Hot deformation behavior and processing map of Mg-12Gd-1MM-0.6Zr magnesium alloy

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Abstract. The hot deformation behavior of Mg-12Gd-1MM-0.6Zr (wt.%) magnesium alloy was tested by Gleeble-1500D hot simulator with reduction of 60% and strain rates from 0.001 to 1 s$^{-1}$ at the temperature range from 753 to 793 K. The results show that the flow stress is influenced by both, temperatures and strain rates. At constant temperatures, flow stress is increased with strain rate, while at constant strain rates, it decreased with temperature. The constitutive equation of Mg-12Gd-1MM-0.6Zr alloy during hot compression was constructed by the linear regression analysis. Average activation energy and stress exponent were 227.94 kJ/mol and 2.87, respectively. The processing map was plotted and analyzed via the dynamic material model. The most proper ranges for hot deformation temperature and strain rate were found to be 763 to 783 K and 0.01 to 0.1 s$^{-1}$, respectively.

1. Introduction
Mg alloys are widely used in aerospace and automobile industry for their several advantages, including low density, high specific strength, high specific stiffness and so on [1]. The addition of rare-earth elements can result in good high temperature properties and creep resistance [2]. At present, researchers developed many promising rare-earth Mg alloys, such as WE43 [3]. But few Mg alloys contain cheap misch-metal (MM) elements, which can form thermo-stable phases with Mg, and the comprehensive properties of Mg alloy can be improved because of the interaction of MM elements. It has been reported [4] that Mg alloy can obtain better comprehensive properties than cast Mg alloy after deformation. Therefore, many researchers studied the thermal deformation behavior of Mg alloys. Xiao et al. [5] studied the hot deformation behavior and processing map of Mg-10Gd-4.8Y-0.6Zr (wt.%) Mg alloy, and concluded that the optimal parameters of the alloy were temperatures from 723 to 773K and strain rates from 0.1 to 1s$^{-1}$. By studying the thermal deformation behavior of Mg-7Gd-2.5Nd-0.5Zr (wt.%) Mg alloy, Xin et al. [6] calculated the thermal activation energy of the alloy was 235.958 kJ/mol, and the appropriate hot deformation temperatures of the alloy were 723 773 k. At present, there are few studies on the thermal deformation behavior and processing map of Mg-Gd-MM-Zr Mg alloy. Based on this background, the present work is focused on the thermal deformation behavior of Mg-12Gd-1MM-0.6Zr (wt.%) Mg alloy, which composition was optimized in the previous research. The change rule of the
flow stress during deformation at elevated temperatures was analyzed. Furthermore, material constant was calculated, the flow stress constitutive model of the alloy was established and the processing maps, according to DMM (dynamic materials model) model, which provided a basis for the formulation and optimization of the thermal processing process of this alloy, were achieved.

2. Experimental procedure
The Mg-12Gd-1MM-0.6Zr alloy was gravity-cast in a medium frequency electromagnetic induction melting furnace under the protection of mixed tetrachloromethane and argon gas. Pure Mg was melted in an iron crucible, and the dried pure metals Gd and MM ((Ce48%, La30%, Nd19%, Pr1%, wt.%) were added at 750 °C to the melt. Upon heating to 850 °C, Mg-30% Zr (wt.%) master alloy was added. After holding the melt on this temperature for 10 minutes, mechanical stirring was initiated. Melt was cooled down to 750 °C and quenched in water below the crucible. For a quick solidification. The final dimensions of the cast ingot are 110 mm × 300 mm after removal of ingot scale. The alloy composition was tested by ICP-AES, which results are shown in table 1.

| Table 1. Chemical composition of Mg-12Gd-1MM-0.6Zr alloy (wt. %) measured via ICP-AES. |
|----|----|----|----|
| Experimental alloy | Gd | Ce* | Zr | Mg |
| Mg-12Gd-1MM-0.6Zr | 12.34 | 1.08 | 0.40 | Bal |

*The content of cerium element was used to represent the content of MM in the alloy.

After casting, a homogenization treatment at 793K for 48 h was conducted, finished by water quenching. Ingot was cut into a small cylinders of 10 mm × 15 mm. The hot compression experiment of constant temperature and strain rate was carried out on gleeble-1500d thermal simulation machine. In the thermal simulation experiment, the heating rate was 5 K/s and the holding time was 2 min. During the deformation process, strain sensors and temperature sensors were used to measure axial strain and temperature changes of the samples. The test deformation temperature was set as 753, 773, and 793 K the deformation rate was 0.001, 0.0, 0.1, and 1s⁻¹, the true strain was 0.916, and one trial was conducted per condition without lubrication. After thermal deformation, the experimental material was quenched into water immediately. Then, the microstructure of the initial condition (as-cast and homogenized) and deformed samples was analyzed by optical microscopy and XRD. Finally, flow curves were drawn after simple smoothing.

3. Results and analysis

3.1. Initial microstructure
Figure 1 depicts the microstructure and XRD patterns of Mg-12Gd-1MM-0.6Zr alloy. The as-cast microstructure is composed of α–Mg phase and eutectic structure Mg₅Gd. Mg₁₂MM phase has high solidification temperature. Thus, Mg₁₂MM phase was formed first when the ingot solidified, and then wrapped in Mg₅Gd phase which was formed later. After homogenization annealing at 793K for 48h, Mg₅Gd phase was completely dissolved, and the remaining Mg₁₂MM phase was not completely dissolved.
Figure 1. The microstructure and XRD pattern of Mg-12Gd-1MM-0.6Zr alloy.

3.2. Flow curve

Figure 2. Flow curves of Mg-12Gd-1MM-0.6Zr alloy under different deformation conditions (a) 753K (b) 773K and (c) 793K.
Flow curves of Mg-12Gd-1MM-0.6Zr alloy under different conditions obtained from the thermal compression test are shown in figure 2. It can be found that at the same strain rate, the flow stress decreases with the increase of deformation temperature. At the same deformation temperature, the flow stress increases with the increase of strain rate, indicating that the alloy has obvious positive strain rate sensitivity during thermal compression. When the deformation temperature increases, the thermal activation is enhanced, and the atomic kinetic energy increases accordingly, which results in a decrease of the critical shear stress of dislocation slip. In addition, the dynamic softening increases with temperature. All those result in the decrease of the flow stress of the alloy. When the strain rate increases, dislocation density in unit time increases, dislocation climbing is blocked, and the critical shear stress of the alloy increases, leading to the increase of flow stress. It can be seen that the deformation temperature and strain rate have a great influence on the thermal deformation behavior of Mg-12Gd-1MM-0.6Zr alloy.

It can be seen in figure 1 that the flow curve of Mg-12Gd-1MM-0.6Zr alloy has obvious steady-state rheological characteristics, that is, the flow stress increases with the increase of strain and gradually descend until smooth when it reaches a certain peak. At the initial stage of deformation, the grain boundary and the second relative dislocation in the alloy act as a hindrance which leads to the continuous increase of the dislocation density and forming work hardening. While the work hardening rate is greater than the softening rate caused by dynamic recovery and dynamic recrystallization, the deformation resistance increases rapidly with the increase of true strain. As the stress continues to grow, the dislocation density in the alloy reaches a certain degree, the recrystallization ratio increases, the softening effect of recovery and recrystallization is greater than the work hardening, and the flow stress decreases with the increase of true strain. When the work-hardening rate and dynamic softening rate reach a balance, the flow stress curve enters the steady rheological stage.

3.3 Constitutive equation
The constitutive relation is the relationship between the flow stress of the material and parameters of hot working process. The flow deformation behavior of metal at high temperature can be expressed by the Arrhenius equation [7].

In the case of low stress, the relationship between strain rate and flow stress can be described as follows

\[ \dot{\varepsilon} = A_1 \sigma^{n_1} \]  

Under higher stress conditions, the relationship can be described as follows:

\[ \dot{\varepsilon} = A_2 \exp(\beta \sigma) \]  

In all stress states, it can be described as:

\[ \dot{\varepsilon} = A \left[ \sinh(\alpha \sigma) \right]^n \]  

Among them, A1, A2, A, n1, n, \( \alpha \) and \( \beta \) all are constants independent of deformation temperature. n1, \( \alpha \) and \( \beta \) satisfy the relational expression \( \alpha = \beta / n_1 \). R represents the gas constant, and its value is 8.314J/(K·mol). T is the absolute temperature (K). \( \dot{\varepsilon} \) is the strain rate. \( \sigma \) can represent either the flow stress corresponding to the strain at any time or the maximum flow stress.

The deformation of metal at high temperature is a thermal activation process. Sellars and Tegart [8] proposed a hyperbolic sine function including thermal activation energy Q and deformation temperature T, which modified the Arrhenius relation to describe such thermal activation steady-state deformation behavior. Therefore, formula (3) can be reduced to:

\[ \dot{\varepsilon} = A \left[ \sinh(\alpha \sigma) \right]^n \exp(-Q/RT) \]
According to the results of the thermal compression test of Mg alloy, the coefficients in the constitutive equation are obtained by linear regression. The logarithm of equations (1) and (2) can be obtained as follows:

\[
\begin{align*}
  n_1 & = \frac{\partial \ln \dot{\varepsilon}}{\partial \ln \delta} \\
  \beta & = \frac{\partial \ln \varepsilon}{\partial \delta}
\end{align*}
\]  

(5) \hspace{1.5cm} (6)

Figure 3 shows the relationship between strain rate, deformation temperature and maximum flow stress. According to the experimental results, the logarithms of equations (1) and (2) are taken to draw the relationship between \( \ln \sigma - \ln \dot{\varepsilon} \) (figure 3a) and \( \sigma - \ln \dot{\varepsilon} \) (figure 3b), while \( n_1 \) and \( \beta \) values are obtained (\( \alpha = \beta / n_1 \) is used to calculate the value of \( \alpha \)). The relationships between \( \ln \dot{\varepsilon} - \ln(\sinh(\alpha \sigma)) \) (figure 3c) and \( 1000/T - \ln(\sinh(\alpha \sigma)) \) (figure 3d) are obtained, according to formula (4) after the \( \alpha \) value is substituted into formula (4). The obtained coefficients are listed in table 2.

| Coefficient | \( n_1 \) | \( \beta \) | \( \alpha \) | \( n \) | \( Q(J/mol) \) | \( A \) |
|-------------|--------|--------|--------|--------|----------|--------|
| Value       | 3.960  | 0.116  | 0.029  | 2.871  | 227944.845 | 6.01 \times 10^{18} |

Therefore, the flow stress constitutive model of Mg-12Gd-1MM-0.6Zr alloy can be described as:

\[
\dot{\varepsilon} = 6.01 \times 10^{18} [\sinh(0.029\sigma)]^{2.87} \exp(-227944.845/RT)
\] 

(7)

Figure 3. Relationship between peak stress, strain rate, and deformation temperature.
Figure 4. Comparison of calculated and measured peak stresses.

As shown in figure 4, the peak stress calculated by equation (7) is compared to the measured peak stress tested under the corresponding conditions. The average relative error between the calculated results and the measured values is 8.7 %, which meets the requirements of engineering calculation. Therefore, the constitutive model can well predict the stress of materials when deformation at strain rates from 0.001 to 1 s⁻¹ at the temperature range from 753 to 793 K.

3.4. Processing map

Prasad et al. [9] proposed the dynamic material model (DMM) based on the large plastic deformation continuum mechanics, physical system model and irreversible thermodynamics. The model can reveal the process of large plastic deformation and microstructure of materials and illustrate the dissipative behavior of external energy through material plastic deformation. The material in thermal deformation can be considered as a nonlinear energy dissipation body. Its power dissipation and dissipation characteristics are based on the rheological dynamic constitutive equation of the material. Under a certain deformation temperature (T) and strain (\(\dot{\varepsilon}\)), the dynamic constitutive equation has the following form:

\[
\sigma = K \dot{\varepsilon}^m \tag{8}
\]

where \(\sigma\) is flow stress, \(\dot{\varepsilon}\) is strain rate, \(K\) is material strain constant and \(m\) is sensitive factor of strain rate. The energy \(P\) obtained by the material in the processing process in unit time and in unit volume can be divided into two parts: the dissipation amount \(G\) and the dissipation coordination \(J\), whose mathematical expression is:

\[
P = \sigma \dot{\varepsilon} = G + J = \int_0^{\dot{\varepsilon}} \sigma d\dot{\varepsilon} + \int_0^\sigma \dot{\varepsilon} d\sigma \tag{9}
\]

Among them, \(G\) represents the energy consumed by plastic deformation, and \(J\) represents the energy required by the evolution of microstructure. According to equations (8) and (9), the distribution of \(P\) between \(G\) and \(J\) is determined by the strain rate sensitivity index \(m\), as shown in equation (10).

\[
\left[\frac{\partial}{\partial \varepsilon} \right]_{\dot{\varepsilon}, T} \Rightarrow \left[\frac{\dot{\varepsilon}}{\dot{\varepsilon}} \frac{\partial \sigma}{\partial \varepsilon} \right]_{\dot{\varepsilon}, T} = \frac{\partial (\dot{\varepsilon} \sigma)}{\partial (\dot{\varepsilon} \varepsilon)} \Rightarrow m = m \tag{10}
\]

When the material is in an ideal linear dissipation state \((m=1)\), \(J\) has the maximum value \(J_{max}=P/2\). The power dissipation factor \(\eta\) is a dimensionless parameter that describes the ratio of the energy consumed by the microstructure change to the total linear energy consumed during the thermal
deformation process, its mathematical expression is as follows:

$$\eta = \frac{j}{J_{\text{max}}}$$  \hspace{1cm} (11)

According to equations (8), (9) and (11):

$$\eta = \frac{2m}{1+m}$$  \hspace{1cm} (12)

It is generally believed that the high $\eta$ value region in the thermal processing map corresponds to the optimum processing performance region. Based on the principle of irreversible thermodynamics of large plastic deformation, Prasad et al. [10] derived the criterion for the flow instability of materials:

$$\xi(\dot{\varepsilon}) = \frac{\partial \lg \left( \frac{m}{\dot{\varepsilon}} \right)}{\partial \lg \dot{\varepsilon}} + m < 0,$$  \hspace{1cm} (13)

where $\xi(\dot{\varepsilon})$ is a function of deformation temperature and strain rate. The area marked as negative on the power dissipation diagram becomes the instability diagram. The rheological instability diagram drawn by the rheological instability criterion is superimposed on the drawn power dissipation diagram, and the thermal processing map, which can directly display the processing safety zone, and the rheological instability zone is obtained.

In order to ensure the accuracy of the strain rate sensitive factor $m$, cubic spline function to fitting the relationship between $\lg \sigma$ and $\lg \dot{\varepsilon}$ was used, and the strain rate sensitivity factor $m$ is calculated according to equation (12). Figure 5 is a thermal processing map of the Mg-12Gd-1MM-0.6Zr alloy at an equivalent logarithmic strain of 0.7. Contour represents the value of power dissipation factor $\eta$, and the grey portion (Domain 1) indicates the instability domains. In the safe region on the processing map, the greater the power dissipation efficiency $\eta$, the better the material processing performance. It can be concluded from figure 5 that at constant strain rate and increasing deformation temperature, the $\eta$ value first increases and then decreases. When deformation temperature is constant, the strain rate decreases continuously, and the $\eta$ value increases continuously which indicate the better processing property of the alloy. When the strain rate is more than 0.1 s$^{-1}$, instability region appeared on the flow map. The red portion (Domain 2) in figure 5 shows the optimum processing area, so the optimum parameters of Mg-12Gd-1MM-0.6Zr alloy for deformation are temperatures of 763-783K, and the strain rates of 0.01-0.1s$^{-1}$.

A suitable processing area and an instability area in figure 5 were selected to observe the microstructure, as shown in figure 6. Dynamic recrystallization occurs in Mg-12Gd-1MM-0.6Zr Mg alloy when deformation at 773 K and 0.1s$^{-1}$ (belongs to Domain 2), and the recrystallized grain size is
about 14μm; when deformation at 793k and 1s⁻¹ (belongs to Domain 1), the Mg₁₂MM phase softened, which caused the material instability. These features indicate that the processing map is accurate.

![Microstructure of deformed Mg-12Gd-1MM-0.6Zr Mg alloy](image)

**Figure 6.** Microstructure of deformed Mg-12Gd-1MM-0.6Zr Mg alloy.

### 4. Conclusions

In this study, the hot deformation behavior, constitutive equation, and processing map of Mg-12Gd-1MM-0.6Zr (wt.%) Mg alloy via hot compression tests with strain rates from 0.001 to 1 s⁻¹ at the temperature range of 753 to 793 K has been studied. The main conclusions can be summarized as follows:

1. When the deformation temperature is constant, the flow stress of Mg-12Gd-1MM-0.6Zr alloy decreases with the decrease of strain rate. When the strain rate is constant, the flow stress increases with the decrease of temperature, indicating that the Mg-12Gd-1MM-0.6Zr alloy is temperature- and strain-rate-sensitive material.

2. Under the experimental conditions of this paper, the average thermal activation energy of the alloy is 227.94kJ/mol, and the stress index n is 2.87. The constitutive equation of thermal compression deformation for Mg-12Gd-1MM-0.6Zr alloy can be expressed as: \( \dot{\varepsilon} = 6.01 \times 10^{18} \sinh(0.029\sigma)^{2.87} \exp(-227944.845/RT) \).

3. According to the analysis of the thermal processing map of the alloy, when the deformation temperature is constant, as the strain rate decreases, the power dissipation factor \( \eta \) increases continuously, and the material processing range increases. When the strain rate is constant, the value of \( \eta \) first increases and then decreases. The optimum parameters for hot processing of Mg-12Gd-1MM-0.6Zr alloy were temperatures of 763-783K and strain rates of 0.01-0.1 s⁻¹.

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