treatments [17] and grain refinement [15,18–20]. However, among the aforementioned approaches, texture and grain size give significant contributions to the mechanical asymmetric behavior.

In addition, it has been demonstrated that superplastic forming of Mg alloys plays a crucial role and represents a good opportunity for shaping of these materials into complex geometries and into curved parts, for subsequent use in a wide range of applications [21]. Thus, in order to expand their applicability, considerable effort has been devoted to improve the formability of Mg alloys.

Superplasticity generally occurs at high temperatures (above ~0.5 T_m, where T_m is the absolute melting temperature of the material), where the plastic flow mechanism is mainly diffusion-controlled. It is accepted that fine grains with size of less than 10 μm are required to obtain superplasticity since the dominant flow process is grain boundary sliding (GBS) [22,23]. Accordingly, several investigations have been carried out on tailoring the microstructure through various plastic deformation methods aimed at improving the superplastic ductility such as extrusion [24], equal channel angular pressing (ECAP) [25] and differential speed rolling (DSR) [26]. Thermo-mechanical processing techniques such as rolling and extrusion are generally used to achieve small grain sizes. However, these techniques cannot attain a grain size smaller than ~2–3 μm. In addition, the mentioned methods generally cause a strong texture in which the basal planes in most grains are distributed parallel to the rolling or extrusion directions, giving rise to deterioration of ductility.

In the last decades, microstructural refinement induced by severe plastic deformation (SPD) techniques has been attracting great attention owing to the possibility of achieving very high mechanical properties in Mg alloys. As a bulk processing method, ECAP is one of the most efficient SPD techniques being able to produce metallic materials with ultra-fine grained (UFG) structures by introducing massive strain during deformation [19,27–34]. Alloys with UFG structure featuring high-angle and non-equilibrium grain boundaries were found to be capable of GBS and exhibit marked superplasticity properties even at high strain rates [35] and/or at low temperatures [36].

In the present work, the possibility of fine tuning texture and properties in a Mg alloy was investigated in order to extensively improve plasticity. An UFG ZM21 Mg alloy with an equiaxed grain structure and particular basal plane orientation was produced through a multi-stage ECAP method showing very high superplasticity and limited mechanical asymmetry.

2. Experimental procedure

2.1. Materials and method

In the present work, a commercial ZM21 alloy provided by SHL-ALUBIN company (Mg–1.78Zn–0.89Mn, wt.%) in the form of extruded bar was investigated. Cylindrical specimens of 10 mm in diameter with a length of 100 mm were machined from the extruded bars for further processing. ECAP was conducted using a die featuring two cylindrical channels of 10 mm in diameter, intersecting at an angle of 110° and with an outer arc of curvature of 20°. According to the Iwahashi equation [37], this geometry involves an equivalent shear strain of 0.76 per each pass. ECAP processing consisted of two stages, at 200 and 150 °C, where 4 passes were performed. Table 1 summarizes the conditions investigated for the ZM21 alloy at the different ECAP temperatures. All the samples were subjected to repetitive pressings and rotated by 90° in the same direction between each pass according to the procedure designated as route B_C in the literature [38]. Samples were sprayed with MoS_2 lubricant and pressed into the ECAP die at a speed of 30 mm/min.

Table 1
ECAP processing conditions of the investigated samples.

Sample code	Sample condition
A0	As-extruded ZM21 Mg alloy
A4	ECAP treated for 4 passes at 200 °C
B4	A4 + ECAP treated for 4 passes at 150 °C

2.2. Microstructural and texture characterization

Microstructural examination of the all samples was characterized by Electron Backscattered Diffraction (EBSD) technique in a plane perpendicular to the pressing direction. In particular, distribution of grain size, crystallographic texture and grain orientation maps of the ZM21 alloy in the as-received condition and after ECAP process were obtained using EBSD technique interfaced with a Field Emission Gun Scanning Electron Microscope (FEG-SEM). All the data were then processed with TSL OIM™ software. The typical scan area was 30 μm × 30 μm with a 0.2 μm step size for the ECAP-processed specimens, while, in order to achieve good statistical data due to the presence of coarser grains, a larger scan area was selected for as-received alloy samples. Prior to EBSD analysis, all specimens' surfaces were carefully prepared by standard mechanical polishing followed by low-angle ion milling. The fracture surfaces of all samples at different temperatures of tensile tests were also examined using FEG-SEM.

2.3. Mechanical characterization

Mechanical properties were evaluated by tensile and compression tests. Standard deviations were calculated in each case out of a population of three experiments. Following ASTM E8-04 [39] tensile specimens with a gage length of 12 mm and a diameter of 4 mm were machined along the longitudinal direction of the ECAP billets and tested at room temperature (RT) and at 150 °C. Moreover, characterization of the superplastic behavior was carried out at 200 °C with strain rates ranging from $5.0 \times 10^{-4}\ s^{-1}$ to $1.0 \times 10^{-2}\ s^{-1}$. Compression tests were carried out at room temperature at a strain rate of $1 \times 10^{-3}\ s^{-1}$on cylindrical specimens with diameter of 10 mm and height of 20 mm according to ASTM E9-09 specifications [40], cut along the axial direction of the as-extruded and ECAP processed billets.

3. Results and discussion

3.1. Microstructural characterization

Fig. 1 shows EBSD maps and corresponding grain size distribution of all the investigated samples. The color code of all the figures is given by the reference sector at the bottom left corner of the images. As seen in Fig. 1a and b, the extruded samples (A0) exhibit a heterogeneous microstructure with a bimodal grain size distribution with different coarse-grain sizes (5–60 μm) and contain considerable fraction of grains ranging from 10 to 30 μm. However, after the first stage of ECAP (A4 samples) the initial grain structure was remarkably refined, resulting in a microstructure consisting of fine and equiaxed grains with a size distribution from 0.3 to 3 μm and an average grain size of 0.7 μm (Fig. 1c and d). Thus, it should be noted that ECAP process led to a considerable grain refinement in the alloy through dynamic recrystallization (DRX) mechanism. Interestingly, as depicted in Fig. 1e and f, with an additional 4 ECAP passes at 150 °C (second stage of processing, B4 samples) not only no further grain refinement occurred, but also a slight grain growth was observed, from 0.7 to 0.9 μm. The observed grain coarsening can be attributed to the relatively high interpass time spent at processing temperature and lower propensity to recrystallization given by the small grain size structure. A