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Effects of technical parameters of continuous semisolid rolling on microstructure and mechanical properties of Mg-3Sn-1Mn alloy

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Abstract: A sloping semisolid rheo-rolling process of Mg–3Sn–1Mn alloy was developed, and the effects of process parameters on the microstructure and mechanical properties of Mg–3Sn–1Mn alloy strip were studied. The results show that the primary grain average diameter of the strip increases with the increase of the roll speed. The primary grain average diameter decreases firstly and then increases with the increase of the vibration frequency, and the tensile strength and elongation of the strip increase firstly and then decrease with the increase of the vibration frequency. The primary grain average diameter increases with the increase of casting temperature, and the tensile strength and elongation of the strip decrease correspondingly. When the casting temperature is 670 °C, the roll speed is 52 mm/s, and the vibration frequency is 60 Hz, Mg–3Sn–1Mn alloy strip with good properties is produced. The mechanical properties of the present product are higher than those of Mg–3Sn–1Mn alloy casting with the addition of 0.87% Ce (mass fraction).

Key words: Mg-3Sn-1Mn alloy; semisolid; continuous rheo-rolling forming; microstructure; mechanical properties

1 Introduction

Magnesium resource in the earth is very rich, and magnesium alloys have many application merits such as low density, high strength and non-toxicity, so magnesium alloys are respected to be used in many fields, especially in automobile [1-3]. The wrought magnesium alloys are used commonly based on Mg-Al alloys that have reasonable formability at elevated temperatures. But Mg-Al alloys have relatively poor mechanical properties at high temperatures, mainly due to the thermal instability of Mg₁₇Al₁₂ phase [4–6]. In order to develop new low-cost wrought magnesium alloys with good creep and mechanical properties at high temperature, Mg-3Sn-1Mn (mass fraction, %) alloys are of particular interest. However, the plasticity of magnesium alloy is not good, and the production cost of magnesium alloy materials is high. Manufacturing magnesium alloy materials becomes a hot subject all over the world [7,8]. Semisolid rolling is a near-net shape metal forming process. Semisolid metal is rolled

directly to strip by this process, so this technique has outstanding advantages of saving energy, good product quality and low cost. Preparing good quality slurry is the first key procedure in this process [9,10]. Many slurry preparation processes were developed, such as mechanical stirring [11], magneto hydrodynamic (MHD) stirring [12], melt conditioner direct chill casting [13], bubble stirring [14]. Among them, sloping plate process has the advantages of high efficiency and low cost, and attracts much attention all over the world [15]. The principle is to cast the liquid metal onto a sloping plate, and then the melt is cooled and stirred by sloping plate and transforms into semisolid slurry with fine spherical primary grains. MOTEGI [16] manufactured the semisolid billet of Al-Si-Mg alloy by this process. The slurries of stainless steel, A356, A2017 and AZ91 alloys were also prepared in the previous studies [15]. HAGA [17] developed the rheo-rolling process of aluminum alloys by using the sloping plate device, and the rolling speed and the plasticity of the alloy were greatly improved. KAPRANOS et al [18], GUO et al [9] and GRIMMIG et al [19] also carried out the similar

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research work on this technique, which accelerated the development of this process. Previous studies adopted stationary plates that usually caused slurry adhesion on the plate. In this work, a vibrating sloping plate was adopted to prepare semisolid Mg-3Sn-1Mn alloy, and the semisolid alloy was directly rolled into the strip by using a modified rolling mill. The effects of technical parameters of continuous semisolid rolling on the microstructure and mechanical properties of Mg-3Sn-1Mn alloy were studied.

2 Experimental

Figure 1 shows the schematic diagram of continuous semisolid rolling. The melt was cast onto the vibrating sloping plate surface and then nucleated rapidly under strong cooling provided by the cooling plate. The primary grains grew into fine spherical structures gradually under stirring of the vibration and melt flow. Subsequently, the slurry flowed into the roll gap. The two broadsides of roll gap were constrained by the convex and concave rolls, so semisolid rolling was achieved. The roll speed of semisolid alloy is higher than that of the conventional liquid roll casting, and the microstructure and mechanical properties of the product are better than that produced by the conventional roll casting. The roll diameter of experimental machine is 400 mm, and the maximum roll speed is 366.67 mm/s. The rolls are cooled by circulating water. The cross section size of the strip is 4 mm×160 mm. Table 1 shows the chemical composition of experimental material of Mg-3Sn-1Mn alloy. The liquidus and solidus temperatures of the alloy are 645 and 620 °C, respectively. The Mg-3Sn-1Mn alloys were prepared by adding pure Mg and Sn (>99.9%, mass fraction), and 99.9% Mn (mass fraction), which were melted in a resistance furnace and protected by argon. When the melt temperature approximately reached 750 °C, the melt was homogenized by mechanical stirring and refined. After complete mixing, the melt was held at 750 °C for 30 min and carried to the testing machine, and then cast onto the vibrating sloping plate surface under the protection by argon. The casting temperature varied from 650 to 750 °C, and the roll speed varied from 40 to 80 mm/s. In order to study the effects of technical parameters of continuous semisolid rolling on the microstructure and mechanical properties of Mg-3Sn-1Mn alloy, the strips produced under different process parameters were taken for the microstructure observation. The specimens were polished and etched by a solution of 15 mL HCl+56 mL C₂H₅OH+47 mL H₂O, and the microstructures were observed by an Olympus PMG51 metallographic microscope. The mechanical properties of the strips were measured by using a CMT5105 tensile machine.



Fig. 1 Schematic diagram of continuous semisolid rolling

 Table 1 Chemical composition of Mg-3Sn-1Mn alloy (mass fraction, %)

Sn	Mn	Ca	Ni	Fe	Mg
3	1	0.5	0.01	0.01	Bal.

3 Results and discussion

3.1 Effect of roll speed on microstructure of Mg-3Sn-1Mn alloy

The microstructures of Mg-3Sn-1Mn alloy strips at center part produced at different roll speeds are shown in Fig. 2. The relationships between solid fraction, average grain size and roll speed are shown in Fig. 3. The results reveal that the microstructures are mainly composed of spherical or rosette primary grains and some small secondary crystallization grains. The secondary crystallization grains transform from remnant liquids in the roll gap and distribute between the primary grains under high roll speed. The primary grain size increases and solid fraction decreases with the increase of the roll speed. It is commonly believed that heterogeneous nucleus forms on the plate surface and dendrite breakage causes the microstructure refinement during the sloping plate process [20,21]. The recent studies revealed that the temperature distribution in most of the melt is homogenous, and the cooling rate of the melt on the water-cooling sloping plate can reach 1000 K/s and is much higher than that of the conventional casting (usually less than 100 K/s). Therefore, eruptive nucleation can happen in the whole melt [15]. The heterogeneous nucleation and eruptive nucleation cause fine microstructure of the strip finally. The nucleation rates of heterogeneous nucleation and eruptive nucleation are all affected by cooling and stirring time of the melt during the progress. The cooling and stirring time decreases with the increase of roll speed, so the nucleation rate decreases with the increase of roll speed correspondingly. Therefore, the strip grain size decreases with the increase of roll speed. At the same time, since the cooling time decreases with the increase of roll speed, the liquid fraction increases with the increase of



Fig. 2 Cross-sectional microstructures of Mg-3Sn-1Mn alloy strips at center part produced at different roll speeds (vibration frequency 40 Hz, casting temperature 670 °C): (a) 52 mm/s; (b) 87 mm/s



Fig. 3 Relationship between solid fraction, average grain size and roll speed (vibration frequency 40 Hz, casting temperature $670 \,^{\circ}\text{C}$)

roll speed correspondingly. The roll speed of 52 mm/s is suggested in this work.

3.2 Effect of vibration frequency on microstructure and properties of Mg-3Sn-1Mn alloy

It is found that the slurry adhesion on stationary plate surface usually happens. In order to avoid this phenomenon, a vibrating sloping plate was adopted. The slurry adhesion was avoided effectively. In addition, vibration can promote stirring strength and is helpful to establishing homogenous solute and temperature fields that are the ideal conditions for eruptive nucleation. Moreover, vibration can accelerate the dendrite breakage and heterogonous nucleus liberation, which also causes grain refining. Figure 4 shows the microstructures of Mg-3Sn-1Mn alloy strip on the cross-section produced at different vibration frequencies. Figure 5 shows the relationship between the average grain size and vibration frequency. Figure 6 shows the effect of vibration



Fig. 4 Cross-sectional microstructures of Mg–3Sn–1Mn alloy strip produced at different vibration frequencies (casting temperature 670 °C, roll speed 52 mm/s): (a) 60 Hz; (b) 80 Hz



Fig. 5 Relationship between average grain size and vibration frequency (casting temperature 670 °C, roll speed 52 mm/s)



Fig. 6 Effect of vibration frequency on mechanical properties of Mg-3Sn-1Mn alloy strip (casting temperature 670 °C, roll speed 52 mm/s)

frequency on the mechanical properties of Mg-3Sn-1Mn alloy strip. It is found that the primary grain size decreases firstly and then increases with the increase of vibration frequency, and the tensile strength and elongation of the strip increase firstly and then decrease with the increase of vibration frequency. The critical value is 60 Hz. Stirring strength of vibration can affect the grain size of the alloy. The vibration frequency

and vibration amplitude have a linear relationship established by the regression of test value as follows:

$$A = -0.015 f + 1.7 \tag{1}$$

where A represents the vibration amplitude and f is the vibration frequency. Through Eq. (1), it can be seen that the vibration amplitude decreases with the increase of vibration frequency. When the vibration frequency is lower than 60 Hz, the frequency vibration will dominate the stirring strength of the alloy. So, the grain size decreases with the increase of frequency vibration. Once the vibration frequency is higher than 60 Hz, the vibration amplitude will determine the stirring strength. So, the grain size increases with the increase of vibration frequency and the decrease of vibration amplitude. The vibration frequency is suggested to set at 60 Hz in this work.

3.3 Effects of casting temperature on microstructure and properties of Mg-3Sn-1Mn alloy

The cross-sectional microstructures of Mg-3Sn-1Mn alloy strips produced at different casting temperatures are shown in Fig. 7. Figure 8 shows the grain size comparsion of Mg-3Sn-1Mn alloy strips produced at different casting temperatures. It is found that the primary grains become coarse with the increase of casting temperature gradually. The grain size of Mg-



Fig. 7 Cross-sectional microstructures of Mg-3Sn-1Mn alloy strips produced at different casting temperatures (vibration frequency 20 Hz, roll speed 70 mm/s): (a) 670 °C; (b) 690 °C; (c) 710 °C; (d) 730 °C



Fig. 8 Grain size comparison of Mg–3Sn–1Mn alloy strips produced at different casting temperatures (vibration frequency 20 Hz, roll speed 70 mm/s)

Figure 9 shows the relationship between the mechanical properties of the strips and casting temperature. The tensile strength and elongation of the strips decrease with the increase of casting temperature. The reason is that the grain size becomes larger with the increase of casting temperature. As mentioned before, heterogeneous nucleation and eruptive nucleation on the sloping plate are the main reasons for fine microstructure formation. Heterogeneous nucleation and eruptive nucleation on the sloping plate are closely related to casting temperature. Generally, nucleation rate increases with the decrease of casting temperature. Therefore, the grain size of the strip also decreases with the decrease of casting temperature. But if the casting temperature is too low, the flow ability of alloy is not good, which will affect the stability of the process, so the casting temperature of 670 °C is suggested.



Fig. 9 Relationship between mechanical properties of strips and casting temperatures (vibration frequency 20 Hz, roll speed 70 mm/s)

3.4 Microstructure and properties of product

When the casting temperature was 670 °C, the roll speed was 52 mm/s, and the vibration frequency was 60 Hz, Mg-3Sn-1Mn alloy strip with a cross-section size of 4 mm×160 mm was produced by the proposed process, and the strip surface quality is good, as shown in Fig. 10. From Fig. 10, it can be seen that the inner microstructure of Mg-3Sn-1Mn alloy strip is mainly composed of fine spherical or rosette grains, and the average grain size is 47 μ m. It is reported that the addition of 0.87% Ce (mass fraction) to the Mg-3Sn-1Mn alloy can refine the grains and improve the tensile and elongation of the alloy [22]. The ultimate tensile strength of Mg-3Sn-1Mn alloy casting with the addition of 0.87% Ce was 165 MPa, and the elongation was 3.9% [22]. In the present work, the ultimate tensile strength of Mg-3Sn-1Mn alloy is 175 MPa, and the elongation to failure is 5.6%, as shown in Fig. 11. Therefore, the ultimate tensile strength and elongation are improved respectively by 24.8% and 84.6% in comparison with Mg-3Sn-1Mn alloy casting



Fig. 10 Mg-3Sn-1Mn alloy strip and its microstructures obtained at optimized parameters: (a) Mg-3Sn-1Mn alloy strip; (b) Microstructure of product on longitudinal section; (c) Microstructure of product on cross-section



Fig. 11 Stress-strain curve of Mg-3Sn-1Mn alloy strip

with the addition of 0.87% Ce (mass fraction). The present technique can be used to produce Mg-3Sn-1Mn alloy strip with better mechanical properties. Additionally, the strip produced by the rheo-rolling usually needs deep rolling, so the final properties can be improved significantly.

4 Conclusions

1) The primary grain size of Mg-3Sn-1Mn alloy strip increases and the solid fraction decreases with the increase of roll speed.

2) The primary grain size decreases firstly and then increases with the increase of vibration frequency, and the tensile strength and elongation of strip increase firstly and then decrease with the increase of vibration frequency.

3) The primary grain size of Mg–3Sn–1Mn alloy strip increases with the increase of casting temperature, and the tensile strength and elongation of the strip decrease correspondingly.

4) When the casting temperature is 670 °C, the roll speed is 52 mm/s, and the vibration frequency is 60 Hz, Mg-3Sn-1Mn alloy strip with good quality is produced by the proposed process. The mechanical properties of the present product are better than that of Mg-3Sn-1Mn alloy casting with addition of 0.87% Ce (mass fraction).

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工艺参数对连续流变轧制成形 Mg-3Sn-1Mn 合金板材组织和力学性能的影响

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摘 要:开发了 Mg-3Sn-1Mn 合金板材倾斜板连续流变轧制成形工艺,并研究工艺参数对合金板材微观组织和 力学性能的影响。结果表明:随着轧辊转速的增加,板材的初生晶粒平均直径增大;随着倾斜板振动频率增加, 板材的初生晶粒平均直径先减小后增大,板材的抗拉强度和伸长率先增加后降低。随着浇注温度的升高,板材的 初生晶粒平均直径逐渐增大,板材的抗拉强度和伸长率逐渐降低。当浇注温度为 670 °C、轧辊转速为 52 mm/s、 倾斜板振动频率为 60 Hz 时,制备了组织性能较好的 Mg-3Sn-1Mn 合金板材,其力学性能优于添加 0.87%Ce(质 量分数)的 Mg-3Sn-1Mn 合金热轧板材的力学性能。

关键词: Mg-3Sn-1Mn 合金; 半固态; 连续流变轧制成形; 微观组织; 力学性能

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