

Available online at www.sciencedirect.com

Transactions of Nonferrous Metals Society of China

 γ^2

www.tnmsc.cn

Trans. Nonferrous Met. Soc. China 20(2010) s397-s401

Effect of Al5Ti1B master alloy on microstructures and properties of AZ61 alloys

MA Xu-liang($1^{1, 2}$ WANG Xiang(2^2 , LI Xin-lin() 2^2 , YANG Lei $($

1. School of Materials Science and Engineering, Harbin University of Science and Technology, Harbin 150080, China; 2. Center for Biomedical Materials and Engineering, Harbin Engineering University, Harbin 150001, China

Received 23 September 2009; accepted 30 January 2010

Abstract: AZ61 alloys with different levels of Al5Ti1B master alloy additions were prepared by conventional casting method. The effects of Al5Ti1B contents and holding time on microstructures and microhardness of AZ61 alloys were studied by XRD, OM and microhardness testing techniques. The results show that when the addition level of Al5Ti1B master alloy is less than 0.5% (mass fraction), the average grain size of the alloys decreases with the increase of Al5Ti1B content at the same holding time. But the grain size increases somewhat with further addition of Al5Ti1B. The average grain size of the alloys decreases with the increase of the holding time as it is less than 30 min at the same addition level of Al5Ti1B. It is considered that TiB₂ particles can serve as the heterogeneous nucleation sites of *α*-Mg during solidification, and heterogeneous nucleation is the main reason for the grain refinement of AZ61 alloys. The microhardness of the refined AZ61 alloys with 1.0% Al5Ti1B addition is increased by about 8%. **Key words:** AZ61 alloys; Al5Ti1B master alloy; grain refinement; microhardness

1 Introduction

l

֦

l

Magnesium alloys are widely used for structural, aerospace and automotive applications due to their high specific strength and low density[1−2], but their mechanical properties and performance still do not meet the needs of some important parts in vehicles and other applications. Therefore, many ways are investigated in order to further improve the mechanical properties of Mg based alloys[3−5]. Grain refinement is one of important methods used to improve the mechanical properties of magnesium alloys[6]. These methods include melt superheating, rapid cooling, carbon inoculation, melt agitation, and addition of solute elements such as Ca, Sr and RE[7−9]. Small amount of addition of Ca is found to be very effective in refining the microstructures of Mg-Al alloys. However, the brittleness will increase if Ca content is higher than 0.2%[10]. Adding pure Sr to magnesium alloys will produce serious burning loss, thus, the amount of Sr is difficult to control[5]. The superheating method requires extra energy to heat and hold the melt up to 150−260 ˚C above the melting point for a certain period. At this temperature, melt oxidation may become excessive[11]. Among the added agents of

grain refining, adding master alloy is an important research direction for its convenient operation, less pollution and fine grain refinement efficiency[12]. It is well known that Al5Ti1B master alloy is an effective grain refiner in aluminum alloys, and it can also refine ZA84 magnesium alloy[13]. However, whether Al5Ti1B alloy has a similar function in AZ61 alloy when it is used in Al alloys has not yet been reported. It is therefore the objective of this work to study the grain refinement efficiency of Al5Ti1B master alloy in AZ61 alloy and the mechanical properties of AZ61 alloy.

2 Experimental

Al5Ti1B master alloy was fabricated by LSM Company of England. The AZ61 alloy was prepared by melting pure Mg (99.9%, mass fraction), Al (99.9%, mass fraction), Zn (99.95%, mass fraction) and Al-10Mn master alloy in an electrical resistance furnace under the protection of RJ−2 flux. Al5Ti1B master alloy was added into the melt at 730 ˚C. The melt was held for different time and then poured into a mild steel mold that was preheated to 250 ˚C with a size of *d*25 mm×100 mm. The holding time was 5, 10, 20, 30 and 50 min, respectively. The chemical compositions of the

Foundation item: Project(2010RFQXG117) supported by the Special Fund for Technological Innovation Program of Harbin, China **Corresponding author:** WANG Xiang; Tel: +86-451-82518173; E-mail: wangxiang@hrbeu.edu.cn

experimental AZ61 alloys are listed in Table 1. All samples were subjected to solution-treatment at 400 ˚C for 8 h followed by quenching into water in order to reveal the grain boundaries. For microstructural observation, the samples were polished and etched in a solution of 3% HNO₃+97% alcohol. The phase identification was carried out via X-ray diffraction (XRD) using Rikagu D/max−RB diffractometer. The characterization of the grain size was conducted on an OLYMPUS−PMG311U optical microscope(OM). The microhardness of the alloys was examined with 1 N load for 20 s by microhardness instrument.

Table 1 Chemical compositions of experimental alloys (mass fraction, %)

| Alloy No. | Al | Zn | Mn | Al5Ti1B | Mg |
|----------------|-----|-----|-----|---------|------|
| 1 | 6.5 | 1.0 | 0.3 | | Bal. |
| \overline{c} | 6.5 | 1.0 | 0.3 | 0.3 | Bal. |
| 3 | 6.5 | 1.0 | 0.3 | 0.5 | Bal. |
| 4 | 6.5 | 1.0 | 0.3 | 0.8 | Bal. |
| 5 | 6.5 | 1.0 | 0.3 | 1.0 | Bal. |

3 Results and discussion

3.1 Effects of Al5Ti1B addition on microstructures of AZ61 alloys

Fig.1 shows the XRD patterns of AZ61 alloys with different levels of Al5Ti1B master alloy addition. From Fig.1, it is found that all the existing phases are *α*-Mg and β -Mg₁₇Al₁₂, which confirms that the addition of Al5Ti1B does not result in the formation of other new phases.

Fig.1 XRD patterns of AZ61 alloys with different contents of Al5Ti1B: (a) AZ61; (b) 0.3% Al5Ti1B; (c) 0.5% Al5Ti1B; (d) 0.8% Al5Ti1B; (e) 1.0% Al5Ti1B

The typical microstructures of as-cast AZ61 alloys non-refined and refined with Al5Ti1B are shown in Fig.2. It is clear that both samples display a similar equiaxed dendritic structure. In addition to the petal-like primary

α-Mg, there is $β$ -Mg₁₇Al₁₂ phase distributed in the interdendritic region. For the unrefined AZ61 alloy, β -Mg₁₇Al₁₂ phases are coarse and distributed at the interface of *α*-Mg in the form of semi-continuous reticulation (as shown in Fig.2(a)). But for the refined AZ61 alloy, the coarse reticular β -Mg₁₇Al₁₂ phases become uncontinuous, discrete and fine, and mainly distribute at the grain boundary and in some grains (Fig.2(b)). This indicates that the solute distribution in the refined AZ61 alloy is more homogeneous than that in the non-refined alloy. The variation of morphology and distribution of β -Mg₁₇Al₁₂ phase is attributed to the refinement of *α*-Mg. The volume of melt with high solute levels is decreased and the amount of grain boundary is increased by the process of grain refinement, which makes the liquid with eutectic composition distribute more homogeneously and the amount of eutectic phase per unit area of grain boundary decreases at the final stage of solidification.

Fig.2 Microstructures of AZ61 alloys: (a) Non-refined; (b) Refined with 0.5%Al5Ti1B

Fig.3 shows the effects of Al5Ti1B content on the microstructures of AZ61 alloys with solution-treatment. The holding time for all the as-cast AZ61 alloys melt is 30 min. It is evident that $β$ -Mg₁₇Al₁₂ phases are dissolved completely into *α*-Mg phase after solution-treatment, and the microstructure is single supersaturated *α*-Mg phase. The black particle phases distributed at the grain boundary and in the matrix irregularly are A1-Mn compounds. It can also be seen from the figure that the addition of Al5Ti1B master alloy can refine the grain of

AZ61 alloys to some extend. The average grain size for the unrefined AZ61 alloy is about 400 µm and it decreases gradually with increasing Al5Ti1B addition, and a minimum of 50 µm for the grain size can be obtained at an optimum addition level (0.5% Al5Ti1B), while the grain size increases somewhat with further addition of Al5Ti1B. For example, the grain size is about 100 µm at the addition level of 1.0%. There is some gurgitation in the grain size distribution, but the extent of gurgitation for the refined alloy is less than that for the unrefined alloy.

3.2 Effects of holding time on microstructures of refined AZ61 alloys

Fig.4 shows the effects of holding time on microstructures of refined AZ61 alloys with 0.5%Al5Ti1B master alloy addition after solutiontreatment. It can be seen that the grain size decreases gradually with increasing the holding time during certain holding time, which reveals that the refinement

efficiency can be exerted after a certain gestation time. The grain size of refined AZ61 alloys is minimum when the holding time is 30 min, and there is an ascending trend with increasing the holding time because of the decline of the refinement efficiency of Al5Ti1B master alloy. The inoculant needs a gestation stage to work after it is added into the molten alloy. The heterogeneous particles distribute uniformly in the liquid and wet sufficiently with molten alloy, and the optimum refinement efficiency can be obtained in the process of gestation. But refinement efficiency fades with prolonging the holding time because of the dissolution and aggregation of heterogeneous particles.

3.3 Effects of Al5Ti1B content on microhardness of AZ61 alloys

Fig.5 shows the effects of Al5Ti1B content on the microhardness of as-cast AZ61 alloys. The holding time for all the AZ61 melt is 30 min. It can be seen that the microhardness of AZ61 alloys increases to some extent

Fig.6 shows the XRD pattern of A5Ti1B master alloy. It is indicated that this master alloy consists of $Al₃Ti$ and TiB₂ particles. $Al₃Ti$ dissolves quickly when the master alloy is added into the AZ61 liquid alloy as the added titanium content into the melt is within the solubility limit. $TiB₂$, with high melting point, is presumed to serve as the heterogeneous nuclei of *α*-Mg.

Fig.5 Effects of Al5Ti1B content on microhardness of AZ61 alloys

because of the addition of Al5Ti1B master alloy. The average microhardness of refined AZ61 alloy with 1.0% Al5Ti1B addition is approximately 8% higher than that of non-refined AZ61 alloy. All these reveal that the

According to the heterogeneous nucleation theory, the lattice parameter of TiB₂ is close to that of α -Mg, suggesting a low contact angle between α -Mg and TiB₂, as required if $TiB₂$ is to act as an effective refiner. In addition, $TiB₂$ should be as stable as possible in the molten metal, possess a maximum of surface area, and have optimum surface character (perhaps be rough or pitted). Both $TiB₂$ and α -Mg are of the hexagonal close-packed structures[15], so there is a good lattice mismatch between them. When the A15Ti1B master alloy is added into $AZ61$ alloy, $TiB₂$ distributes uniformly in the melt and can be employed as the heterogeneous nuclei for *α*-Mg during solidification, which results in the refinement of grain. And the amount of effective nucleation site increases with the increase of the master alloy addition. However, the grain size cannot decrease continuously with the increase of master alloy addition, and there is a limited value above which smaller grain cannot be obtained. The reason is that the melt cannot provide the necessary energy needed for the formation of nucleus when more nucleating sites are provided into the melt. On the other hand, some nuclei will be melted again because of the over-high local melt temperature resulting from the release of latent heat during solidification. All these result in the phenomenon that there exists an utmost value of the refinement.

Fig.6 XRD pattern of Al5Ti1B master alloy

5 Conclusions

1) The microstructure of as-cast AZ61 alloys consists of α -Mg and β -Mg₁₇Al₁₂ phases. The addition of Al5Ti1B master alloy does not result in the formation of other new phases.

2) The addition of Al5Ti1B master alloy refines the grain of AZ61 alloys to some extent. The average grain size is about 400 µm for the unrefined AZ61 alloy and it decreases gradually with increasing Al5Ti1B addition. A minimum of 50 μ m for the grain size can be obtained at an optimum addition level (0.5% Al5Ti1B). The grain

size increases with further addition of Al5Ti1B. Microstructures of AZ61 alloys with Al5Ti1B addition are refined firstly and then coarsened with the increase of holding time. This reveals that the refinement efficiency of Al5Ti1B fades increasingly after it is up to the optimum. Ti B_2 , with high melting point and hexagonal close-packed structure, is presumed to serve as the heterogeneous nuclei of *α*-Mg. The grain refinement of *α*-Mg mainly results from the existence of a large number of heterogeneous nuclei.

3) The microhardness of refined AZ61 alloys can be increased by about 8% with 1.0% addition of Al5Ti1B.

References

- [1] LIU Yan-hui, LIU Xiang-fa, LI Ting-bin, BIAN Xiu-fang, ZHANG Jun-yan. Grain refining effect of Al-Ti-C master alloy on Mg-Al alloys [J]. The Chinese Journal of Nonferrous Metals, 2003, 13(3): 622−625. (in Chinese)
- [2] WANG Ying-xin, ZENG Xiao-qin, DING Wen-jiang. Effect of Al-4Ti-5B master alloy on the grain refinement of AZ31 magnesium alloy [J]. Scripta Materialia, 2006, 56: 269−273.
- [3] CHEN Jing-yang, GUAN Shao-kang, LIN Dun-wen. Effects of Al3Ti4B master alloy on microstructure and properties of Mg-7Al-0.4Zn-0.2Mn alloys [J]. The Chinese Journal of Nonferrous Metals, 2005, 15(3): 478−484. (in Chinese)
- [4] DAS G, LIU Z F. Investigation on the microstructural refinement of an Mg-6%Zn alloy [J]. Mater Sci Eng A, 2006, 419: 349−356.
- [5] YANG Ming-bo, PAN Fu-sheng, CHENG Ren-ju, TANG Ai-tao. Effect of Mg-10Sr master alloy on grain refinement of AZ31 magnesium alloy [J]. Mater Sci Eng A, 2008, 491: 440−445.
- [6] CAO P, QIAN M, STJOHN D H. Effect of iron on grain refinement of high-purity Mg-Al alloys [J]. Scripta Materialia, 2004, 51: 125−129.
- [7] LIU Hong-mei, CHEN Yun-ze, TANG Yong-bai, HUAN De-ming, TU Ming-jing, LI Ming-zhao, ZHAO Min, LI Yi-guo. Microstructure and properties of Mg-Al alloys with carbon and boron inoculation [J]. Foundry, 2006(6): 615−618. (in Chinese)
- [8] LU L, DAHLE A K, STJOHN D H. Grain refinement efficiency and mechanism of aluminum carbide in Mg-Al alloys [J]. Scripta Materialia, 2005, 53: 517−522.
- [9] DU J, YANG J, KUWABARA M, LI W F, PENG J H. Effect of strontium on the grain refining efficiency of Mg-3Al alloy refined by carbon inoculation [J]. Journal of Alloys and Compounds, 2009, 470: 228−232.
- [10] DU J, YANG J, KUWABARA M, LI W F, PENG J H. Improvement of grain refining efficiency for Mg-Al alloy modified by the combination of carbon and calcium [J]. Journal of Alloys and Compounds, 2009, 470: 134−140.
- [11] ZHANG M X, KELLY P M, OIAN M. Crystallography of grain refinement in Mg-Al based alloys [J]. Acta Materialia, 2005, 53: 3261−3270.
- [12] HAN Guang, LIU Xiang-fa, DING Hai-min. Grain refinement of Mg-Al based alloys by a new Al-C master alloy [J]. Journal of Alloys and Compounds, 2009, 467: 202−207.
- [13] PENG Zhuo-kai, ZHANG Xin-ming, CHEN Jian-mei, XIAO Yang, JIANG Hao, DENG Zhen-zhen. Effects of Mn, Zr on microstructure and properties of Mg-Gd-Y alloys [J]. The Chinese Journal of Nonferrous Metals, 2005, 15(6): 917−922. (in Chinese)
- [14] LEE Y C, DAHLE A K, STJOHN D H. Role of solute in grain refinement of magnesium [J]. Metall Mater Trans A, 2000, 31: 2895−2905.
- [15] LI Jian-guo, HUANG Min, MA Mo. Performance comparison of AlTiC and AlTiB master alloys in grain refinement of commercial and high purity aluminum [J]. Trans Nonferrous Met Soc China, 2006, 16: 242−253.

(Edited by CHEN Wei-ping)