Materials and Design 34 (2012) 637–648

Contents lists available at ScienceDirect

Materials and Design

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

Grain refinement of AZ91D magnesium alloy by Al–Ti–B master alloy and its effect on mechanical properties

T.J. Chen *, R.Q. Wang, Y. Ma, Y. Hao

Key Laboratory of Gansu Advanced Nonferrous Metal Materials, Lanzhou University of Technology, Lanzhou 730050, China

ARTICLE INFO

Article history:
Received 1 December 2010
Accepted 11 May 2011
Available online 18 May 2011

Keywords:
A. Non-ferrous metals and alloys
E. Mechanical
F. Microstructure

ABSTRACT

The effects of grain-refining parameters on grain size of AZ91D alloy have been investigated using an Al–Ti–B master alloy as refiner and an adequate refining technique has been developed. Simultaneously, the ultimate tensile strength (UTS) and hardness of the as-cast, solutionized and solution-aged alloys have also been examined at room temperature. The results indicate that the Al–Ti–B master alloy is an effective grain refiner for AZ91D alloy. Increasing pouring temperature or cooling rate from holding temperature to pouring temperature is beneficial for obtaining small grains. But for addition amount, holding temperature or holding time, there is an optimal value. The grain size can be decreased from 422 μm to 79 μm after being refined according to the obtained technique. Accordingly, both the UTS and hardness are obviously enhanced. Solution treatment can further improve the UTS but decrease the hardness while solution-age treatment leads these two properties to increase. Compared with the un-refined as-cast alloy, the UTS and hardness of the solution-aged alloy are increased by 71% and 53% respectively. Correspondingly, the fracture mode during tensile testing changes with the treatments. These changes in mechanical properties and fracture mode are attributed to the different contributions of dissolution or precipitation of β phase to grain bonding strength, solution strengthening and precipitation strengthening. In addition, the microstructure sensitivity of the alloy refined by this refiner to casting thickness is quite low compared with that of the alloy refined by SiC particles.

1. Introduction

Magnesium alloys have wide applications in the fields of automobile, aerospace, electronic products and portable tools due to their attractive engineering properties such as low density, high specific strength and stiffness, improved damping property, electromagnetic shielding capacity, excellent machinability and good castability [1]. Among the commonly used magnesium alloys, AZ91D alloy is the most dominative one. However, its mechanical properties are relatively lower than those of another family of light alloys, aluminum alloys, which seriously limits its applications. It is well known that achievement of a fine grain size generally can improve mechanical properties of most metals and alloys. Thus, a fine grain size is very important for the service performance of cast products [2].

However, for AZ91D alloy, one aluminum bearing magnesium alloy, there is still no a commercially used grain refining technique although several methods have been developed [3,4]. These methods mainly include superheating [2,4], the Elfinal process [2], carbon inoculation [2–6], melt vibration or shearing [7,8] and addition of alloying elements or other additives such as Ca, Sr [9], Nd [10], Mn [11], Al_8Mn_5 [12], ZnO [13], Al–B–C [14], Al–Ti–C [15], B [16], Ti [17], TiB_2 [18] and Al–Ti–B [19]. Comparatively, the introduction of Ti, B, TiB_2 or Ti–B can be in form of Al or Mg based master alloys, and thus is very easy and reliable. Furthermore, Al and Mg are the main alloying elements of AZ91D alloy and a little addition does not change the composition or pollute the melt. More importantly, the resulting refining effect is very significant. The grain size can be decreased from several hundred micrometers to dozens of micrometers [16–19]. So, this kind of refining technique should have large application potential. However, the existing investigations are relatively scarce and all of them have focused on the refining mechanisms, and the detailed effects of refining parameters on microstructure have not been involved [16–19]. The existing results indicate that AlB_2 particles that formed from the reaction with Al in the melt are the heterogeneous nucleation substrates of α-Mg when B is added [16]. Ti is a strong constitutional cooling element and thus Ti atoms can restrict the growth of α-Mg and can also accelerate nucleation when Ti is used as the effective constituent of refiner [17]. When both B and Ti are simultaneously used, not only the TiB_2 particles can act as the nucleation substrates, but also the extra Ti atoms may restrict the growth of dendrites and accelerate nucleation [17–19]. So it can be expected that the Ti–B (or TiB_2–Ti) containing refiner should have more effective

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* Corresponding author. Tel.: +86 931 2976573; fax: +86 931 2976578.
E-mail addresses: chentj@lut.cn, chentj1971@126.com (T.J. Chen).

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doi:10.1016/j.matdes.2011.05.020

refining role than the sole B or Ti containing refiners. Furthermore, Al–Ti–B master alloys have been commercially used for producing aluminum alloy castings and they can be purchased from the present markets.

Therefore, in this work, the effects of refining parameters on the grain size of AZ91D magnesium alloy have been investigated using an Al–Ti–B master alloy as refiner and a reliable technique has been developed. Simultaneously, the hardness and ultimate tensile strength (UTS) of the as-cast and heat treated alloys refined according to this technique have been examined.

2. Experimental process

The used material in this work is commercial AZ91D alloy and the refiner is an Al–5Ti–1B master alloy that is commercially applied to aluminum alloys. A quantity of AZ91D alloy was remelted under protection of RJ-2 flux. A given amount of the Al–Ti–B master alloy (addition amount) was then added at a given temperature (addition temperature), followed by holding for a given time (holding time). Subsequently, the melt was cooled to a given temperature (pouring temperature) in a given cooling rate (cooling rate), and finally poured into a permanent mold with a given cavity diameter (ingot size). That is to say that five refining parameters, such as addition amount, addition temperature, holding time, pouring temperature, cooling rate and ingot size, were considered in this work. It should be noted that when a parameter varied, the other four parameters remained unchanged. The detailed parameters are presented in Table 1.

Table 1 shows that three ingot sizes of Φ16 mm, Φ45 mm and Φ70 mm were employed. For the ingots with 16 mm diameter, one small specimen with 10 mm length was directly cut from each of them. For the ingots with 45 mm and 70 mm diameters, two specimens with Φ16 × 10 mm were machined from their edge and center regions respectively to examine their microstructure uniformity. All of the small specimens were finished and polished by standard metallographic technique. Then they were etched by aqueous solution containing glycerol, nitric acid, hydrochloric acid and acetic acid and observed on an optical microscope (OM). To delineate the grain boundaries and quantitatively examine the grain size, the specimens then were solutionized at 420 °C for 8 h and water-quenched. Following, they were processed again according to the above procedures for preparing metallographic specimen and also observed on the OM. The obtained images were analyzed by Image-Pro Plus 5.0 software. The diameter of a round with equivalent area to a grain is taken as the size of this grain. On each specimen, three typical images were examined. The average of all of the rounds' diameters is taken as the grain size of this specimen. Some of the specimens were also observed or analyzed on an electron scanning microscope (SEM) with energy disperse spectroscopy (EDS) or an electron probe electron probe microanalyzer (EPMA).

Based on the above experiments, a grain refining technique was developed. To examine the mechanical properties of the refined alloy, some ingots with 16 mm diameter were prepared according to this technique. The hardness and UTS of the three-state alloys such as as-cast sate, solutionized state (solutionized at 420 °C for 24 h) and solution-aged state (solutionized at 420 °C for 24 h and then aged at 200 °C for 16 h) were examined at room temperature. The tensile testing bars have dimensions of a gauge of 40 mm and a diameter of 8 mm. The tensile testing was carried out on a universal material testing machine at a nominal strain rate of $3.32 \times 10^{-3}\ s^{-1}$. The average of five tests was taken as the final UTS. The hardness was examined on a Brinell hardness tester. The resulting value was the average of six tests. Fracture surface morphologies and side-views of the fracture surfaces were observed on the SEM and OM respectively. In addition, the Al–Ti–B master alloy was analyzed by an X-ray diffractometer (XRD) and the EPMA.

3. Results and discussion

3.1. Microstructure characteristics of Al–Ti–B master alloy and the related grain refining mechanisms

Fig. 1 gives the XRD result of the used Al–Ti–B master alloy. It indicates that this refiner is consisted of three phases such as α-Al, $TiAl_3$ and TiB_2. From the back-scattered image shown by Fig. 2a, it can be seen that it includes three structures with different morphologies and sizes: gray matrix, large-sized polygonal white particles (most of them are larger than 20 μm) and small-sized white particles (their size is about 2–3 μm). Fig. 2b–d indicates that these three structures are rich in Al, Ti and B respectively. Together with the XRD result, it can be suggested that the gray matrix is α-Al, the large white particles are $TiAl_3$ and the small white particles are TiB_2.

The existing investigations indicate that TiB_2 particles have a same hexagonal structure with α-Mg and their lattice constants are $a = 0.30,\ 054$ nm and $c = 0.32,\ 528$ nm while those of α-Mg are $a = 0.3202$ nm and $c = 0.521$ nm [18]. The smallest disregistry between TiB_2 and α-Mg is only 5.6% and the crystallographic orientation relationship is $(0001)_{Mg}\|(0001)_{TiB_2}$, much less than 15% – the critical value for a particle to act as heterogeneous nucleation substrate [18,19]. In addition, TiB_2 particles possess melting temperature higher than 3000 °C and exist as solid particles in the molten alloy [20]. Therefore, TiB_2 particles can act as heterogeneous nucleation substrate of α-Mg theoretically. The existing experimental results from SEM observation and EDS analysis also demonstrate that there is always a small TiB_2 particle in the center region of each fine equiaxed dendrite and this TiB_2 particle is suggested to be the nucleus of the dendrite [18,19]. In addition, the size of nucleation substrates is also an important factor which decides their nucleation potency [14]. The size of TiB_2 particles in

Table 1
Processing parameters used in this work.

Processing parameters	Addition amount (wt.%)	Holding time (min)	Holding temperature (°C)	Cooling rate (°C s^{-1})	Pouring temperature (°C)	Ingot diameter (mm)
Addition amount	0, 0.05, 0.2, 0.3, 0.5, 0.7, 1.0	30	750	0.7	705	16
Addition temperature	0.3	30	690, 720, 750, 780	0.7	705	16
Holding time	0.3	10, 20, 30, 60, 90	750	0.7	705	16
Cooling rate	0.3	30	750	0.05, 0.3, 0.7	705	16
Pouring temperature	0.3	30	750	0.7	690, 705, 780	16
Ingot diameter	0.3	30	750	0.7	705	16, 45, 70