Journal of Alloys and Compounds 643 (2015) 28–33

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

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

Enhanced mechanical properties and rolling formability of fine-grained Mg–Gd–Zn–Zr alloy produced by equal-channel angular pressing

Fumin Lu [a], Aibin Ma [a,b,*], Jinghua Jiang [a,c,*], Jing Chen [a], Dan Song [a], Yuchun Yuan [a], Jianqing Chen [a], Donghui Yang [a]

[a] College of Mechanics and Materials, Hohai University, Nanjing 211100, China
[b] Jiangsu Collaborative Innovation Center of Advanced Micro/Nano Materials & Equipment, Nanjing University of Science and Technology, Nanjing 210094, China
[c] Jiangsu Key Laboratory of Advanced Structural Materials and Application Technology, Nanjing 226000, China

ARTICLE INFO

Article history:
Received 14 January 2015
Received in revised form 11 April 2015
Accepted 18 April 2015
Available online 24 April 2015

Keywords:
Mg–Gd–Zn–Zr-alloy
Equal-channel angular pressing (ECAP)
Grain refinement
Rolling
Mechanical property

ABSTRACT

A fine-grained Mg–1.8Gd–1Zn–0.1Zr (at.%) alloy with long-period stacking ordered (LPSO) phase was obtained via solid-solution(SS) treatment plus multi-pass equal-channel angular pressing(ECAP). The effects of post-ECAP rolling on microstructure change and deformation characteristic of the Mg alloy were investigated. The results showed that the fine-grained alloy after 16 ECAP passes at 658 K had a yield strength of 334.4 MPa with an elongation of 22.5%. Grain refinement with LPSO formation simultaneously improved the strength and ductility of the ECAPed alloy, indicating a good plastic formability. The ECAPed Mg sheet was easily rolled at 773 K from 1.5 mm to 0.24 mm in thickness without edge cracking. After rolling, the fine-grained Mg alloy exhibited higher tensile strength with appropriate elongation. The post-ECAP rolling has been successfully used in the high productivity of Mg thin sheet with good mechanical properties.

1. Introduction

Lightweight design in aerospace, automobiles and electronics industries has become more and more important, owing to urgent demands for energy saving and environment protection. During the past decades, Mg alloys have received a tremendous amount of attention due to their low density and high specific strength [1,2]. However, their applications are limited since conventional Mg alloys are still insufficient in mechanical properties and corrosion resistance. The development of new casting Mg alloys with excellent mechanical properties for near-net shape products are strongly desired. The production and engineering application of Mg thin sheets required the better formability at lower temperature and reduced anisotropy of mechanical properties [3,4].

Rare-earth (RE) alloying (including Y [5–7] and Gd [8,9]), heat treatment and plastic deformation (such as extrusion and rolling [10,11]) have been found to be the effective approach to enhance the strength, ductility and formability of Mg alloys. Recently, many Mg–Zn–RE system alloys containing Y and/or Gd have been developed and exhibit good mechanical properties after plastic forming. Peng et al. [12] reported that the Y addition to Mg–7Gd alloy could refine the microstructure and increase the precipitation with improving aging–hardening behaviors. Yamasaki et al. [13] found that a hot extruded Mg–2.3Zn–14Gd (wt.%) alloy could precipitate 14H-type long-period stacking order (LPSO) structure during aging at 623 K, thus simultaneously improving the proof strength and elongation. Many previous investigations discover that the evident grain refinement, the precipitation of intermetallic phases and LPSO structures are important factors for superior mechanic properties of the newly-developed Mg–Zn–RE system alloys [14,15].

Grain ultrafining by severe plastic deformation (SPD) is a promising direction for improving the formability of Mg alloys. At present, equal-channel angular pressing (ECAP) is the most developed SPD method to produce bulk fully-dense ultrafine-grained (UFG) metallic materials [16,17]. It is usual that multiple ECAP passes are required to obtain the sound UFG microstructure with uniform second phases. In general, the ECAP-fabricated (ECAPed) metals demonstrate the mechanical advantages of superior strength at room temperature and enhanced superplasticity at relatively low temperatures near $0.5T_m$ (melting temperature in Kelvin). These maybe endow the high productivity if the ECAPed UFG metals are used to produce basic parts in many branches of mechanical engineering. Thus, an interesting attempt has been made to examine the effect of rolling after ECAP on the

* Corresponding authors at: College of Mechanics and Materials, Hohai University, Nanjing 211100, China. Tel.: +86 25 8378 7239; fax: +86 25 8378 6046.
E-mail addresses: aibin-ma@hhu.edu.cn (A. Ma), jinghua-jiang@hhu.edu.cn (J. Jiang).

http://dx.doi.org/10.1016/j.jallcom.2015.04.118
0925-8388/

microstructure and mechanic properties of ECAPed UFG Al–Mg alloys [18,19]). While a few works were reported on the microstructure and mechanical properties of the semi-products from Mg alloys subjected to ECAP followed by rolling.

The combined process of ECAP and conventional rolling can be expected to fabricate UFG Mg sheets or plates with good mechanical properties and high productivity. In view of the practical interesting of the ECAPed UFG Mg–Zn–RE system alloys, we designed a new Mg–Gd–Zn–Zr alloy and then studied microstructure changes and mechanical behaviors of the Mg alloy subjected to ECAP plus rolling.

2. Experimental

A new Mg–1.8Gd–1Zn–0.1Zr (at.%, hereinafter referred to as GZ11 K) alloy was melted from high purity Mg (>99.95%), high purity Zn (>99.95%), Mg–31Gd (wt.%) and Mg–33Zr (wt.%) master alloys in an electric resistance furnace. After holding at 1013 K for 30 min with a shielding gas mixture of SF_6 and CO_2, the melt was cast into a preheated mild steel mold of 200 mm × 22 mm × 100 mm in size. The casting ingots were solution-treated at 773 K for 24 h and quenched to room temperature in water. Then the solution-treated alloy was cut into the ECAP billets with dimensions of 40 mm × 19.5 mm × 19.5 mm.

The ECAP process was performed using a rotary die with $\varphi = 90°$ (inner arc of curvature) and $\psi = 0°$ (outer arc of curvature) [20]. The billets were subjected to 16 passes of ECAP at 658 K with the plunger speed of 12 mm/min. After ECAP, the rolling samples (dimensions: 30 mm × 10 mm × 1.5 mm) were taken from the center of the ECAP billets with the rolling plane parallel to the pressing direction. The rolling process was performed at the rolling rate of 20 mm/s and under room temperature (RT), 673 K and 773 K, respectively. During hot rolling, the samples were reheated in a furnace and kept for 5 min prior to each rolling pass. The reduction per rolling pass was 10%. After each pass of rolling, the samples were rotated by 180° with a constant perpendicular direction.

The tensile tests were carried out at room temperature and an extension rate of 0.18 mm/min using a TF50S electronic universal testing machine. The dog-bone flat specimens with the dimensions of 10 mm × 5 mm × 2 mm were machined from the ECAPed and ECAP + Rolling samples with the tensile axis parallel to the pressing or rolling direction.

The microstructures of the as-cast, ECAPed and ECAP + Rolling samples were investigated by an optical microscopy (OM, Olympus BX51), a scanning electron microscope (SEM, JEOL 6500) with energy dispersive spectroscope (EDS) and a transmission electron microscope (TEM, JEOL-2000 EX). The samples for OM and SEM observation were prepared by a conventional mechanical polishing technique and etched with a picric (5 g) – acid(10 ml) – ethanol(80 ml) – H_2O(10 ml) solution at room temperature. All of the samples were cut along the pressing/rolling direction and then observed by OM/SEM. A linear intercept method was used to measure the average grain size of these alloys. Thin foils for TEM observations were prepared with a twin-jet method. The phase constitutes were analyzed by X-ray diffraction (XRD, BRUKER D8). The tensile fracture surface of the as-cast, ECAPed and ECAP + Rolling samples were also observed by SEM.

3. Experimental results and discussion

3.1. Effect of ECAP on microstructures

Fig. 1(a and b) shows OM and SEM micrographs of the as-cast GZ11 K alloy. It can be seen that the as-cast alloy are mainly composed of equiaxed dendrites α-Mg matrix with networks eutectic compounds at the grain boundaries. As shown in our previous paper [21], the XRD and EDS analysis of the as-cast alloy suggest that the intergranular eutectic compound is the second phase of Mg_5(Gd, Zn). Some fine lamellar phases uniformly distribute from grain boundaries to the interior of α-Mg grains. Many previous investigations discovered the 14H-LPSO structure in Mg–Zn–Gd alloys [22–24]. And likewise, the lamellar phases in the as-cast GZ11 K alloy were identified by TEM as the 14H LPSO structure (Fig. 1(c)). After solution treatment at 773 K, some globular Mg_5(Gd, Zn) phases were remained in the α-Mg matrix and the lamellar 14H LPSO structure also existed (Fig. 1(d)).

Fig. 2 presents optical, SEM and TEM micrographs of the ECAPed GZ11 K alloys at 658 K for 16 passes. The deformed grains of the 16-pass sample are relative fine and homogeneous. The original lamellar 14H LPSO structure also existed but formed kink bands (indicated by red circle in Fig. 2c) in the deformed matrix. Moreover, plenty of equiaxed small grains having grain size of 1–2 μm could be found in the α-Mg matrix (Fig. 2d). During the warm ECAP process, the dislocations can continuously evolve into dislocations tangles, dislocation cells, dislocation walls and subgrain boundaries, finally form new grain boundaries to create new refined grains [21,25]. It indicates that besides grain refinement of the α-Mg phase, the redistribution of broken Mg_5(Gd, Zn) phases and the fine-lamellae 14H-LPSO structure are important feature of microstructure evolution of the alloy subjected to multi-pass ECAP process.

3.2. Reduction rate and microstructure after rolling

Table 1 lists the total reduction of the SS samples and the 16-pass ones rolled at different temperature without edge crack. It is obvious that the total reduction of two kinds of GZ11 K samples increase with raising the rolling temperature, but the increment of the SS sample is less than the ECAPed one. Although the total reduction of the 16-pass sample after rolling at room temperature is minimum (only about 13%), the value after rolling at 773 K is maximum. Table 1 implies that grain refinement of the new Mg alloy affects its governing deformation mechanism during the rolling at different temperature. The better plastic formability of the fine-grained Mg alloy at higher rolling temperature reveals the combined deformation of grain boundary sliding and dislocation viscous glide [20].

Fig. 3 presents optical micrographs of the rolled GZ11 K samples at the total reduction without edge crack. After room-temperature rolling, the remained eutectic compounds of the SS alloy were almost distributed along α-Mg grain boundaries (in Fig. 3(a)). A comparison of Fig. 3(b) with Fig. 3(c) shows that raising the rolling temperature is propitious to fine crystal of the Mg alloy sheets. After hot rolling at 773 K, the smaller equiaxed α-Mg grains were formed in the deformed matrix. The rolling at 773 K obviously reduced the grain size of the α-Mg matrix, resulting in a more homogenous microstructure. Compared with the SS + Rolling sample, the average grain size of the ECAP + Rolling sample is considerably fine. It indicates that the previous warm-ECAP processing is propitious to high mechanical properties of the rolled Mg alloy sheet.

3.3. Mechanical properties

Fig. 4 presents engineering stress–strain curves of the Mg alloy in the state of SS, 16-pass ECAP and 16-pass ECAP + RT rolling. Table 2 illustrates mechanical properties of the SS sample and the 16-pass one with/without RT rolling. For the purpose of comparison, two similar alloys reported in the previous work [13,26] are also listed.

It is obvious that performing 16 passes of ECAP and post-rolling at room-temperature increase the U.T.S. value of the SS sample from 256.6 MPa to 374 MPa and 410.8 MPa, respectively. Grain refinement via multi-pass ECAP process simultaneously increased both strength and ductility of the Mg alloy, which resulted in the significant improvement of reduction in size after subsequent hot rolling. The RT rolling further improved the U.T.S. value of the Mg alloy, and the elongation of the 16-pass sample after RT rolling reached 9.92%. In general, the sample rolled at a higher temperature has a lower U.T.S. value but a higher reduction. The fine-grained Mg sheet for 16 ECAP passes was successfully rolled at 773 K into a thickness of 0.24 mm without edge cracking, resulting in a high reduction of 77% (in Table 1). This fact indicates that ECAP can develop the fine-grained microstructure with the better plastic capacity at high temperature, and that the governing deformation mechanism may be altered by post-rolling at different temperature