

Effects of melt temperature on as-cast structure and mechanical properties of AZ31B magnesium alloy

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Abstract: The effects of melt temperature on the as-cast structure and mechanical properties of AZ31B magnesium alloy were studied through a home-made electrical resistance furnace. The results show that the equiaxed dendrite size of AZ31B magnesium alloy under water-cooled metal cooling is linearly decreased with increasing the melt temperature below 850 °C, whereas the size changes little over 850 °C. The second phases in the microstructure are firstly refined; however, they are coarsened when the melt temperature exceeds 850 °C. With the increase of melt temperature, the tensile strength, yield strength and elongation of AZ31B magnesium alloy samples are rapidly enhanced first, then are slightly declined. When the melt temperature is increased to 850 °C, the tensile strength, yield strength and elongation reach 260 MPa, 75.4 MPa and 27.57%, respectively. The tensile strength, yield strength and elongation are increased by 15%, 13% and 61%, respectively compared with 750 °C. DSC analysis shows that the nucleation temperature is decreased, the critical nucleus radius is lessened with the increase of melt temperature, which increase the degree of supercooling and heterogeneity nucleation rate in melt, and the growth rate becomes larger with the increase of the degree of supercooling. That is the reason why the grain size is first rapidly decreased, and then is little increased.

Key words: AZ31B magnesium alloy; as-cast structure; mechanical properties; melt temperature

1 Introduction

As the lightest structural material, magnesium alloys taking on high specific strength, super-duper damping and so on, show a powerful attraction in the automotive, aerospace, electronics and other industrial sectors [1,2]. Because magnesium is the close-packed hexagonal structure and dislocation glide is anisotropic, the ductility and formability are poor at room temperature and magnesium alloy applications are restricted. The grain refinement is an important way to improve the deformation capacity [3], so the grain refinement of magnesium alloys has attracted increasing attention.

The methods of refining as-cast structure of magnesium alloys include deteriorating, alloying, exerting external fields and melt superheating. The studies show that the addition of carbon or carbon modifier, such as $MgCO_3$, C_2Cl_6 or CaC_2 , can effectively refine Mg-Al-based alloys, and it is thought that a large number of dispersed Al_4C_3 particles in the

melt become the crystalline cores [4]. The grain refinements are obtained through adding a small amount of Ca, Sr or traces of Ti, B, Sb, Sn and RE into aluminum-containing magnesium alloys [5–9], or adding Zr into magnesium alloys without containing aluminum [10]. Among these methods, the method of adding carbon or carbon modifier is low price, whereas the use of a large amount of carbon modifier can cause serious environmental pollution. The semi-solid structure with thin and spherical primary phase was prepared through the electromagnetic stirring by MAO et al [11]. XU et al [12] studied the effects of the alternative electromagnetic field on the solidification structure of ZK60 magnesium alloy and it is found that the solidification structure is refined. WANG et al [13] and FANG et al [14] researched the grain refinement of magnesium alloy under external field and achieved some results. However, the external field equipment is relatively complex, and it is difficult to apply to the production.

Melt superheating treatment is an effective method of refinement grain. In the past decades, the effects of

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melt superheating on aluminium alloy and Mg–1.5Si–1Zn alloy have been studied [15,16], and it is found that melt superheating treatment can refine the as-cast structure, whereas the refinement mechanism is not clear so far [17,18]. Because magnesium alloy is easy to oxidize and burn, the researches on the effects of melt superheating on the solidification structure and mechanical properties are few. The aim of this work is to explore the influence of melt superheating temperature on the solidification structure and mechanical properties of AZ31B magnesium alloy, to obtain the optimum treatment temperature, and to analyze the mechanism of grain refinement.

2 Experimental

The commercial AZ31B magnesium alloy was taken as raw materials, of which chemical composition is shown in Table 1. AZ31B magnesium alloy was melt in a home-made electrical resistance furnace with a mild steel crucible, and the melt was protected by the mixture gas of 99.5% CO₂ and 0.5% SF₆ in volume fraction. The schematic diagram of electrical resistance furnace is shown in Fig. 1. In order to accurately control the melt temperature, two thermocouples were designed in the electrical resistance furnace. One was used for measuring the furnace cavity temperature, and the other for measuring the melt temperature. The melt temperature is increased to 750, 800, 850 and 900 °C, respectively, after that the crucible with the protective gas was moved to a prepared cooling device to cool.

Table 1 Chemical composition of AZ31B magnesium alloy (mass fraction, %)

Si	Fe	Cu	Mn
≤0.005	≤0.05	≤0.05	≤0.2~0.5
Ni	Zn	Al	Mg
≤0.05	0.5~1.5	2.5~3.5	Bal.

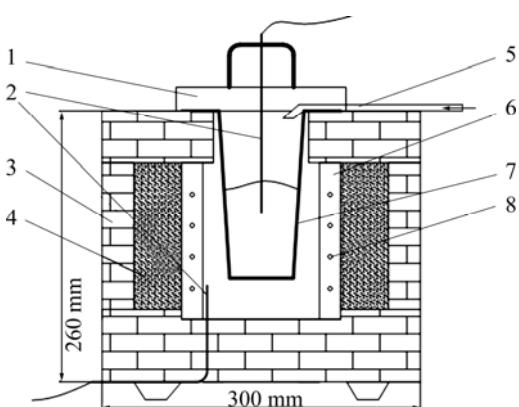


Fig. 1 Schematic diagram of melting device of magnesium alloy: 1—Furnace lid; 2—Heat electric couple; 3—Refractory brick; 4—Mineral flux; 5—Gas-guide tube; 6—Internal furnace; 7—Crucible; 8—Resistance wire

The as-cast ingots were cut along the longitudinal surface, and three tensile samples of $d8 \text{ mm} \times 40 \text{ mm}$ are obtained on each as-cast ingot, as shown in Fig. 2. The tensile tests were done on a electronic universal testing machine (CMT5305) at the stretching speed of 2 mm/min. The microscopic specimens were intercepted at the end of the tensile samples. The microscopic specimens were etched with a solution containing 5.5 g picric acid, 5 mL glacial acetic acid, 90 mL alcohol and 10 mL water after they were shined and polished, and then the microstructures were observed by a metallographic microscope (Axiovert200MAT) and a scanning electron microscope (S-3000N). The grain sizes were measured through the average linear intercept method. A rotating X-ray diffraction instrument of Japan Science (D/max-RB 12 kW) was applied to determining the phases in AZ31B magnesium alloy. A synthesis thermal analyzer (Setaram) was used to study the effects of the melt temperature on the nucleation temperature and solidification range during the solidification process of AZ31B magnesium alloy.

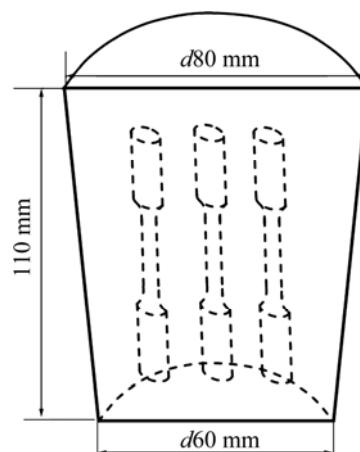


Fig. 2 Schematic diagram of casting and sampling location

3 Results and analysis

3.1 Effects of melt temperature on as-cast structure of AZ31B magnesium alloy

The as-cast structures of AZ31B magnesium alloy under different melt temperatures are shown in Fig. 3, and XRD patterns are shown in Fig. 4. It can be seen from Fig. 3 that the as-cast microstructure of AZ31B magnesium alloy is typical dendrite, and many spherical non-equilibrium eutectic phases are distributed in the interdendritic and at the grain boundary. When the melt temperature reaches 750 °C or 800 °C, the equiaxed dendrite is very coarse, as shown in Figs. 3(a) and (b), whereas the equiaxed dendrite is obviously refined at 850 °C (Fig. 3(c)). As the melt temperature continues to rise, the equiaxed dendrite changes little (Fig. 3(d)).

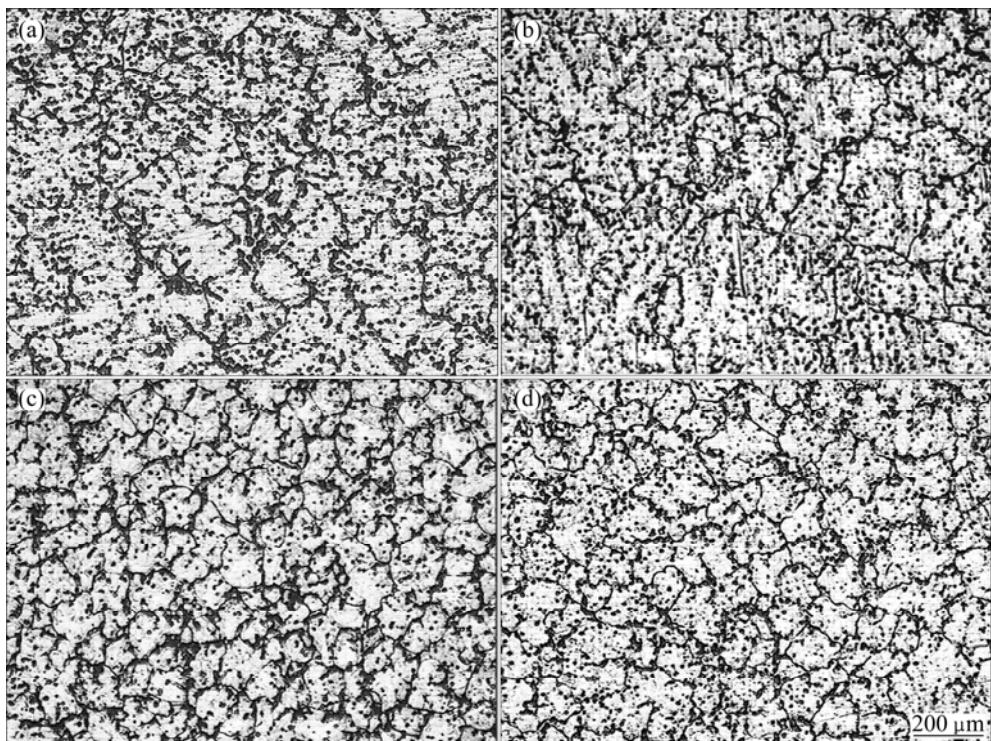


Fig. 3 Optical structures of as-cast AZ31B magnesium alloy under different melt temperatures: (a) 750 °C; (b) 800 °C; (c) 850 °C; (d) 900 °C

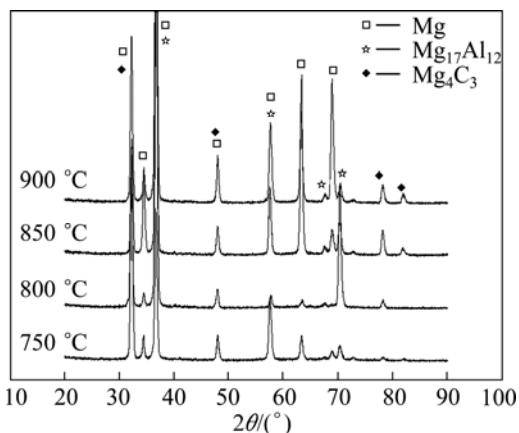


Fig. 4 XRD patterns of AZ31B magnesium alloy under different melt temperatures

It can be seen from Fig. 4 that there mainly exist α -Mg, $Mg_{17}Al_{12}$ and Al_4C_3 in AZ31B magnesium alloy. The peak value of Al_4C_3 is smaller when the melt temperature is lower, which shows that the amount of Al_4C_3 is very small. The peak value of Al_4C_3 becomes larger with increasing the melt temperature, which indicates that the amount of Al_4C_3 is enhanced. The peak values of MnAl and MgO are not found in the XRD patterns, which suggests that the amount of MnAl and MgO is too small.

SEM images of AZ31B magnesium alloy are shown in Fig. 5 at different melt temperatures. It can be seen

that the gray area is α -Mg matrix, and the white second phases distributed on the grain boundary and inside the grains are mainly $Mg_{17}Al_{12}$. The second phases mainly contain the lath-shaped, rod-shaped and spheroidal phases. When the melt temperature is 750 °C, the second phases are very coarse, and many of them exist in lath shape. The spheroidal second phases are obviously refined with the increase of melt temperature, and the number of lath-shaped second phases is reduced, and at the same time, the lath-shaped second phases are changed into needle-shaped phase. The spheroidal and needle-shaped second phases are both coarsened when the melt temperature rises to 900 °C.

3.2 Effects of melt temperature on grain size of AZ31B magnesium alloy

The effects of melt temperature on grain size of AZ31B magnesium alloy are shown in Fig. 6. It can be found that the grain sizes are rapidly decreased first, then change little with the increase of melt temperature. When the melt temperature is 750 °C, the average grain size of equiaxed dendrite is (247 ± 12) μ m, whereas the average grain size is reduced to (95 ± 5) μ m when the melt temperature is enhanced to 850 °C. The grain size is decreased by 61.5% compared with 750 °C. When the melt temperature is increased to 900 °C, the average grain size is (99 ± 5) μ m, which is decreased by 60% compared with 750 °C and increased by 4% compared

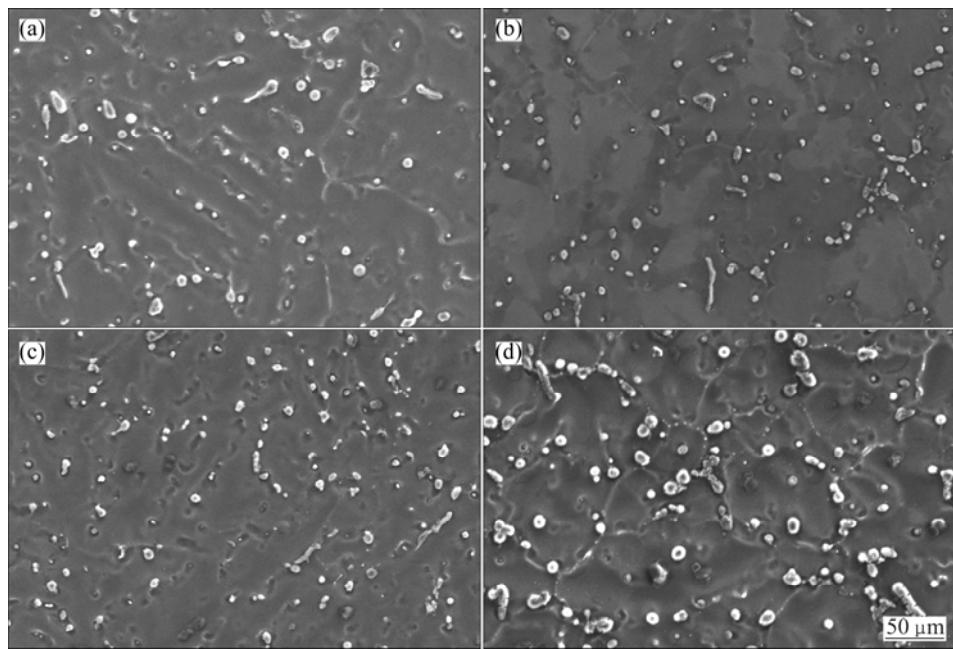


Fig. 5 SEM images of AZ31B magnesium alloy at different melt temperatures: (a) 750 °C; (b) 800 °C; (c) 850 °C; (d) 900 °C

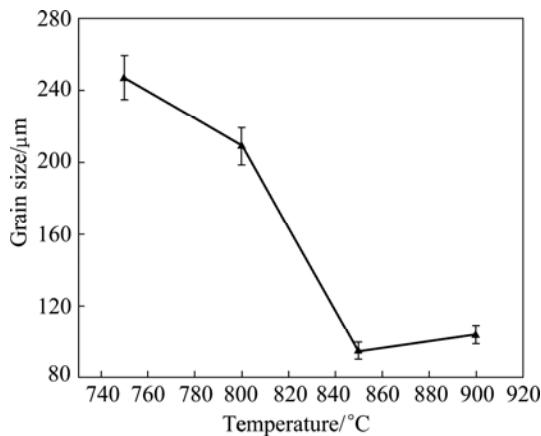


Fig. 6 Effects of melt temperature on grain size

with 850 °C. This indicates that the grain can be refined by enhancing the melt temperature. Whereas it is incorrect that the higher the melt temperature, the better the refine effect. After the melt temperature exceeds 850 °C, the refinement effect with increasing the melt temperature is very little.

The effects of the melt temperature on the size of the second phases are shown in Fig. 7. It can be seen that the size of the second phases is rapidly decreased, then is fast increased with the enhancement of melt temperature. The average size of the second phases is about 11.5 μm when the melt temperature is 750 °C. The average size of the second phases is about 8.5 μm when the melt temperature is increased to 850 °C, which is decreased by 26%. The average size of the second phases is yet increased to 12.5 μm as the melt temperature is increased to 900 °C, and it is increased by 47% compared with 850 °C.

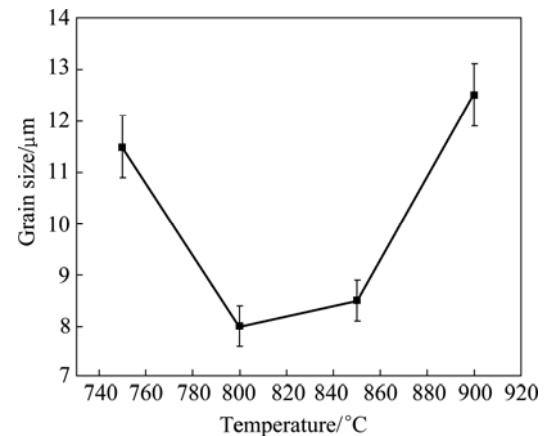


Fig. 7 Effects of melt temperature on the second phase size

3.3 Effects of melt temperature on mechanical properties of AZ31B magnesium alloy

The mechanical properties of AZ31B magnesium at different temperatures are list in Table 2. The effects of the melt temperature on the mechanical properties of AZ31B magnesium alloy are shown in Fig. 8. It can be found that the mechanical properties are first increased, then are slightly decreased with the increase of melt temperature. When the melt temperature reaches 850 °C, the tensile strength, yield strength and elongation of AZ31B are 260 MPa, 75.4 MPa and 27.57%, respectively, which are increased by 15%, 13% and 61%, respectively, compared with 750 °C. This indicates that the tensile strength, yield strength and elongation of AZ31B are greatly improved when the melt temperature is increased from 750 to 850 °C, especially the increase amplitude of the elongation is the largest. This means that the plasticity of AZ31B magnesium alloy at room

temperature is significantly improved, so the deformation ability is advanced to a large extent.

Table 2 Tensile properties of AZ31B magnesium alloy

Temperature/°C	σ_b /MPa	σ_s /MPa	δ /%
750	226	65.1	17.16
800	232	67.1	17.2
850	260	76.5	27.57
900	250	75.6	23.98

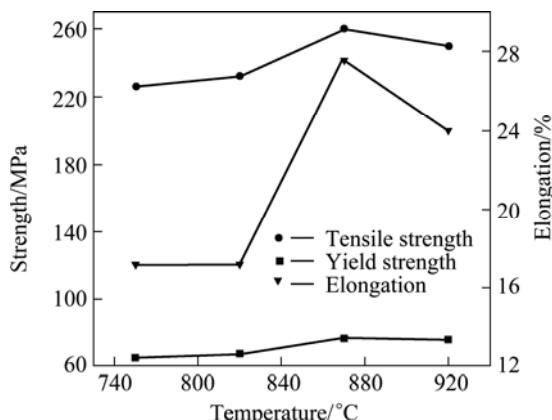


Fig. 8 Effects of melt temperature on tensile properties of AZ31B magnesium alloy

The tensile strength, yield strength and elongation of AZ31B are decreased by 4%, 1% and 11%, respectively, when the melt temperature is increased from 850 to 900 °C. The decrease amplitude of elongation is the largest among them. It can be seen from Fig. 5 and Fig. 7 that the second phases are coarsened when the melt temperature is increased to 900 °C, in despite of the fact that the size of equiaxed dendrite changes little, the mechanical properties of AZ31B magnesium are still reduced.

In summary, the mechanical properties of AZ31B magnesium alloy can be obviously improved with increasing the melt temperature. Under the experimental conditions, the mechanical properties of AZ31B magnesium alloy are the best at 850 °C. The continued increase of melt temperature will result in the decrease of the mechanical properties.

The effects of the grain sizes on the mechanical properties of AZ31B magnesium alloy are shown in Fig. 9. It can be found that the tensile strength, yield strength and elongation of AZ31B are increased with decreasing the grain sizes. The smaller the grain size, the larger the increasing amplitude of the mechanical properties. This proves that the mechanical properties of magnesium alloy can be obviously enhanced through refining the grain, especially the elongation, so the deformation ability at room temperature is greatly improved.

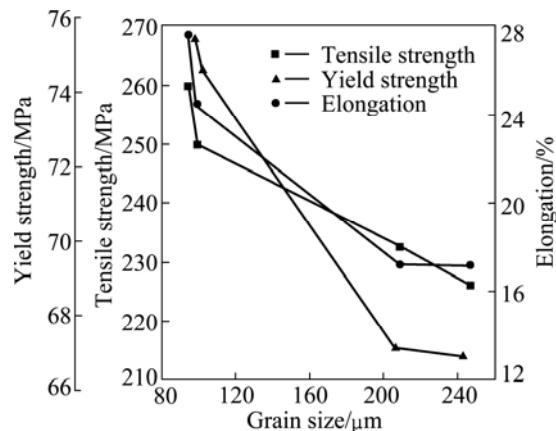


Fig. 9 Effects of grain size on tensile properties of AZ31B magnesium alloy

From the view of the grain boundary coordination deformation, the reasons that the grain refinement can improve the plasticity of magnesium alloy at room temperature are as follows: the dislocation slipping process can be shortened and the deformation is more diffuse and uniform through refining the grain; grain refinement can make grain boundary more mobile and grain rotation more easy; grain rotation blocks the deformation of grain in the soft orientation and accelerates the deformation of grain in the hard orientation; grain refinement can activate the non-basal face slipping of prismatic face and pyramidal face located in magnesium alloy.

3.4 Discussion

After AZ31B magnesium alloys are heated from 400 to 750, 800, 850 and 900 °C at a rate of 10 °C/min, respectively, they are cooled at a rate of 5 °C/min to room temperature, and the DSC curves are shown Fig. 10. It can be found that the melt temperature has a significant impact on the solidification process.

The nucleation temperature is 627 °C when the melt temperature is 750 °C. With the increase of melt temperature, the nucleation temperature is gradually decreased. When the melt temperature is increased to 850 °C, the nucleation temperature is decreased by 15 °C compared with 750 °C. When the melt temperature is increased from 850 °C to 900 °C, the nucleation temperature is only decreased by 2 °C.

The melt structure and nature are important factors for determining the solidification structure and properties. The melt structure of the metals or alloys is micro-uniform and there exist a number of atomic clusters with different compositions and structures, whereas it is the atomic cluster of the largest size to play a major role in the solidification process [19]. The melt temperature is one of the most important factors affecting

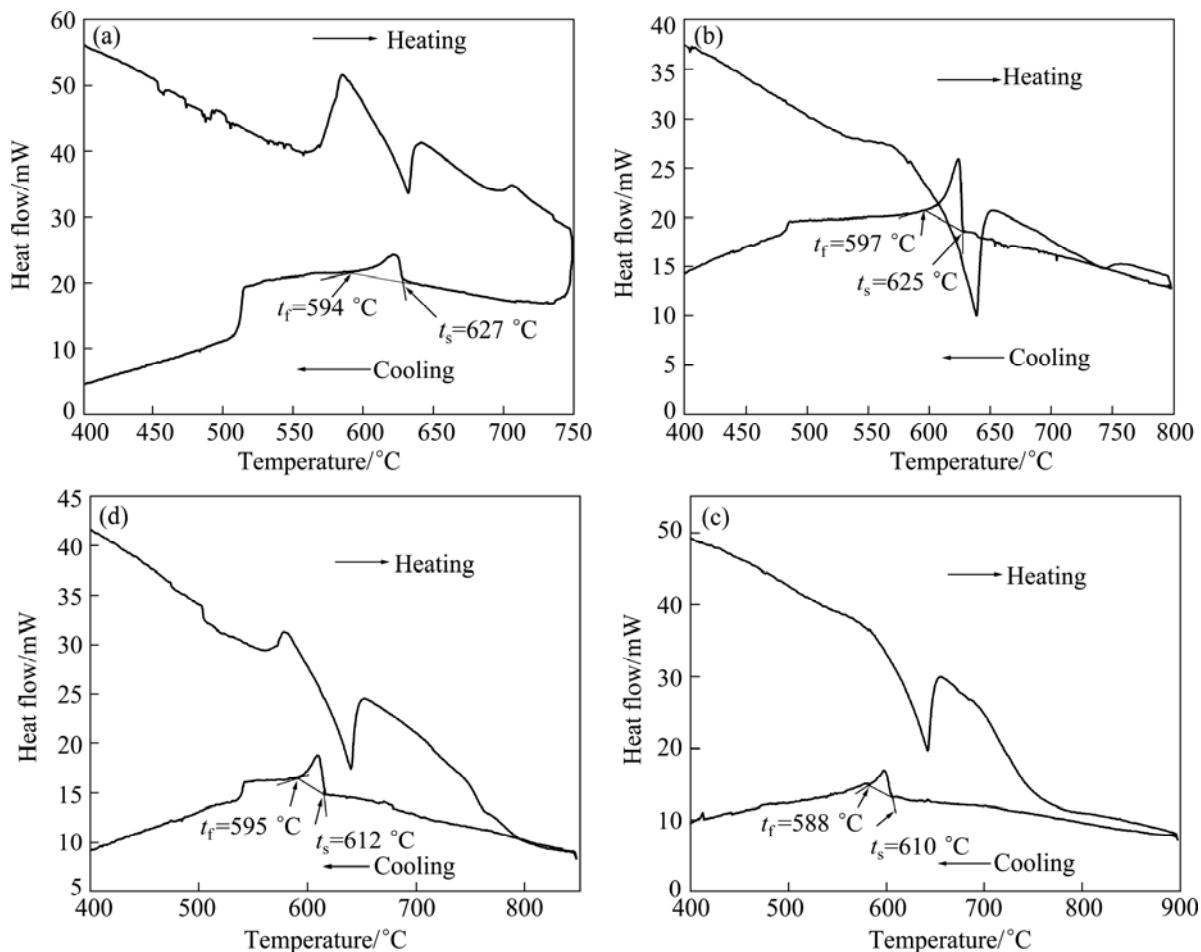


Fig. 10 DSC curves of AZ31B magnesium alloy at different melt temperatures (t_s —Onset temperature; t_f —Final temperature): (a) 750 °C; (b) 800 °C; (c) 850 °C; (d) 900 °C

the size of atomic clusters. The atomic number in the largest size clusters is lessened with increasing the melt temperature, and the decreasing amplitude decreases with the increase of melt temperature; for heterogeneous nucleation, the atomic number in the critical nuclei is increased with the increase of melt temperature, and the intersecting point is defined with the nucleation temperature under the equilibrium state. The schematic diagram of the determination of nucleation temperature is shown in Fig. 11. The diffusion rate of atoms is far less than the rate of temperature change, so the changes of melt structure lag the temperature changes, which can influence the nucleation temperature [20]. For example, when the melt is cooled from t_1 to t_s , the nucleation temperature of equilibrium state is t_s , whereas the atomic number of the largest size clusters cannot be instantly increased to the equilibrium value n_s , the atomic number is increased to n_1 , less than n_s ; in order to make the atomic number of the largest size clusters equal to the atomic number of critical nucleus, the temperature must be reduced to t'_1 , namely, the nucleation temperature is decreased to t'_1 . When the melt is cooled from t_2 to t_s ,

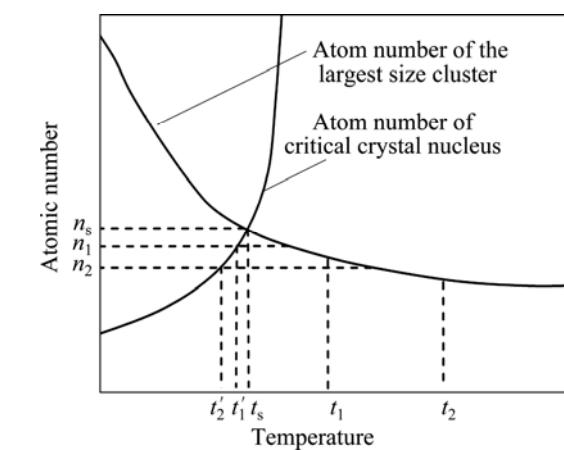


Fig. 11 Schematic diagram of nucleation temperature determination

because t_2 is higher than t_1 , it is more difficult that the atomic number of the largest size clusters reaches the equilibrium value n_s . The atomic number only reaches n_2 , so the temperature must be reduced to t'_2 to let the atomic number of the largest size clusters equal to the atomic number of critical nucleus, namely, the nucleation

temperature is decreased to t'_2 . This illustrates that the higher the melt temperature, the lower the nucleation temperature, and the larger the degree of supercooling.

In summary, the change of melt structure is delayed than the temperature change during the solidification, inducing that the nucleation temperature is decreased with the increase of melt temperature. So the nucleation degree of supercooling is increased and the critical crystal nucleus is decreased, which makes the nucleation rate increase and the solidification structure refine. Whereas the higher the melt temperature, the smaller the increase extent of degree of supercooling. So, the refinement effect is gradually decreased. The refinement effect is very obvious when the melt temperature is below 850 °C, nevertheless the refinement effect is not distinct when the melt temperature is above 850 °C.

According to the relationship between the growth rate and the degree of supercooling, the larger the degree of supercooling is, the faster the growth rate is [21], so the larger the single grain size is, which is one of the reasons leading to the results shown in Fig. 6. Namely, with the increase of degree of supercooling, the grain size is first rapidly decreased, and then is little increased.

4 Conclusions

1) The equiaxed dendrite size of AZ31B magnesium alloy under water-cooled metal cooling is linearly decreased with increasing the melt temperature below 850 °C, whereas the size changes little over 850 °C. The second phases in the microstructure are firstly refined; however, they are coarsened when the melt temperature exceeds 850 °C.

2) With the increase of melt temperature, the tensile strength, yield strength and elongation of AZ31B magnesium alloy samples are rapidly increased first, then are slightly declined. When the melt temperature is increased to 850 °C, the tensile strength, yield strength and elongation reaches 260 MPa, 75.4 MPa and 27.57%, respectively, which are increased by 15%, 13% and 61%, respectively compared with 750 °C.

3) DSC analysis shows that the nucleation temperature is decreased, and the critical nucleus radius is lessened with the increase of melt temperature, which increases the degree of supercooling and heterogeneity nucleation rate in melt, and the growth rate becomes larger with the increase of the degree of supercooling. That is the reason why the grain size is first rapidly decreased, and then is increased little.

References

- [1] POLMEAR I J. Magnesium alloy and applications [J]. Mater Sci Tech, 1994, 10(1): 1–16.
- [2] KU K. Magnesium alloys and their applications [M]. Oberursel: DGM Information Gesellschaft, 1992.
- [3] KUROTA K, MABUCHI M, HIGASHI K. Review processing and mechanical properties of fine-grained magnesium alloys [J]. J Mater Sci, 1999, 10: 2255–2259.
- [4] JIN Qing-lin, EOM J P, LIM S G, PARK W W, YOU B S. Effects of C_2Cl_6 addition on grain refinement and mechanical properties of AZ31 magnesium alloy [J]. Metals and Materials International, 2003, 9(5): 453–458.
- [5] YANG Ming-bo, PAN Fu-sheng, CHENG Ren-ju, TANG Ai-tao. Comparation about efficiency of Al–10Sr and Mg–10Sr master alloys to grain refinement of AZ31 magnesium alloy [J]. J Mater Sci, 2007, 42: 10074–10079.
- [6] WANG Ying-xin, ZENG Xiao-qin, DING Wen-jiang, LUO A A, SACHDEV A K. Grain refinement of AZ31 magnesium alloy by titanium and low-frequency electromagnetic casting [J]. Metallurgical and Materials Transactions A, 2007, 38(6): 1358–1366.
- [7] WANG S C, CHOU C P. Effect of adding Sc and Zr on grain refinement and ductility of AZ31 magnesium alloy [J]. Journal of Materials Processing Technology, 2008, 197: 116–121.
- [8] SRINIVASAN A, PILLAI U T S, PAI B C. Microstructure and mechanical properties of Si and Sb added AZ91 magnesium alloy [J]. Metallurgical and Materials Transactions A, 2005, 36(8): 2235–2243.
- [9] SUN Yang-shan, WENG Kun-zhong, YUAN Guang-yin. Effects of Sn addition on microstructure and mechanical properties of magnesium alloys [J]. The Chinese Journal of Nonferrous Metals, 1999, 9(1): 55–60. (in Chinese)
- [10] PAN Fu-sheng, YANG Ming-bo, SHEN Jia, WU Lu. Effects of minor Zr and Sr on as-cast microstructure and mechanical properties of Mg–3Ce–1.2Mn–0.9Sc (wt.%) magnesium alloy [J]. Materials Science and Engineering A, 2011, 528: 4292–4299.
- [11] MAO Wei-min, ZHEN Zi-sheng, CHEN Hong-tao. Microstructure of semi-solid AZ91D alloy solidified during electromagnetic stirring [J]. Chinese Journal of Materials Research, 2005, 19(3): 303–307. (in Chinese)
- [12] XU Guang-ming, BAO Wei-ping, CUI Jian-zhong, GU Xiao-feng. Microstructure of Mg alloy ZK60 in different magnetic fields [J]. Journal of Northeastern University: Natural Science, 2004, 25(1): 48–50. (in Chinese)
- [13] WANG Bin, YANG Yuan-sheng, ZHOU Ji-xue, TONG Wen-hui. Microstructure refinement of AZ91D alloy solidified with pulsed magnetic field [J]. Transactions of Nonferrous Metals Society of China, 2008, 18(3): 536–540.
- [14] FANG Can-feng, ZHANG Xing-guo, JI Shou-hua, JIN Jun-ze, CHANG Yu-bao. Microstructure and corrosion property of AZ61 Magnesium alloy by electromagnetic stirring [J]. Transactions of Nonferrous Metals Society of China, 2005, 15(3): 536–539.
- [15] JIE Wan-qi, CHEN Zhong-wei, REIF W, MULLER K. Superheat treatment of Al–7Si–0.55Mg melt and its influences on the solidification structure and the mechanical properties [J]. Metallurgical and Materials Transactions A, 2003, 34(3): 799–806.
- [16] GU Z H, WANG H Y, ZHENG N, ZHA M, JIANG L L, WANG W, JIANG Q C. Effect of melt superheating treatment on the cast microstructure of Mg–1.5Si–1Zn alloy [J]. J Mater Sci, 2008, 43: 980–984.
- [17] CAO Peng, QIAN Ma, DAVID H S. Mechanism for grain refinement of magnesium alloys by superheating [J]. Scripta Materialia, 2007, 56: 633–636.
- [18] MOTEGI T. Grain-refining mechanisms of superheat-treatment of and carbon addition to Mg–Al–Zn alloys [J]. Materials Science and Engineering A, 2005, 413: 408–411.
- [19] WANG Guang-hou. Recent progress on cluster physics [J]. Progress in Physics, 1994, 14(2): 121–171.

[20] JIAN Zeng-yun, ZHOU Jing, CHANG Fang-e, JIE Wan-qi. Research on the hysteresis of atom cluster size variation in Ga melt from the nucleation undercooling [J]. *Acta Metallurgica Sinica*, 2009, 45(9): 1146–1152. (in Chinese)

[21] LIU F, SOMMER F, BOS C, MITTEMEIJER E J. Analysis of solid state phase transformation kinetics: Models and recipes [J]. *International Materials Reviews*, 2007, 52(4): 193–212.

熔体温度对 AZ31B 镁合金铸态组织及力学性能的影响

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摘要: 采用自制的电阻炉研究熔体温度对 AZ31B 镁合金凝固组织与拉伸性能的影响。结果表明: 在温度低于 850 °C 水冷时, 金属型 AZ31B 镁合金铸锭等轴枝晶的尺寸随着熔体温度的升高呈直线下降, 超过 850 °C 后变化不大。组织中第二相呈现出先细化后粗化的变化规律。AZ31B 镁合金试样的抗拉强度、伸长率和屈服强度随着熔体温度的提高而先快速增大后略有减小, 熔体温度为 850 °C 时试样的抗拉强度达到 260 MPa, 屈服强度达到 75.4 MPa, 伸长率达到 27.57%, 比熔体温度为 750 °C 时的分别提高了 15%、13% 和 61%。DSC 分析表明, 升高熔体温度使凝固开始点温度降低, 临界晶核半径减小, 从而增加了熔体中的过冷度, 提高了熔体中非均匀形核率, 是镁合金晶粒细化和拉伸性能提高的主要原因。

关键词: AZ31B 镁合金; 铸态组织; 力学性能; 熔体温度

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