Journal of Alloys and Compounds 491 (2010) 165–169

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

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

Grain refining efficiency of a new Al–1B–0.6C master alloy on AZ63 magnesium alloy

Guolong Ma, Guang Han, Xiangfa Liu*

Key Laboratory for Liquid-Solid Structural Evolution and Processing of Materials, Ministry of Education, Shandong University, Jinan 250061, PR China

ARTICLE INFO

Article history:
Received 14 August 2009
Received in revised form 23 October 2009
Accepted 26 October 2009
Available online 30 October 2009

Keywords:
Al–1B–0.6C master alloy
Al_3BC
Grain refinement
AZ63 magnesium alloy

ABSTRACT

A new Al–1B–0.6C master alloy for grain refinement of Mg–Al-based alloys has been fabricated by melt reaction in the present work. Through XRD and EPMA detection, it is found that Al–1B–0.6C master alloy mainly contains Al_3BC particles with the size distribution from 2 μm to 8 μm. The reaction between Al_4C_3 and B in Al melt forms the new Al_3BC phase. Grain refinement experiments reveal that the master alloy can refine AZ63 (Mg–6Al–3Zn–0.4Mn) alloy efficiently. At 760 °C, the grain size is sharply reduced from 710 μm to 70 μm with the addition of 2 wt.% Al–1B–0.6C master alloy, and the grain morphology of α-Mg transits from a characteristic sixfold symmetrical shape to a petal-like shape. The grain refining mechanism is attributed to the presence of Al_3BC particles in the master alloy which can act as effective nucleating substrates of primary α-Mg.

1. Introduction

The use of magnesium alloys for structure parts in automotive industry is receiving increased attention due to their low density, high specific strength and high specific stiffness, which can meet the demands of light weight and low energy consumption.

However, the hexagonal close-packed (HCP) crystal structure of magnesium has only three slip systems at room temperature leading to bad plastic deformation capability and restricting the use of magnesium alloys seriously. Grain refinement is an important method to improve plastic deformation capability and strength, following the improvement of mechanical properties and workability [1–3].

Mg–Al-based alloys are the most common and economic commercial magnesium alloys to which many grain refining methods have been developed, including superheating, Elfinal, carbon inoculation and addition of micro-alloying elements [4–16]. Among them, carbon inoculation is known to be the most effective grain refining method for operating at a low temperature and less fading with long-time holding. The refining mechanism widely accepted is that Al_4C_3 particles nucleate primary α-Mg gains [4,6–9]. C_2Cl_6 is the most useful grain refiner for commercial application, but it can release harmful volatile matters during refining process leading to environment pollution. Thus, the researchers pay more attention to the inorganic carbon-containing refiner recently. The research shows that some carbon-containing agents, including carbon powders [6], Al_4C_3 [7], SiC [10,11] and carbon-bearing master alloys [12,13], can refine Mg–Al-based alloys successfully.

Meanwhile, some researchers begin to study the grain refinement effect of boron on Mg–Al-based alloys. Nishino et al. [17] suggested that Al–4%B master alloy containing AlB_2 and β-AlB_{12} compounds gave rise to significant grain refinement on AZ91 alloys; Wang et al. [18] investigated the effect of a new Al–4Ti–5B master alloy on the grain refinement of AZ31 alloys and found that the master alloy refined AZ31 alloys effectively. The possible mechanism of boron refining method [17,18] is that B reacts with other elements, such as Ti and Al, generating TiB_2 and AlB_2 particles which have the same crystal structure and similar lattice constants with α-Mg and can be nucleating substrates during solidification of primary α-Mg.

Though much research work has been carried out to refine the grain size of Mg–Al-based alloys, a reliable, eco-friendly and commercial master alloy still remains to be developed. In the present work, a new Al–1B–0.6C master alloy has been developed by melt reaction using the grain refinement effect of C and B to reinforce the grain refinement capability of the master alloy. Also, its microstructure, phase identification and refining performance have been studied; the possible refining mechanism was discussed, too.

2. Experimental procedure

The Al–1B–0.6C master alloy was fabricated by adding graphite preforms and Al–3B master alloy preheated to 100–200 °C into Al melt at 1100–1300 °C using a clay-bonded graphite crucible in an intermediate frequency induction furnace. After being held for about 10 min, the melt was poured into a permanent mold and a sample with dimensions of Ø70 mm × 35 mm was achieved. Phase identification and microstructure analysis of the master alloy were studied by X-Ray Diffractor (XRD)

---

* Corresponding author. Tel.: +86 531 88392006; fax: +86 531 88395414.
E-mail address: xfliu@sdu.edu.cn (L. Xiangfa).

0925-8388/$ – see front matter © 2009 Elsevier B.V. All rights reserved.
doi:10.1016/j.jallcom.2009.10.221

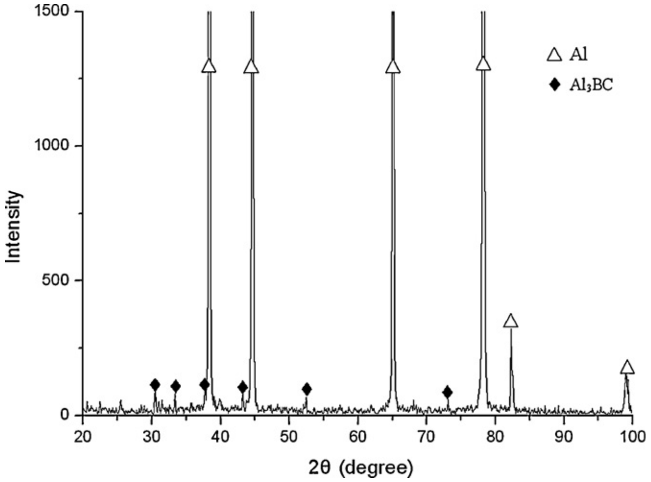

Fig. 1. XRD spectrum of Al–1B–0.6C master alloy.

(Rigaku D/max-rB, Japan) and electron probe micro-analyzer (EPMA) (JXA-8800R, Japan).

For grain refinement test, a commercial AZ63 magnesium alloy was melted in an electric resistance furnace using a stainless steel crucible under the protection of flux. The melts were heated to 730 °C and 760 °C, respectively, and then added into 1 wt.% or 2 wt.% of the Al–1B–0.6C master alloy. After being held for 30 min, the melts were stirred for 30 s and then cast into a cylindrical steel mold with the size of Ø25 mm × 60 mm. The grain refinement test was repeated for three times to inform the accuracy of the final results. In order to reveal grain boundaries, the ingots were held at 420 °C for 12 h in a furnace followed by quenching in cold water (T4 solution heat treatment). All specimens taken from the same position were sectioned, mounted, polished and then etched in a solution of picric and acetic acid. Microstructures were investigated by high scope video microscope (HSVM) (KH-2200MD2, Japan). The mean liner intercept method was used to measure the average grain sizes of the ingots. More than ten cross-lines for each sample (more than thirty cross-lines in total for three samples) were used to counter the number of grains for each grain refining case and the final result was the average of all measured values of grain size.

3. Results and discussion

3.1. Microstructure and reaction process of Al–1B–0.6C master alloy

Fig. 1 shows the XRD spectrum of the new Al–1B–0.6C master alloy; peaks of Al and Al_3BC can be seen in the spectrum. The optical micrograph of the Al–1B–0.6C master alloy is illustrated in Fig. 2. It is shown that there mainly exits a light brown phase distributing uniformly in the Al matrix with the size from 2 μm to 8 μm. To identify the chemical constituent of these particles, EPMA line scan analysis was conducted and the result is presented in Fig. 3. High concentration of Al, B and C enriched in these particles indicates that these compounds are Al_3BC together with considering the XRD result. It is noticed that there exist some smaller particles (marked by arrow) with high concentration of Al and C which are deduced to be Al_4C_3 phase but not shown in the XRD spectrum due to small amount.

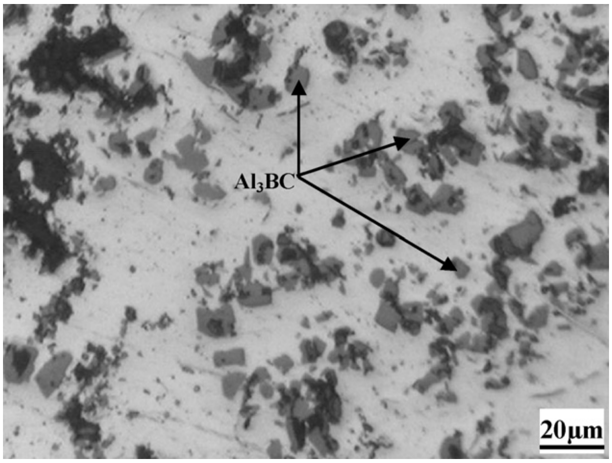

Fig. 2. Optical micrograph of Al–1B–0.6C master alloy.

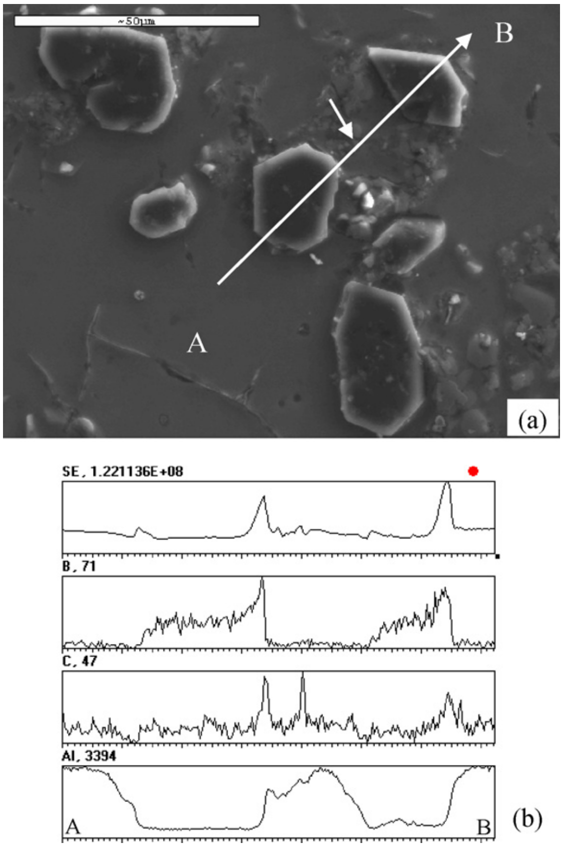

Fig. 3. EPMA line analysis of Al–1B–0.6C master alloy: (a) second electron image and (b) EPMA line analysis along A–B across the particles.

Based on the above analysis results, the chemical reaction process during the preparation of the Al–1B–0.6C master alloy is proposed and the conclusions are as follows: (i) after addition of the graphite preforms into Al melt at the temperature of 1100–1300 °C, carbon in preforms reacted with the molten Al and formed Al_4C_3 particles, as shown in reaction (1), and the presence of Al_4C_3 phase presented in Fig. 3 further confirmed this process; (ii) the added Al–B master alloy dissolved in the molten Al and the solute B atoms would react with Al_4C_3 particles generated in the former process which produced the Al_3BC phase, as shown in reaction (2):

$$4[\mathrm{Al}] + 3\mathrm{C} \rightarrow \mathrm{Al_4C_3} \tag{1}$$

$$\mathrm{Al_4C_3} + 3[\mathrm{B}] + 5[\mathrm{Al}] \rightarrow 3\mathrm{Al_3BC} \tag{2}$$

It is observed the interior parts of these particles have less concentration of Al, B and C than that of the edge parts from Fig. 3 and this can be explained through the chemical reaction process. After the addition of Al–3B master alloys, the B atoms would dissolve from AlB compounds existing in the master alloy and then diffused to neighbor fields. Due to the short holding time and the presence of Al_4C_3 particles, AlB compounds did not dissolve into the melt completely while the solute B atoms had reacted with Al_4C_3 phases creating Al_3BC phase layer tightly around the diffusion zone of B which could absorb more solute B