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Effect of La_2O_3 addition on the microstructure of partially remelted Mg-9Al-1Zn alloy

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The effect of La_2O_3 addition on the microstructure of partially remelted Mg-9Al-1Zn (AZ91D) alloy was studied. The results indicate that small amounts of La_2O_3 additions to AZ91D alloy refined the partially remelted microstructure and caused the formation of new phase- $LaAl_4$ in the microstructure. Moreover, the grain size of the partially remelted alloys is decreased with the increasing of La_2O_3 addition. In as-cast microstructure the $LaAl_4$ phases have two morphologies: needle-like and particle-shape. The presence of more $LaAl_4$ phases, especially, the particle-shape $LaAl_4$ which dispersedly distributed within the grains can induce more dislocations, which can result in the occurring of a large amount of recrystallizations. Moreover, these $LaAl_4$ phases can also restrain the growth and combination of the recrystallizations and partially remelted initial grains during the subsequent heat-treatment.

1. Introduction

Semisolid forming is an effective net-shape forming process, which combines the elements of both casting and forging, showing many advantages over the conventional process [1–4]. The key factor controlling both the flow of a slurry and product properties is the equiaxed, non-dendritic microstructure which behaves thixotropically and can be formed to the net shape [5, 6]. As a result, the major effort of all the semisolid technologies is focused on the generation of globular microstructures [7, 8]. In general, the smaller and rounder the grains, the better will be the rheology of the semisolid alloy [8]. Several processes can generate the non-dendritic globular microstructure. One of the processes is strain-induced melt activation (SIMA), which has been the subject of many studies. Comparing with other starting semisolid material producing methods such as magnetohydrodynamic stirring, mechanical stirring, spray stirring and semisolid isothermal heat treatment [2, 8–12], SIMA has several advantages. It omits the procedure of molten metal treatment, and is applicable for both low and high melting alloys [4]. SIMA process consists of four discrete stages [12, 13]. First the alloy is cast in convenient sizes to obtain a typical dendritic microstructure. Subsequently, it is hot worked so a directional microstructure is introduced and the thickness of the casting is decreased. The third stage involves the introduction of a critical level of stored energy in the alloy by cold working. Finally, the deformed alloy is partially remelted, and held isothermally for a short time. In SIMA, the strain is mainly introduced into the materials through deformation.

As the lightest metal structure material, magnesium alloy has low density, high-damping characteristic, excellent machineability and castability, etc [14, 15]. More and more magnesium alloy products are used in automobile, communication and aerospace industries [14]. However, the range of applications for this alloy is limited by the low strength and poor creep resistance at temperatures in excess of 120°C [16–19]. Rare earths (RE) elements are usually as alloying additions to magnesium alloys [20] for it is useful in increasing the mechanical properties of magnesium alloy [21, 22]. At present, most attention has been focused on investigations into the microstructure and properties of as-cast magnesium alloy containing RE [16–22]. However, there have been very few partially remelted studies of RE-containing Magnesium alloys up to now. In this paper, we examine the influences of La_2O_3 addition and variation of its concentration on the partially remelted microstructure of AZ91D alloy as produced by the SIMA method.

2. Experimental procedures

2.1. Experimental materials

In the present work, AZ91D magnesium alloy was adopted as the matrix and different La_2O_3 additions

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TABLE I Chemical composition of the studied alloys (wt.%)

Alloys	Mg	Al	Zn	Mn	Fe	Nd	La_2O_3
AZ91D	Balance	9.075	0.680	0.210	0.005	0.005	0
AZ91D-1wt % La_2O_3	Balance	8.674	0.618	0. 235	0.005	0.005	1
AZ91D-2wt % La_2O_3	Balance	8.855	0.647	0.232	0.003	0.025	2
AZ91D-4 wt % La_2O_3	Balance	9.052	0.646	0.282	0.004	0.030	2

were made. Chemical compositions, as measured with an ARL 4460 Metals Analyzer, of the studied alloys are listed in Table I. It should be mentioned that the weight fractions of 1, 2 and 4 wt% La_2O_3 in the AZ91D alloys are the designed content.

2.2. Experimental procedure

The La_2O_3 was added as preform into the AZ91D. The preform was made from commercial powders of aluminum (98.0% purity), magnesium (>98.0% purity), and La_2O_3 (>98.0% purity), and the average particle sizes of them are less than 106 μm, 106 μm and 5μm respectively. Magnesium and aluminum powders with an weight ration of 10:1 mixed with 1, 2 and 4 wt% La_2O_3 powders by ball milling for 6 h, then were pressed into cylindrical preforms (20 mm diameter and 15 mm length) by using a stainless steel die with two plungers.

The AZ91D were melted in a graphite crucible electric resistance furnace. The preforms with 1.0, 2.0 and 4.0 wt% La_2O_3 were added respectively when the temperature of the melt was about 700°C. Then the melt was held at 700°C for 30 min and stirred uninterruptedly to make sure that La_2O_3 dissolve completely. Then the molten alloys were poured into the metal mold to form square billets. Subsequently, the billets were cut into samples with the size of 12.0 mm × 12.0 mm×13.1 mm, and compressed by 20.6% in height on a hydraulic machine. After compressing, the samples were heated to 570°C in the vertical tube furnace with a heating rate of 5°C/min under the protective atmosphere of flowing CO_2 to prevent oxidation, and held at that temperature for 5 min.

2.3. Differential thermal analysis

To study the influence of La_2O_3 addition on the remelting characteristics of the materials, 50 mg of AZ91D and AZ91D-4 wt% La_2O_3 are heated at 20°C/min to about 800°C under an argon atmosphere in the DTA apparatus. The eutectic and liquidus temperatures of the AZ91D alloy and AZ91D- 4 wt% La_2O_3 alloy are all 437 and 599°C, respectively. The results indicate that the La_2O_3 addition has no influence on the eutectic and liquidus temperatures of the AZ91D alloy. Based on the DTA experiments, the isothermal holding temperatures during the partially remelted are selected at 570°C, ranging between the eutectic and liquidus temperatures.

The microstructure observations were examined by scanning electron microscopy (Model JSM-5310) and optical microscopy. Phases of the alloys were analyzed by X-ray diffraction (XRD) (Modal D/Max 2500PC Rigaku, Japan).

3. Results and discussion

3.1. Alloy composition

Fig. 1a and b shows the SEM microstructure and XRD pattern of partially remelted AZ91D-4 wt% La_2O_3 alloy. After La_2O_3 was added, a needle-like or particle-shape intermetallic phase was observed. Results of XRD analysis show the intermetallic phase in AZ91D-xwt% La_2O_3 alloys was $LaAl_4$ (Fig. 1b). In addition to the major Mg (α) and $LaAl_4$ phases, the $Mg_{17}Al_{12}$ (β) eutectic phase was also detected as expected in the alloy by XRD. As can be seen in Fig. 1a, $LaAl_4$ phases are almost uniformly distributed within the grains and at the grain boundaries.

3.2. Grain size

Fig. 2 shows the partially remelted microstructures of the AZ91D-xwt.% La_2O_3 alloys after isothermal holding at the predetermined temperature of 570°C.The microstructure of AZ91D alloy is composed of globule α-Mg primary phase and irregular β-$Mg_{17}Al_{12}$ precipitation phase along grain boundaries (Fig. 2a). As can be seen in the pictures, the addition of La_2O_3 fined

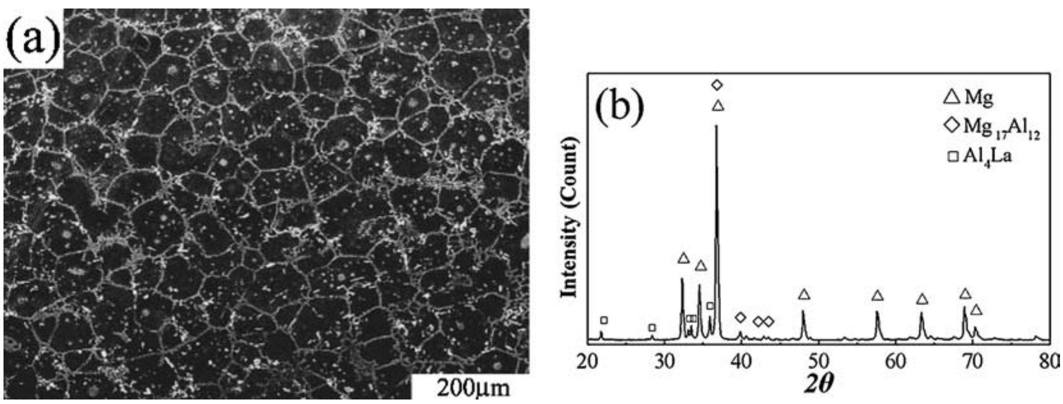

Figure 1 (a) SEM microstructure and (b) XRD pattern of the partially remelted AZ91D-4 wt% La_2O_3 alloy.

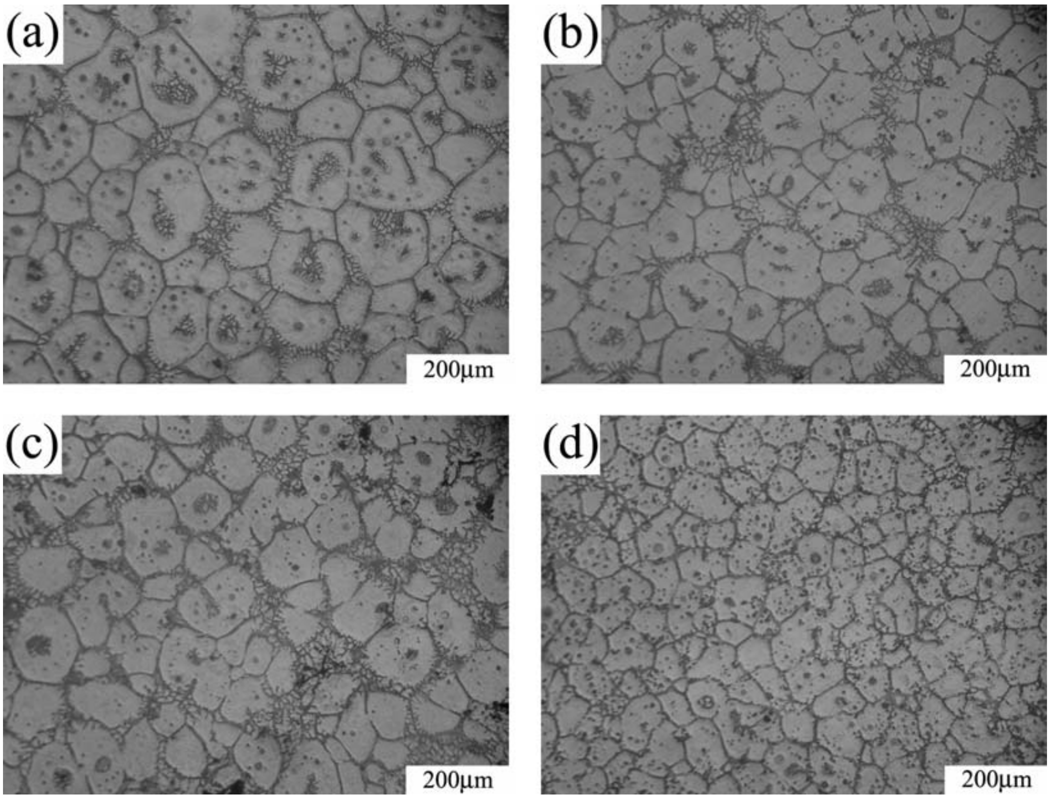

Figure 2 Partial remelting microstructures of the AZ91D matrix alloy and AZ91D-xwt.% La_2O_3 alloys at the predetermined isothermal holding temperature of 570°C: (a) AZ91D matrix alloy; (b) AZ91D-1 wt% La_2O_3 alloy; (c) AZ91D-2 wt% La_2O_3 alloy; (d) AZ91D-4 wt% La_2O_3 alloy.

grains, and finer grains were achieved with increasing of La_2O_3 content. After 4 wt% addition of La_2O_3, the grain is of the order of ∼100 μm (Fig. 2d), which is more than 50% smaller than that of the AZ91D alloy. The results are important for practical applications because the small globules will favor the flow of the slurry and make the intricate parts be produced easily.

It is well known that although the initial grain size does not determined the globules generated after a sufficiently long isothermal holding, the initial size of the globules in the semisolid is mostly affected by the initial grain size [22]. Recrystallization after pre-deformation prior to partially remelted can give rise to the favourable small initial grain sizes. Recrystallization is influenced not only by the predeformation [23] but also by the presence of $LaAl_4$ particles in this matrix.

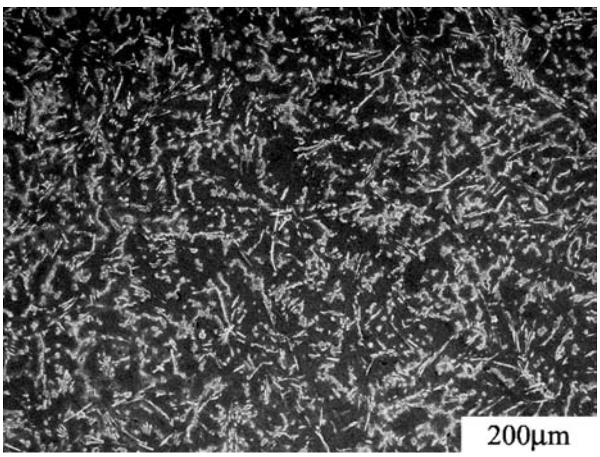

Figure 3 SEM microstructure of the as-cast AZ91D-4 wt% La_2O_3 alloy.

Fig. 3 shows the as-cast microstructure of the AZ91D-xwt.% La_2O_3 alloys after compression. In as-cast microstructure, the morphologies of $LaAl_4$ phases are also the needle-like and particle-shape. It has been shown elsewhere [20, 21] that $LaAl_4$ phase forms concurrently with the formation of α-Mg dendrites and before the formation of $Mg_{17}Al_{12}$ during the initial casting solidification. So some of $LaAl_4$ phases distributed at the grain boundaries and some distributed within the grains (Fig. 3). Since magnesium and its alloy have high affinity with oxygen, oxygen of the La_2O_3 perhaps reacts with Mg of the alloy to form the MgO which agglomerated or floated in the master alloy and was removed from the melting alloy as slag during the stir casting in a graphite crucible electric resistance furnace.

The presence of $LaAl_4$ phases gives the main reason why La_2O_3 refine the partially remelted microstructure. First, particle-shape $LaAl_4$ phase which dispersedly distributed in the grains can cause stress concentration and accelerate the dislocation multiplication. Secondly, these particle-shape $LaAl_4$ phases can also retard the climbing and the cross slipping of the dislocation, which results in the increasing of the amounts of dislocation. More dislocations can induce more recrystallization. Thirdly, these particle-shape $LaAl_4$ phases can also retard the combination and growth of recrystallization grains, which results in the decreasing of the size of recrystallization. Moreover, the effect of particle-shape $LaAl_4$ phases on the dislocation and rcrystallization increases with the increasing of the La_2O_3 addition. As a result, the presence of more particle-shape $LaAl_4$ results in the occurring of more and finer recrystallization grains. These recrystallization grains can easily give rise to the small initial grains which evolve towards the small globular partially remelted microstructure. The $LaAl_4$ phases distributed in the intergranular regions make the grains isolate with respect to each other, which restrains the combination and growth of the grains, and fines the partially remelted globule microstructure. La deoxidized from La_2O_3 reacts with Al within the molten metal to form the $LaAl_4$.

4. Conclusion

(1) Addition of La_2O_3 to AZ91D alloy results in the formation of $LaAl_4$ in the partially remelted microstructure. The new intermetallic phases show needle-like or particle-shape morphology and distribute at the grain boundaries and within grains.

(2) Small amounts of La_2O_3 addition to AZ91D alloy cause significant refinement of partially remelted microstructure. With the increasing of La_2O_3 addition, the size of grain is finer.

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