



Effect of compression ratio on microstructure evolution of Mg–10%Al–1%Zn–1%Si alloy prepared by SIMA process

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Abstract: A new Mg–10%Al–1%Zn–1%Si alloy with non-dendritic microstructure was prepared by strain induced melt activation (SIMA) process. The effect of compression ratio on the evolution of semisolid microstructure of the experimental alloy was investigated. The results indicate that the average size of α -Mg grains decreases and spheroidizing tendency becomes more obvious with the compression ratios increasing from 0 to 40%. In addition, the eutectic Mg₂Si phase in the Mg–10%Al–1%Zn–1%Si alloy transforms completely from the initial fishbone shape to globular shape by SIMA process. With the increasing of compression ratio, the morphology and average size of Mg₂Si phases do not change obviously. The morphology modification mechanism of Mg₂Si phase in Mg–10%Al–1%Zn–1%Si alloy by SIMA process was also studied.

Key words: Mg–Al–Zn–Si alloy; compression ratio; microstructure evolution; eutectic Mg₂Si phase; strain induced melt activation (SIMA) process

1 Introduction

Magnesium alloys have been recognized as the most promising lightweight structural materials in aerospace, marine, automotive, electronics, medical industries and other fields due to their excellent properties [1,2]. The Mg–Al–Si series alloys (e.g. AS21 and AS41) are the most common commercial magnesium alloys, which have the best combination of the excellent castability and high strength [3,4]. However, the undesirable fishbone shaped eutectic Mg₂Si phase tends to form in Mg–Al–Si series

alloys in the traditional solidification process, which can give detrimental effects on the microstructure and mechanical properties of the alloys [5–7]. Therefore, the application of Mg–Al–Si series alloys will be greatly promoted if the fishbone shaped eutectic Mg₂Si phase can be modified by using a simple and effective method.

It is well known that the semisolid metal (SSM) processing has broad development space in modifying the microstructure and mechanical properties for magnesium alloys [8,9]. At present, the methods of obtaining semisolid material with non-dendritic microstructure include mechanical

stirring [10], electromagnetic stirring [11], rapid solidification [12] and semisolid isothermal heat treatment [13]. Among all conventional techniques of SSM, the strain induced melt activation (SIMA) process has some inherent advantages in comparison with other methods [14,15]. The SIMA process is relatively simple and does not require complex equipment; in addition, it omits the procedure of molten metal treatment, and is suitable for both low and high melting point alloys [16].

In recent years, many studies have been performed to modify the eutectic Mg_2Si phase in Mg–Al–Zn–Si alloy to obtain an increased strength and ductility. YANG et al [17] studied the effect of semisolid isothermal heat treatment on the microstructure of Mg–6Al–1Zn–0.7Si alloy, where the eutectic Mg_2Si phase could be modified from the Chinese script shape to granule and/or polygon shapes. CAO et al [18] studied the effects of isothermal process parameters on the semisolid microstructure of Mg–8%Al–1%Si alloy, where the Chinese script shaped eutectic Mg_2Si phase could be modified to polygon shape by semisolid isothermal heat treatment. MA et al [19] studied the effect of holding temperature on microstructure of an AS91 alloy during semisolid isothermal heat treatment, where the eutectic Mg_2Si phase changed from the initial Chinese script shape to granule and/or polygon shapes during the isothermal heat treatment. But less work has been carried out on the Mg–Al–Zn–Si alloy prepared by SIMA process.

The purpose of the present work is to investigate the microstructure evolution of Mg–10%Al–1%Zn–1%Si alloy prepared by SIMA process, and particular attention is paid to the effect of compression ratio on semisolid microstructure of the experimental alloy, in order to develop a simple and effective method for modification of fishbone shaped Mg_2Si phase in the experimental alloy. It is expected that the preliminary study results can be significant in promoting the preparation of high quality Mg–Al–Zn–Si alloys.

2 Experimental

The Mg–10%Al–1%Zn–1%Si alloy was prepared by the following materials: commercial pure Mg, pure Al, pure Zn and pure Si powder. The experimental alloy was melted in an electric resistance furnace under a protective atmosphere of

SF_6/CO_2 and then cast into a preheated permanent mould to get ingots. Table 1 listed the actual chemical composition of the experimental alloy, which was measured by using an optical emission spectrometer (ICP-OES, Avio200, USA). Subsequently, the ingots were machined into the cylinder samples with dimensions of $d16\text{ mm} \times 20\text{ mm}$, and then were subjected to deformation by compression at $350\text{ }^\circ\text{C}$ in the 200 t hydraulic pressure machine, and the compression ratios were 0, 8%, 16%, 24%, 32% and 40%, respectively.

Table 1 Chemical composition of Mg–10%Al–1%Zn–1%Si alloy (wt.%)

Al	Zn	Si	Fe	Ni	Sn	Mg
10.123	0.985	0.976	0.00147	0.00137	0.00136	Bal.

The differential scanning calorimeter (DSC, NETZSCH STA449C, Germany) was applied to obtaining the semisolid holding temperature of the experimental alloy. The sample of around 40 mg was heated at $15\text{ }^\circ\text{C}/\text{min}$ to $700\text{ }^\circ\text{C}$ under an argon protective atmosphere in the DSC apparatus, and the DSC curve of the experimental alloy is shown in Fig. 1. Based on the DSC result, the isothermal holding temperature was selected at $560\text{ }^\circ\text{C}$, which was between the eutectic and liquidus temperature.

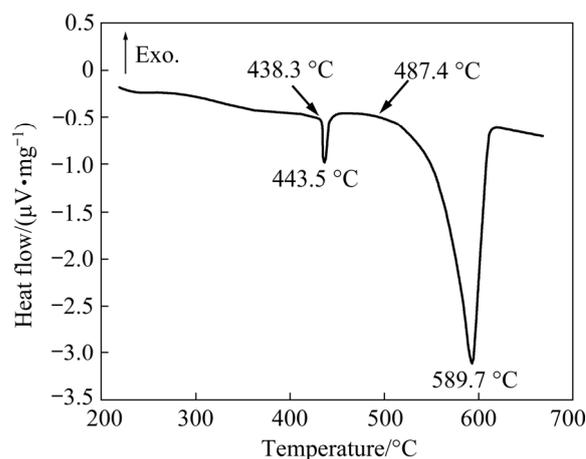


Fig. 1 DSC curve of as-cast Mg–10%Al–1%Zn–1%Si alloy

After being compressed, the samples were held at $560\text{ }^\circ\text{C}$ for 30 min in an electric resistance furnace under a protective atmosphere of SF_6/CO_2 , and then were taken out immediately for cold water quenching. Metallographic samples were polished in accordance with standard procedures and etched

with 3 vol.% HNO₃ in alcohol. Microstructure and phase analyses were examined by using optical microscopy (OM, Olympus PMG3, Japan) and X-ray diffraction (XRD, Rigaku Dymax, Japan). In this work, quantitative metallography analyses including the average size and shape factor of solid grains were performed by using the MIP image analyzing software.

3 Results and discussion

3.1 As-cast microstructure

Figure 2 shows the as-cast microstructure and XRD pattern of Mg–10%Al–1%Zn–1%Si alloy. As shown in Fig. 2(a), the as-cast microstructure of Mg–10%Al–1%Zn–1%Si alloy comprise α -Mg dendrites, (α -Mg+Mg₂Si) eutectic with fishbone shaped Mg₂Si phases embedded in the α -Mg matrix and β -Mg₁₇Al₁₂ phases precipitated discontinuously at grain boundaries. It can be seen from Fig. 2(b) that the microstructures of Mg–10%Al–1%Zn–1%Si alloy consist of α -Mg, β -Mg₁₇Al₁₂ and Mg₂Si phases, which agrees with the compositions of the

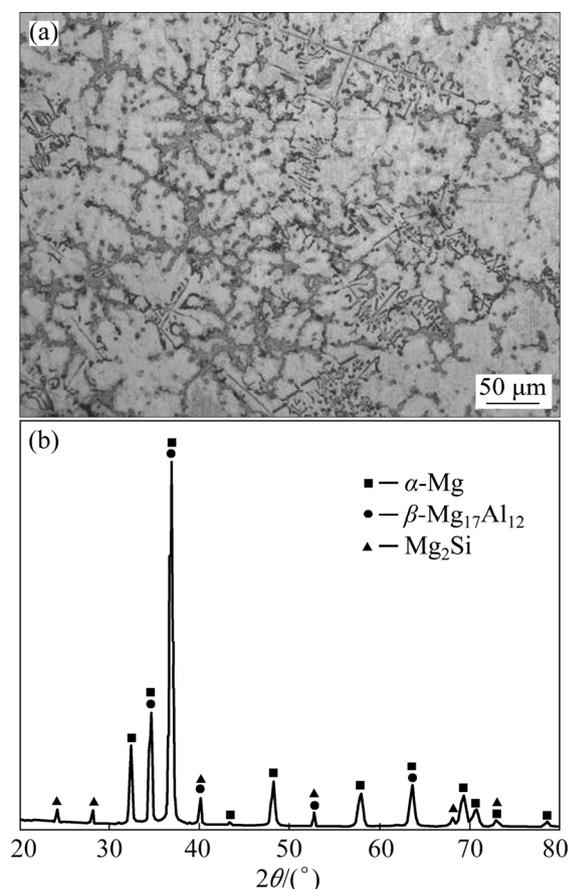


Fig. 2 Microstructure (a) and XRD pattern (b) of as-cast Mg–10%Al–1%Zn–1%Si alloy

common Mg–Al–Si based alloys [20]. In general, the Mg₂Si phases in Mg–Al–Si based alloys are prone to forming coarse Chinese script shape at low solidification rate [21]. Therefore, under the permanent mould casting in this work, the Mg–10%Al–1%Zn–1%Si alloy is formed at low solidification rate, so the Mg₂Si phase in experimental alloy exhibits coarse fishbone shaped morphology as well.

3.2 As-deformed microstructures

Figure 3 shows the as-deformed microstructures of Mg–10%Al–1%Zn–1%Si alloy with different compression ratios from 0 to 40%. It can be seen from Fig. 3(a) that the α -Mg dendrites distinctly exhibit the spontaneous growth characteristics, which are formed during the metallurgical solidification process. After being deformed at lower compression ratios, most of the primary α -Mg dendrites, (α -Mg+Mg₂Si) eutectic with fishbone shaped Mg₂Si and β -Mg₁₇Al₁₂ phases still maintain their shapes, as shown in Figs. 3(b, c). With the compression ratios increasing, β -Mg₁₇Al₁₂ and Mg₂Si phases are ruptured and distribute over the grain boundaries like strings, and present a slight orientation along the deformation direction, as shown in Fig. 3(d). When the compression ratios continue to increase, two phases approach the same deformation degree, and the evident fibroid orientation is vertical to the compression direction, as shown in Fig. 3(e). When the compression ratio reaches 40%, the vertical spacing is nearly invariable, and the microstructure has no obvious difference, as shown in Fig. 3(f).

3.3 Semisolid microstructures

Figure 4 shows the semisolid microstructures of Mg–10%Al–1%Zn–1%Si alloy with different compression ratios after isothermal holding at 560 °C for 30 min. As shown in Figs. 4(a, b), when the compression ratios are smaller than 16%, the microstructure consists of many α -Mg grains with large size and irregular shape, although the α -Mg grains have undergone spheroidization obviously (Fig. 4(b)). With increasing the compression ratio, α -Mg grains become more spheroidal gradually. The high angle boundaries of recrystallized grains are penetrated by liquid, thus leading to the entire fragmentation of α -Mg grains into smaller ones. Through careful examination, the microstructure

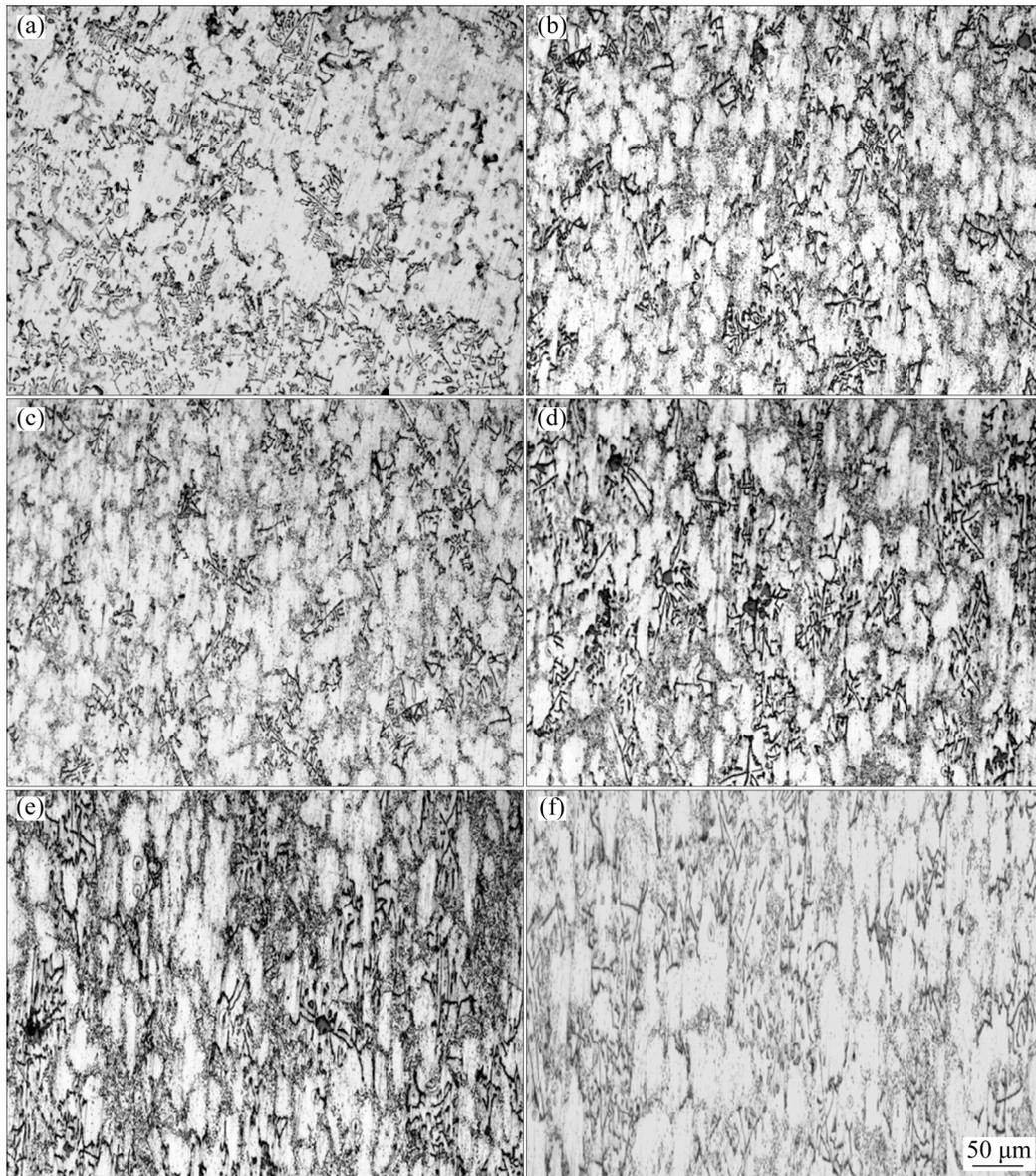


Fig. 3 As-deformed microstructures of Mg–10%Al–1%Zn–1%Si alloy with different compression ratios: (a) 0; (b) 8%; (c) 16%; (d) 24%; (e) 32%; (f) 40%

reveals that these newly formed α -Mg grains are still slightly irregular (Fig. 4(c)). With further increase of compression ratio, the microstructures consist of fine and nearly spheroidal α -Mg grains uniformly distributing in a liquid phase (Figs. 4(d, e)). As shown in Fig. 4(f), such microstructures are sufficient for semisolid processing by inducing enough strain. By comparing Figs. 4(a–f), it is found that the spheroidization and refinement of α -Mg grains are particularly clear for higher compression ratios. The higher the compression ratio is, the greater the overall α -Mg grain boundary and sub-grain boundary area are, which leads to greater potential for the development of

recrystallisation nuclei, and therefore, finer recrystallised grains occur. During isothermal holding treatment, a finer recrystallised grain size results in a finer α -Mg grain size with the same processing parameters [22].

Figure 5 shows the effects of compression ratios on the average size and shape factor of α -Mg grains after isothermal holding at 560 °C for 30 min. It can be observed that average size of α -Mg grain gradually decreases from 89.12 to 43.96 μm with increasing the compression ratio. However, the values of shape factor increases with the increasing of compression ratio, which indicates that the degree of spheroidization is improved. Because the

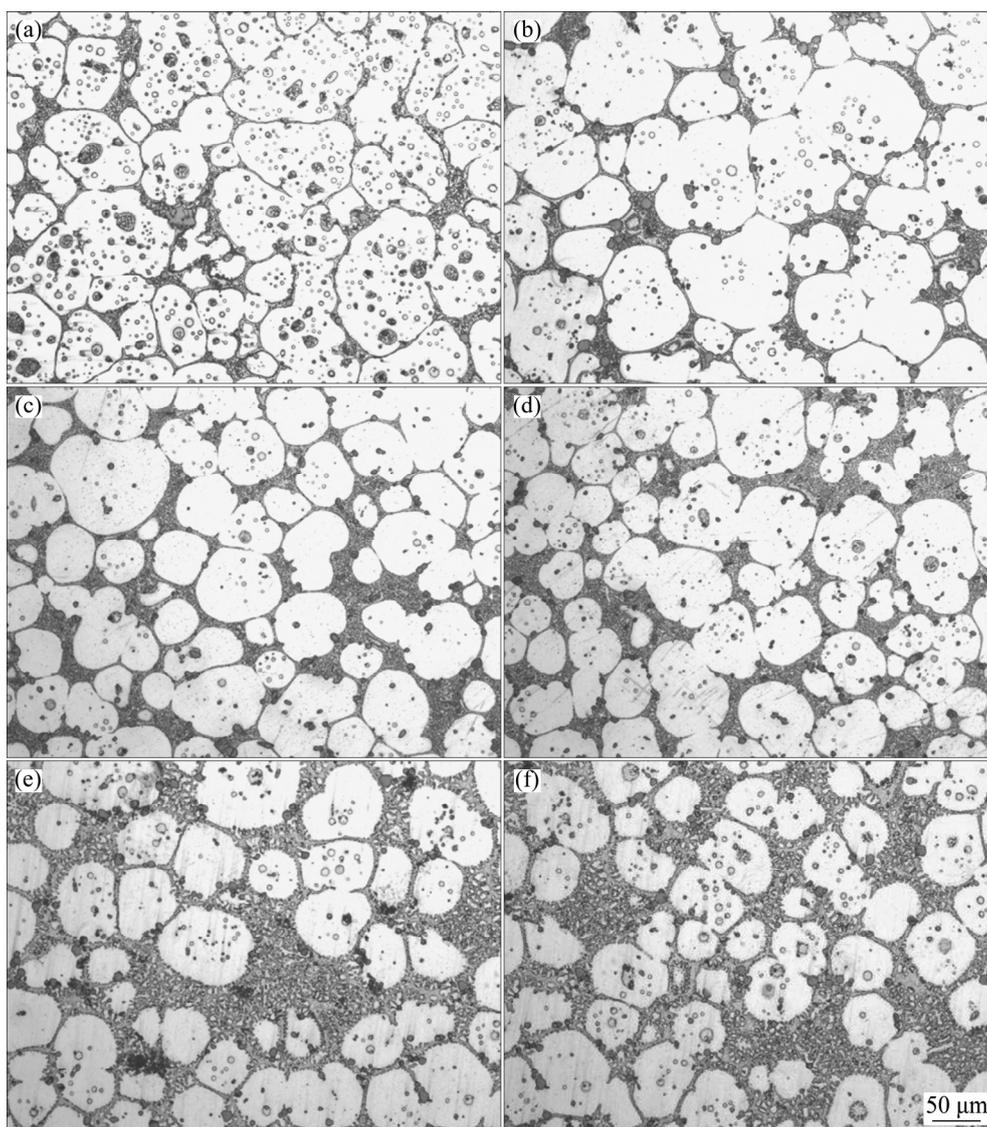


Fig. 4 Semisolid microstructures of Mg–10%Al–1%Zn–1%Si alloy with different compression ratios after isothermal holding at 560 °C for 30 min: (a) 0%; (b) 8%; (c) 16%; (d) 24%; (e) 32%; (f) 40%

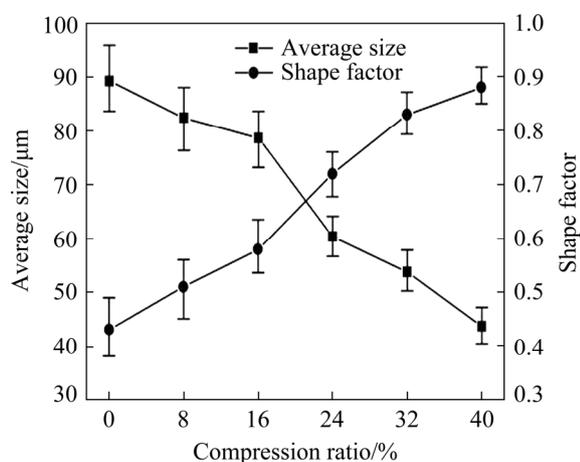


Fig. 5 Effects of compression ratios on average size and shape factor of α -Mg grains after isothermal holding at 560 °C for 30 min

state of as-deformed Mg–10%Al–1%Zn–1%Si alloy is more thermodynamically unstable during isothermal holding, the atomic diffusion capacity increases. Spheroidization is a diffusion-controlled process, so the shape factor of α -Mg grains is improved.

Figure 6 shows the morphologies of Mg_2Si phase in semisolid microstructures of Mg–10%Al–1%Zn–1%Si alloy with different compression ratios after isothermal holding at 560 °C for 30 min. The microstructures of all the samples show that the initial fishbone shaped Mg_2Si phase has transformed into globular shape with an average grain size of 16–17 μm , instead of polygon shape during semisolid isothermal heat treatment [17,20].

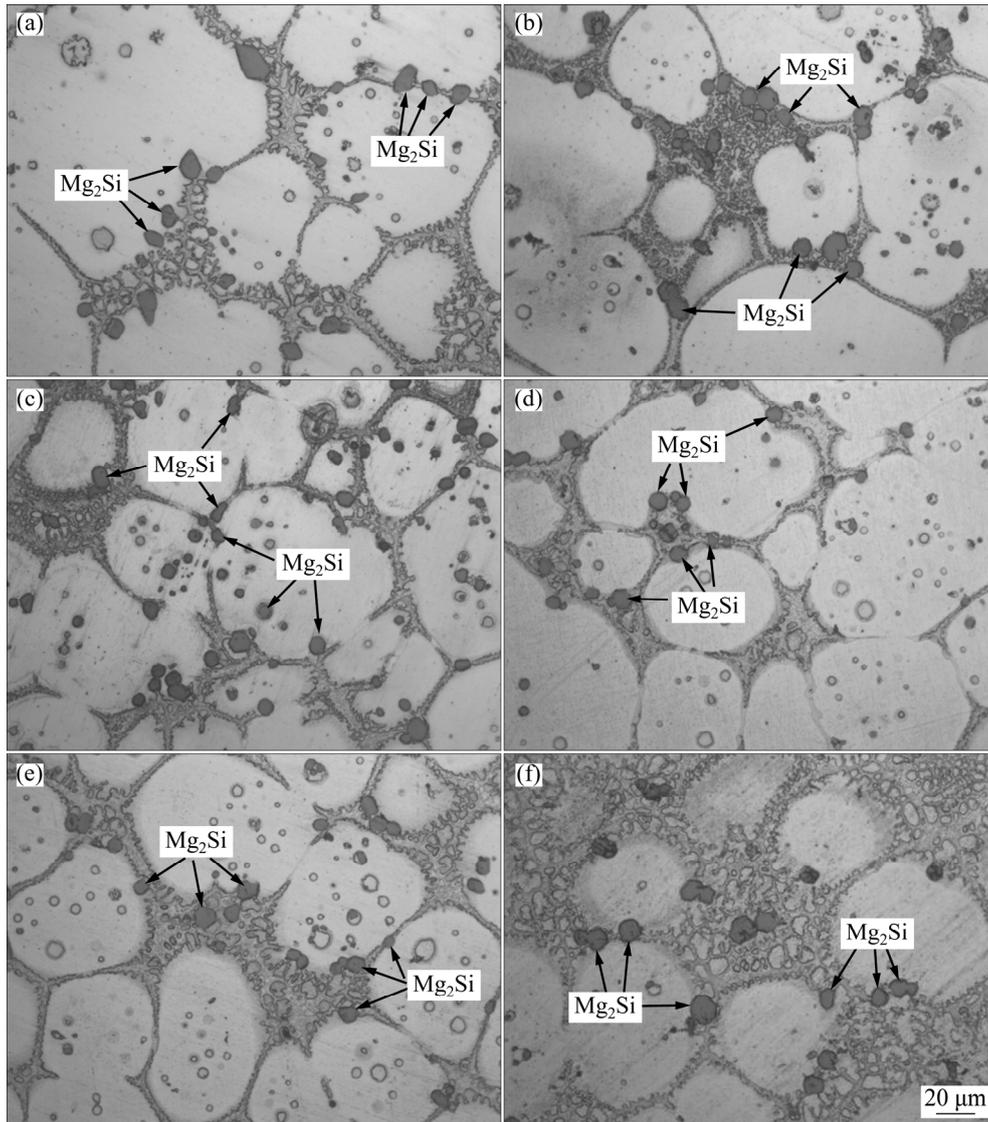


Fig. 6 Morphologies of Mg_2Si phase in semisolid microstructures of Mg-10%Al-1%Zn-1%Si alloy with different compression ratios after isothermal holding at 560 °C for 30 min: (a) 0; (b) 8%; (c) 16%; (d) 24%; (e) 32%; (f) 40%

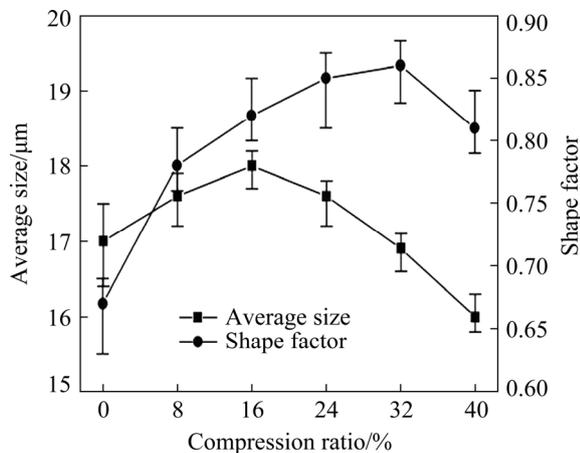


Fig. 7 Effects of compression ratios on average size and shape factor of Mg_2Si phases after isothermal holding at 560 °C for 30 min

With the increasing of compression ratio, the morphology and average size of Mg_2Si phases do not change obviously, as shown in Fig. 7. It can also be observed that most of the globular Mg_2Si phases are present in the liquid phases distributing at the α -Mg grain boundaries, but a few of ones are found inside the α -Mg grains (as shown in Fig. 6 by the arrows).

Figure 8 shows the sketch of modification mechanism of fishbone shaped Mg_2Si phase. Actually, the fishbone shaped Mg_2Si phase has the dendrite characteristics. There always exist concave pits with large curvature and high Si concentration at the bottom of Mg_2Si dendrite arms (Positions A and B shown in Fig. 8(a)). Therefore, the Si atoms

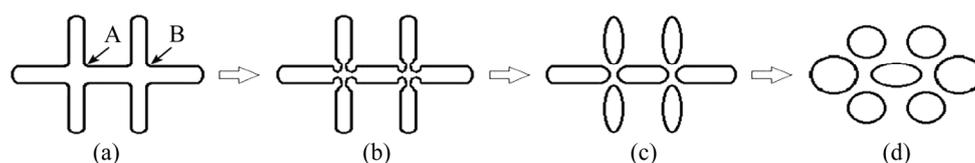


Fig. 8 Sketch of modification mechanism of fishbone shaped Mg_2Si phase in larger curvature site: (a) Mg_2Si phase; (b) Dissolving; (c) Breaking; (d) Spheroidizing

would diffuse from these positions with higher Si concentration to flat interface with lower Si concentration during the semisolid isothermal heat treatment, leading to the breaking of local Si concentration balance. In order to keep the balance of Si concentration, the Mg_2Si phase in these positions would dissolve gradually to provide the deficiency of Si concentration, as shown in Fig. 8(b). Consequently, under the effect of Si atoms diffusion and interface tension, the concave pits with larger curvature at the bottom of the Mg_2Si dendrite arm are dissolved and eventually broken, as shown in Fig. 8(c). Finally, the globular shape Mg_2Si phases which have close curvature radius at different positions would be formed during the isothermal heat treatment, as shown in Fig. 8(d).

Figure 9 shows SEM images of Mg–10%Al–1%Zn–1%Si alloy as-cast and as-deformed with 40% compression ratio after isothermal holding at 560 °C for 30 min. And the EDS results of the Positions A, B, C, D and E in Fig. 9 are listed in Table 2. From Table 2, the discontinuous phase is β - $Mg_{17}Al_{12}$ (Position A) and the fishbone shaped phase is Mg_2Si (Position B) in Fig. 9(a). Meanwhile, after isothermal holding at 560 °C for 30 min with 40% compression ratio, the globular shaped phase appears, which is identified as Mg_2Si ((Position C) in Fig. 9(b)). The Positions D and E are identified as matrix near Mg_2Si phases in the as-cast and as-deformed with 40% compression ratio after isothermal holding treatment. The Si contents in the matrix near Mg_2Si phases at Positions D and E are 0.22 and 0.23 wt.%, respectively, which indicates that little Si dissolves in the matrix after isothermal holding and Si is still in the form of Mg_2Si phases. Therefore, it can be concluded that the volume fraction of Mg_2Si phases keeps constant, which indicates that the SIMA process can modify the fishbone shaped Mg_2Si phases in the Mg–10%Al–1%Zn–1%Si alloy.

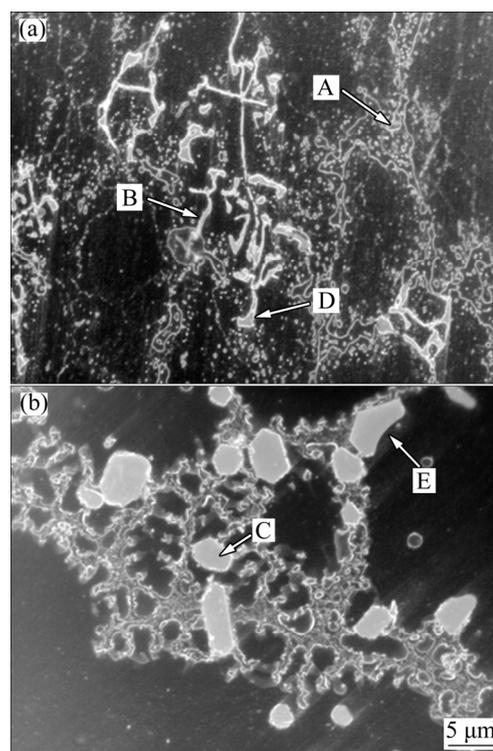


Fig. 9 SEM images of Mg–10%Al–1%Zn–1%Si alloy: (a) As-cast; (b) As-deformed with 40% compression ratio after isothermal holding at 560 °C for 30 min

Table 2 EDS results of Positions A, B, C, D and E in Fig. 9 (wt.%)

Position	Mg	Al	Zn	Si	Total
A	76.63	21.75	1.62	–	100
B	64.57	–	–	35.43	100
C	65.35	–	–	34.65	100
D	93.34	6.02	0.42	0.22	100
E	92.45	6.85	0.47	0.23	100

4 Conclusions

(1) The Mg–10%Al–1%Zn–1%Si alloy was prepared by SIMA process and the effect of compression ratio on the microstructure evolution of the experimental alloy was investigated. With the compression ratios increasing from 0 to 40%, the average size of α -Mg grains decreases from 89.12

to 43.96 μm and spheroidizing tendency becomes more obvious.

(2) The morphology of eutectic Mg_2Si phase transforms completely from the initial fishbone shape to globular shape in $\text{Mg-10\%Al-1\%Zn-1\%Si}$ alloy prepared by SIMA process. With the increasing of compression ratio, the morphology and average size of Mg_2Si phases do not change obviously.

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压缩变形量对 SIMA 法制备 Mg-10%Al-1%Zn-1%Si 合金显微组织演变的影响

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摘 要: 采用应变诱发熔化激活法(SIMA)制备具有非枝晶显微组织的 Mg-10%Al-1%Zn-1%Si 合金, 并且研究压缩变形量对 Mg-10%Al-1%Zn-1%Si 合金半固态显微组织演变的影响。研究表明: 随着压缩变形量从 0 提高到 40%, α -Mg 晶粒的平均尺寸减小并且其球化趋势变得更为明显。此外, 在 SIMA 法制备的 Mg-10%Al-1%Zn-1%Si 合金中的共晶 Mg₂Si 相从初始鱼骨状完全转变成为近球状; 随着压缩变形量的增大, 共晶 Mg₂Si 相的整体形貌和平均尺寸没有发生明显变化。并且, 研究 SIMA 法制备的 Mg-10%Al-1%Zn-1%Si 合金中共晶 Mg₂Si 相的形貌转变机制。

关键词: Mg-Al-Zn-Si 合金; 压缩变形量; 显微组织演变; 共晶 Mg₂Si 相; 应变诱发熔化激活法(SIMA)

(Edited by Bing YANG)