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Effect of Process Parameters on the Microstructure of Semi-solid ZL101 Aluminum Alloy

Shujian Cheng[a], Hua Hou[a]*, Yuhong Zhao[a], Yuchun Jin[a]

[a]*Material Science and Engineering College, North University of China, Taiyuan 030051, China*

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Aluminum alloy is the important alloy which has been widely used. To reduce waste, it is necessary to thixoform these alloys in near net shape. Semi-solid slurry is the key in process. In this work, the semi-solid slurry of ZL101 aluminum alloy was prepared by using serpentine channel. Effect of process parameters on the microstructure of semi-solid ZL101 aluminum alloy produced by serpentine channel was investigated. The results show that, the morphology of primary α-Al grains transformed from rosette to spheroid with decreasing pouring temperature. Satisfied semi-solid slurry of ZL101 aluminum alloy was prepared with pouring at 630°C to 670°C. Increasing curve number can improve the morphology of primary α-Al grain and decrease grain size with same pouring temperature. Desired slurry can be obtained with the lower pouring temperature when serpentine channel is preheated. It can make the primary nucleus gradually evolve into spherical and near-spherical grains with the effect of "self-stirring" in serpentine channel and chilling.

Key *Words: Semi-solid processing, Al-alloy, Microstructure, Slurry*

1 Introduction

Many researchers have examined the benefits of semi-solid processing (SSP) over the last several decades. Semi-solid processing is a promising technology based on the thixotropic behavior of materials in semi-solid state, which takes place at temperatures between solidus and liquidus. The process depends on material behaving in a thixotropic way in semi-solid state [1-4] . The required spheroidal microstructure can be obtained by a number of routes, such as mechanical stirring [5,6], electromagnetic stirring [7,8]. However, the former readily pollutes metal and the latter has low electromagnetic stirring efficiency. Recently, a controlled nucleation method has been reported, which is simple, practical and less expensive because of no application of stirring. The preparation methods include new rheocasting [9,10], vertical pipe[11,12], vibrating wavelike sloping plate process [13-15], inverted cone pouring channel [16,17] and cooling slope processes [18-21]. These techniques have similarities, such as, pouring the alloy melt through a plate or tube without stirring. Therefore, they greatly reduce energy consumption and save cost of production.

The serpentine channel process (SCP) was presented based on controlled nucleation method. During the period of preparing semi-solid slurry, the direction of alloy melt changes several times in field of gravity, so alloy melt has function of "self-stirring" [21-25]. With effect of chilling and stirring in serpentine channel, the primary nucleus gradually evolved into spherical and near-spherical grains. In this work, an innovative processing technique of semi-solid metal slurry, namely serpentine channel process, is introduced, and effect of process parameters on microstructure of semi-solid ZL101 aluminum alloy slurry were investigated.

*e-mail: houhua@263.com

2 Experimental procedure

2.1 Materials and apparatus

In the experiment, a commercial ZL101 aluminum alloy was used. Its chemical composition (mass fraction, %) is Si 6.6%, Mg 0.28%, Fe <0.16%, Mn <0.10%, Zn <0.10% and Al balanced. The liquidus and solidus are 617°C and 556°C, which were tested by differential scanning calorimetric (DSC) method, as shown in Fig. 1.

The schematic of SCP equipment and slurry preparation process is shown in Fig. 2. The serpentine channel is made of stainless steel and consists of two symmetrical blocks locked together by sleeve. The collective crucible, with size of d 69 mm×150 mm, is made of stainless steel. The melting apparatus is a crucible resistance furnace. The temperatures of liquid aluminum alloy, semi-solid slurry and inner wall of the serpentine channel were measured by Ni−Cr/Ni−Si thermocouple. The temperature accuracy is ±1°C.

2.2 Methods

Before serpentine channel process, as-cast ZL101 aluminum alloy were machined into feedstocks with mass of 1kg. The alloy was melted in a crucible resistance furnace at 720−730°C, and then incubated for 15 min. The alloy melt was cooled to chosen pouring temperature and casted into collective crucible via serpentine channel, and then rapidly quenched in cold water to obtain the high temperature solidification microstructure. The collective crucible was at room temperature. The temperature of serpentine channel were 100°C or 25°C before pouring. The processing parameters and characteristic parameter of semi-solid slurry are listed in Table 1.

Specimens for optical microscopy observation were cut from the center of quenched slurries and then roughly

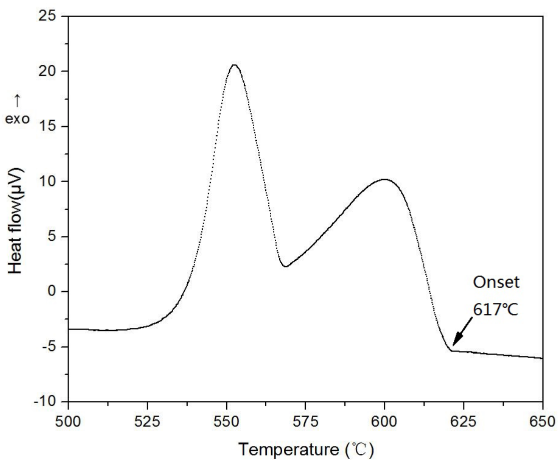

Fig. 1 DSC cooling curves of ZL101 aluminum alloy

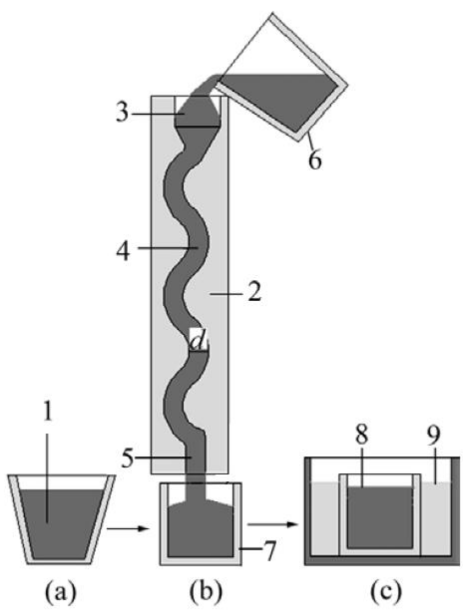

Fig. 2 Schematic diagram of preparing semi-solid ZL101 aluminum alloy slurry by serpentine channel: (a) Melt; (b) Preparation of semi-solid slurry; (c) Cooling. 1—K-type thermocouple; 2—Serpentine channel; 3—Pouring cup; 4—Serpentine bend; 5—Diversion pipe; 6—Melting crucible; 7—Collective crucible; 8—Slurry; 9—Cold water

ground, finely ground, polished and etched with 0.5% HF aqueous solution finally. Microstructures of specimens were investigated by ZIESS optical microscope. The professional image analysis software (Image-Pro Plus) was adopted to analyze equivalent particle diameter (*D*) and average shape factor (S_f) of primary α-Al grains which were calculated by the following equations:

$$D = \frac{\sum_{N=1}^{N} \sqrt{4A/\pi}}{N} \quad (1)$$

$$S_f = \frac{\sum_{N=1}^{N} \frac{4\pi A}{P^2}}{N} \quad (2)$$

Where *A* and *P* are the area and perimeter of a particle, respectively. The S_f varies from 0 to 1, when the value of S_f is close to 1, particle shape approaches to a circle.

3 Results and discussion

3.1 As-cast and SCP microstructures

ZL101 aluminum alloy semi-solid slurry is obtained with effect of chilling and stirring in serpentine channel. The microstructures of quenched slurry were prepared by serpentine channel and conventional casting, as shown in Fig. 3. The white phase is primary α-Al and the black matrix is the eutectic phase [26,27].

Fig. 3(a) shows the microstructure of conventional casting. The particles morphology is typical dendrites and the length is more than 100μm. Fig. 3(b) shows the microstructure of semi-solid slurry prepared by SCP. It can be seen that non-dendritic primary α-Al particles are uniform throughout entire cast. The phenomenon indicates that collective crucible was filled completely and solidification of remaining liquid might have occurred throughout entire component [19]. In addition, there are two main constituents in the microstructure: the relatively large α-Al globules (marked as α_1-Al) with particle diameter about 30-80μm, and the microstructure is composed of numerous small α-Al particles (marked as α_2-Al) of approximately 15μm diameter. There are two distinct solidification stages in the SCP. Solidification of slurry in serpentine channel is referred to as the first solidification, and solidification of slurry in collective crucible is referred to as the secondary solidification. The temperature declines rapidly in serpentine channel. A large number of nucleus are created in slurry, which grow larger α_1-Al particles in serpentine channel. The particles morphology is almost spherical and they are uniformly distributed throughout the microstructure. When alloy melt is poured into collective crucible, heterogeneous nucleation recurs in the remaining liquid. All the nucleus grow and solidify within a short time, forming the smaller α_2-Al particles, creating the ‘‘secondary solidification’’. Solidification time is very short because of quenching, and as a result, the primary α_2-Al particles are very small and less spherical in shape.

Equivalent particle diameter and average shape factor of primary α-Al grains are listed in Table 1. The equivalent particle diameters and the average shape factors of primary α-Al grains are 83, 75, 70, 62μm and 0.42, 0.47, 0.52, 0.59, which produced by 3 curves serpentine channel with pouring temperature at 690, 670, 650, 630°C, respectively. Fig. 4(a) ~ (d) show the microstructures of ZL101 aluminum alloy semi-solid slurry poured at 690, 670, 650, 630°C by serpentine channel with 4 curves, respectively. The equivalent particle diameters and the average shape factors of primary α-Al grains are 75, 68, 63, 59μm and 0.49, 0.56, 0.64, 68, respectively. The equivalent particle diameters and the average shape factors of primary α-Al grains are 65, 63, 58, 55μm and 0.53, 0.58, 0.67, 0.72, which produced by 5 curves serpentine channel with pouring temperature at 690, 670, 650, 630°C, respectively. Desired quality of slurry can be also achieved with lower pouring temperature when serpentine channel is preheated, as shown in Fig. 8.

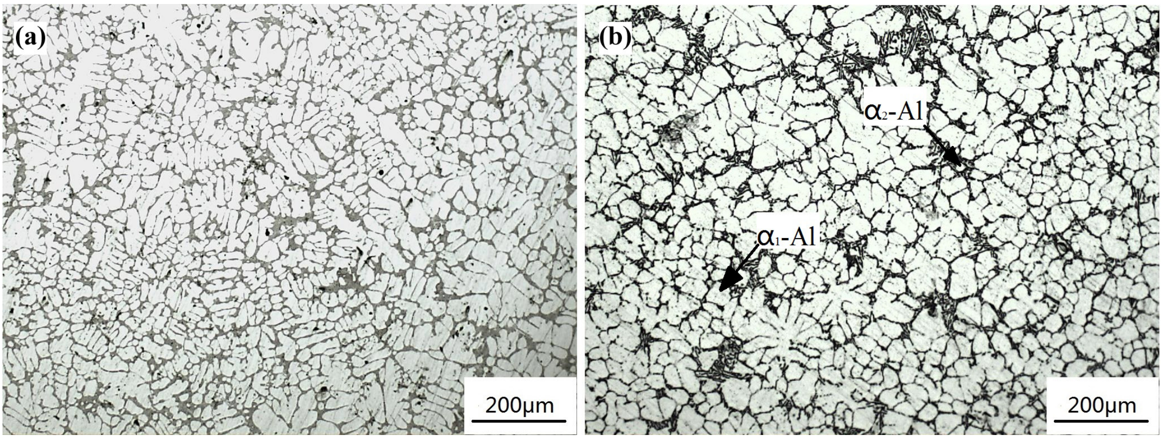

Fig. 3 Microstructures of ZL101 aluminum alloy slurry prepared by two methods (a) conventional casting; (b) serpentine channel

Table 1. Processing parameters and characteristic parameter of semi-solid slurry

Sample No.	Curve number	Curve diameter/mm	Pouring temperature/°C	Serpentine channel temperature/°C	Pouring time/s	Slurry mass/kg	Equivalent particle diameter /μm	Average shape factor
A1	3	25	690	25	2	1.03	83	0.42
A2	3	25	670	25	2	0.99	75	0.47
A3	3	25	650	25	2	1.02	70	0.52
A4	3	25	630	25	2	1.04	62	0.59
B1	4	25	690	25	2	1.02	75	0.49
B2	4	25	670	25	2	0.98	68	0.56
B3	4	25	650	25	2	1.00	63	0.64
B4	4	25	630	25	2	0.99	59	0.68
C1	5	25	690	25	2	1.04	65	0.53
C2	5	25	670	25	2	1.06	63	0.58
C3	5	25	650	25	2	0.98	58	0.67
C4	5	25	630	25	2	0.98	55	0.72

3.2 Microstructural evolution with increasing pouring temperature

Figs. 4 shows the microstructures of semi-solid ZL101 aluminum alloy slurry prepared by serpentine channel with 4 curves at different temperature .The characteristic parameter of semi-solid slurry is listed in Table 1. As shown in Figs. 4(a) ~ (d), primary α-Al grains are more and more spherical under the condition of decreasing pouring temperature using same serpentine channel. Fig. 4(a) shows that when pouring temperature is 690°C, the morphology of primary α-Al grains is irregular. A similar microstructure can be seen and more non-dendritic primary α-Al grains can be found in Fig. 4(b). Fig. 4(c) ~ (d) show that the primary α-Al grains are mainly near-spherical and spherical with pouring temperature decreased. Declining the pouring temperature leaded to equivalent particle diameter of the primary α-Al grains decreases and average shape factor increases, as shown in Fig. 5. The initial temperature of serpentine channel is low and it has chilling effect, which decreases the critical energy of nucleation and critical nucleus radius increases the nucleation ratio. Therefore, a large number of the primary α-Al nuclei will be generated. When pouring temperature is high, the inner wall of serpentine channel will be heated, resulting in chilling effect being weakened and nucleation ratio decreased. And grains have more time to grow. If pouring temperature is lower, the temperature of alloy melt in serpentine channel will decline rapidly below to liquidus. It is helpful for nucleation [28]. However, when pouring temperature is too low, the thickness of solidified shell in serpentine channel increases markedly, and the chilling effect of serpentine channel will be weakened. So semi-solid slurry can be prepared with satisfied quality when the pouring temperature is between 630 and 670°C.

3.3 Microstructural evolution with increasing curve number

Fig. 6(a) ~ (c) shows the microstructure produced by serpentine channel with different number of curves. Primary α-Al grains are smaller and more spherical made by 5 curves serpentine channel, as shown in Table 1. It can be seen from Fig. 6(a) that a small number of fine rosettes or dendrites primary α-Al grains apart from near-spherical or spherical. In addition, almost all of the primary α-Al grains are near-spherical or spherical which made by 5 curves, as shown Fig. 6(c). As the number of curves was increased, solid grains refining and the degree of spheroidization improving [29].

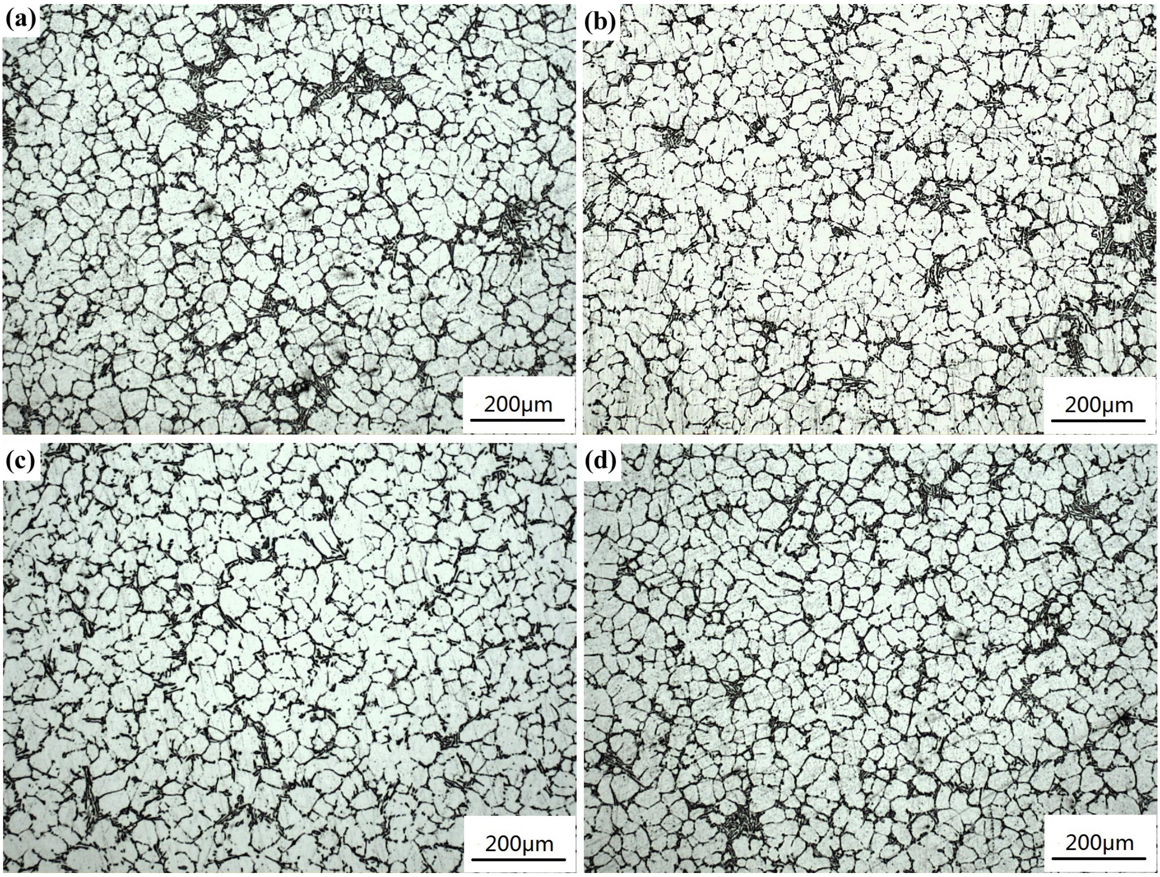

Fig. 4 Microstructures of semi-solid ZL101 aluminum alloy slurry prepared by serpentine channel with 4curves at different temperatures (a) 690°C; (b) 670°C; (c) 650°C; (d) 630°C

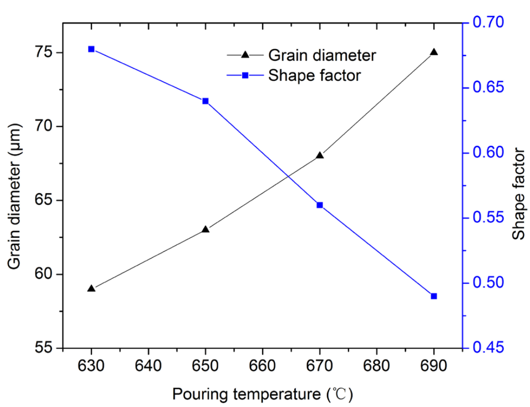

Fig. 5 Effects of pouring temperature on D and S_f

However, the hanging slurry increases with increasing curve number. The variation of equivalent particle diameters and average shape factors with different number of curves is shown in Table 1. Graphs about equivalent diameter and average shape factor of the primary α-Al grains prepared by three kinds of the serpentine channels with 3, 4, 5 curves respectively are shown in Fig. 7, which indicates that at the given pouring temperature of serpentine channel, equivalent particle diameter of the primary α-Al grains decreases and average shape factor of the primary α-Al grains increases with the curve number increasing. The inner wall of serpentine channel which acts as the concave nucleation substrate has a favorable effect on heterogeneous nucleation, so many primary α-Al nucleus form in a heterogeneous nucleation pattern. More crystal nucleus can be generated as the area of the heterogeneous nucleation substrate enlarges with increasing number of curves. Meanwhile, the self-stirring of alloy melt is strengthened with curve number increasing, which has a favorable effect on breaking of dendrites and refining primary α-Al grains. It can be seen from this group of experiments that better slurry can be obtained with 5 curves serpentine channel, but the hanging slurry is aggrandized. Therefore, the serpentine channel with 4 curves is appropriate.

3.4 Effect of serpentine channel temperature on the microstructure

Fig. 8 shows the microstructures of semi-solid slurry prepared by serpentine channel with different temperature. Primary α-Al are composed of vast spherical and a small number of fine rosettes grains. The serpentine channel was preheated at 100°C or 25°C. Comparing Fig. 8(a) with Fig. 8(b), equivalent particle diameter increases and average shape factors decreases at the same condition with the

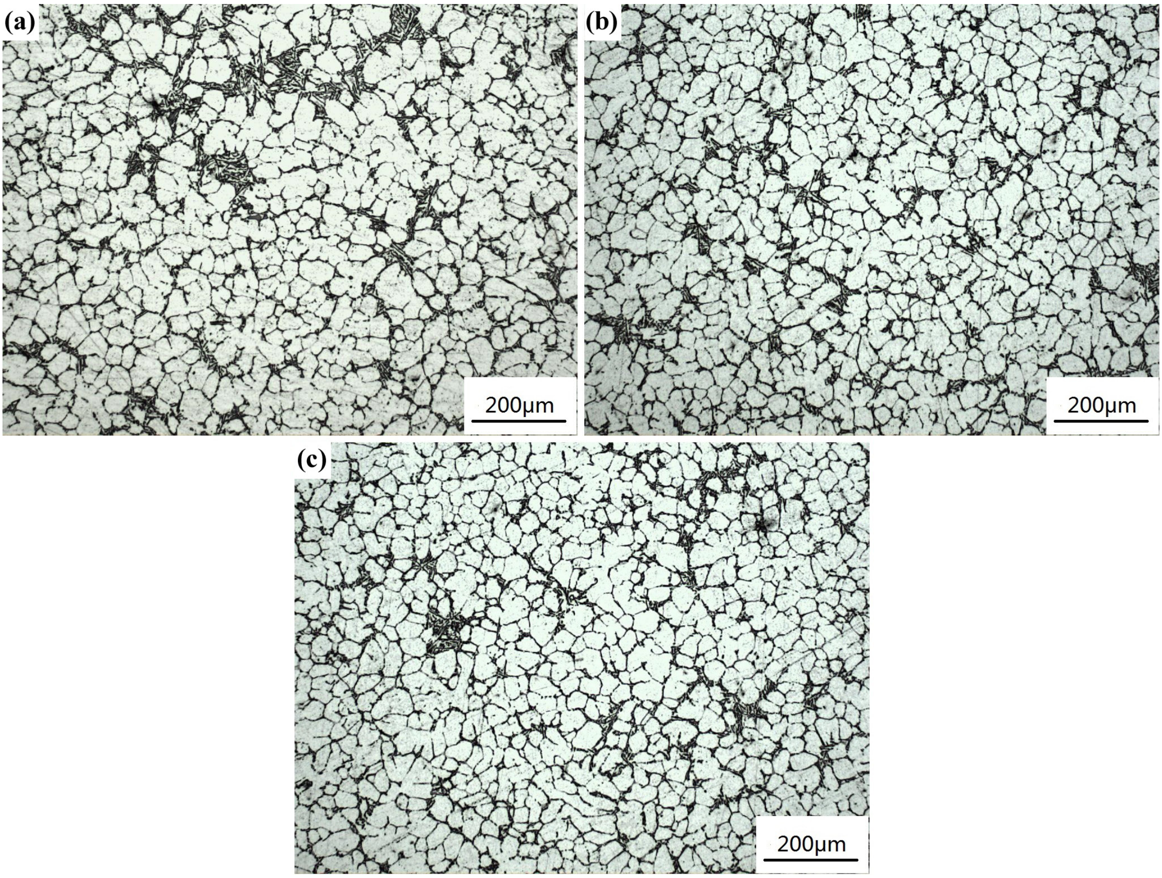

Fig. 6 Microstructures of semi-solid ZL101 aluminum alloy slurry prepared by serpentine channel with different curve numbers at 630°C (a) 3 curves; (b) 4 curves; (c) 5 curves

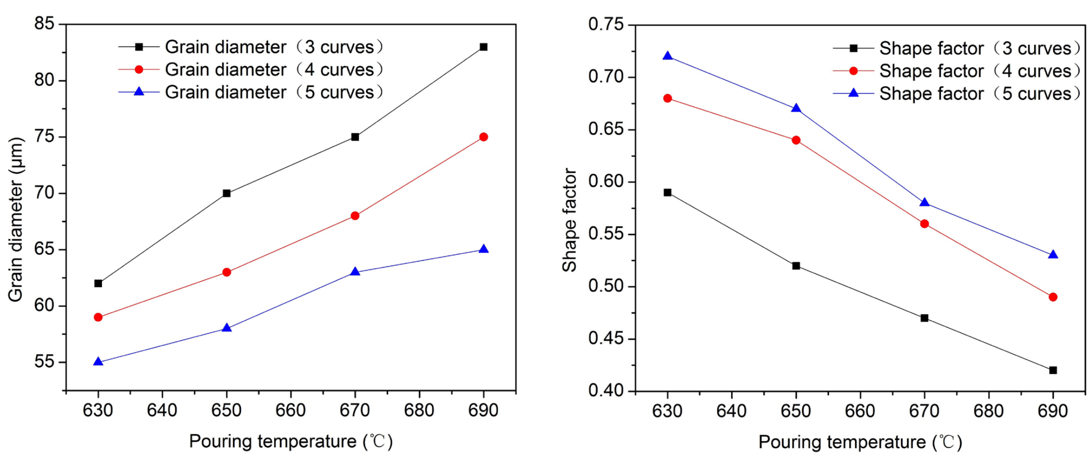

Fig. 7 Effects of curve number on *D* and S_f

decreasing serpentine channel temperature. It can be seen in the experiment that efficiency is higher when the serpentine channel is preheated. Super-cooling is little when serpentine channel temperature is high, which is not good for refining grains. The number of crystal nuclei can be enlarged with decreasing pouring temperature, and getting favorable slurry.

The temperature of serpentine channel increases when slurry is produced abidingly. Cooling serpentine channel contributes to getting large super-cooling and receiving desired slurry. But it is difficult to control the serpentine channel temperature through cooling process which increased cost. Investigating the relationship between pouring temperature and serpentine channel temperature is more important. It is good for continuous production and saving cost. When the alloy melt flows through serpentine channel, its temperature continuously drops and viscosity increases. At the same

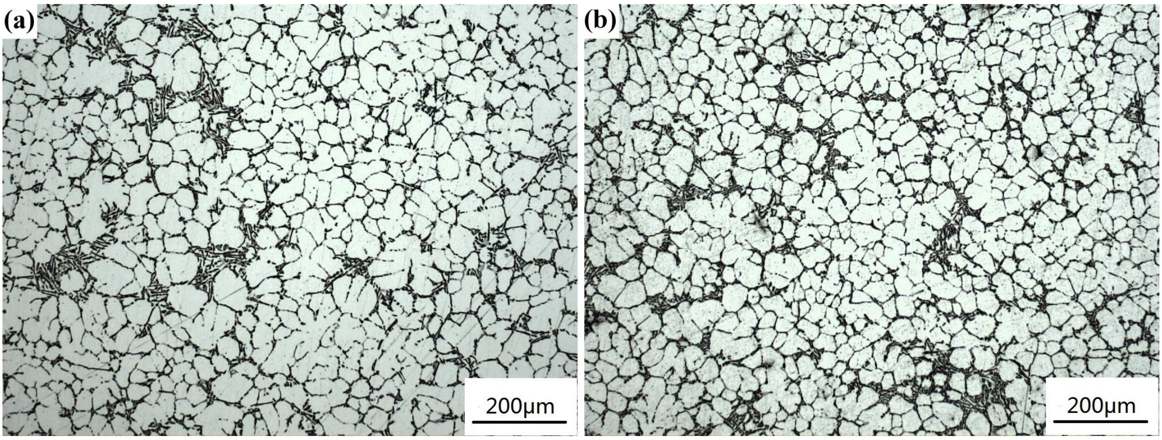

Fig. 8 Microstructures of semi-solid ZL101 aluminum alloy slurry prepared by serpentine channel with different temperature: (a) 100°C; (b) 25°C

time, the friction between alloy melt and inner wall varies with the viscosity, resulting in displacements in different points of the alloy melt, which will lead to shearing forces. What's more, the shearing forces are diverse in different points of the alloy melt, so primary α-Al grains in the alloy melt will self-rotate. It can be seen in the experiment that lowing pouring temperature can solve the problem and receive good slurry.

4 Conclusions

The semi-solid ZL101 aluminum alloy slurry with desired quality can be generated by serpentine channel. Here the microstructural evolution of such materials in the semi-solid state has been analysed.

The equivalent particle diameter of primary α-Al grains decreases and the average shape factor of primary α-Al grains increases with the pouring temperature decreasing, and desired pouring temperature is in the range of 630 and 670°C.

Increasing curve number of serpentine channel can decrease the equivalent particle diameter of primary α-Al grains and increase the average shape factor of primary α-Al grains. Considering about quality of slurry and efficiency, 4 curves serpentine channel is appropriate.

Higher temperature of serpentine channel makes the grains coarse. Semi-solid slurry meets requirements of thixomolding. It ensures that continued access to slurry and reduce the preparation cost.

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