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Effect of high shear rate on solidification microstructure of semisolid AZ91D alloy

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Abstract: To investigate the effects of rotation speed and shearing time on morphology of semisolid AZ91D alloy, experimental work was undertaken using a twin-screw slurry maker. The results show that increasing the rotation speed and reasonable time can give rise to substantial grain refinement during continuous shearing stage, which can be attributed to the increasing of effective nucleation rate caused by the extremely uniform temperature due to high shear rate and high degree of turbulence. Comparing with low rotation speed at the same thermal condition, the analysis indicates that the microstructures obtained at high rotation speed are homogenous spherical and fine grains instead of dendritic or rosette and exhibits uniform distribution in the eutectic matrix.
key words: semisolid; high shear rate; solidification; AZ91D; rotation speed

1 Introduction

Over the years, semisolid metal (SSM) processing has been established as a unique manufacturing route for the production of near-net-shape components of high integrity and performance. Different methods can be used to obtain thixotropic structures. One approach that has received a lot of attention in the past few decades is to apply agitation to the melt during solidification to achieve grain multiplication. Different agitations have been introduced into the production of semisolid slurries with thixotropic microstructures since the inception of semisolid processing. Among them, a specific shear rate from external source was imposed on semisolid processing [1−3]. Several techniques have been used successfully to achieve thixotropic structures by applying a specific shear rate, for example, electromagnetic stirring[4], new rheocasting (NRC) process[5], gas bubble purging[6], etc. These methods have been recognized as low shear rate with no more than 1 000 s^{-1} to produce semisolid slurry. However, high shear rate of metal alloys during solidification can produce fine spheroidal microstructure suspended in the liquid matrix. Among them, the twin screw slurry maker is characterized by high shear rate and high degree of turbulence[7]. FAN etal[8−9] studied the influence of various process parameters such as rotation speed and shearing time in the fabrication of thixotropic microstructures under high shear rate.

In the work, numerical and experimental investigation was made to give an explanation of the formation and evolution of solidification microstructures under high shear rate. So the twin screw slurry maker developed by BCAST[10] at Brunel University for introducing high shear rate was used to investigate solidification behavior of semisolid AZ91D alloy. Experimental work was undertaken to investigate the effects of rotation speed and shearing time on the solidification microstructure of AZ91D alloy.

2 Experimental

Commercial AZ91D alloy was used in this investigation because of its wider solid-liquid range and good fluidity. Its chemical composition (mass fraction) were Al 8.3%, Zn 0.5%, Mn 0.2%, Mg bal. Differential thermal analysis(DTA) result shows that the temperature of liquidus of AZ91D alloys is 873K. AZ91D magnesium alloy was melted at 943 K in an electric resistance furnace and protected by N_2+0.4% SF_6 mixture gas. A predetermined dose of liquid alloy from the melting furnace was fed into the slurry maker. The liquid alloy

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with high shear rate was continuously cooled to a fixed temperature. Shearing temperature was controlled by setting the barrel temperature. Finally, the slurry was immediately transferred into a copper mould, and a slice of the alloy in the mould was sampled. All samples were prepared by a standard procedure, and etched in an aqueous solution of 60% (volume fraction) ethylene glycol, 20% acetic acid, 1% concentrated HNO_3. Microstructure examination was performed by a Zeiss optical microscope with a quantitative image analysis system. A close observation of microstructure can be given by the average gain diameter $D=2(A/\pi)^{1/2}$ and the shape factor $F=4\pi A/P^{2}$, where A and P are average area and average perimeter of primary phase, respectively.

3 Results

3.1 Effect of rotation speed on semisolid microstructure

The slurry maker was operated at a shearing temperature of 867 K and the shearing time of 30 s. The rotation speed of the twin-screw varied between 100 and 800 r/min, Rotation speed has a direct and positive influence on microstructure evolution. A coarser grain structure at 200 r/min is shown in Fig.1(a). When rotation speed increases to 400 r/min, the coarser microstructure will disappear and the microstructure will be mainly dominated by a fine equiaxed grain structure, as shown in Fig.1(b). Finally, the primary solid grains has a finer size, spherical morphology and a uniform distribution at 800 r/min, as shown in Fig.1(c). It is clear that intensive shearing does make the grains refiner. Fig.2 shows the effects of rotation speed on the average grain diameter and grain density of primary grains. It is evident that the grain density increases and the average grain diameter decreases with increasing of rotation speed, indicating that high shear rate suppresses, to some degree, the formation of the primary phase. However, a further increasing of shear rate cannot improve nucleation, as indicated by the constant particle density in Fig.2.

3.2 Effect of shearing time on semisolid microstructure

Shearing time is another key process factor on microstructure evolution. The morphology of primary phases still retained spherical between 10 and 150 s at the shearing temperature of 867 K and rotation speed of 500 r/min, but the distribution of average grain diameter and grain density of primary phases varied with stirring time. Fig.3 shows the effect of shearing time on volume fraction and grain density of primary phases. It is interesting to note that the critical value for the distribution of volume fraction is similar to that of grain density. That is to say,

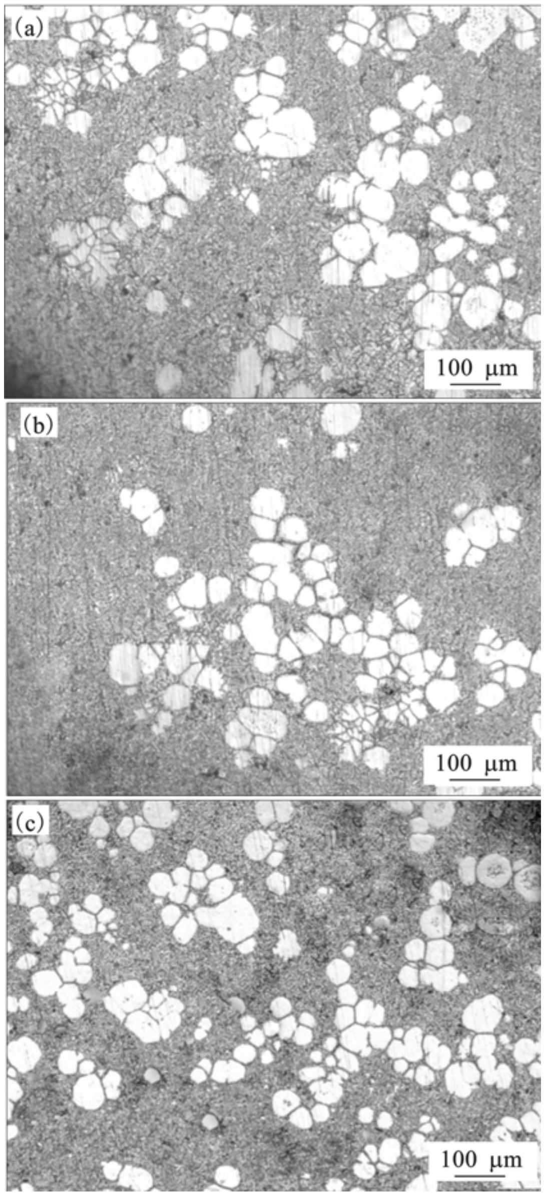

Fig.1 Microstructures of AZ91D alloy under different rotation speed: (a) n=200 r/min; (b) n=400 r/min; (c) n=800 r/min

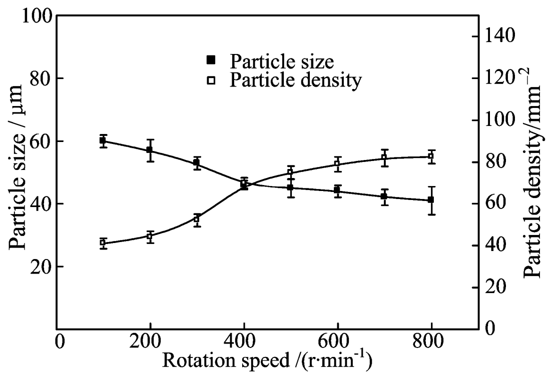

Fig.2 Effect of rotation speed on average grain diameter and grain density of primary phases

grain density increases continuously and reaches the peak at 30 s. The volume fraction also increases gradually

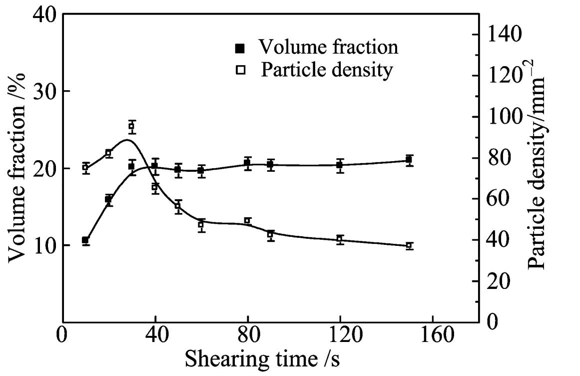

Fig.3 Effects of shearing time on volume fraction and particle density of primary phases

before 30 s, however, it is almost constant value after 30 s. After several experiments were performed between 10 and 150 s, it is confirmed that there is fine grains in the shearing time range of 20–40 s. The experimental results show that the optimum shearing time is 30s at the shearing temperature of 867 K and rotation speed of 500 r/min.

4 Discussion

When the rotation speed is not exerted in the melt, i.e. quiescent, it is similar to the conventional casting processes. Temperature at the edge of the melt is different from the center. Namely, temperature is high at the edge of the melt but low in the center. As a result, temperature field is not completely homogeneous in the melt. Heterogeneous nucleation took place in the undercooled liquid closed to the wall. The majority of the nuclei was transferred by the convection to the overheated liquid region and dissolved, which can form a small grain density. The distance between the primary grains is long and the diffusion fields overlap takes place at later times in solidification. Therefore, primary grains have more chance and space for unstable growth, and dendritic microstructures or rosette microstructures can be obtained.

The finite element platform was exploited to perform the simulation for identifying the main characteristics of flow pattern. Velocity vectors generated by the simulations for the twin-screw maker at 867 K and 100 r/min for 30s are featured in Fig.4. Rotation speed only affect velocity values, that is, velocity values increases with increasing of rotation speed. However, rotation speed cannot affect the shape of the velocity distribution. The velocity in the nip region is about 2.8 m/s corresponding to a rotation speed of 100 r/min and the calculated values of the Reynolds number is $Re=V_tH\rho/\mu$ for the melt is 10 800 with a channel depth H (10mm) and apparent viscosity μ =0.005 Pa·s. Fig.5 shows the velocity in the nip region under different rotation speed. This suggests that rotation speed with no less than 100 r/min in the semisolid state can lead to high degree of turbulence. At the same time, shear rate is calculated as $r=\pi n(D/\delta-2)$, where n and D are the rotation speed and the outer diameter of the screw, respectively, and δ is the gap between the screw flight and the inner surface of the barrel. A shear rate of 3 200 s^{-1} corresponds to a rotation speed of 100 r/min, outer diameter of 54 mm and the gap of 0.5 mm, which is much higher than 1 000 s^{-1}. Therefore, high shear rate and high degree of turbulence make the twin-screw slurry maker very powerful for dispersive mixing[11].

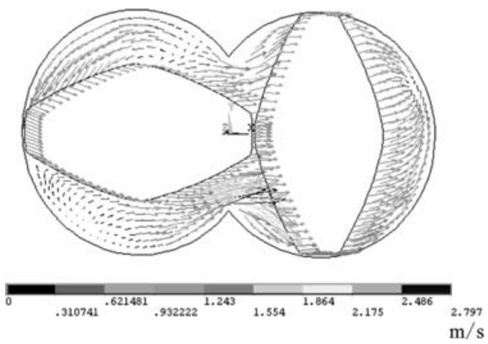

Fig.4 Velocity vectors of melt at rotation speed of 100 r/min

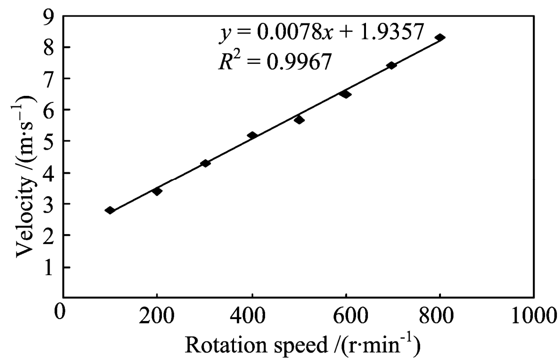

Fig.5 Velocity in the nip region under different rotation speed

Conversely, rotation speed is exerted in the melt, and the temperature of the melt might be different with different rotation speed. rotation speed has a vital effect on the temperature field. The isothermal line shift outward and the effect region of high temperature at edge becomes narrow, which can be attributed to dispersive mixing due to high shear rate and high degree of turbulence. Rotation speed brings hotter melt streams descending along the edge towards the center zone that is actively cooled. As seen from varying curve of the temperature, distribution becomes shallow with the increasing of rotation speed in Fig.6. It can be observed that larger rotation speed provides a more uniform temperature in the melt.

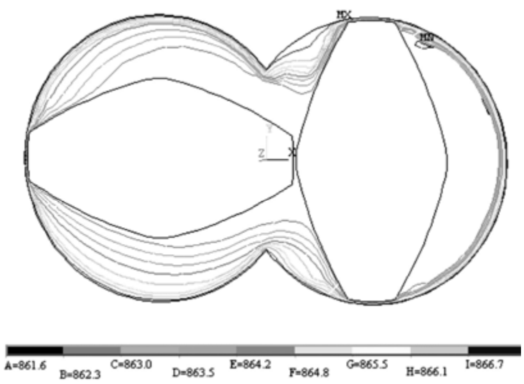

Fig.6 Temperature distribution of melt at 300 r/min

As a result, high shear rate and high degree of turbulence can make the twin-screw maker powerful for dispersive mixing. Original nucleation agents of AZ91D can be piled up in clusters, but they may obtain the extra energy resulted from the fluctuation of the melt caused by dispersive mixing. Nucleation agents tend to separate from the clusters and be broken up into smaller heterogeneous particles. Once the smaller particles are diffused fully and distributed uniformly in the melt, the nucleation sites and grain densities are certainly promoted. FAN et al [9] has already manifested that MgO particles can act as potent nucleation sites for the primary α-Mg phase. High rotation speed can effectively disperse the oxide films and oxide skins into individual MgO particles of 100–200 nm in diameter.

At the same time, the temperature field is extremely uniform and below its liquidus throughout the entire alloy melt. It is vital to avoid any chance for recalescence due to high shear rate and high degree of turbulence. Nucleation will occur in the entire volume of the liquid and each nucleus will survive. Many studies have already manifested that high grain density is beneficial to the spherical growth of primary phases. The probability of overlap of diffusion fields of the adjacent grains increases, which leads to the decreasing of concentration gradient in front of solid-liquid interface and increasing of their interface stability. In addition, the distribution of grains and spacing among them is uniform. Under this situation, the growth will be limited and stable, and ideal globular microstructures can be obtained. Moreover, a higher rotation speed provides a larger shape factor for gains.

It is important to mention that a mathematical model (1) developed based on the results of the present research with good chances is extrapolated for the effect of the rotation speed and solidification velocity on shape factor, but this topic has been contemplated for future publications.

$$S = 1 - \frac{4}{3 + \sqrt{1 + \frac{4KD_l n}{\upsilon^2}}} \qquad (1)$$

where S is the shape factor; n is the rotation speed; K is the proportional coefficient; D_l is the liquid diffusion coefficient and υ is the solidification velocity.

In order to express the morphology simply and clearly, the grain shape was simplified into a hex-angle-star shape, and Matlab was selected to describe the morphology of semisolid AZ91D alloy under different rotation speed in Fig.7(K=0.01[12], $D_l = 5.8\times10^{-9}\ \mathrm{m^2\cdot s^{-1}}$[13]).

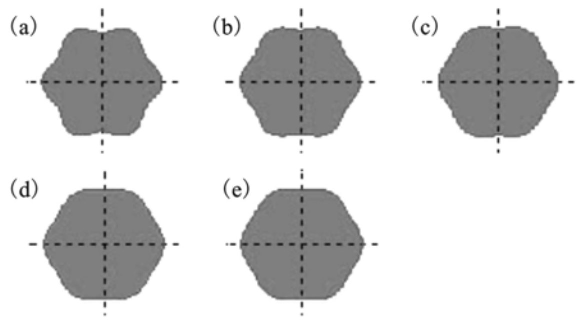

Fig.7 Simulation result for solidification velocity and rotation speed on structure evolution: (a) 200 r/min; (b) 400 r/min; (c) 600 r/min;(d) 800 r/min;(e) 1 000 r/min

During the continuous shearing stage of the primary solidification, grain density increased continuously and reached the peak at 30 s. The increased grain density during the continuous cooling can be explained by a continuous nucleation mechanism. That is, all the nuclei can survive due to the uniform temperature and the composition fields created under high shear rate. This is named as continuous effective nucleation. However, grain density decreased gradually during isothermal shearing. Moreover, grain density decreased significantly with prolonged exposure to shearing time. The main reason for decreasing in primary solid grain density is coarsening effect, coarsening acts in opposite of grain refinement effect. As the starting grains are fairly spherical in shape, Ostwald ripening between different grains is the primary mechanism for reduction in surface energy. Primary phases with smaller sizes were dissolved in liquid as a result of increasing diffusion flux from interfaces with high roundness toward interfaces with low roundness. A growing particle can be described by the classical LSW relationship[14].

$$d^3 - d_0^3 = kt \qquad (2)$$

where d is the initial particle size; d_0 is the size at time t and k is the coarsening rate constant.

The coarsening rate constant k can be well adopted from the experimental data in Fig.8, where k=3.6113× 10^3 μm^3/s. It is apparent that the k value at 500 r/min is smaller than that at 10 r/min and 99 r/min for the same thermal condition[13]. In other words, the growth of primary Mg particles is suppressed by the increasing of shear rate during isothermal shearing. This slow coarsening rate can be attributed to a unique solidification behavior at high shear rate. As the particles are close to spherical and the particle size distribution is narrow, the driving force for coarsening is substantially reduced.

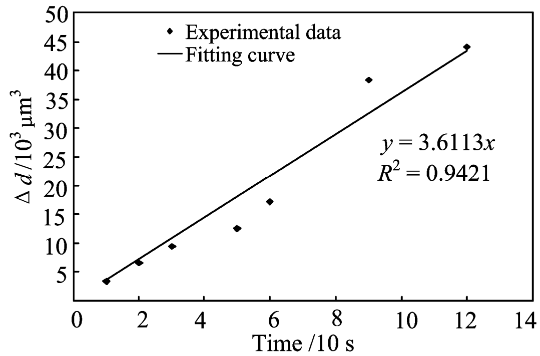

Fig.8 Experimental results and fitting curve

5 Conclusions

1) Grain density increases and average grain diameter decreases with increasing of rotation speed, indicating that high rotation speed suppressed, to some degree, the formation of the primary phase. However, a further increasing of rotation speed cannot improve it.

2) Grain density increases continuously and reaches the peak at 30s, volume fraction also increases gradually before 30 s. However, it was almost constant value after 30 s.

3) Comparing with low rotation spee with the same thermal condition, the analysis indicates that the microstructures obtained at a high low rotation speed, are homogenous spherical and fine grains instead of dendritic or rosette and exhibited uniform distribution in the eutectic matrix.

4) During isothermal shearing, the k coarsening rate constant value at high rotation speed is smaller than that at low rotation speed for the same thermal condition. The results suggested that the growth of primary particles is lower at high rotation speed than that at low rotation speed.

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