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## Study on the effect of solution treatment on hot deformation behavior and workability of Mg-7Gd-5Y-0.6Zn-0.8Zr magnesium alloy



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## **ABSTRACT**

The effect of solution treatment on hot deformation behavior and workability of Mg-7Gd-5Y-0.6Zn-0.8Zr alloy has been studied by isothermal compression test in the temperature range from 350 °C to 500 °C and the strain rate range from  $0.001s^{-1}$  to  $1s^{-1}$ . The precipitation of LPSO phases inside the deformation grains of solution treated alloy increased when the strain rate reduced ( $\leq 0.01$ s<sup>-1</sup> in particular), and decreased when the deformation temperature rose beyond 475  $\degree$ C. The precipitation of LPSO phases and existence of stacking faults hindered the growth of dynamically recrystallized grains and caused insufficient DRX. After solution treatment, the deformation activation energy increased by 34.65KJ/mol compared to the as-cast alloy due to increased solute amount of REs in the Mg matrix. Besides, solution treatment narrowed the flow instability region of Mg-7Gd-5Y-0.6Zn-0.8Zr alloy at low temperature and high strain rate due to more homogenous deformation, but weakened the hot working ability at high temperature due to higher sensitivity of deformation microstructure to temperature. The processing maps of as-cast and solution treated alloy exhibited two similar dynamic recrystallization (DRX) regions, i.e. Region I (390–440 °C, 0.001–0.01s<sup>-1</sup>) and Region II (440–465 °C, 0.01–0.1s<sup>-1</sup>). Besides, the as-cast alloy possessed a wider workability region (Region III:  $465-500$  °C and 0.03-1s<sup>-1</sup>) than the solution treated alloy (Region III: 465–490  $^{\circ}$ C and 0.3-1s<sup>-1</sup>) at high temperature.

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## 1. Introduction

Among the lightest engineering metallic materials, magnesium and its alloys show a great potential for lightweight application in the fields of automobile, aerospace and electronics industries  $[1-3]$  $[1-3]$  $[1-3]$ , whereas the low strength and ductility limit their processing and wide utilization. In recent years, many researches have been conducted to improve the strength, ductility and corrosion resistance of Mg alloys  $[4-6]$  $[4-6]$  $[4-6]$ . In particular, the addition of RE elements can improve the mechanical properties of Mg alloys effectively, so RE-containing magnesium alloys, especially Mg-Gd-Y-Zr alloys, are attracting more and more attention in recent years [\[7\].](#page--1-0) Compared with the available commercial magnesium alloys, the Mg-Gd-Y-Zr alloys possess higher specific strength and creep resistance  $[8,9]$ . Besides, the introduction of Zn element into the Mg-RE-Zr alloys can enhance the precipitation of long period stacking ordered (LPSO) phases, contributing to the refinement of Mg matrix grains during hot deformation. Moreover, the LPSO phases usually act as hardening phases in the Mg-RE alloys, coordinating with the short-fiber reinforcement mechanism when they reach critical length [\[10,11\].](#page--1-0) Chen et al. [\[12\]](#page--1-0) pointed the existence of LPSO phase could inhibit the formation of micro-cracking and improve the ductility due to the highly coherent interface. However, as one of the HCP metals, plastic deformation proceeds at room temperature almost entirely by the basal slip with the lowest critical resolved shear stress, exhibiting heterogeneous deformation in magnesium al-loys [\[13,14\]](#page--1-0). Hence, it is essential for magnesium alloys to be processed at high temperatures, at which non-basal slip is activated to improve the workability. Moreover, the same material with different initial states may possess different hot processing windows. Prasad and Rao [\[15\]](#page--1-0) revealed the anisotropy of hot workability of as-rolled AZ31magnesium sheet in various directions. Similarly, the same material with different microstructural features may also show different workability. Kim et al.



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 $[16-18]$  $[16-18]$  $[16-18]$  found the Mg-Zn-Y alloy with different phase constitutions or alloying ways (Ca or CaO) presented different working windows. The Mg-RE-Zn alloys often show different microstructures after heat treatment, so it is necessary to study the effect of heat treatment on deformation behavior and workability of Mg-RE-Zn alloys.

The aim of this present study is to investigate the effect of solution treatment on hot deformation behavior of Mg-7Gd-5Y-0.6Zn-0.8Zr alloy. Besides, in order to obtain the hot work window of Mg-7Gd-5Y-0.6Zn-0.8Zr alloy, both the processing maps of Mg-7Gd-5Y-0.6Zn-0.8Zr alloy before and after solution treatment were established and discussed.

#### 2. Experimental procedures

The Mg-7Gd-5Y-0.6Zn-0.8Zr alloy was produced from highpurity Mg (99.99%), pure Gd, Y, Zn and Zr in an electric resistance furnace under a mixed protective atmosphere of  $CO<sub>2</sub>$  and  $SF<sub>6</sub>$  with the ratio of 100:1. The melting temperature was kept at 750  $\degree$ C and a large alloy ingot with 100 mm in diameter and 1000 mm in length was prepared by semi-continuous casting with the casting speed of 400 mm/min, followed by annealing treatment at 320  $\degree$ C for 2 h and naturally cooled in air  $\sim$  20 °C. The chemical composition of the Mg-RE-Zn-Zr alloy was determined by XRF analysis, conducted on a PW4400 X-ray spectrometer and the result was automatically calculated by the  $IQ + software$ . The solution treatment was performed at 500 $\degree$ C for 10 h, followed by cooling down in the air about 20 $\degree$ C. The cylindrical specimens for isothermal compression were spark machined from the as-cast and solution treated ingot with 8 mm in diameter and 12 mm in height.

The isothermal compression test was conducted in the temperature range from 350 °C to 500 °C with 25 °C intervals and the strain rate range from  $0.001s^{-1}$  to  $1s^{-1}$  on a Gleeble-1500D thermal simulator. The specimens were heated to deformation temperature at a heating rate of 10  $\degree$ C/s and held for 2min before hot compression. Subsequently, the specimens were compressed to a true strain of 0.7 in low vacuum atmosphere of  $1 \times 10^{-3}$  Torr and then quenched into water of about 20 $\degree$ C. In order to reduce the friction, graphite paper was used as lubricant between the crosshead and specimen during hot compression. After hot compression, the specimens were sectioned parallel to the compression axis for microstructural observation using an Olympus-PMG3 optical microscope (OM). The electron backscattered diffraction (EBSD) measurement was carried out on a Quanta 200FEG scanning electron microscope with the step size of 0.8  $\mu$ m, and the data with the confidence (CI) value higher than 0.1 were analyzed using the software of TSL-OIM. The TEM analysis was conducted on a Talos F200x transmission electron microscope operated at 200 KV. The phase analysis was carried out by X-ray diffraction (XRD, Cu Ka radiation) with a scanning speed  $10^{\circ}/\text{min}$  and a step size of 0.026 $^{\circ}$ . The XRD data were collected in the  $2\theta$  diffraction angle range of  $20-80^\circ$  and indexed using the 2014 ICDD database. The grain size of as-cast and solution treated specimens were measured by the linear intercept method using optical micrographs at three individual fields. The as-cast and solution treated specimens for OM observation were mechanically ground, polished, and then etched by the solution of 5.5 g picric acid, 5 ml acetic acid, 10 ml  $H_2O$  and 90 ml alcohol for about 10s. The specimens for EBSD analysis were first mechanically ground and polished, and then electropolished in a solution of perchloric acid and alcohol with the ratio of 1:9 for 90 s at the temperature of about  $-20$  °C. The specimens for TEM analysis were mechanically ground to about 50 µm, followed by ion-milling under the angle of  $10^{\circ}$  for 20min and then ion-milled to perforation under the angle of  $4^\circ$  at an ion accelerating voltage of 3 kV.

#### 3. Results

### 3.1. Initial microstructure

The actual chemical composition (wt.%) of the present Mg-RE-Zn-Zr alloy was determined as follows: Gd-6.84, Y-5.06, Zn-0.62,  $Zr-0.80$  and the rest Mg. [Fig. 1](#page--1-0)a-d shows the OM microstructures and SEM microstructures of Mg-7Gd-5Y-0.6Zn-0.8Zr alloy before and after solution treatment with the average grain size of  $36.46 \pm 4.15$  µm and  $37.55 \pm 5.01$  µm, respectively. It is evident that the dendritic second phases existed at the grain boundaries of the as-cast alloy, which dissolved into the Mg matrix without evident grain growth after solution treatment. The XRD analysis indicated that the main second phase in as-cast alloy was  $Mg_3(Gd, Y, Zn)$ , which disappeared after solution treatment, as shown in [Fig. 1e](#page--1-0). However, amounts of stacking faults occurred in the matrix of solution treated alloy, as shown in the insertion of [Fig. 1](#page--1-0)b. Besides, the LPSO phases, cuboid-shaped particles rich in REs and globular particles rich in Zr, as shown in [Fig. 1](#page--1-0)a-d, were not detected by XRD due to their low volume fractions in the as-cast and solution treated alloy.

### 3.2. Stress-strain behavior

[Fig. 2](#page--1-0) shows the flow stress-strain curves of as-cast and solution treated Mg-7Gd-5Y-0.6Zn-0.8Zr alloy deformed at different temperatures and strain rates. For both the as-cast and solution treated alloy, the flow stress decreased with increasing temperature and decreasing strain rate, indicating that the flow stress was sensitive to deformation temperature and strain rate. Actually, the flow stress-strain curves showed two typical deformation characteristics in the hot compression experiment. When deformed at 400–425 °C with the strain rate lower than  $0.01s^{-1}$  or the temperature higher than 425 $\degree$ C, the true stress-strain curves exhibited single peak stress at the initial deformation stage, followed by the decrease of flow stress to a relatively steady value. It represents typical deformation softening characteristic resulting from sufficient dynamic recrystallization (DRX). Under the other deformation conditions, the flow stress decreased continuously beyond the peak value due to incomplete dynamic recrystallization or flow instability [\[19,20\].](#page--1-0)

Compared with the as-cast Mg-7Gd-5Y-0.6Zn-0.8Zr magnesium alloy, the solution treated alloy exhibited higher peak flow stress because of solution strengthening under the same deformation condition, as shown in [Fig. 3](#page--1-0). The peak stress increased by 4-30 MPa after solution treatment (see [Fig. 3a](#page--1-0)). Noticeably, the variation of relative difference of peak stress with deformation temperature and strain rate was quite complex (see [Fig. 3](#page--1-0)b). The relative difference of peak stress increased until to 475  $\degree$ C, and then decreased at 500 °C under the same strain rates. Moreover, the relative difference of peak stress was more pronounce at low strain rate ( $\leq$ 0.01s<sup>-1</sup> in particular). For instance, the peak stress increased by 131% after solution treatment at 475°C/0.001s $^{-1}$ . The variation of relative difference of peak stress under different deformation temperature and strain rates may be associated with the precipitation of secondary phase during hot deformation, which will be discussed in the following section. Besides, at higher deformation temperatures and lower strain rates, the peak strain decreased for both as-cast and solution treated alloys (see Fig.  $3c-d$ ), indicating that dynamic softening took place more easily and dynamic recrystallization proceeded more sufficiently.

## 3.3. Kinetic analysis

Usually, the effect of deformation temperature and strain rate on

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