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# Effect of initial as-cast microstructure of AZ91D magnesium alloy on its semisolid microstructure

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ABSTRACT

The effect of initial as-cast microstructure of AZ91D alloy on its semisolid microstructure has been investigated. The uniformities of semisolid microstructures of cast ingots with different diameters have also been examined. The results indicate that the initial as-cast microstructure has large effect on the semisolid microstructure: the finer the as-cast microstructure, the smaller and more spheroidal the primary particles in the resulting semisolid microstructure. For the alloys with grain sizes of 70–100 μm, one dendrite in the as-cast microstructure frequently evolves into one spheroidal primary particle in the semisolid microstructure during partial remelting. But for the alloys having grain sizes of larger than 100 μm, one dendrite always separates into two or more particles with large-size difference and irregular morphologies. The microstructure inhomogeneity of cast ingots must result in the inhomogeneity of their semisolid microstructures and these two inhomogeneities are enhanced as the ingot diameter increases. The microstructure inhomogeneity can be decreased to some extent after being partially remelted. But to obtain a uniform semisolid microstructure, it is necessary to achieve a uniform as-cast microstructure.



## 1. Introduction

Thixoforming is a relatively new and powerful technology for forming near net shaped products of alloys in semisolid state. Millions of aluminium alloy components are produced using this process every year in the world [1]. But for magnesium alloys, the lightest structural metal material, the investigations on this aspect are obviously laggard to those of aluminum alloys. So far, only one technique, thixomoulding, has been employed in practice [2]. Therefore, it is necessary to pay more attention to magnesium alloys.

It is well known that the key procedure of thixoforming is to prepare semisolid microstructure with small and spheroidal primary particles. There are several methods to fabricate this kind of nondendritic semisolid microstructure. Alternatively, grain refining process produces the desired microstructure by adding grain refiner during traditional casting and a following heat treatment in mushy zone, i.e., it does not need special treating procedures required by other methods, such as stirring, spraying and deformation [3,4]. So it is a relatively simple method and has large potential in practice application. The existing investigation indicates that only the alloys with fine equiaxed dendrites can generate the desired semisolid microstructure after being partially remelted, but for those with developed dendrites, the solid particles are always large and irregular [3]. The reason that leads to this result can be verified through studying the microstructural evolution processes of the alloys with different as-cast microstructures during partial remelting. Unfortunately, most of the existing investigations have focused on the previously deformed magnesium alloys [3–10]. Only a few papers have involved the as-cast ones with relatively fine dendrites and emphasized on the morphology change and coarsening behavior of the solid primary particles or the Mg2Si particles in the Si containing alloys in semisolid states [11–15]. The investigation on the alloys with different as-cast microstructures is very scarce. The details how a dendrite with a given morphology and size in the as-cast microstructure evolves into a solid particle with a given morphology and size in the semisolid microstructure and the relations between these two microstructures are still not clear. In addition, the sizes of the used specimens in the existing investigations are mostly smaller than 20 mm in diameter [4–15]. It is expected that the as-cast microstructure difference in the different regions of a cast ingot increases as the ingot diameter increases and this as-cast microstructure difference must result in the semisolid microstructure difference. But the investigation about this is also very scarce.

Therefore, in the present work, the effects of initial as-cast microstructures of AZ91D alloys with different grain sizes on the semisolid microstructures have been investigated in order to verify

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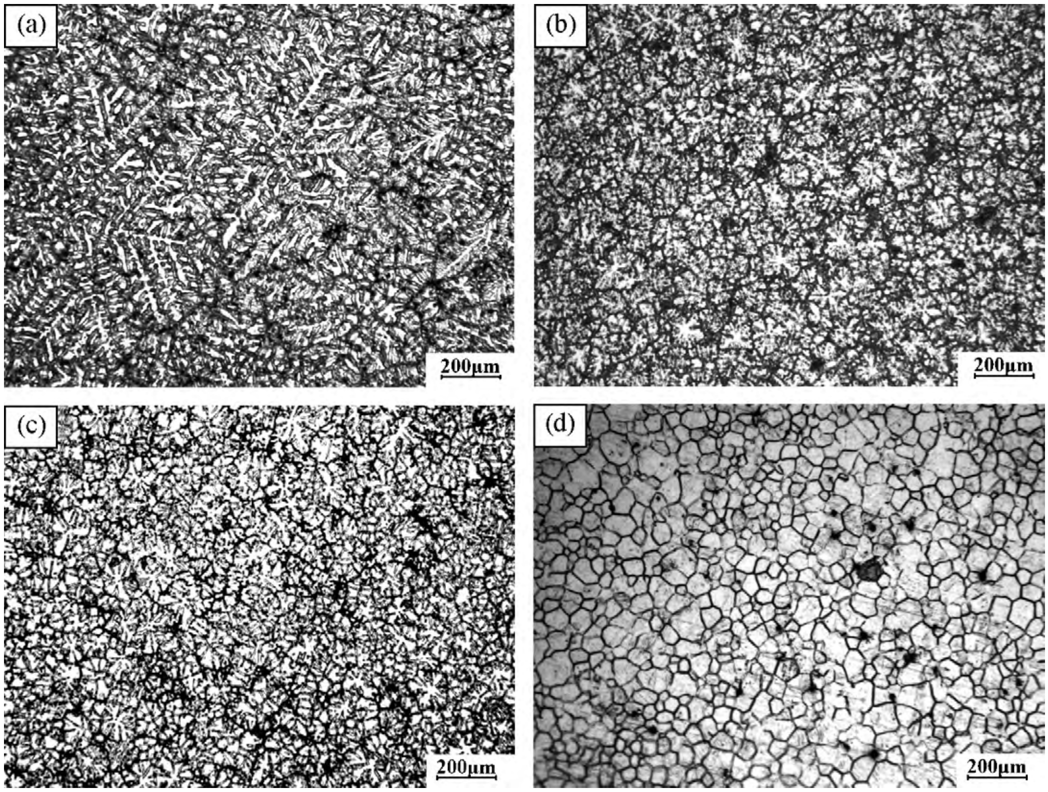


**Fig. 1.** As-cast microstructures of (a) the not refined, and refined AZ91D alloys by (b) 0.2%, (c) 0.6% SiC particles, and (d) the (b) alloy solution-treated at 420 °C for 8 h.

the relations between these two-state microstructures. In addition, the uniformities of semisolid microstructures of the cast rods with different diameters have also been discussed.

## 2. Experimental procedure

The alloy used in this work is commercial AZ91D alloy and it contains 9.04% Al, 0.6% Zn, 0.31% Mn and some trace elements or impurities (<0.002% Cu, <0.001% Fe, <0.001% Ni and <0.001% Be) (the percentages in this paper all refer to weight percentage). Previous investigation indicates that the alloys refined by different amounts of SiC particles have large difference in microstructure or grain size [16]. So in this work, the alloys (cast rods with 16 mm in diameter) refined by different amounts of SiC particles were used to verify the effect of initial as-cast microstructure on semisolid microstructure. The cast rods with three diameters of 16 mm, 45 mm and 70 mm were employed to examine the uniformities of semisolid microstructures. The details about preparation of the AZ91D alloys can be found in Ref. [16].

All of the specimens used for partial remelting have same dimensions of Ø 16 mm × 10 mm. The rods with 16 mm diameter were directly cut into specimens with such dimensions. But for the rods with 45 mm and 70 mm diameters, two specimens (Ø 16 mm × 10 mm) were machined from the center and edge of each rod, respectively. All of the specimens were heated for 20 min at 580 °C and then water-quenched quickly. In Ref. [17], the mechanism of microstructural evolution of the AZ91D alloys with relatively fine grains (refined by 0.2% SiC particles and having grain size of 70 μm) has been detailedly reported. To verify the mechanism of the alloy with developed dendrites (having grain size of 311 μm), some specimens from the alloy without refining were heated at 580 °C for different durations ranging of 0–20 min and then also water-quenched. One cross section of each of the water-quenched specimens was finished and polished by standard metallographic techniques. Subsequently, they were etched by aqueous solution containing glycerol, nitric acid, hydrochloric acid and acetic acid and observed on an optical microscope (OM). The size and shape factor of primary particles in the semisolid microstructures were quantitatively examined. The area $A_i$ and perimeter $P_i$ of each primary particle were obtained and the average particle size $D$ was calculated from the formula:

$$D = \frac{[\Sigma 2(A_i/\pi)^{1/2}]}{N} \quad (1)$$

where $N$ is the total particle numbers. The shape factor $F$ was calculated from the formula:

$$F = \frac{(\Sigma P_i^2 / 4\pi A_i)}{N} \quad (2)$$

If the particles are perfectly spherical, the shape factor has a value of 1; it increases for less spheroidal particles [18]. On each sample, three images with magnification of 200 times were examined.

To examine the grain sizes of the as-cast alloys, the specimens (also cut from the above rods and having dimensions of Ø 16 mm × 10 mm) were solution-treated at 420 °C for 8 h to delineate grain boundaries, and then processed according to the above procedures for preparing metallographic specimen and observed on the OM. The grain sizes were also calculated from the formula (1).

## 3. Results and discussion

### 3.1. Effect of initial as-cast microstructure on semisolid microstructure

Fig. 1(a)–(c) shows the typical microstructures of the as-cast AZ91D alloys refined by different amounts of SiC particles. The result indicates that the primary dendrites gradually change from the very developed dendrites of the not refined alloy (i.e., the alloy refined by 0% SiC particles) to the small and uniform equiaxed dendrites as the addition amount of SiC particles increases to 0.2% (Fig. 1(a) and (b)). But when the amount exceeds 0.2%, the primary dendrites slowly become developed again (comparing Fig. 1(b) and (c)). The reason why the dendrites change in such behavior with the amount of SiC particles can be found in Ref. [16]. Fig. 1(d) presents the microstructure of the alloy refined by 0.2% SiC particles and followed by a solution treatment at 420 °C for 8 h. It shows that this treatment can obviously delineate the grain boundaries and this alloy has relatively fine grains. Table 1 gives the quantitative examination result about the alloys' grain sizes, which shows that the alloys with different grain sizes ranging from 311 μm to 70 μm can be obtained through adjusting the addition amount of SiC particles.

Fig. 2 presents the typical semisolid microstructures of the alloys refined by different amounts of SiC particles. It shows that the primary particles gradually change from the very large and irregular structures to the small and quite spheroidal particles as the

**Table 1**
Grain sizes of the AZ91D alloys refined by different amounts of SiC particles.

| Addition amount of SiC (wt.%) | 0 | 0.05 | 0.1 | 0.15 | 0.2 | 0.4 | 0.6 | 0.8 | 1 |
|---|---|---|---|---|---|---|---|---|---|
| Grain size (μm) | 311 | 180 | 169 | 101 | 70 | 75 | 81 | 87.5 | 93.5 |

amount of SiC particles increases from 0% to 0.2%. They then slowly become large and irregular again when the amount exceeds 0.2%. The quantitative examination result about the primary particle size and shape factor is show in Fig. 3. For convenience of comparison, the grain size data of the as-cast microstructures have also been included in Fig. 3. It indicates that the change tendencies of the primary particle size and morphology are well consistent to that of the grain size of the as-cast microstructures: the finer the grains in the as-cast microstructure, the smaller and more spheroidal the primary particles in the semisolid microstructure.

Comparing the primary particle sizes of the semisolid alloys and the grain sizes of the corresponding as-cast alloys shown in Fig. 3, it can be found that the primary particle sizes are always smaller than the grain sizes for the coarse-grained alloys (refined by less than 0.15% SiC particles) while the particle sizes are comparative to the grain sizes for the fine-grained alloys (refined by 0.15% and more). Together with Table 1, it can be concluded that the primary particle sizes are equivalent to the grain sizes for the alloys having grain sizes of 70–100 μm (i.e., the grain sizes within the shadow region shown in Fig. 3), but for the alloys having grains larger than 100 μm, the primary particles are smaller than the grains.

The authors' previous investigation indicates that for the AZ91D alloy refined by 0.2% SiC particles and having grain size of 70 μm, as shown by (1)–(3) shown in Fig. 4, one equiaxed dendrite in the as-cast microstructure experiences three stages, the initial coarsening, structural separation and spheroidization, to evolve into one spheroidal particle in the semisolid microstructure during partial remelting at 580 °C [17]. The (2) stage of structural

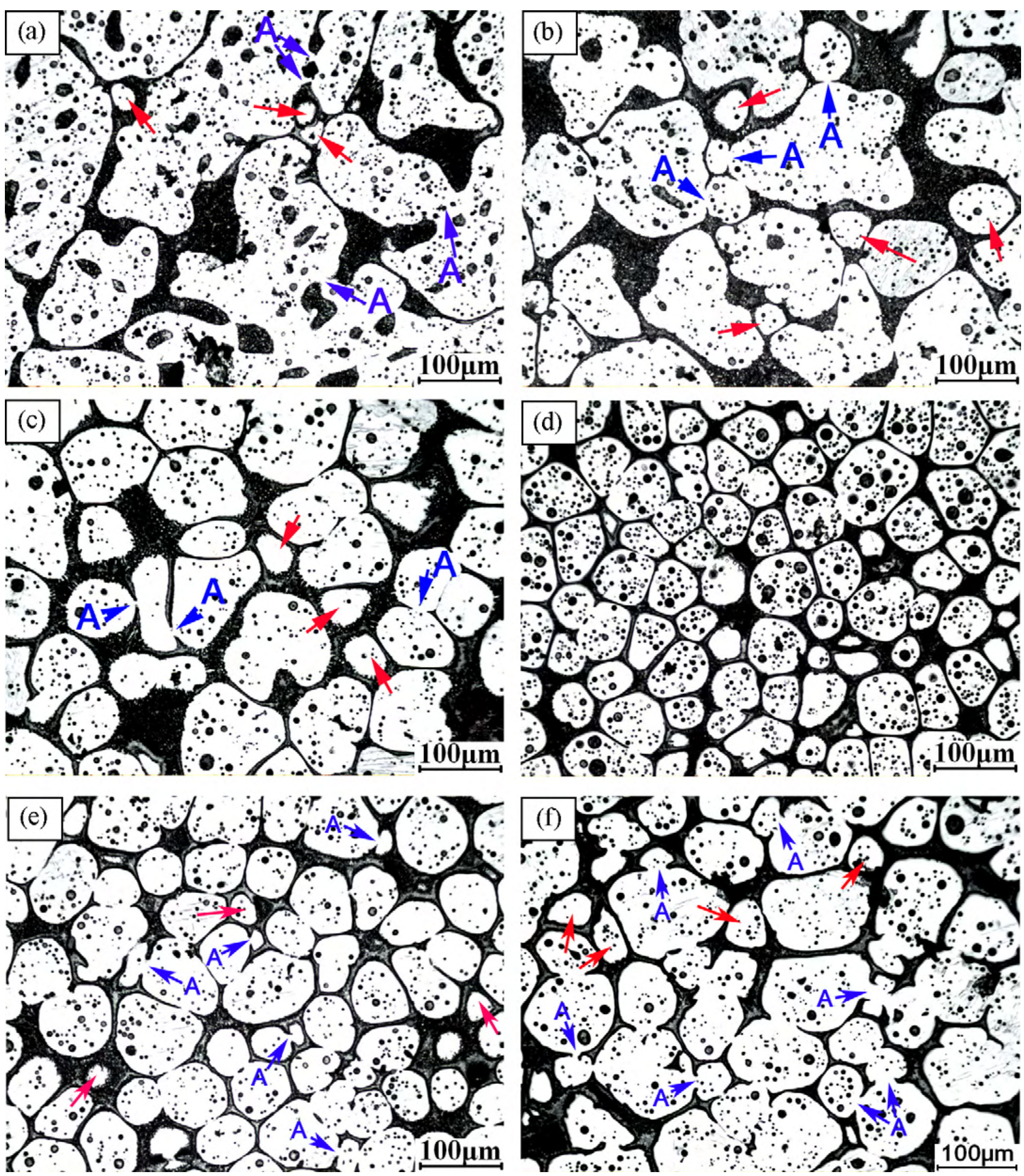


**Fig. 2.** Semisolid microstructures of (a) the not refined, and refined AZ91D alloys by (b) 0.05%, (c) 0.15%, (d) 0.2%, (e) 0.4% and (f) 1.0% SiC particles.

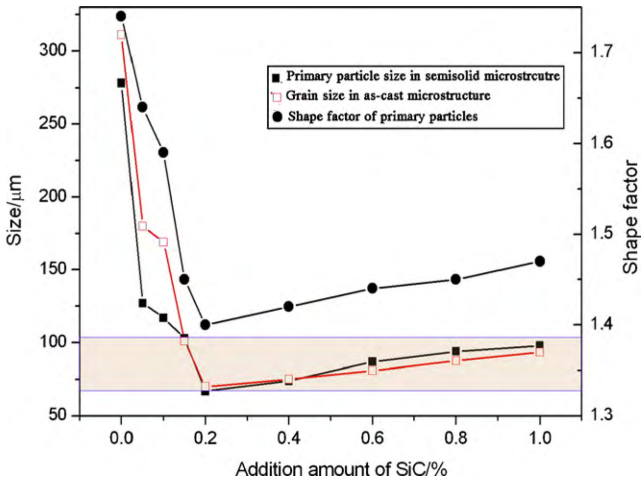


**Fig. 3.** Variations of the primary particle size and shape factor in semisolid microstructure and grain size in as-cast microstructure with the addition amount of SiC particles.

separation includes two steps, the melting of the residual eutectic (i.e., (a) step in Fig. 4) and the subsequent penetration of the first formed liquid along the original boundaries ((b) step). But for the coarse-grained alloys, their microstructural evolution operates in a different way during the (2) stage. Herein, the alloy without refining is taken as an example to explain this way. As shown by Fig. 1(a) and Fig. 5(a), the primary dendrites are quite developed and some of secondary arms on which third arms are also developed have grown relatively longer than the others, which leads the general morphologies of the dendrites irregular, not being similar to the regularly equiaxed morphologies of the fine equiaxed dendrites. In addition, there is more amount of eutectic or more serious solute segregation between the dendrite arms and at arm roots. During the initial coarsening stage, as show by the circled regions in Fig. 5(b), there is much amount of eutectic is left around them. Then the residual eutectic melts during the initial period of the structural separation stage, leading most part of the boundaries of coarsened arms to be surrounded by liquid phase (the boundary along the dot line in Fig. 5(c)). Following, the other part of the boundaries and even the necked roots are penetrated by the first formed liquid or they directly melt due to relatively low melting point from the serious solute segregation during the later period of the structural separation stage (see the boundary marked by curved arrow in Fig. 5(d)), separating the coarsened arms from their parent dendrite (particle A might have separated from its parent dendrite B as shown in Fig. 5(e)). Finally, the separated particles evolve into irregular structures after being spheroidized (Fig. 5(f)). Namely, the original one dendrite separates into two or more irregular particles with large difference in size. Based on this standpoint, it can be suggested that as shown by arrows in Figs. 2(a)–(c), (e), (f) and 5(f), the small-sized particles maybe originate from the separated dendrite arms. In addition, it can be seen that there are lots of agglomerates in which two particles are always connected by a thin neck (as shown by A). They should result from the not complete separation of the arms from their parent dendrites. These two phenomena can also demonstrate that the separation of arms from their parent dendrites has really occurred. Therefore, the microstructure evolution of the coarse-grained alloys can be schematically illustrated by Fig. 6. It can be found that the general evolution stages are identical to those of the fine-grained alloys, also including the three stages of (1)–(3). The difference lies in the (2) stage: the developed arms separate from their parent dendrites during the (b) step for the coarse-grained alloys, but there is no this behavior for the fine-grained alloys. It is just because of the separation of dendrite arms that the average sizes of the primary particles in the semisolid microstructures are always smaller than the original grain sizes for the coarse-grained alloys (Fig. 3). In addition, because the resulted solid particles are very irregular (Fig. 5(e)), the final particle shape factors are still large although they become spheroidal to some extent after being spheroidized for the same time with the fine-grained alloys (Fig. 5(f)).

But for the fine-grained alloys, the solute distribution is quite uniform and most of the solute-rich β phase between the dendrite arms dissolves into the original α grains during the initial coarsening stage. So it is difficult for the dendrite arms to melt at their roots during the subsequent structural separation stage (Fig. 4). Namely, one original dendrite in the as-cast microstructure evolves into one primary particle in the semisolid microstructure. It can be expected that the size of the resulted primary particles should be smaller than that of the original dendrites due to the partial remelting during spheroidization stage. But as shown by Fig. 2(d), the primary particles are quite small, and thus the solid/liquid interfacial energy is relatively large. In addition, the distance between the particles is also relatively short. So the coarsening, especially Ostwald ripening, is quite active under this condition. That is to say that the decrease of the particle size from partial remelting and the increase from coarsening are in a competition condition during the spheroidization stage [19]. Due to the comprehensive effect of these two reverse factors, the resulting particle size is usually comparative to the original grain size.

However, it should be noted that if the dendrite arms separate from their parent dendrite is not only determined by the dendrite size, but also depended the morphology of the dendrite. According to the evolution mechanism discussed above, it can be expected that if the dendrite size is quite small (less than 100 μm), but its one or more arms are relatively developed, i.e., its morphology is not so equiaxed. The distribution of Al-rich eutectic β phase should be quite nonuniform between the arms. So it is possible that the developed arms separate from the dendrite during partial remelting. Similarly, if one large-size (larger than 100 μm) dendrite have a very equiaxed shape, it is possible that all of its arms do not separate and finally it evolves into a large-size spheroidal particle. That is to say that the separation of a dendrite's arms is a relative phenomenon, but not an absolute thing. Generally, the possibility that one large-sized dendrite separates into two or more particles should be obviously lager than that of a small-sized dendrite. Most

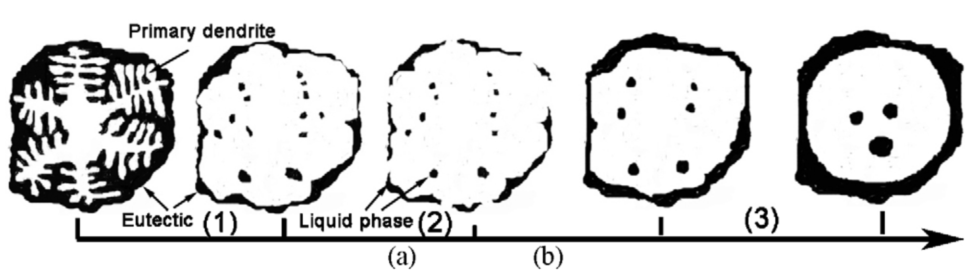


**Fig. 4.** Illustration of the evolution process of one fine equiaxed dendrite during partial remelting.

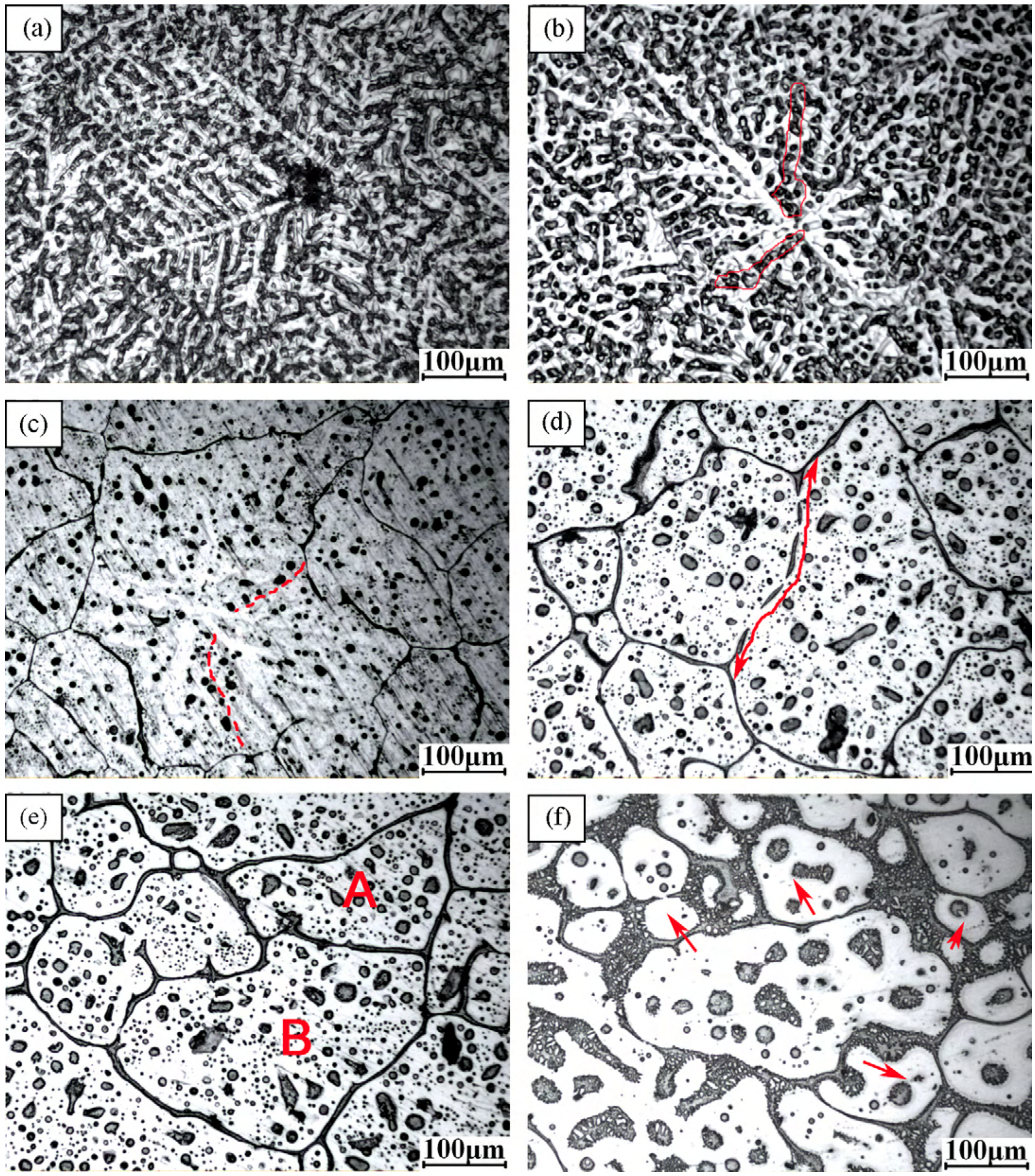


**Fig. 5.** Microstructures of the not refined AZ91D alloys heated at 580 °C for different durations and then water-quenched. (a) 0 min, (b) 2 min, (c) 3 min, (d) 5 min, (e) 7 min and (f) 20 min.

of the particles in the semisolid microstructure of a fine-grained alloy are resulted from their respective original dendrites in the as-cast microstructure. But for a coarse-grained alloy, quite a few of the particles, especially the small-sized particles may originate from the separated arms.

As shown by Figs. 2 and 3 or described above, the smaller the grains in the as-cast microstructure, the smaller and more spheroidal the primary particles in the semisolid microstructure. But it should be noted that the coarsening, especially the mergence of dendrites or primary particles, will become more and more active as the grain size continuously decreases because the boundary or solid/liquid interfacial energy becomes more and more large. So, one primary particle in the final semisolid microstructure may originate from two or more original dendrites in the as-cast

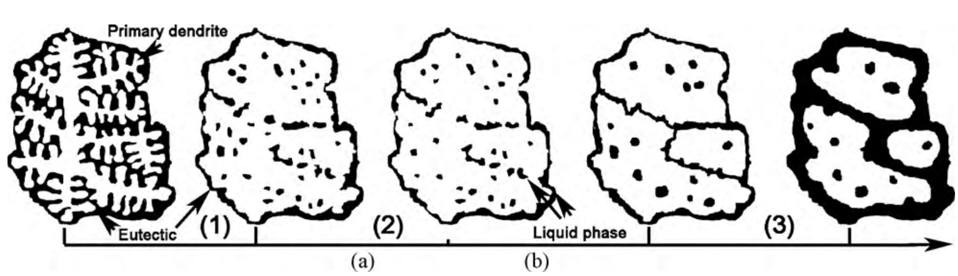


**Fig. 6.** Illustration of the evolution process of one developed dendrite during partial remelting.

microstructure. Due to this reason, the primary particle size of the semisolid microstructure will not decrease and maintain a constant as the as-cast microstructure are refined to a given degree. This has been demonstrated by the investigations on the grain-refined ZA27 alloy and equal channel angular pressed AZ91D alloy [20,21]. But the results imply that this condition does not occur in the present work. The reason should be that the as-cast microstructure of the AZ91D has not been refined to this given degree.

Based on the above discussion, it can be concluded that for the AZ91D alloys with grain sizes of 70–100 μm, one primary dendrite in the as-cast microstructures frequently evolves into one primary particles in the semisolid microstructures after being partially remelted, and the finer the dendrites, the smaller and more spheroidal the primary particles. But for the alloys having grain sizes of larger than 100 μm, one dendrite always evolves into two or more particles with large-size difference and irregular morphologies due to the separation of the dendrite arms from their parent dendrites.

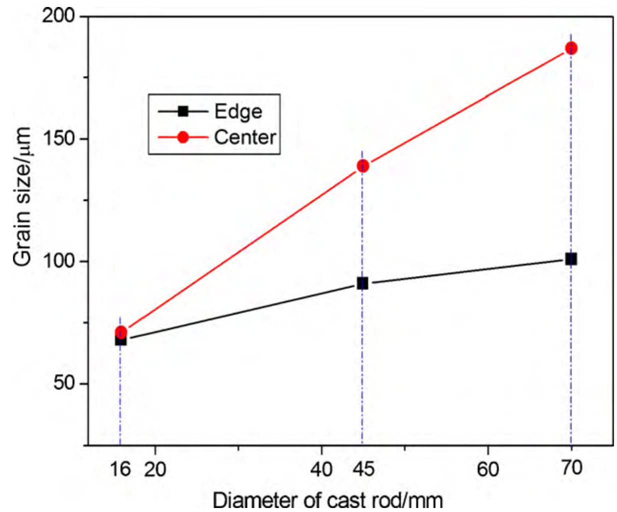


**Fig. 7.** Primary particle sizes of different regions of the cast AZ91D alloy ingots with different diameters after being heated for 20 min at 580 °C.

### 3.2. *Uniformity of semisolid microstructures of cast ingots with different diameters*

Because the cooling rates during solidification in different positions of a cast ingot are different, the resulting microstructures are also different. For a rod ingot, the microstructure at the edge is always finer than that in the center. In the present work, three kinds of rods with diameters of 16 mm, 45 mm and 70 mm are employed. The quantitative examination results of the microstructures are presented in Fig. 7. It shows that the difference in grain size between these two regions increases as the diameter increases. Fig. 8 gives the semisolid microstructures in these two regions of the three rods, which indicates that the primary particle sizes in the centers are larger than those in the edges and the difference in particle size between these two regions also increases as the diameter increases. Namely, this change tendency is also completely consistent to that of the grain size of the as-cast microstructure: the finer the as-cast microstructure, the smaller the primary particles. Fig. 9 presents the primary particle sizes of these semisolid microstructures. For convenience of comparison, the grain sizes of the corresponding as-cast microstructures are also included. It again indicates that for the fine-grained specimens (alloys having grain sizes of 70–100 μm as shown by the shadow region), the primary particle size is basically equivalent to the grain size, but for the coarse-grained specimens, the former is obviously smaller than the latter. This phenomenon can be well interpreted by the reasons provided in the above section.

In addition, Fig. 9 shows that the difference in semisolid microstructure between these two regions of a rod is smaller than that in as-cast microstructure. For example, for the rod with diameter of 45 mm, the grain size of the as-cast microstructure in the center is 53% larger than that in the edge, but the primary particle size of the semisolid microstructure in the center is only 30% larger than that in the edge. That is to say that partial remelting can uniformize the microstructure to some extent. According to the discussion in above section, this should be attributed to the

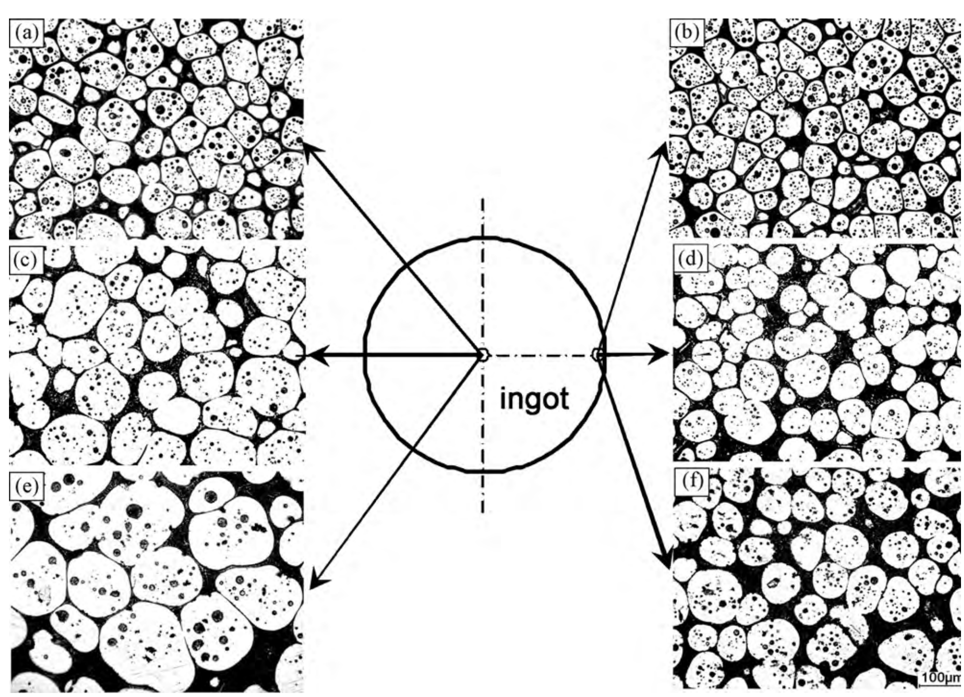


**Fig. 8.** Semisolid microstructures of different positions of the AZ91D alloy rods with different diameters. (a) and (b) Ø 16 mm, (c) and (d) Ø 45 mm, (e) and (f) Ø 70 mm.

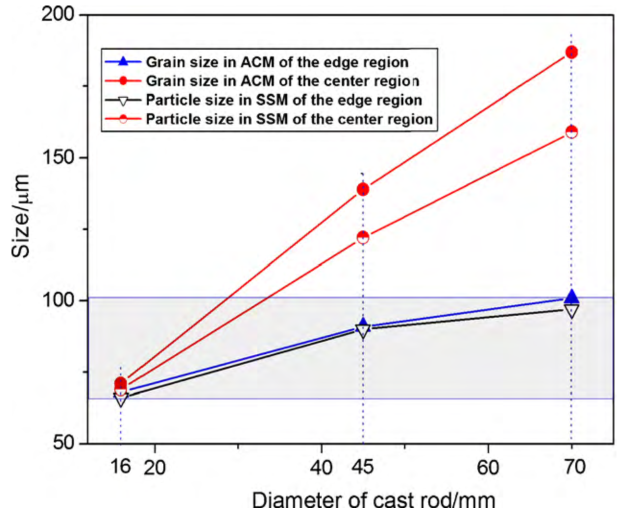


**Fig. 9.** Variations of the grain sizes in the as-cast microstructures (ACM) and primary particle sizes in the semisolid microstructures (SSM) in different regions with the diameter of cast rod.

separation of dendrite arms from their parent dendrites for the coarse microstructure in the center and the coarsening of the primary particles for the fine microstructure in the edge. However, it can be found that this homogenization is very limited. Therefore, to obtain a uniform semisolid microstructure, it is the first to achieve a uniform as-cast microstructure, which is especially important for large-sized ingot.

## 4. Conclusions

(1) The initial as-cast microstructure of AZ91D alloy has large effect on its semisolid microstructure. The finer the as-cast microstructure, the smaller and more spheroidal the primary particles in the resulting semisolid microstructure.
(2) For the alloys with grain sizes of 70–100 μm, one dendrite in the as-cast microstructure frequently evolves into one spheroidal primary particle in the semisolid microstructure after being partially remelted. But for the alloys having grain sizes of larger than 100 μm, one dendrite always becomes into two or more particles with large-size difference and irregular morphologies.
(3) The inhomogeneity of the as-cast microstructure must result in the inhomogeneity of the semisolid microstructure and both these inhomogeneities are intensified as the ingot diameter increases.
(4) The microstructure inhomogeneity can be decreased to some extent after being partially remelted. But to obtain a uniform semisolid microstructure, it is the prerequisite to achieve a uniform as-cast microstructure.

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