Effect of compression deformation on the distortion energy and semi-solid microstructure of Mg-6Al-0.9Gd alloys

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Abstract. In order to investigate the effect of distortion energy on the microstructure of magnesium alloys prepared by strain induced melt activation (SIMA) process and partial remelting, Mg-6Al-0.9Gd magnesium alloys were deformed at 200 ℃ with 0-20% compression ratios, and the relationship between deformation and eutectic melting activation energy was established based on differential scanning calorimetry (DSC) analysis, thereby determining the distortion energy at different compression ratios. The results showed that distortion energy increased with increasing compression ratio, which contributed to the morphological transition from dientritic to equiaxed structure, and accelerated the formation of liquid phase during subsequent partial remelting. All things (grain size, liquid fraction and distribution in the semi-solid microstructure) considered, the optimal compression ratio for this paper was 20%.

1. Introduction

The Mg-Al series alloys are the most common commercial magnesium alloys due to their good castability and high strength at room temperature, and have received specific attention in the automobile industry [1]. However, their application ranges were restricted due to poor high-temperature strength and creep resistance especially at temperature above 120℃ [2]. Previous investigations indicated that addition of rare earth elements (RE, e.g. Gd, Y, Nd, Sm, Dy) to Mg-Al alloys can effectively improve their high temperature strength and creep resistance by generating thermally stable precipitates Al₁₁₁RE₃ or Al₂RE phases and suppressing the formation of β-Mg₁₇Al₁₂ phases [3].

To date, almost 90% of total magnesium alloy components are produced by casting processes, especially high pressure die casting (HPDC) [4]. In recent years, semi-solid processing had become popular for industrial and commercial production, and exhibited significant advantages over HPDC, such as longer die life due to lower temperature, better mechanical properties due to less entrapped air, less porosity and fine microstructure, and near-net shape processing [5]. It is well known that the key of semi-solid processing is to prepare semi-solid slurry with a non-dendritic, spheroidal microstructure. Many methods such as electromagnetic stirring [6], gas bubbling process [7], recrystallization and partial melting [8], and strain induced melt activation (SIMA) have been used to prepare semi-solid slurry [9]. In the SIMA process, the billet with dendritic crystal is pre-deformed (compression, forging, extrusion, e.t.), and then reheated to the semi-solid state temperature followed by holding isothermally...
to make the dendritic crystal sphericized. Finally, the semi-solid billet is formed into required component. Through the pre-deformation, a certain number of distortion energy accumulates in the deformed billet, which contributes to the microstructure transition from dendritic crystal to equiaxed grains at low temperature in a short time, preventing microstructure coarseness during holding process at elevated temperature [10]. Therefore, it is of great significance to study the correlation between distortion energy and the semi-solid microstructure of the deformed metals. However, there are only limited amount of reports about the quantitative examination of distortion energy, because it cannot be experimentally measured directly [11]. Considering the eutectic melting activation energy when atom transits from solid phase to liquid phase during melting can be determined by calculation [12], it is necessary to investigate the relationship between distortion energy and eutectic melting activation energy. In present work, Mg-6Al-0.9Gd alloys were subjected to hot compression deformation, and the microstructure of the compressed specimens with different compression ratios was observed. The relationship between eutectic melting activation energy and distortion energy was examined through DSC analysis, thereby determining the accumulated distortion energy at different compression ratios. The effect of distortion energy on the semi-solid microstructure was discussed. This work is expected to provide theoretical guidance in semi-solid processing of Mg alloys.

2. Experimental procedures

High-purity Mg (99.95 wt. % purity), high-purity Al (99.98 wt. % purity), Mg-30%Gd master alloy were used as raw materials to prepare Mg-6Al-0.9Gd magnesium alloys. Cylindrical specimens with the same volume but different dimensions (ø25.2mm×61.3mm, ø24.5mm×64.8mm, ø23.8mm×68.7mm) were machined from the obtained ingot casting. These specimens were heated to 200 °C and held for 60 min, and then compressed into ø26.6 mm×55mm cylindrical specimens using closed-die forging in a WE-600 universal materials tester. The compression ratio of the specimens was defined as \( \varepsilon = (H_0 - H) / H_0 \), where \( H_0 \) and \( H \) are specimen height before and after compression deformation, respectively. Hence, the compression ratios of the three cylindrical specimens are 10%, 15%, and 20%, respectively.

Both the samples for metallographic observation and DSC tests were machined from the section center of the compressed specimens. In order to examine the effect of compression ratio on the solidus and liquidus temperatures, DSC tests were performed on a NETZSCH STA 449F3 thermal analyzer under high-purify argon gas atmosphere. The samples with 3 mm in diameter and about 30 mg in weight were heated to 800 °C at 5 K/min, 10K/min, and 15K/min, respectively, and then cooled to room temperature. In order to study the effect of compression deformation on the semi-solid microstructure, the compressed specimens were subjected to a isothermal heat treatment at 550 °C for 15 min, and subsequently quenched in cold water. The metallographic specimens were mechanically ground, polished and etched with a 4 vol. % nitric acid-ethanol solution, and then examined by using an Olympus GX71 optical microscope (OM). Grain size \( D = \sqrt{4A/\pi} \), where \( A \) is the area of solid grain), and liquid phase fraction were measured from the resulting optical micrographs using an image analysis system.

3. Results and discussion

3.1. Deformed microstructure

Figure 1 shows the microstructure of Mg-6Al-0.9Gd alloys at different deformation ratios. Figure 1 (a) shows the original cast microstructure, which is characterized by randomly distributed coarse dendritic crystal. Figure 1 (b) shows the microstructure of the compressed specimen at 10% compression ratio. It can be seen from Figure 1 (b) that the microstructure still presents dendritic morphology, and only partial dendrites were broken. When compression ratio reaches 15%, some dendrite arms were broken into fragments, some dendrite arms become thinner and longer due to compression deformation, and the space between adjacent dendrite arms decreased obviously, as shown in Figure 1 (c). When compression ratio rises to 20%, the number of fractured dendrites further increased, and finer particles were formed in some areas, as shown in Figure 1 (d). During compression deformation, some large
dendritic crystals were squashed, bent, and broken into fine grains. The grains rearranged and rotated under flow stress, forming wavy stripes and causing plastic deformation of the whole specimen.

![Figure 1. Optical microstructure of Mg-6Al-0.9Gd alloys at different compression ratio](image)

(a) 0%, (b) 10%, (c) 15%, and (d) 20%.

The microstructure of the Mg-6Al-0.9Gd alloys gradually changed with increasing compression ratio can be ascribed to following aspects: on the one hand, at the early stage of compression deformation, some dendrite arms were stretched due to plastic deformation, which cause the increase of intragranular defects. On the other hand, the dendritic crystals which are not easy to be deformed plastically were fractured, which introduced intergranular defects. The accumulation of micro defects resulted in the increase of distortion energy.

The difference in morphology of dendritic crystals became greater and greater with increasing compression ratio, and this can be due to the difference between α-Mg dendritic crystals and β-Mg₅₇Al₁₂ phase. The former are soft phases, and the latter are hard phases at grain boundary. When deformation was small, the soft dendritic crystals underwent slight deformation. With the increase of deformation, the soft dendritic crystals were stretched, and suffered from obvious plastic deformation. However, the hard β-Mg₅₇Al₁₂ phases which are not easy to be deformed plastically were ruptured.

### 3.2. Eutectic melting activation energy

The compression deformation caused the accumulation of distortion energy in the deformed Mg-6Al-0.9Gd alloys. When eutectic reaction occurs during subsequent partial remelting, the thermodynamic behavior of the deformed samples will vary with different deformation. Figure 2 shows the DSC curves obtained at a heating rate of 10 K/min for the deformed Mg-6Al-0.9Gd alloys with different compression ratios, in which $T_p$ is peak melting temperature, i.e. the temperature corresponding to the maximum deflection of the DSC curve, and $T_{on}$ is onset melting temperature, i.e. the temperature found by extrapolating the baseline (prior to the peak) and the tangent at the inflection point on the leading side of the peak to their intersection [13], as shown in Figure 2 (d). It can be seen that all the
endothermic peaks in the DSC curves are sharp with narrow temperature range. Combining the microstructure of the deformed Mg-6Al-0.9Gd alloys and phase diagram [14], it can be inferred that these endothermic peaks correspond to eutectic reaction, i.e. α-Mg + β-Mg17Al12 → L.

Onset melting temperature $T_{on}$ refers to the minimum temperature at which endothermic effect occurs, and is one of the most important data in DSC analysis. Table 1 lists the onset melting temperatures determined from DSC curves. Onset melting takes place at the early stage of eutectic structure transformation. From Table 1, it can be found that at the same heating rate, $T_{on}$ decrease somewhat with increasing compression ratio. Under the same compression ratio, $T_{on}$ increases with increasing heating rate.

The peak melting temperatures $T_p$ under different conditions are shown in Table 2. From Table 2, it can be noticed that the peak melting temperature of the original material is 710.14 K when heating rate is 10K/min. As the compression ratio increased from 10% to 20%, the peak temperature decreased from 709.96 K to 709.37 K, i.e. at the same heating rate, $T_p$ decreases with increasing compression ratio. This is because with the increase of compression ratio, the coarse dendritic crystals were squashed, bent, and broken into fine particles, and the whole specimen was deformed, releasing heat. The larger the compression ratio is, the more the released heat is. Hence, the required heat of the deformed alloys during remelting decreased, i.e. $T_p$ decreased consequently. Under the same compression ratio, $T_p$ increases with increasing heating rate. This is due to that with the increase of heating rate, generated heat flow per unit time increases, $T_p$ becomes higher and higher consequently. In addition, the atoms in the alloys move faster, thermal inertia increases, energy fluctuates, hence $T_p$ increase.

![Figure 2](image.png)

**Figure 2.** DSC curves of Mg-6Al-0.9Gd alloys with different compression ratio

(a) 0%, (b) 10%, (c) 15%, and (d) 20%, heating rate is 10K/min.
Eutectic melting activation energy can be determined using Kissinger formula [15],

\[
d\left[\ln\left(\frac{\varphi}{T_p^2}\right)\right]/d(1/T_p) = -E/R
\]

where \(\varphi\) is heating rate (K/min), \(T_p\) is the peak melting temperature obtained from differential thermal analysis, \(E\) is eutectic melting activation energy (kJ/mol), and \(R\) is gas constant (8.314 kJ/mol). Figure 3 shows the relationship between \(\ln(\varphi/T_p^2)\) and \(1/T_p\), which is fairly linear. The eutectic melting activation energy \(E\) can be determined by calculating the slope of the fitted straight lines according to (1). Figure 4 shows the eutectic melting activation energy of the deformed Mg-6Al-0.9Gd alloys under different compression ratios.

**Table 1.** Ton of the Mg-6Al-0.9Gd alloys at different compression ratio, (K).

| Heating rate (K/min) | Compression ratio (%) | 0       | 10     | 15     | 20     |
|---------------------|-----------------------|---------|--------|--------|--------|
| 5                   | 707.33                | 706.90  | 706.52 | 705.83 |
| 10                  | 708.45                | 707.09  | 706.72 | 706.11 |
| 15                  | 707.88                | 707.24  | 706.91 | 706.28 |

**Table 2.** \(T_p\) of the Mg-6Al-0.9Gd alloys at different compression ratio, (K).

| Heating rate (K/min) | Compression ratio (%) | 0       | 10     | 15     | 20     |
|---------------------|-----------------------|---------|--------|--------|--------|
| 5                   | 709.51                | 709.37  | 709.21 | 708.99 |
| 10                  | 710.14                | 709.96  | 709.62 | 709.37 |
| 15                  | 710.92                | 710.77  | 710.37 | 710.05 |

**Figure 3.** The relationship between \(\ln(\varphi/T_p^2)\) and \(1/T_p\).
When compression ratio increases from 0 to 20%, the eutectic melting activation energy of the Mg-6Al-0.9Gd alloys decreases from 3236.91 kJ/mol to 2963.10 kJ/mol. This is due to that with the increase of compression ratio, the density of serious defects (e.g. grain boundary, dislocation, and vacancy) gradually increases, the disorder degree of crystal structure increases consequently, which causes the reduction of eutectic melting activation energy.

The distortion energy stored in the deformed sample can be regarded as the difference between the eutectic melting activation energy of the sample before and after deformation [16]. Table 3 shows the obtained distortion energy based on eutectic melting activation energy corresponding to different compression ratios. It can be found that with the increase of compression ratio, eutectic melting activation energy decreases from 3236.91 kJ/mol to 2962.99 kJ/mol, while the distortion energy accumulated to 273.92 kJ/mol after 20% deformation. In other word, partial eutectic melting activation energy transformed into distortion energy stored in the deformed alloys.

When compression ratio is 10%, the distortion energy stored in the alloys is very little, and the dendritic crystals have the tendency of fracture, but no obvious deformation has taken place. When compression ratio reaches 15%, the dendrite arms become long and thin due to plastic deformation, and the density of the generated defects increases, causing the distortion energy to increase. When compression ratio rises to 20%, the number of fractured dendrites increases, which is a process of stress relief, causing the distortion energy to decrease. However, the soft dendrite arms were further stretched and become longer and thinner, causing the distortion energy to increase consequently. The variation of distortion energy is consistent with the evolution of microstructure.

Table 3. The activation energy and distortion energy of the deformed Mg-6Al-0.9Gd alloys at different compression ratios.

| Compression ratio(%) | 0%   | 10%   | 15%   | 20%   |
|----------------------|------|-------|-------|-------|
| Activation energy(kJ/mol) | 3236.91 | 3089.80 | 3001.52 | 2962.99 |
| Distortion energy(kJ/mol) | 0 | 147.11 | 235.39 | 273.92 |

3.3 Effect of deformation on semi-solid microstructure

Figure 5 shows the microstructure of Mg-6Al-0.9Gd alloy at different compression ratios with isothermal holding temperature of 550 °C for 15 min. It is found that the microstructure of the compressed alloys has changed into equiaxed grains surrounded by liquid phase at grain boundary. Under compression ratio 10%, some dendrite arms were melted and the liquid phase precipitated at
grain boundaries, mainly segregated at triple junctions, as shown in Figure 5 (a). Then the grain boundaries were irregular and chaotic due to the incipient melting of eutectic phase there. Moreover, there exist some small entrapped liquid pool inside the solid particles. When the compression ratio increases to 15%, the liquid pool inside some small grains connected with the liquid phase at grain boundaries, forming network of liquid phase as shown in Figure 5 (b). When the compression ratio reaches 20%, the amount of liquid phase increased obviously and penetrated most grain boundaries as shown in Figure 5 (c), and it is beneficial for improving the liquidity of the alloy during semi-solid processing.

Figure 5. Optical micrographs of the deformed alloys at different compression ratios after isothermal heat treatment at 550 °C for 15 min, (a) 10%, (b) 15%, and (c) 20%.

Figure 6 shows the variations of liquid fraction and average grain size with compression ratio. With the compression ratio varying from 10 % to 20 %, the average grain diameter decreases from 55.08 μm to 49.11 μm, and the liquid fraction increases from 13.19 vol.% to 29.86 vol.% During the compression deformation, distortion energy was stored in the alloys, and the energy accumulated with increasing compression ratio. It is well known that the distortion energy stored in the alloys provide driving force for recrystallization, which can be verified by the occurrence of many strain-free equiaxed grains as shown in Figure 6. The initial recrystallized grain size during partial remelting depends mainly on the amount of stored energy, and decreases with increasing distortion energy. The smaller the grain size was, the more the grain boundary area was, thus more melting paths were provided for the penetration of liquid phase. When the remelting temperature rises to above solidus, the deformed alloys with more grain boundary have more solid-liquid interfacial energy, and thus have higher diffusion rate. Consequently, the formation rate of liquid phase in the compressed alloys was enhanced, and the liquid fraction in the alloys increased with increasing compression ratio under the same condition.
4. Conclusions
1) The distortion energy stored in the deformed Mg-6Al-0.9Gd alloys increased with increasing compression ratio. When compression ratio increased from 10% to 20%, the distortion energy increased from 147.11 kJ/mol to 273.92 kJ/mol.
2) With the increase of deformation, the defect density increased, and the distortion energy was stored in the deformed Mg-6Al-0.9Gd alloys in forms of dislocation multiplication.
3) The optimum compression ratio should be 20%, at which the average diameter of equiaxed grain reached the minimum, and the fraction of liquid phase raised to the maximum after a isothermal treatment at 550 °C for 15 min.

5. References
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