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Effects of semi-solid isothermal process parameters on microstructure of Mg-Gd alloy

SU Gui-hua(苏桂花), CAO Zhan-yi(曹占义), LIU Yong-bing(刘勇兵), WANG Yu-hui(王玉慧), ZHANG Liang(张 亮), CHENG Li-ren(程丽任)

Key Laboratory of Automobile Materials, Ministry of Education,
Department of Materials Science and Engineering, Jilin University, Changchun 130025, China

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Abstract: The effects of semi-solid isothermal process parameters on the microstructure evolution of Mg-Gd rare earth alloy produced by strain-induced melt activation (SIMA) were investigated. The formation mechanism of the particles in the process of the isothermal treatment was also discussed. The results show that the microstructure of the as-cast alloy consists of α-Mg solid solution, Mg_5RE and $Mg_{24}RE_5$ (Gd, Y, Nd) phase. After being extruded with an extrusion ratio of 14:1 at 380 ˚C, the microstructure of Mg-Gd alloy changes from developed dendrites to near-equiaxed grains. The liquid volume fraction of the semisolid slurry gradually increases with elevating isothermal temperature or prolonging isothermal time during the partial remelting. To obtain an ideal semisolid slurry, the optimal process parameters for the Mg-Gd alloy should be 630 ℃ for isothermal temperature and 30 min for the corresponding time, respectively, where the volume fraction of the liquid phase is 52%.

Key words: magnesium alloy; Mg-Gd alloy; rare earth; strain-induced melt activation; semisolid; isothermal treatment

1 Introduction

The semisolid forming (SSF) has become a widely accepted metal process, since it possesses the merits of traditional methods for casting and forging, such as the reductions of macrosegregations and porosity, low operating temperature and low forming force[1−3]. Many methods such as magnetohydrodynamic stirring (MHD), mechanical stirring, spray casting, semi-solid isothermal heat treatment and strain-induced melt activation (SIMA) can be used to produce semisolid materials. Among them, SIMA is an effective technology with significant commercial advantages of simplicity and low equipment cost in fabrication process, and has been demonstrated to be applicable to most engineering alloys, including aluminum, magnesium and copper alloys[4−6]. Whereas, magnesium alloys mainly used in SSM processing are limited to a few commercial Mg-Al alloys such as AZ91D, AM50 and AM60[7−8]. And their application is often restricted due to their inherent deficiencies, such as poor high temperature strength and creep resistance.

It is well known that the addition of RE elements in Mg alloys can bring about high strength especially at elevated temperatures. More recently, gadolinium and other RE have been found to raise the heat resistance because of the existence of rare earth phases with high thermal stability. Moreover, the mechanical properties of the alloys are higher than those of the conventional Mg alloys particularly at elevated temperature[9−14].

Based on the above analysis, the Mg-8Gd-2Y-1Nd-0.3Zn-0.6Zr (mass fraction, %) alloy was prepared, and the microstructure evolution process of the alloy during isothermal heat treatment was discussed. The results could be useful for the development of new semisolid magnesium alloys.

2 Experimental

The alloy ingot with a nominal composition of Mg-8Gd-2Y-1Nd-0.3Zn-0.6Zr (mass fraction, %) was prepared in an electric-resistant furnace under an anti-oxidizing flux protection by using high purity Mg

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Corresponding author: CAO Zhan-yi; Tel: +86-431-85095852; Fax: +86-431-85095876; E-mail: caozy@jlu.edu.cn

(99.9%), Zn (99.9%), the Mg-20RE (RE=Gd, Y and Nd, mass fraction, %) and Mg-30Zr (mass fraction, %) master alloys. The obtained ingot with a diameter of 80 mm was homogenized at 520 ˚C for 10 h, and then quenched into hot water at about 60 ˚C, at last a sheet with a size of 50 mm×8 mm in the cross section was got by hot extrusion. The extrusion ratio was 14:1 and the corresponding temperature was 380 ℃.

After being extruded, the samples were heated to the required temperature and held for predetermined time in the electric resistance furnace with the protective atmosphere of CO_2 and SF_6, and then were quenched into water at the room temperature. According to Mg-Gd binary alloy diagrams, the solidus and the liquidus temperatures for the Mg-8Gd magnesium alloy are 600 ˚C and 643 ˚C, respectively. To study the effects of temperature, the samples were heated at the temperatures of 600, 620, 630 and 640 ˚C, respectively, for 15 min. And to investigate the effects of time, the samples were held at 630 ˚C for 3, 9, 15, 30, 45 and 60 min, respectively.

The microstructures were characterized using an optical microscope (OLYMPUS-PMG3), a scanning electron microscope (SEM) (Model JSM-5310, Japan) equipped with an energy-dispersive spectrometer (EDS) (Model Link-Isis, Britain). Quantitative metallographic analyses including the volume fraction, the size and morphology of solid phase were taken using the self-programmed analysis software for characterizing the evolution of the microstructures during the isothermal treatment.

3 Results and discussion

3.1 Microstructure of extruded alloy

Fig.1 shows the optical micrographs of the as-extruded alloy on the extrusion direction (ED)−transverse direction (TD) plane. It can be seen from Fig.1 that dynamic recrystallization during the extrusion process makes the coarse, dendritic microstructure of the direct chill cast billet transform into a fine and equiaxed grain structure. However, there are elongated and unrecrystallized grains orientated in the extrusion direction as shown in Fig.1(a). The grain size distribution tends to be heterogeneous, and the small recrystallized grains of about 7 μm in size are embedded in a matrix with large grains of approximately 25 μm in diameter. Lots of irregular-shaped particles distributed in the matrix are observed in Fig.1(b) and these particles are mainly RE-rich particles[15]. According to the composition of the eutectic phases under the as-cast condition, it can be concluded that the RE-rich particles are the remained eutectic phases of Mg_5RE and $Mg_{24}RE_5$ (RE=Gd, Y, Nd) that will be broken into small particles

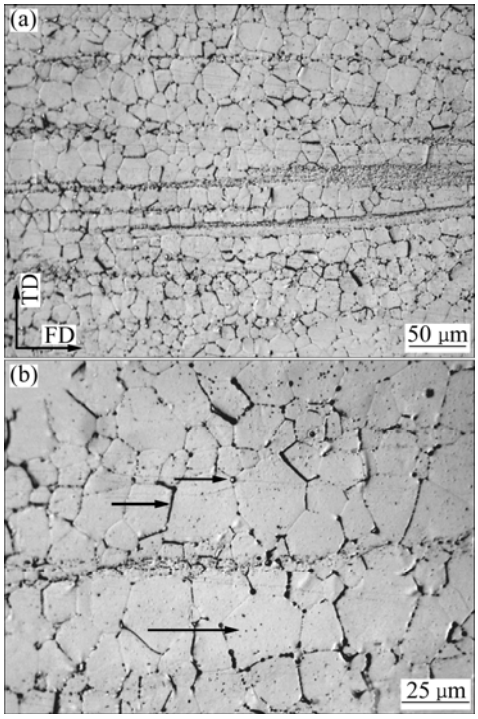

Fig.1 Optical micrographs of as-extruded alloy on extrusion direction (FD)−transverse direction (TD) plane: (a) In low magnification; (b) In high magnification

during the extrusion process[13].

3.2 Effect of isothermal temperature on semisolid microstructure

The quenched microstructures of the as-extruded alloy after isothermal treatment at different temperatures for 15 min, are illustrated in Fig.2. It can be seen that the microstructure mainly consists of spheroidal recrystallized particles with some internally entrapped liquid. With the temperature elevating from 600 ˚C to 640 ˚C, the volume fraction of solid particles gradually decreases from 83% to 39% and the average size of the solid particles reduces from 93 μm to 88 μm, as shown in Fig.3, which is obtained using the self-programmed analysis software. The further observation indicates that the boundary liquid film among solid particles is relatively thicker; the particles become more spheroidal; and the amount of particles reduces with increasing partial remelting temperature.

3.3 Effect of isothermal holding time on semisolid microstructure

Fig.4 shows the microstructures of the as-extruded alloy heated at 630 ˚C for different times. It can be observed that the fraction of liquid phases increases, the amount of the semisolid particles reduces, the size of the particles grows larger and the shape of the particles

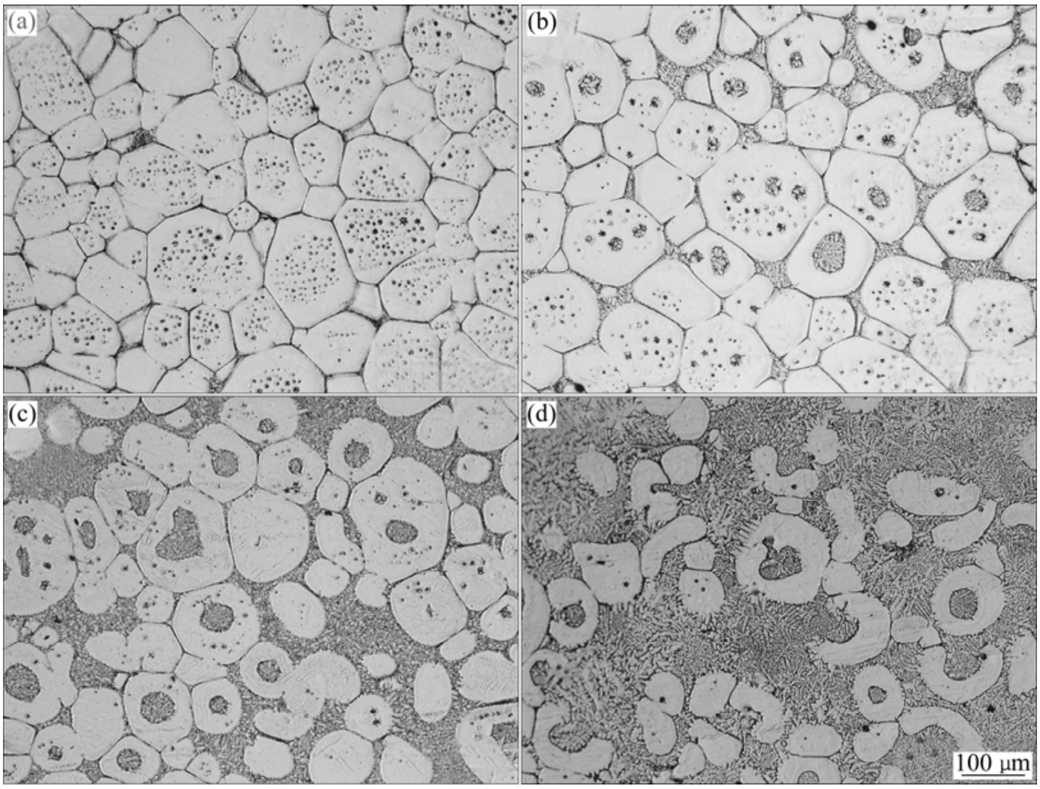

Fig.2 Microstructures of as-extruded alloy heated at different temperatures for 15 min: (a) 600 ˚C; (b) 620 ˚C; (c) 630 ˚C; (d) 640 ˚C

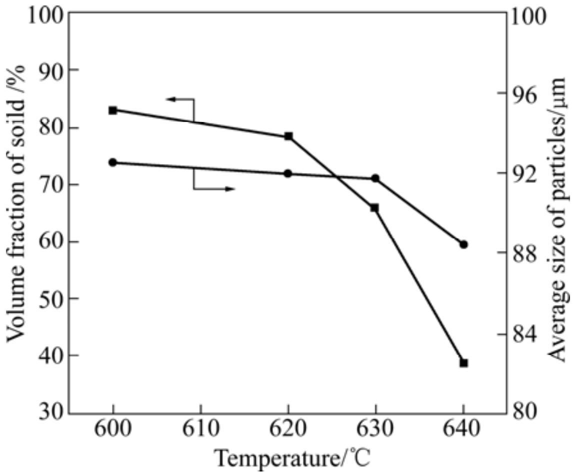

Fig.3 Effects of temperature on volume fraction of solid and average size of particles

becomes more globular with prolonging isothermal time at 630 ˚C. Much of the small liquid inclusions develop in the early stage of remelting. These inclusions continuously grow with increasing holding time in the semisolid state, accompanied with the reduced amount. The variations of the average size of the particles and the solid volume fraction in the semisolid microstructure as a function of isothermal holding time at 630 ˚C are illustrated in Fig.5. With the isothermal holding time varying from 3 min to 60 min, the solid volume fraction gradually reduces from 78% to 22%, and the average size of the semisolid particles increases gradually from 74 μm to 105 μm in the first 30 min and it will remain unchanged although the processing time is extended.

3.4 Evolution mechanisms

The microstructure of as-cast alloy is composed of dendritic α-Mg, eutectic β-phase Mg_5RE and $Mg_{24}RE_5$ (RE=Gd, Y, Nd), which distribute both at grain boundaries and within grains. After predeformation, much energy stores in the alloy, which increases the density of vacancies and dislocations, although most of the deformation energy is released as heating energy. Banded structures are typical for extruded products of the studied alloys as shown in Fig.1. After the short holding time or the low holding temperature, in order to decrease the free energy, the vacancies will combine, and the dislocations will climb and cross slip, which results in the occurrence of recovery and recrystallization. Moreover, low melting point phase melts which distributes in the recrystallization grain boundaries or within grain as a result of the holding temperature that is higher than the solidus temperature (see Fig.2(a) and Fig.4(a)). With increasing holding time, many small liquid droplets within grain combine and form several big liquid drops in order to reduce the interfacial energy. After full recrystallization and then increasing the holding time, Ostwald ripening and coalescence play an important role to make the average size of the solid

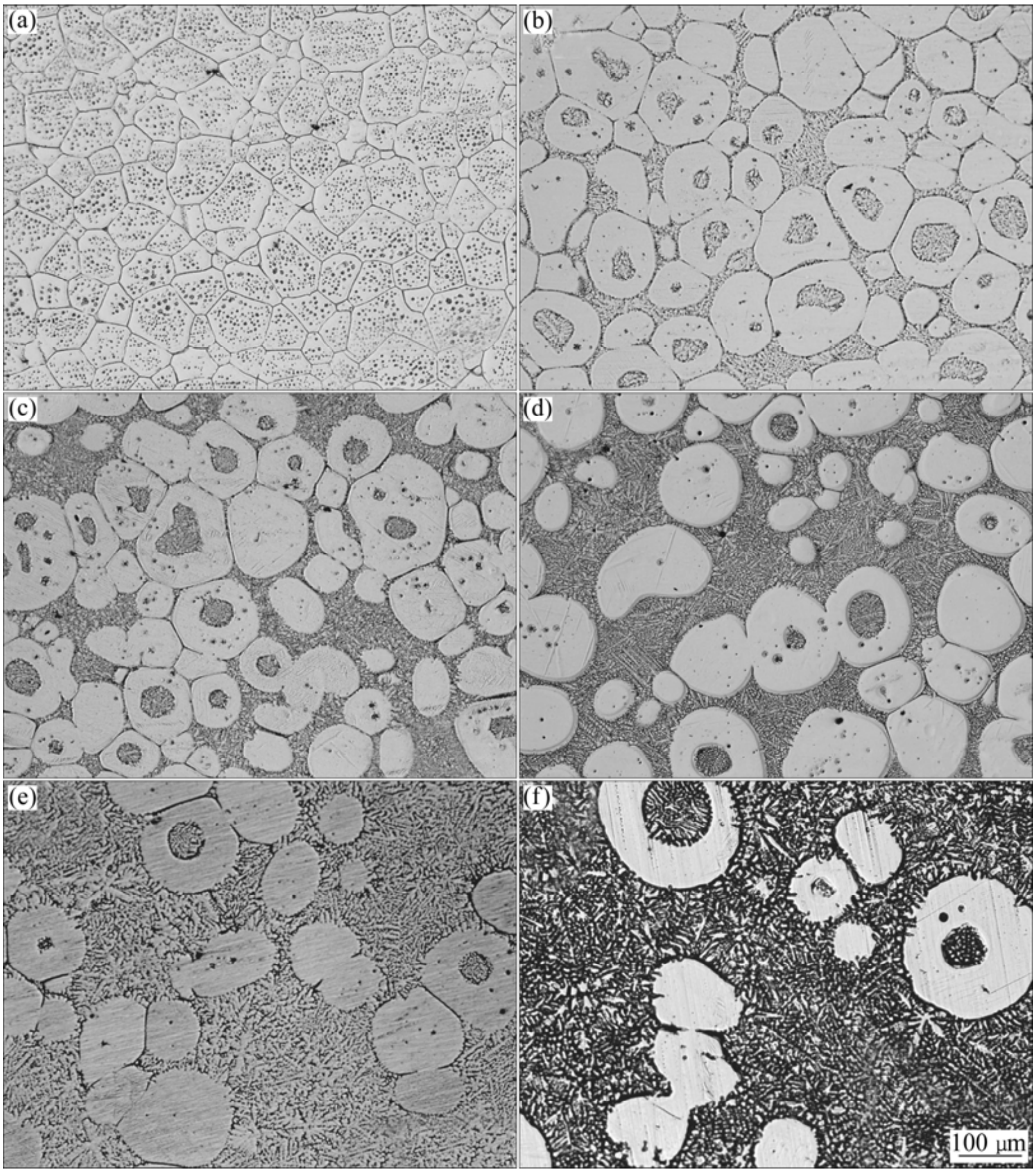

Fig.4 Microstructures of as-extruded alloy heated at 630 ℃ for different times: (a) 3 min; (b) 9 min; (c) 15 min; (d) 30 min; (e) 45 min; (f) 60 min

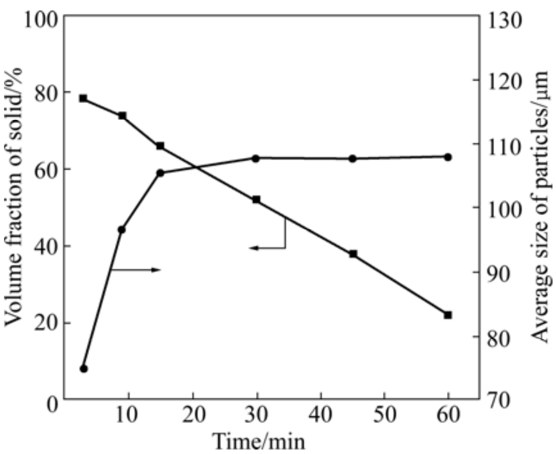

Fig.5 Effects of time on volume fraction of solid and average size of particles

particles increase. Ostwald ripening involves the growth of large particles at the expense of small particles, and it is governed by the Gibbs–Thompson effect which alters the chemical potential of solutes at the particle/liquid interface, depending on the curvature of the interface[16]. The reduction of interfacial energy between the solid phase and liquid phase provides the driving force for grain coarsening. Note that there are still a small number of irregular-shaped grains, which probably result from the coalescence of two spheroidal grains. Moreover, these irregular-shaped grains possibly originating from the extruded grains have not yet been completely broken up. When the holding time is long enough, which makes the solid volume fraction lower down, the Ostwald ripening mechanism also begins after the structural coarsening. The larger grain gradually becomes spheroidal to lower the solid/liquid interfacial energy.

It is clear that the high semisolid isothermal temperature reduces the volume fraction of solid and accelerates the spherical evolution of the solid particles, long isothermal time makes the semisolid particles more globular, but the size of the particles would grow larger. By analyzing the experimental data presented in this study, it is found that the optimal process parameters should be isothermal temperature of 630 ˚C, and time of 30 min. In this case the semisolid particles of the slurry are small and globular, and the solid volume fraction is also suitable.

4 Conclusions

1) With elevating isothermal temperature during the partial remelting, the liquid fraction in the semisolid slurry would be gradually enlarged and the solid particles would become small, and spheroidize with the consideration of the remelting mechanism and the specific surface energy.

2) With prolonging isothermal holding time during the partial remelting, the volume fraction of liquid phase gradually enlarges. The average diameter of the solid particles would increase, and the amount of the solid particles would decrease, by considering the Ostwald ripening and coalescence mechanism.

3) The optimal process parameters should be isothermal temperature of 630 ˚C, and time of 30 min.

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