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# Influences of melt treatment on grain sizes and morphologies of AZ91D alloy

QI Xiang-chao(齐祥超)[1], TANG Jing-lin(唐靖林)[1, 2], LI Shuang-shou(李双寿)[1, 2], ZENG Da-ben(曾大本)[1]

1. Department of Mechanical Engineering, Tsinghua University, Beijing 100084, China;
2. Key Laboratory for Advanced Materials Processing Technology, Ministry of Education, Tsinghua University, Beijing 100084, China

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**Abstract:** Influences of the three melt treatment processes (namely, refinement with carbon inoculation, electromagnetic stirring and "refinement & electromagnetic stirring" processing) on the microstructures of AZ91D alloy were explored experimentally. The results indicate that the micron carbon powders inoculation processing with 0.1% (mass fraction) addition level makes the grain size of the primary phase ($\alpha$-Mg) decrease to approximate 40% that of the initial primary crystal, from about 530 μm to 200 μm around. The electromagnetic stirring processing not only decreases the grain sizes sharply, but also transfers the grain shape of $\alpha$-Mg from coarse dendritic to nearly spheralitic with the shape factor from about 0.1 to approximate 0.8. And the "refinement & electromagnetic stirring" processing improves grain shape of $\alpha$-Mg further and refines grain size to about 72 μm, less than 15% that of the initial primary crystal.

**Key words:** melt treatment; electromagnetic stirring; grain size; grain morphology; AZ91D alloy

## 1 Introduction

The primary crystal morphology and grain size are the two important factors influencing the mechanical properties[1−2], and also the key technological parameters in magnesium alloys processing. The researches[1−6] indicate that the melt modification treatment of alternatives (carbon powders, carbon compounds, Sr, and so on) added into the Mg-Al alloys melts can refine the grain size of $\alpha$-Mg effectively. Since the semisolid metal processing(SSM) is invented by Flemings[7], the SSM with special advantages has been the main processing to improve the metal microstructures and mechanical properties, extensively applied to the light metals such as aluminum alloys and zincum alloys [7−10]. However, the exploration of magnesium alloy semisolid processing to enhance the properties is still in the laboratory research stage because the magnesium alloy has the characteristics of active chemical property, being flammable and explosive, special primary crystal structure, and so on.

The results such as the grain size decreasing sharply, approximately spherical grain shape and mechanical properties enhancement, have been achieved in the former experiments[11−12] on the aluminum alloys. Based on these, the new experiments on the comparison between refinement with carbon inoculation and electromagnetic stirring processing, and on the contrast between electromagnetic stirring and "refinement & electromagnetic stirring" processing are done to explore the influences on the microstructures of $\alpha$-Mg. The influencing rule and mechanism of the three melt treatment processes (refinement with carbon inoculation, electromagnetic stirring and "refinement & electromagnetic stirring" processing) on the grain size and grain shape of $\alpha$-Mg are analyzed. And the aim of this article is to investigate the effective method for fast semisolid slurry preparation of magnesium alloys with refined grains and sound spherical microstructures, providing evidences for a new rheoforming processing of magnesium alloys.

## 2 Experimental

The experimental materials are the AZ91D alloy

**Corresponding author:** TANG Jing-lin; Tel/Fax: +86-10-62772502; E-mail: jixc02@mails.tsinghua.edu.cn

and alternatives. The alternatives include the micron carbon powders, hexachlorethane and Al-10Sr master alloy. The electromagnetic stirring device is the independently developed reversible multilevel-speed electromagnetic stirrer. The mould and crucible are made of low carbon steel.

The AZ91D alloy was heated to 450 ℃ in the air, and then up to 730 ℃ in the protective gas (98.5% $N_2$ and 1.5% $SF_6$, volume fraction). The micron carbon powders or hexachlorethane were put into the AZ91D alloy melt, and the melt was agitated for 3−5 min and kept at 720 ℃. About every other 10 min, the melt was poured into the mould, which was heated up to about 200 ℃.

After being heated up to 730 ℃ and kept for more than 15 min, the melt was poured into the 650 ℃ crucible and cooled to 630 ℃ around. Then the crucible was put into the electromagnetic stirring device and the melt was stirred (frequency of 30 Hz, voltage of 220 V, the stirring directions changed every other 3 s and double- direction stirring, different stirring times).

In the "refinement & electromagnetic stirring" processing, the only difference is that the Al-10Sr master alloys were added into the former melt for the melt modification treatment before the melt was stirred in the electromagnetic stirring device. The other steps were the same as the electromagnetic stirring processing.

All the samples were in the air-cooling state. The samples were subjected to a solution treatment of holding at 405 ℃ for 12 h and water-cooling. The grain size and grain shape of each sample were measured from the central region of a longitudinal section, which was cut through the axis, according to the intercept method described in the ASTM standard E112. The samples for optical microscopy(OM) were prepared by the standard technique of grinding with SiC abrasive paper and polishing with the diamond abrasive, and etching. And the microstructures of samples were examined by OM for quantitative metallography.

## 3 Results and discussion

The microstructures of AZ91D alloy remelted are shown as Figs.1 and 2. The microstructures are very clear, similar to the hexagram, but it is hard to measure the grain size (Fig.1). The grain borders are much clearer after solution treatment, just as shown in Fig.2. Therefore, the samples in air-cooling state are used to observe the primary crystal morphology and the samples after solution treatment are used to measure the grain size.

The shape factor ($F_c$) is imported to describe the particle morphological evolution quantificationally. The equation of shape factor[13] is

$$F_c = \frac{4\pi S}{P^2} \tag{1}$$

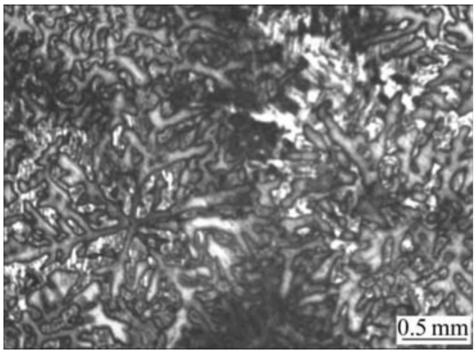


**Fig.1** Microstructure of AZ91D alloy in air-cooling state

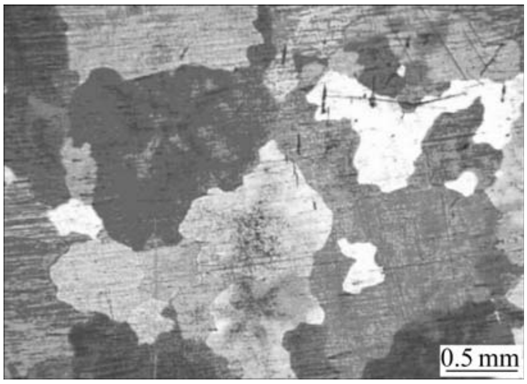


**Fig.2** Microstructure of AZ91D alloy after solution treatment

where $S$ is the surface area of particle measured, and $P$ is the surface circumference of particle measured. The equation indicates that the shape factor is in the range of 0−1, and the closer the shape factor is to 1, the rounder the particle is. When the factor is 1, the morphology of particles is spherical. The shape factor of the dendrites is about 0.1[13].

### 3.1 Carbon inoculation

The relations between the grain size and the holding time in the refinement with carbon inoculation processing (Figs.3 and 4) are investigated.

The micron carbon powder addition level is 0.1% (mass fraction) of AZ91D alloy melt. Fig.3 shows the relation between the grain sizes and holding time after the carbon powders are added. It can be seen that after the AZ91D alloy is remelted, the grain size is the largest, about 530 μm. When the micron carbon powders are put into the melt and the melt is agitated for 3−5 min, the grain size becomes smaller, which indicates that the carbon powders have effect on refining the grain. Within 20 min, the grain size decreases with the holding time. When the holding time is more than 20 min, the grain sizes stay at a steady level, which is approximate 40% that of the initial primary crystal, about 200 μm.

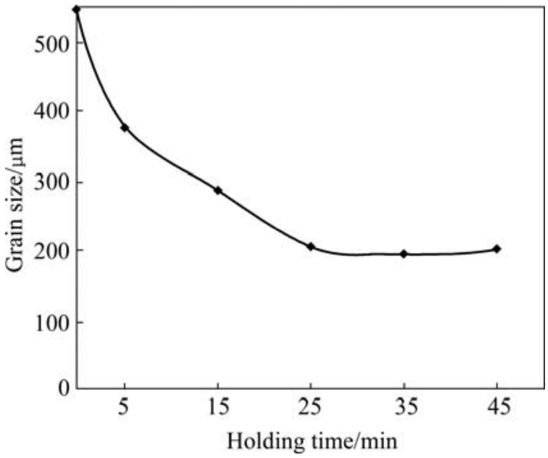


**Fig.3** Relation between grain size of $\alpha$-Mg and holding time (Addition level of micron carbon powder: 0.1%, mass fraction)

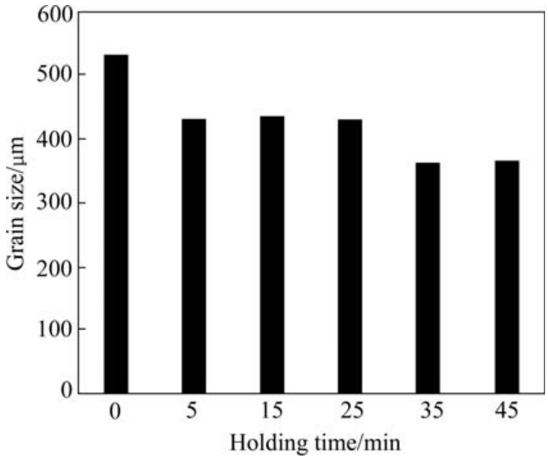


**Fig.4** Relation between grain size of $\alpha$-Mg and holding time (Addition level of hexachlorethane: 0.25%, mass fraction)

The refinement processing with hexachlorethane inoculation is also explored. The results are shown in Fig.4 when the addition level is 0.25% (mass fraction). When the holding time is more than 30 min, the grain size decreases to about 350 μm and keeps at that level.

MURTY et al[14] and MAXWELL and HELLAWELL[15] indicated that the grain amount increases linearly with the nucleation particles increasing when the nucleation particles are less. There are much more crystal grains when the holding time is more than 20 min of carbon powders and 30 min of hexachlorethane refinement in comparison with the initial primary crystal grain remelted only. It is widely accepted that the aluminum carbide ($Al_4C_3$) is the compound largely responsible for AZ91D alloy refining effects[3, 16−17], as the heterogeneous nucleation particle. According to the crystallology theory, if the crystal matrix mismatch is less than 9%[18], one particle could be the basic nucleation site for another. The crystal matrix mismatch between $\alpha$-Mg particle and aluminum carbide particle is only 3.8%[18] and their crystal textures are the same (close-packed hexagonal lattice). Hence, the $Al_4C_3$ particle can be the basic nucleation site for $\alpha$-Mg primary particle. But the $Al_2CO$ particle crystal texture is also the close-packed hexagonal lattice, with the matrix mismatch of 0.9%[18], and could be the basic nucleation site for $\alpha$-Mg primary particle, too. $Al_2CO$ particle contains the oxygen element since it would be introduced into the melt in the form of oxygen gas and aqueous vapour or anhydrous alcohol during the polishing. There are black particle in most grain crystal macrostructures (Fig.5), providing the evidence of heterogeneous nucleation. But it is unsure which particle is the basic nucleation site of $\alpha$-Mg primary particle, and the further research is still needed to reveal the refinement modification mechanism of AZ91D alloy with carbon inoculation.

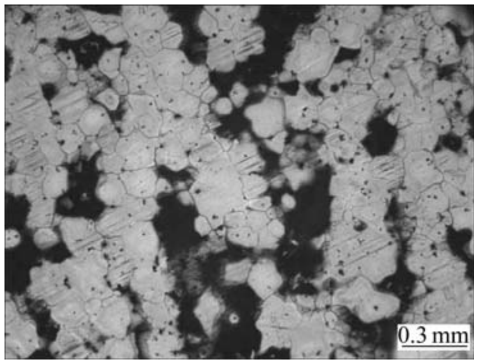


**Fig.5** Grain morphology by refinement with micron carbon powder addition after solution treatment

According to above experiments, carbon can make the grain size of AZ91D finer effectively. And it is clear that the grains by refinement with carbon powders addition (Fig.5) are much finer than those of hexachlorethane addition (Fig.6). Compared with those of only remelted treatment (Fig.2), grain sizes decrease much by about 60%. The shape factor is in the range of 0.1−0.2 after carbon powders or hexachlorethane is added

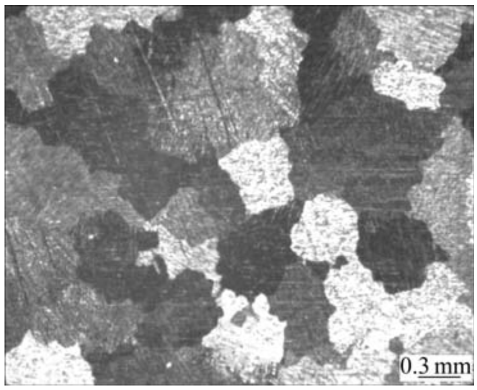


**Fig.6** Grain morphology by refinement with hexachlorethane addition after solution treatment

in AZ91D melt, indicating that the carbon inoculation processing influences the particles morphology in a little degree.

### 3.2 Electromagnetic stirring processing

In the experiments, the independently developed reversible multilevel-speed electromagnetic stirrer is applied to prepare the semisolid slurry. The stirring direction and the changing time can be adjusted in this electromagnetic stirrer compared with the traditional electromagnetic stirrer. The grain refinement samples are gained by the electromagnetic stirring with different stirring time. When the stirring time is 30 s, the grain size is less than 150 μm and there are simple dendrites in the grain crystal microstructures (Fig.7). But when the stirring time is more than 90 s, the grain size is 80 μm around and the morphology changes to nearly sphericitic (Fig.8) from coarse dendritic. There is a core in almost every grain crystal. The result demonstrates that electromagnetic stirring processing can refine the AZ91D microstructures more effectively than the carbon inoculation processing.

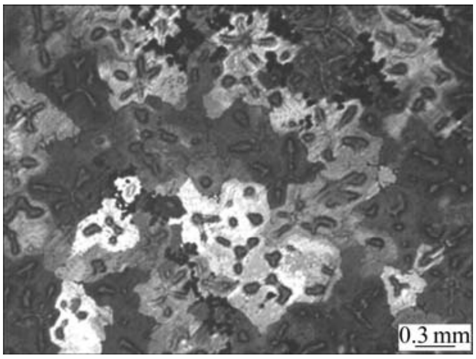


**Fig.7** Microstructure of AZ91D alloy after stirring for 30 s

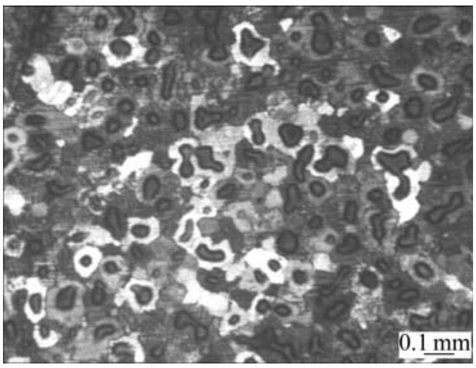


**Fig.8** Microstructure of AZ91D alloy after stirring for 90 s

The shape factor is about 0.4 when the stirring time is 30 s, while the shape factor is 0.8 approximately when the stirring time is over 90 s. The grain microstructures by electromagnetic stirring are much sounder and finer than those by refinement with carbon inoculation, and also than those only by remelting, indicating that the electromagnetic stirring processing improves the microstructures in a greater degree.

The mechanism is mainly that the electromagnetic stirring changes the conditions of melt solidifying. In the electromagnetic stirring processing, the melt flows intensely and the heat transfer conditions are altered[19]. So there is a uniform solute and temperature distribution and the nucleating area becomes much larger. Therefore, there are much more crystal nucleus at the same time. FAN[7] indicated that forced convection promotes the finer particles with a non-dendritic morphology. FLEMINGS[9], YOSHIKI et al[20−22] and MAO et al[19] reported that the reason of refinement is the collapse of $\alpha$-Mg dendrite arms due to the shock wave resulting from cavitation, which is the bursting of cavities caused by the stirring. When the electromagnetic stirring time is short (no more than 30 s), there are many half-baked dendrites (Fig.7), which supports the dendrite arm collapse theory. And when the electromagnetic stirring time is more than 90 s, there is a big core in almost grain crystals. The core is not the heterogeneous nucleus because the size of these cores is in the same level with the grain. But, these cores stand a good chance of the collapse arms engulfed into solute as the nucleation site. And the different phase positions between the inner and boundary in one grain crystal prove this point (Fig.8).

The grain crystals collide and friction with each other or the crucible wall under the intense electromagnetic stirring, making themselves sound and fine (Fig.7), so the shape factor increases to 0.8 around from about 0.1. Secondly, the electromagnetic stirring changes the solute distribution to influence the growth of $\alpha$-Mg primary dendrite, refining the $\alpha$-Mg primary particle.

The experiment results state that the samples with fine grain and sound spherical microstructure could be prepared by the electromagnetic stirring processing in a short time.

### 3.3 "Refinement & electromagnetic stirring" processing

The primary exploration on the "refinement with Sr addition and electromagnetic stirring" processing was done. Sr with different addition level in 0.005%−0.1% (mass fraction) range was added into the AZ91D alloy melt. The grain size is refined to 370 μm, reaching the best refinement effect when addition level of Sr is 0.01% and other conditions are the same as those of carbon inoculation processing, revealing that the refining effect of Sr addition is less than that of carbon powder inoculation.

The result of "refinement with Sr addition and electromagnetic stirring" processing is shown in Fig.9. The grain size decreases with the addition level increasing when the addition level is lower than 0.02%. When the addition level is 0.02%, the refining effect is the best with grain size smaller than that of electromagnetic stirring processing, decreasing the grain size to less than 15% that of the initial primary crystal, about 72 μm. The grain morphology is much more spherical (Fig.10) with the shape factor of 0.85 around.

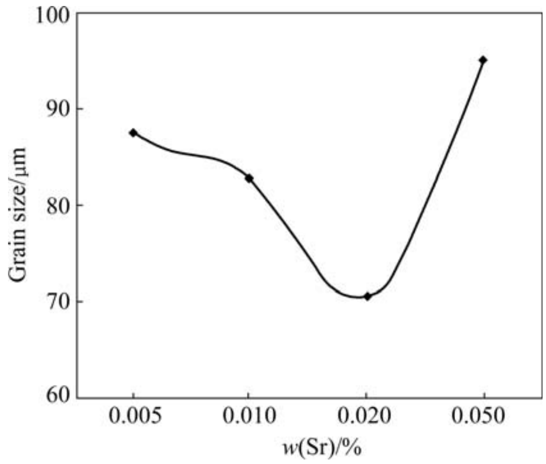


**Fig.9** Relation between grain size and Sr addition by "refinement with Sr addtition and electromagnetic stirring" processing

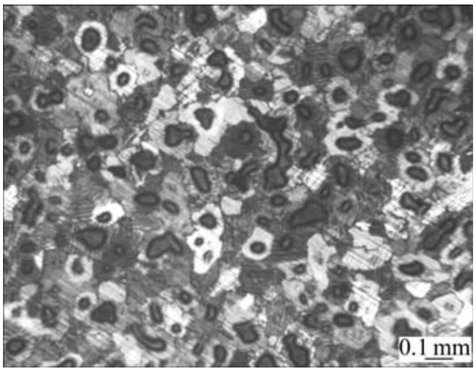


**Fig.10** Microstructure of AZ91D alloy with "refinement with Sr addition and electromagnetic stirring" processing

The former experiments state that compared with the refinement with Sr addition processing, the carbon powder inoculation processing could refine the grain size much more effectively. If the problem of the way of carbon powder inoculation is solved, the "refinement with carbon powders addition and electromagnetic stirring" processing will be investigated immediately, the refining effect of which should be much better than that of "refinement with Sr addition and electromagnetic stirring" processing.

According to the above experimental results, when the best refining effect is achieved, Sr addition in the "refinement with Sr addition" processing and the "refinement with Sr addition and electromagnetic stirring" processing is different, which indicates that the electromagnetic stirring process strengthens and changes the refinement mechanism of refinement with strontium element, and the electromagnetic stirring process is the main reason in the "refinement with Sr addition and electromagnetic stirring" processing.

The refinement mechanism is just as same as the electromagnetic stirring processing, but this method enhances the number of heterogeneous nucleus or the uniform degree of solute distribution. As a result, the refining effect is the most evident.

The thixoforming processing has the long technological process and the billet preparation cost is high, about 40% of the product cost[23]. Therefore, presently, the rheoforming processing becomes more and more important. The key technology to the rheoforming processing is semisolid slurry prepared in a short time, that is, the semisolid slurry preparation online technology. The experimental results show that the "refinement & electromagnetic stirring" processing in these experiments shortens the time of semisolid slurry preparation seriously, indicating that the rheoforming processing is feasible. And the rheoforming processing will be explored in the future work. According to the former experiments, on the condition of the certain electromagnetic stirring parameters (frequency of 30 Hz, voltage of 220 V, stirring directions changed every other 3 s, double-direction stirring and electromagnetic stirring time of 90 s), the samples with finer grains and sound spherical microstructures are gained by the "refinement & electromagnetic stirring" processing, conforming to the requests of semisolid slurry. Thus the above exploration primarily proves that the "refinement & electromagnetic stirring" processing is feasible. Fine grains and sound spherical microstructures can be prepared in a much shorter time through adjusting the electromagnetic stirring parameters such as stirring frequency, stirring voltage, stirring mode, changing time of stirring directions.

## 4 Conclusions

1) The refinement with carbon inoculation can refine the microstructure of AZ91D alloy. The grain size is decreased to about 40% that of the initial primary crystal if the carbon powders are put into the melt and the melt is agitated for 3−5 min and kept for more than 20 min at about 720 ℃.

2) The electromagnetic stirring processing is a feasible method to refine AZ91D alloy. This method makes the grain microstructure sound and fine with the shape factor increasing to approximate 0.8 from about 0.1, and the grain size is refined up 80 μm around from

about 530 μm of only remelted state by approximate 80%. The “refinement with Sr addition and electromagnetic stirring” processing makes the grain microstructure much sounder and finer, and the grain size is about 72 μm.

3) The electromagnetic stirring causes an uniform solute and temperature distribution, and the collapse of dendrite arms of $\alpha$-Mg phase, generating more crystal nucleus to refine the grain size and making the grain crystal spherical. The “refinement & electromagnetic stirring” processing enhances the number of heterogeneous nucleus or the uniform degree of solute distribution on the base of electromagnetic stirring processing and provides the evidence for the further research of rheoforming processing.

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