Thermal compression, processing maps and microstructural evolution of as-cast Mg-Gd-Y-Zn-Zr alloy with long period stacking ordered phase

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Keywords: Mg-Gd-Y-Zn-Zr alloy, constitution equation, processing map, microstructural evolution

Abstract
Thermal deformation of Mg-7.5Gd-2.5Y-1.5Zn-0.5Zr (wt%) as-cast alloy containing LPSO phase was been studied in a temperature range from 623 K to 723 K and a strain rate range from 0.001 to 1 s⁻¹. The microstructural evolution at the various strain rates was also analyzed. The results show that the flow stress decreases significantly with the increase of the deformation temperature. But the peak flow stresses of the alloy increase significantly at high deformation temperature, due to existing both the plentiful lamellar LPSO phase and the fine dynamic DRXed grains. Under the high strain rate conditions, the LPSO phase distorted and formed kink bands. The deformational activation energy (Q) of the alloy is about 234.6 kJ·mol⁻¹. This high Q value mainly owe to profuse lamellar LPSO phase hindering the movement of atoms. With the observation of established processing map of the alloy, some good thermal workabilities are observed in following zones: 723 K, 0.001 < ε < 1 s⁻¹; and the zone with middle temperature and moderate strain rate condition, 673 K, 0.01 < ε < 0.1 s⁻¹. The thermal deformation instability zones are mainly located in the region with high strain rate and lower temperature.

1. Introduction

Magnesium alloys have some good properties like high strength-to-weight ratio and great electromagnetic shielding effect [1–3], but also have some defects such as weak mechanic properties and low corrosion resistance. Adding some rare earth (RE) elements into magnesium alloy is usually used to bring excellent solid solution strengthening and outstanding precipitation strengthening. This enhances also significantly the alloy strength. Besides, a lamellar long-period stacking ordered (LPSO) phase with a unique structure was formed on the substrate with introduction of a certain amount of Zn into Mg-Gd/Y alloys [1, 4, 5]. This enhances the strength and improves the deformation plasticity of the alloy [5, 6].

The plastic deformation plays a crucial role in improving magnesium alloy’s mechanical properties. However, because of hexagonal close-packed (hcp) crystal structure with few slip systems, magnesium alloy presents weak deformation capacity at room temperature [7]. The numbers of slip system increases and twin assisted deformation appears with increase of deformation temperature. Constitutive equation is commonly used to calculate the flow stress of materials during the thermal deformation at high temperature. In this method, the flow stress is optimized by the hyperbolic sinusoidal function containing the power relation under low stress and the exponential relation under high stress, respectively [8, 9]. The difference of microstructure, especially phase, plays an important role in the calculation of flow stress. The degree of bonding between different atoms is a vital factor affecting the value of thermal activation energy Q [4, 6, 9, 10]. Many studies on constitutive equations and thermal processing map of Mg-Gd-Y(-Zn)-Zr series alloys have been made in the literature [2, 6, 7]. The values of activation energy (Q) in Mg-Gd-Y (-Zn)-Zr alloys are very distinct, like as-cast
Mg-9Gd-4Y-0.6Zr (wt%) alloy, $Q = 209 \text{ kJ mol}^{-1}$ \cite{11}, extruded Mg-8Gd-3Y-0.5Zr (wt%) alloy, $Q = 131 \text{ kJ mol}^{-1}$ \cite{12}, as-cast Mg-8.9Gd-5.11Y-3.10Zn-0.47Zr (wt%) alloy, $Q = 240.5 \text{ kJ mol}^{-1}$ \cite{13}. Microstructure and the ratio of dynamic recrystallization (DRX) grains in deformed magnesium alloy are the main factors for the difference of the value of $Q$. When the contents of rare earth (RE) elements under 10 wt%, the microstructure and the second phase are very distinct from high contents of RE elements 10 wt%. However, there is few report on the thermal compression deformation behavior of this magnesium alloys, especially for high content of LPSO phase.

The thermal processing map is of great significance for the determination of the molding process and parameters of the specific metal materials. The thermal processing map is based on the dynamic material model (DMM). It has been widely used in the study of thermal workability of materials under some conditions such as deformation temperature, strain force and strain rate \cite{10}. In particular, for these materials that are difficult to deform, it has proved to be a very useful tool in predicting energy dissipation in the process of material processing.

Magnesium alloy has a poor workability at room temperature. The plastic deformation of magnesium alloy tends to be stable with the increase of deformation temperature \cite{2}. Microstructure varies with the change of chemical composition \cite{13}. Therefore, the thermal deformation behavior of magnesium alloys varies significantly with the microstructure. Based on the previous studies, the higher rare earth and Zn content (RE/Zn) ratio is necessary to form a lamellar LPSO phase \cite{14}. In order to better study the effect of different rare earth content on the relative thermal workability of LPSO, we prepared Mg-7.5Gd-2.5Y-1.5Zn-0.5Zr (wt%) alloy. It contains profuse lamellar LPSO phase in the as-cast alloy. The as-cast 7.5Gd-2.5Y-1.5Zn-0.5Zr (wt%) alloy is first homogenized in this paper. Then the compression experiments were carried out at different temperatures and strain rates. The corresponding constitutive equations and thermal processing map were established by using the experimental data. At the same time, the microstructural evolution of the alloys during thermal deformation was analyzed.

### 2. Experimental

An Mg-Gd-Y-Zn-Zr alloy was firstly prepared by using semi-continuous casting. The master alloy Mg-30 wt% Y, Mg-30 wt% Gd, Mg-30 wt% Zr, and high-purity Zn and high-purity Mg were used for preparing the alloy. The composition (wt%) of the alloy used in the test is as follow: Gd, 7.5; Y, 2.5; Zn, 1.5; Zr, 0.5 and balance Mg. The as-cast ingot was homogenized at 520 °C for 12 h. Then the surface oxidation layer is removed by wire cutting and grinding. The thermal compression tests were carried out on Gleeble-3500 thermal-mechanical simulator. The test temperature and strain rate were in the range of: 673–723 K and 0.001 s$^{-1}$–1 s$^{-1}$. Ar gas is used for protection during the test. The compressed specimen is taken from the center part of the ingot, its size is ø8 mm × 12 mm. In order to reduce the friction of deformation, a pad graphite lubricating film is set between the sample and the head, with a heating rate of 2 K s$^{-1}$, and the sample is kept for 2 min at the corresponding test temperature. The true strain for all thermal compression specimens should be 0.55. After thermal compression, the sample is quenched in water to room temperature.

Olympus-OLS4000 laser scanning confocal optical microscope (OM) and FEI Tecnai G2 F30 transmission electron microscope (TEM) were used to observe and analyze microstructure of the alloy. The samples for microstructure before compression test were taken from the center of the ingot, and the deformed specimens were cut parallel to the compression axis. The foils for TEM observation were mechanically polished to approximately 50 μm in thickness, then punched into discs of 3 mm in diameter, and then punching a hole with a Gatan ion Mill.

### 3. Results and discussion

#### 3.1. Flow stress behavior

Figure 1 shows the true stress - true strain curves of the studied alloy Mg-7.5Gd-2.5Y-1.5Zn-0.5Zr (wt%) at the strain-rate of 0.001 s$^{-1}$, 0.01 s$^{-1}$, 0.1 s$^{-1}$ and 1 s$^{-1}$. The curves could be divided into three stages. Firstly, the flow stress went up sharply with the strain. This is because as the strain increases, the density of dislocation enlarge significantly, which causes work hardening. Besides, LPSO phase formed to enhance the strength of the alloy in the Mg matrix with increased temperature and strain rate. It is similar to substructure-strengthen owing to the fiber-like LPSO phase distributed in the grain. And then, the stress reaches the peak value, the strain increased and reached the critical value for dynamic recovery (DRV) which makes the strain continue to increase. Finally, the stress tends to stay in the stable stage. Dynamic recrystallization (DRX) with stress softening occurs gradually and tends to dynamic equilibrium with work hardening \cite{7}. At low strain rate, dynamic recrystallization increases gradually, and the stress decreases gradually at high strain rate. Dynamic recrystallization is restricted
at high strain rate conditions. Meanwhile, work hardening is strengthened sharply makes the stress slightly increase. As shown in figures 1(a) and (d). At high temperature, the non-basal slip system is activated, dislocation climbing and cross slip activity increase, and the flow stress decreases significantly with the increase of temperature at each strain rate. Because of the formation of higher dislocation density at large strain rates [10]. The stress is increased obviously.

Under the condition of low strain, such as 0.001 s⁻¹ and 0.01 s⁻¹, the compressive peak stress decreases sharply with the rise of deformation temperature. Similar phenomena occur in other Mg-Gd-Y alloys [7, 9]. But compared with other magnesium alloys with similar compositions [15], the change of rheological stress of magnesium alloy in this study with strain rate at various temperatures is little, as shown in table 1. Compared with other magnesium alloys with higher rare earth content with similar composition, the decrease of peak stress of Mg-7.5Gd-2.5Y-1.5Zn-0.5Zr alloy at different strain rate is even smaller. This is mainly due to the distribution of the lamellar LPSO phase which is similar to fine fiber on the matrix. The LPSO phase is very stable below the temperature of 773 K. This hinders the growth of DRX during the deformation process. And it increases the difficulty of dislocation slip and climbing and strengthens the matrix.

3.2. Building constitutive equations
The stress and strain data obtained from the compression test are used to determine the parameters of the constitutive equation of the material. The relationship between strain rate and deformation temperature can be

![Figure 1. True stress - strain curves of Mg-7.5Gd-2.5Y-1.5Zn-0.5Zr (wt%) alloy under various compression deformation conditions: (a) \( \varepsilon = 0.001 \) s⁻¹, (b) \( \varepsilon = 0.01 \) s⁻¹, (c) \( \varepsilon = 0.1 \) s⁻¹ and (d) \( \varepsilon = 1 \) s⁻¹.]

Table 1. The decline-ratios of the flow stress of magnesium alloys from 623 K to 723 K at different strain rates/%

| Alloys (wt%)             | 0.001 | 0.01 | 0.1  | 1    | References |
|--------------------------|-------|------|------|------|------------|
| Mg-7.5Gd-2.5Y-1.5Zn-0.5Zr | 56    | 53   | 48   | 41   | In this study |
| Mg-9.3Gd-3Y-0.35Zr       | 80    | 71   | 55   | 44   | [16]       |
| Mg-10Gd-4.8Y-2Zn-0.6Zr   | 75    | 65   | 61   | 50   | [15]       |
expressed by Arrhenius equation, as shown in equation (1):

\[
\dot{\varepsilon} = A \cdot [\sinh(\alpha \cdot \sigma)]^n \cdot \exp\left(\frac{-Q}{RT}\right)
\]  

(1)

where \(A (s^{-1})\), stress multiplier \(\alpha (MPa^{-1})\) and stress exponent \(n\) are the correlation coefficients of materials; \(\sigma\) is the flow stress (MPa); \(\dot{\varepsilon}\) is the strain rate (s\(^{-1}\)); \(R\) is the gas constant (8.314 \(\text{J mol}^{-1} \text{K}^{-1}\)); \(T\) is the temperature of plastic deformation (K); \(Q\) is the activation energy of deformation (kJ mol\(^{-1}\)) and generally represented by the differential form of equation (2):

\[
Q = R \left(\frac{\partial \dot{\varepsilon}}{\partial \ln [\sinh(\alpha \cdot \sigma)]}\right)_{T} \cdot \left(\frac{\partial \ln [\sinh(\alpha \cdot \sigma)]}{\partial (1/T)}\right)_{\dot{\varepsilon}}
\]

(2)

In the process of establishing the constitutive equation, \(Z\) function is introduced to amend the relevant parameters of the equation accurately. \(Z\) represents the Zener-Hollomon parameter incorporating the two control variables \(T\) and \(\dot{\varepsilon}\). For the case of the low-stress level \((\alpha \cdot \sigma < 0.8)\), \(Z\) is a power function of \(\sigma\), as showed in equation (3). For the case of the high-stress level \((\alpha \cdot \sigma > 1.2)\), \(Z\) is an exponential function of \(\sigma\), as showed in equation (4). Based on the semi-empirical formula of Sellars and Tegart [17], \(Z\) is also a hyperbolic sinusoidal function of \(\sigma\), as indicated in equation (5).

\[
Z = A_{1} \cdot \sigma^{n_{1}}
\]

(3)

\[
Z = A_{2} \cdot \exp(\beta \cdot \sigma)
\]

(4)

\[
Z = \dot{\varepsilon} \cdot \exp\left(\frac{Q}{RT}\right) = A \cdot [\sinh(\alpha \cdot \sigma)]^n
\]

(5)

Where \(\beta\) represents a material constant depending of \(\sigma\) and \(T\), supposing \(\alpha = \beta / n \approx \beta / n_{1}\).

In order to simplify the equation, the natural logarithms are taken simultaneously on both sides of the equations (3)–(5), as showed in equations (6)–(8):

\[
\ln \dot{\varepsilon} = \ln A_{1} - \frac{Q}{RT} + n_{1} \ln \sigma
\]

(6)

\[
\ln \dot{\varepsilon} = \ln A_{2} - \frac{Q}{RT} + T \ln \sigma
\]

(7)

\[
\ln \dot{\varepsilon} = \ln A_{3} - \frac{Q}{RT} + n \ln[\sinh(\alpha \cdot \sigma)]
\]

(8)

Figure 2 shows various curves of the peak stress - strain rate under different conditions. From equation (6), it can be known that \(n_{1}\) can be expressed by the slope of the (\(\ln \sigma\)-\(\ln \dot{\varepsilon}\)) relation, and its value is 9.14777, as showed in figure 2(a). Based on equation (7), \(\beta\) can be obtained with the slope of (\(\sigma\)-\(\ln \dot{\varepsilon}\)) curve, which is 0.063807. Therefore, the value of parameter \(\alpha\) is 0.006975. The first and second terms of the activation energy \(Q\) (equation (2)) represent the slope of [\(\ln \dot{\varepsilon} - \ln(\sinh(\alpha \cdot \sigma))]\) and [\(\ln(\sinh(\alpha \cdot \sigma))\)] respectively, as showed in figures 2(c) and (d).

The calculated value of \(Q\) is 234.553095 kJ mol\(^{-1}\). Lastly, the value of \(A\) was determined to be 1.26608 \(\times 10^{16}\) by the slope of equation (5), as showed in figure 3. By substituting \(Q\) in equation (8), the values of \(n\) and \(A\) can be obtained. Therefore, the semi-empirical formula of flow stress for \(\dot{\varepsilon}\), \(\sigma\) and \(T\) can be expressed as follows:

\[
\dot{\varepsilon} = 1.26608 \times 10^{16} \cdot \sinh(0.006975 \cdot \alpha)^{2.48118} \cdot \exp\left(\frac{-234.553095}{RT}\right)
\]

(9)

The self-diffusion activation energy of pure Mg is about between 130 and 140 kJ mol\(^{-1}\) [8]. Mg-7.5Gd-2.5Y-1.5Zn-0.5Zr (wt%) alloy is relatively higher than 100 kJ mol\(^{-1}\). Due to the low diffusion coefficients of Gd and Y elements in Mg matrix [18], and the fact of containing a large scale of lamellar LPSO phase, it impels the increase of values of \(Q\) in this alloys.

3.3. Establishing processing map

When the microstructure changes such as phase transformation, the energy dissipation will occur for the alloy. The energy dissipation diagram can be established by this method, such as equation (10):

\[
\eta = \frac{2m}{m + 1}
\]

(10)

Where \(m\) represents the strain rate sensitivity of flow stress, \(m = (\partial \ln \sigma) / (\partial \ln \dot{\varepsilon})\). The relationship between \(\sigma\) and \(\dot{\varepsilon}\) can be described by third order polynomial fitting method. The expression as shown below:
Where the constants $a$, $b$, $c$ and $d$ are the material parameters depending on temperature.

Considering the deformation limitation of the unavoidable thermal dynamics under the condition of large plastic deformation, a flow unstable region is defined. According to the range defined by $\xi(\dot{\varepsilon})$, the instability of...
the material during the shaping deformation can be obtained by calculation. A criterion for the onset of flow instability defined by equation (12):

\[
\xi(\dot{\varepsilon}) = \frac{\partial \ln \left( \frac{m}{m+1} \right)}{\partial \ln \dot{\varepsilon}} + m \leq 0
\]  

The thermal processing map is related to both the energy dissipation diagram and the rheological instability diagram. It can be obtained based on equation (10) to equation (12) in Mg-7.5Gd-2.5Y-1.5Zn-0.5Zr (wt%) alloy, as shown in figure 4. The instability zone is mainly located on the region of high strain rate and low temperature. The numerical value of \(\eta\) is the energy dissipation caused by microstructural evolution during thermal deformation. In the studied alloy in this paper, the energy dissipation mainly occurs in the low strain and high strain regions, the maximum energy dissipation is 0.49, as displayed in figures 4(c), (d). It is not doubt that larger energy dissipation corresponds to DRX, grains nucleation and growth \([6]\), as shown in figure 5; The lower energy dissipation occupies a large area of the processing map, and the energy dissipation is below 0.2 (as shown in figure 5(b)). A large number of lamellar LPSO phases were formed at a lower deformation temperature of 623 K. Mg-7.5Gd-2.5Y-1.5Zn-0.5Zr alloy exhibits excellent machinability in the following temperature strain region: high temperature area, 723 K, \(0.001 < \varepsilon < 1 \text{ s}^{-1}\); and moderate strain rate and moderate temperature area, 673 K, \(0.01 < \varepsilon < 0.1 \text{ s}^{-1}\).

### 3.4. Microstructural evolution and discussion

Figure 5 shows the metallographic microstructure of the alloy before and after thermal compression test. It’s noteworthy that the microstructure of the elevated temperature samples is relatively similar, especially in the zone of the lower temperature and higher strain rate. Therefore, the microstructure evolution mainly focuses on lower-temperature and higher strain rate conditions, and high temperature conditions. After thermal compression, the matrix grains grow up significantly. In the low temperature and low strain rate zones, and high temperature and high strain rate zones, the grains are obviously deformed. But in the moderate temperature zones, deformation of the grains with a large amount of LPSO phase is not obvious. When the temperature is
low, the lamellar LPSO phase within the grain does not grow completely, and with the increase of temperature, for example, to 673 K, the LPSO phase further grows and helps to block the grain deformation during the thermal compression process, and to some extent hinders the nucleation and growth of the dynamic recrystallized grain. At the same time, the fine dynamic recrystallization grains formed in the process also changed the LPSO phase, which strengthened the alloy matrix. Compared with the formation of the dynamic recrystallized grain, higher temperature and low strain rate is more favorable for the formation of LPSO phase. Figure 6 shows the microstructure observed with TEM after thermal compression of 723 K. It can be seen from the specimen after thermal deformation that the dynamic recrystallized grains formed during thermoplastic deformation are also changed, that is, the formation of LPSO phase with 14 H structure, especially at high strain rate, as shown in figure 6. However, due to the short stay-time in high temperature, and the slow diffusion of rare earth elements and Zn atoms in the matrix, the growth of LPSO phase is not obvious. Under the condition of high strain rate, the LPSO assisting the grain deformation also occurs kink-band deformation at the same time, and then forms the torsional band. This unique deformation mode has remarkable strengthening effect on the matrix [19]. The twisting bands are shown in figures 5(e) and 6(d) (the blue lines in figure 6(d)).

Lamellar LPSO phase remarkably increases at high deformation temperature and high strain rate. Meanwhile, the growth of DRXed grains is restrained at high deformation temperature. It indicates that LPSO phase formed in the deformation process hinders the reproduction of dislocation and formation of DRXed grains. In addition, the peak stresses obtain at high temperatures and lower strain rates. This behavior reveals that DRX takes place more easily at higher temperatures and smaller strain rates conditions. Nevertheless, the growth of masses of DRXed grains is still restricted by lamellar LPSO phase.

When the deformation temperature is higher than 673 K, a mass of LPSO phase forms completely and deforms with the grains in the deformation process, as shown in figure 5. LPSO phase is a stable phase when the temperature is lower 500 °C. Furthermore, due to the compression force, elongated LPSO phase in grains occurs kink deformation. At high strain rates, kink deformation occurs rapidly and kink bands forms to improve the plasticity of the alloy in hot process. LPSO phase provides a deformation-resistance for atoms moving hardly. It restrains the formation of DRX grains and the movement of dislocation under deformation process. Moreover, lamellar LPSO phase also partly offsets the distortion of grains under lower temperature deformation, as shown in figures 5(b)–(d). Therefore, the flow stress of the alloy at elevated temperature is improved remarkably, and the value of the activation energy of the alloy is also high [20].

4. Conclusions

In this paper, the thermal deformation behavior of as-cast Mg-7.5Gd-2.5Y-1.5Zn-0.5Zr (wt%) alloy containing a large number of lamellar LPSO phases at high temperature has been studied in a temperature range from 623 K to 723 K and a strain rate range from 0.001 to 1 s\(^{-1}\). The following conclusions can be drawn:
The flow stress increases significantly with the decrease of deformation temperature and the increase of strain rate; a large number of lamellar LPSO phases are formed in the matrix and dynamic recrystallized grains during thermal deformation, especially at low strain rate.

(2) The activation energy Q of the alloy is obtained to be 234.6 kJ·mol\(^{-1}\), it is higher than most magnesium alloys. The value of Q at 623 K reaches 279.20 kJ·mol\(^{-1}\), and 187 kJ·mol\(^{-1}\) at 723 K. As a result of lamellar LPSO phase provides a deformation-resistance for atoms moving hardly. And the phase partly offsets the distortion of grains under lower temperature deformation. Therefore, the activation energy of the alloy at high temperature is relatively high.

(3) The instable thermal workability region of Mg-7.5Gd-2.5Y-1.5Zn-0.5Zr alloy is located in the region of low temperature and high strain rate. The alloy shows excellent thermal workability in the following regions: 723 K, 0.001 < \(\varepsilon\) < 1 s\(^{-1}\) and 673 K, 0.01 < \(\varepsilon\) < 0.1 s\(^{-1}\).

(4) A mass of lamellar LPSO phase formed in the hot deformation process. And the phase is also precipitated in DRXed grains, especially at lower strain rate deformation conditions. Kink bands with lamellar LPSO phase deformation were observed at high strain rate to enhance the high-temperature strength of the alloy.

**Acknowledgments**

This work is supported by the Youth Science Fund Project of National Natural Science Fund of China (51401070). We also gratefully acknowledge Dr Li You from University of Science and Technology Beijing for the helpful discussions.
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