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Full Length Article

# Effect of applied pressure on microstructures of squeeze cast Mg–15Gd–1Zn–0.4Zr alloy

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## Abstract

Microstructural evolution of Mg–15Gd–1Zn–0.4Zr (GZ151K, wt%) alloys, cast under 0 MPa (gravity cast) and 6 MPa (squeeze cast), were comparatively studied. It is found that the grain size of squeeze cast GZ151K alloy with applied stress 6 MPa is much smaller than that of the gravity cast counterpart. Moreover, the squeezing pressure hinders the transition from $\beta$' precipitates to $\beta_1$ precipitates during subsequent aging process, leading to reduced volume fraction of $\beta_1$ precipitates in the squeeze cast alloy. Thus, the relatively lower volume fraction of $\beta_1$ precipitates in the squeeze cast GZ151K results in higher hardness increment and stronger precipitation hardening effect.

*Keywords:* Mg–Gd–Zn–Zr alloy; Squeeze casting; Microstructure; Precipitation hardening.

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## 1. Introduction

Environment-friendly magnesium (Mg) alloys with high-specific strength, high elastic modulus, and good machine processing property show increasing applications in automobile, electrical, aeronautics, and space industries [1]. However, the poor thermal resistance and low-yield strength (YS) of Mg alloys remain major obstacles for their wider use [2]. Recent research reports suggest that Mg alloys containing rare earth (RE) elements provide a promising solution to solve those above problems [3]. In variety Mg–RE alloy systems, Mg–Gd alloy systems are quite promising, due to their excellent room temperature strengths and elevated creep resistance. Besides, the adding Zn to Mg–Gd alloys has been reported to promote the formation of long period stacking ordered (LPSO) structures [4]. It has been acknowledged that the LPSO phases/structures can not only strengthen but also toughen Mg alloys [5]. Therefore, strong and ductile Mg–Gd–Zn alloys are anticipated via optimizing the compositions of Mg–Gd–Zn alloys and processes to make the most of precipitation hardening and LPSO structures strengthening.

Mg–Gd–Zn–(Zr) alloys were basically fabricated by gravity casting. Under this process, it is difficult to control the defects (shrinkage) effectively in castings. And this conventional casting process is low efficient. In this regard, it is essential to seek other casting options. Squeeze casting is a relatively new casting technology [6]. Yue and Chadwick [7] reported that squeeze casting as a casting process, in which molten metal was solidified under the direct action of a pressure, was more effective to prevent the appearance of both gas porosity and shrinkage porosity as opposed to all other casting processes. In the process of solidification, the application of mechanical pressure can be summarized as: (1) eliminate the gap between the casting and the mold wall to increase the thermal diffusion coefficient; (2) reduce or eliminate shrinkage during

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solidification; (3) change the solvus and liquidus lines in the equilibrium phase diagram [8].

Applied pressure is one of the most important processing parameters in squeeze casting. Goh et al. [9] reported that squeeze casting pressure was critical to affect the microstructures, properties, and solidification behavior of AZ91–Ca alloys. Regarding Mg–RE alloys, Wang's work [10] showed that increasing the applied pressure led to microstructural refinement of Mg–10Gd–3Y–0.5Zr (wt%) alloy, with accompanying enhancement of tensile yield strength, ultimate tensile strength, and elongation to failure.

Although, some researchers investigated the influence of applied pressure on Mg alloy [11–15], the effect of squeeze pressure on microstructures of Mg–Gd–Zn-Zr alloys was not reported previously. The aim of this paper is to comparatively investigate the microstructural changes due to the squeeze pressure in a typical high-strength Mg–15Gd–1Zn–0.4Zr (GZ151K, wt%) alloy.

## 2. Experimental procedures

The GZ151K alloy was produced from high-purity Mg (99.9 wt%), high-purity Zn (99.9%, wt%), Mg–87Gd (wt%), and Mg–30Zr (wt%) master alloys using an electrical resistance furnace under the gas protection of $CO_2$ and $SF_6$ mixture. After the pure Mg was melt, Mg–87Gd and Mg–30Zr master alloys and pure Zn were added into to the molten Mg. The melt was held at 750 °C for 20 min following by mechanical stirring for 5 min. After that, the melt was transferred for squeeze casting. The processes of squeeze casting were divided into the following steps: (1) check the die and increase the mold temperature followed by spray paint; (2) clamp the die; (3) manually pour the melt and seal the die; (4) start pressuring and keep the punch to let the melt to solidify under pressure; (5) open the die and ejection.

On the basis of our previous work, the solution treatment time was optimized. During the solution treatment, the samples were under the protection of sulfur dioxide, after which the samples were quenched into ~90 °C water. The parameters used for solution treatments are as follows: (1) 485 °C × 12 h; (2) 520 °C × 12 h; (3) 520 °C × 36 h; (4) 530 °C × 12 h. Aging treatments were carried out in oil baths at 175 °C, 200 °C, and 225 °C.

The actual chemical composition of GZ151K was measured as Mg–15.21Gd–1.14Zn–0.32Zr (wt%) using an inductively coupled plasma atomic emission spectroscopy (ICP-AES) analyzer. Phase analysis was conducted by X-ray diffractometer (XRD, copper target Rigaku D/max 2550 V) with a scanning speed of 1 °/min. Phase transformation temperature was confirmed by differential scanning calorimetry (DSC, a Netzsch STA449 F3 machine) with a heating rate of 5 °C rose from 25 °C to 560 °C. The microstructure observation was performed using an optical microscope (OM, Zeiss Axio Observer A1 optical microscope) and a scanning electron microscope (SEM, FEI Nova NanoSEM 230) equipped with a backscatter electron (BSE) detector and an energy dispersive X-ray spectrometer (EDS). The crystal structure of the second phase in the alloy was analyzed by selected area electron diffraction (SAED) on a JEOL 2100 transmission electron microscope (TEM) at 200 kV. The grain sizes of the samples were measured from at least ten pieces of optical micrographs (OM), according to ASTM E112-12. The age-hardening testing was carried out by a HVS-30P Vickers hardness tester under a load of 49 N and a duration time of 49 s.

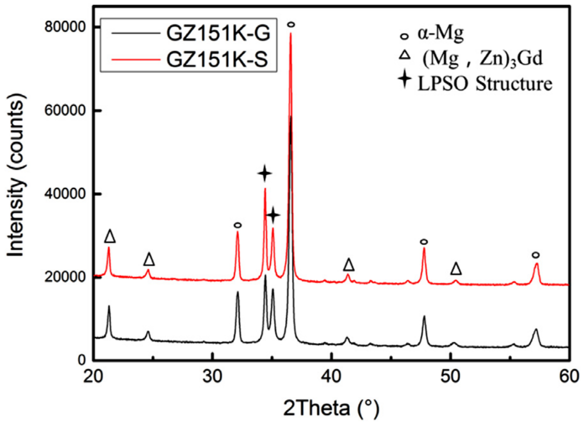

Fig. 1. X-ray diffraction (XRD) patterns of the as-cast GZ151K-G and GZ151K-S samples.*Y*-axis offset of 20,000 was applied on the curve of GZ151K-S.

## 3. Results and discussion

### 3.1. Effect of squeezing cast pressure on the as-cast microstructures

XRD patterns of as-cast GZ151K samples, fabricated by squeeze casting (with applied pressure of 6 MPa) and gravity casting, are shown in Fig. 1, respectively. For clarity of following descriptions, the samples prepared by squeeze casting and gravity casting are designated GZ151K-S and GZ151K-G, respectively. The XRD patterns indicate that both GZ151K-S and GZ151K-G samples are mainly composed of $\alpha$-Mg matrix, (Mg, Zn)$_3$Gd compound and LPSO structure. The squeeze pressure does not change the phases constituents of as-cast samples.

Fig. 2 shows OM and SEM micrographs of the as-cast GZ151K-S and GZ151K-Gd samples. All the phases identified from XRD patterns in Fig. 1 exist in OM and SEM micrographs of GZ151K samples with and without squeeze pressure. The eutectic compounds are distributed along the grain boundaries. Besides, lamellar structures were observed inside grains. The grain sizes of the as-cast GZ151K-G and GZ151K-S were measured as 41 $\mu$m and 28 $\mu$m, respectively. Compared with the GZ151K-G sample, the grain size of the GZ151K-S sample is significantly reduced. The main effect of pressure on microstructural refinement is ascribed to the higher heat transfer coefficient between melt and mold surface [14]. When the pressure applied on the melt, the punch came closer to the melt. Therefore, the interface distance between the casting and die was greatly reduced. The heat transferred

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