Materials Characterization 136 (2018) 310–317

Contents lists available at ScienceDirect

Materials Characterization

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

Effect of high content of manganese on microstructure, texture and mechanical properties of magnesium alloy

Zhengwen Yu[a,b,*], Aitao Tang[b,*], Jiejun He[b,c], Zhengyuan Gao[d], Jia She[b], Jianguo Liu[a], Fusheng Pan[b]

[a] Special Key Laboratory of Oral Disease Research, Higher Education Institution in Guizhou Province, Zunyi Medical University, Zunyi 563003, PR China
[b] College of Materials Science and Engineering, Chongqing University, Chongqing 400044, PR China
[c] College of Materials and Metallurgy, Guizhou Institute of Technology, Guiyang 550003, PR China
[d] School of Mechatronics and Automotive Engineering, Chongqing Jiaotong University, Chongqing 400074, PR China

ARTICLE INFO

Keywords:
Magnesium-manganese alloys
Microstructure
Texture
Mechanical property
PSN

ABSTRACT

In the present investigation, microstructure, texture and mechanical properties of Mg-3Mn alloys fabricated by hot extrusion process with the extrusion temperatures of 250 °C and 300 °C were investigated. The results revealed that the samples exhibited the refined microstructure and weakened basal texture. Refined microstructure of the samples was attributed to the dynamic precipitates suppressing the recrystallization grain growth during extrusion process. Weakened basal texture of the samples was explained as a result of particle stimulated nucleation (PSN) promoted by coarsened Mn precipitates altering the texture of extruded profiles during recrystallization by orienting the c-axis of grains at an angle that favors basal slip activity. As a result, the samples exhibited the high tensile elongation and good yield strength at room temperature.

1. Introduction

Magnesium and its alloys have received great attention in the past decades due to their low density, good thermal conductivity, high electromagnetic shielding characteristics and excellent recyclability. These outstanding properties can significantly contribute to the aspect of weight saving in the design and construction of cell phones, automotive, aerospace components and electronic products [1–3]. However, extensive application of Mg alloys has been limited because of their poor ductility and insufficient strength at room temperature [4]. The poor performance of Mg alloys can be explained as their special *hexagonal close-packed* (hcp) crystal structure and limited number of slip systems at room temperature.

Grain refinement and precipitation hardening have great effect on improving the strength of Mg alloys at room temperature [5–8]. In previous work [9–11], zirconium additions exhibited the obvious effect on refining the microstructure of Mg alloys, such as Mg-Zn and Mg-RE series alloys. Others [12,13] also found that alloying with Mn rather than Zr could effectively refine the microstructure of Mg-RE extrusion profiles, leading to an obvious enhancement of strength. However, recent studies [14–16] figured out that the addition of Mn exhibited the negative effect on refining the microstructure of Mg-1Mn (M1) alloys subjected to the different kinds of extrusion temperature, exhibiting the extremely poor performance at room temperature because of the coarsened microstructure and intensified basal texture. As a result, Mg-Mn alloys have not been widely considered as the structural applications, although the alloys show the ultrahigh damping capacity [17], good corrosion resistance [18] and excellent creep behavior [19].

Recently, investigations [20,21] found that an amount of second phases precipitated prior to extrusion process could potentially be more obvious on improving the mechanical properties of extrusion profiles, because fine precipitates can refine the microstructure of extrusion Mg alloys by impeding the growth of dynamically recrystallized grains during extrusion process. Jung et al. [22] have already proved that the undissolved phases distributed along the recrystallized grain boundaries effectively refined the microstructure of AZ80 alloy during extrusion process, leading to a marked increase in strength and elongation. Additionally, our previous investigation [23] also found that the existence of a high density of fine Mn precipitates prior to extrusion obviously could refine the microstructure of M1 alloy subjected to low extrusion temperature of 250 °C, leading to an obvious improvement in strength and elongation. Therefore, as to further confirm the grain refinement effect of Mn addition on Mg alloys, a high concentration of 3.0 wt% Mn is applied in the present investigation, aiming at understanding how Mn precipitates prior to extrusion affect the microstructure, texture and mechanical properties of as-extruded Mg alloys.

* Corresponding authors at: Chongqing University, Chongqing, PR China.
E-mail addresses: zwyu@zmc.edu.cn (Z. Yu), tat@cqu.edu.cn (A. Tang).

https://doi.org/10.1016/j.matchar.2017.12.029
Received 19 July 2017; Received in revised form 22 November 2017; Accepted 19 December 2017
Available online 20 December 2017
1044-5803/

Table 1
Chemical composition of M3 alloy (wt%).

Alloys	Mn	Fe	Si	Others	Mg
M3	2.971	0.001	0.002	< 0.010	Bal.

2. Experimental Procedure

Commercial pure magnesium (99.98 wt% Mg) and Mg-Mn master alloy containing 4.04 wt% manganese were applied to synthesize the experimental samples. The testing alloy Mg-3.0Mn, hereafter named M3 alloy, was melt in an electric resistance furnace under a mixed atmosphere of CO_2 and SF_6. Molten metal was stirred and held on about 20 min at 740 °C. Then it was poured into a preheated (300 °C) steel mold. The dimensions of the ingots were 100 mm in diameter and 150 mm in length. The billets covered with graphite were homogenized at 500 °C for 24 h and then water quenching immediately. The billets were preheated to 250 °C and 300 °C, respectively, for 2 h and extruded in an XJ-500 horizontal extruder. Solid rods, approximately 16 mm in diameter, corresponding to an extrusion ratio of 25:1, were extruded at the extrusion rate of 4 mm s^{-1} and air cooled.

As to confirm whether the high melting point metal Mn has been added into the M3 alloy, X-ray fluorescence spectrometry (XRF) was employed to inspect the actual chemical composition of the experimental samples shown in Table 1. XRF result indicated that Mn alloying element was successfully added into the experimental samples.

Phase constitution and texture were determined by Rigaku D/max-2500 X-ray diffraction (XRD), using Cu Kα radiation (wave length λ = 0.15406 nm) at 45 kv and 150 mA with a sample tilt angle ranging from 20° to 80°. Texture analyses of the extrusion bars were carried out for the (0002), $(10\bar{1}0)$, $(10\bar{1}1)$ and $(10\bar{1}2)$ planes. The measurements were performed on the cross sections of the specimens. Differential scanning calorimetry (DSC) was tested by NETZSCH STA 449C system. Samples weighted around 30 mg were heated in a flowing argon atmosphere from room temperature to 700 °C for 5 min before being cool down to 100 °C. The cooling curve was recorded at a controlling speed of 10 °C/min.

Samples were ground and polished by standard metallographic preparations. Afterwards, specimens were etched in a solution containing 20 ml acetic acid, 6 g picric acid, 2 ml H_2O and 100 ml ethanol. Zeiss optical microscope was applied to observe the microstructure of extruded profiles.

Transmission electron microscopy (TEM) foils of the samples were prepared by twin-jet electro polishing using a solution of 5.3 g LiCl, 11.6 g $Mg(ClO_4)_2$, 500 ml methanol and 100 ml 2-butoxy-ethanol at approximately −50 °C and 90 V. The specimens were surface cleaned by ion milling using a Gatan Precision ion Polishing System (PIPS) at an operating voltage of 2 kV.

Room temperature mechanical properties of the samples were tested using a universal material testing machine for tension and compression (SANS CMT-5100). Tensile (5 mm diameter and 25 mm gauge length) and compressive (8 mm diameter and 12 mm height) test specimens were machined parallel to the extrusion axis from the extruded rods and pulled to failure with a strain rate of 3 mm/min. At least three samples were taken from different locations of the extruded rods. The tension and compression axes were kept parallel to the extrusion axis.

3. Results

3.1. Phase Component

XRD patterns of both as-cast and as-extruded M3 alloys have been examined shown in Fig. 1. XRD pattern of as-cast M3 alloy shown in Fig. 1a indicates that phase constitutions are α-Mg matrix and Mn precipitates, respectively. None of the Mg-Mn intermetallic compounds are found in this paper. The result is consistence with the previous work [24]. In addition, extrusion process, such as temperature, strain rate, extrusion ratio et al., rarely changes the phase components of Mg alloys [25]. Therefore, cross section of M3 alloy extruded at 300 °C are selected to examine the XRD pattern shown in Fig. 1b. The result demonstrates that the main phases of as-extruded M3 alloy are α-Mg and α-Mn phases. Due to the limited solid solubility of Mn alloying element in Mg matrix shown in Fig. 1c, none of the intermetallic compounds are formed between magnesium and manganese in M3 alloy extruded at both 250 °C and 300 °C. Fig. 1d shows the DSC cooling curve of as-cast M3 alloy in this paper. As clearly observed, the alloy has four main peaks at about 687.2 °C, 678.0 °C, 659.7 °C and 640.1 °C, respectively, corresponding to the pseudo peritectic reaction or phase transformation temperatures.

3.2. Microstructure

Fig. 2 shows the microstructure of both as-cast and as-extruded alloys in the present work. In Fig. 2a, as-cast pure Mg shows the extremely coarsened microstructure with the average grain diameter of ~1481.5 μm. Previous investigation [26] figured out that Mn exhibited the extremely poor grain refinement effect on as-cast Mg alloys with its lowest grain growth restriction factor (GRF) value of ~0.15, compared with those alloying elements, Ti [27], Fe [28], Zr [26] et al., showing the obvious effect on refining the microstructure of as-cast Mg alloys. However, microstructure of as-cast M3 alloy has been refined compared with as-cast pure Mg, and the average grain diameter is ~741.8 μm shown in Fig. 2b.

Metallography of deformed, *e.g.* extruded, rolled, forged, Mg-Mn alloys is difficulty to be revealed due to their poor response to etching [29]. According to the previous investigation [30], there were no significant change in average grain size of Mg-Mn alloys when annealing at 300 °C for 1 h. As to clearly reveal the microstructure of the samples in the present work, therefore, both of the extruded samples are annealed at 300 °C for 1 h, and microstructure of the samples are shown in Fig. 2. The samples exhibit a bimodal microstructure, consisting of equiaxed recrystallized grains and unrecrystallized regions indicated by the arrows in Fig. 2c and Fig. 2d. The volume fraction of unrecrystallized regions in M3 alloy extruded at 250 °C is ~7.8% shown in Fig. 2c, and the average recrystallized and unrecrystallized grain sizes are 1.1 μm and 4.8 μm, respectively. With the extrusion temperature increasing to 300 °C, the volume fraction of unrecrystallized regions, average recrystallized and unrecrystallized grain sizes are ~9.2%, 5.3 μm and 9.4 μm shown in Fig. 2d, respectively.

Fig. 3 shows the SEM images of both as-cast and as-extruded M3 alloys. Obviously, both of the samples show the similar phase constitutions. In Fig. 3a and Fig. 3b, an amount of coarsened Mn-enriched particles, identified by EDS inset of Fig. 3a and marked by the arrows in Fig. 3b, are observed locating among the matrix and along the grain boundary. Fig. 3c and Fig. 3d show the SEM morphologies of M3 alloy extruded at 250 °C. As clearly seen from the figures, a large number of finer Mn-enriched particles confirmed by EDS inset of Fig.3d are observed, and only a small quantity of coarsened particles containing Fe and Si are observed in Fig. 3c. The coarsened particles have been commonly observed in Mg alloys [18]. This can be estimated as the major factor of Mn addition on removing the impurities, and then improving the corrosion resistance of Mg alloys [31].

3.3. Texture

Texture in forms of (0002), $(10\bar{1}0)$, $(10\bar{1}1)$ and $(10\bar{1}2)$ pole figures of M3 alloys extruded at both 250 °C and 300 °C are analyzed to assess the grain orientation evolution shown in Fig. 4. Both of the samples exhibit the unusual fiber texture. In Fig. 4a, M3 alloy extruded at 250 °C shows the texture with ⟨0001⟩ axis of the Mg matrix tilting nearly 40° away from the radial direction, and a maximum texture intensity of 1.606.