Research Article

Tao Lin*, Ji-Xue Zhou*, Cai-Nian Jing*, Yun-Teng Liu, Lin-Lin Zhang, and Xiang-Bin Meng

Improving mechanical properties of ZK60 magnesium alloy by cryogenic treatment before hot extrusion

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Abstract: Few studies of cryogenic treatment were focused on nonferrous alloys, such as magnesium alloy. In this work, the effect of cryogenic treatment (77 K) before extrusion on microstructure and mechanical properties of ZK60 alloy was investigated. The results showed that many fine G.P. zones were formed during the cryogenic treatment and then grew to short fine β_1' precipitates when heating before extrusion. These precipitates pinned dynamic recrystallized grain boundaries in the subsequent extrusion, resulting in fine grains and dispersed spherical precipitates. By the cryogenic treatment before extrusion, the extruded ZK60 alloy showed good tensile strength and elongation balance. Especially, elongation was improved by 29%.

Keywords: ZK60 magnesium alloy, cryogenic treatment, extrusion, mechanical properties

1 Introduction

Magnesium (Mg) alloys are the light metallic materials with low density and high specific strength. These alloys have attracted great interest in reducing the weight of the structural components in the automobile, aerospace, and electronics industries [1–3]. However, magnesium alloys show lower mechanical performance in comparison with aluminum and steel alloys. Many efforts have been directed to enhance their mechanical properties [4–6].

Grain refinement and precipitation strengthening are the two primary methods to improve the strength and ductility for magnesium alloys [7,8]. Commercial magnesium–zinc–zirconium (ZK) alloys were designed and are widely used, which have good strength and ductility due to zinc addition for precipitation strengthening and zirconium addition for grain refinement [7,9–11]. Many works have been done to further enhance their mechanical properties by adding rare earth (RE) or free-RE elements [12–16], using the aging treatment by which Mg–Zn age-hardening precipitates were formed [17–21]. Jung et al. first proposed a pre-aging treatment prior to extrusion (APE) [22]. The main idea was that fine precipitates were expected to be formed by the APE before hot extrusion. These precipitates could pin the dynamically recrystallized grain boundaries and hinder the growth of them during the subsequent hot deformation [23–25]. As a result, the extruded magnesium alloy had fine microstructures and a resultant increase in strength and ductility.

Cryogenic treatment (CT, 77 K) is widely used to improve hardness and wear resistance of machining tools by refining martensite [26,27]. In recent years, cryogenic treatment has been introduced to enhance the mechanical properties of magnesium alloys, i.e., AZ31 [28], AZ91 [29], ZK60 [30,31], Mg–Zn–Gd [32], and AE42 [33]. By this treatment, twinning, precipitate, and finer grains are formed so that hardness, strength, and elongation can be increased [29]. Chen et al. demonstrated that precipitation of fine Mg–Zn particles was

* **Corresponding author: Tao Lin**, Department of Plastic Forming, School of Materials Science and Engineering, Shandong Jianzhu University, Jinan 250101, China, e-mail: lintao19@sdjzu.edu.cn

* **Corresponding author: Ji-Xue Zhou**, Department of Light-weight and High Strength Metal Materials, Qilu University of Technology (Shandong Academy of Sciences), Advanced Materials Institute, Shandong Key Laboratory of High Strength Lightweight Metallic Materials, Jinan 250014, China, e-mail: zhoujx@sas.org

* **Corresponding author: Cai-Nian Jing**, Department of Plastic Forming, School of Materials Science and Engineering, Shandong Jianzhu University, Jinan 250101, China, e-mail: jcn@sdjzu.edu.cn

Yun-Teng Liu, Lin-Lin Zhang: Qilu University of Technology (Shandong Academy of Sciences), Advanced Materials Institute, Shandong Key Laboratory of High Strength Lightweight Metallic Materials, Jinan 250014, China, e-mail: liu_yunteng@126.com, zhlinlin@sas.org

Xiang-Bin Meng: School of Mechanical and Automotive Engineering, Qilu University of Technology (Shandong Academy of Sciences), Jinan 250353, China, e-mail: mengxiangbin84@163.com

induced in the cast ZK60 alloy at a cryogenic temperature [31]. These particles were 20–100 nm, uniformly distributed inside grain and at grain boundaries, which made a beneficial effect on the mechanical properties. Xie et al. investigated the effect of T4, T5, and T6 treatments combined with cryogenic treatment on the microstructure and mechanical properties of the cast ZK60 alloy [34]. Cryogenic treatment before aging would promote more and finer Mg–Zn phases precipitated, leading to higher strength.

Based on the work above, first, by APE, the pre-existing precipitates had a beneficial effect on refining the deformed microstructure [35]. Second, cryogenic treatment can promote finer precipitates formation. So, if cryogenic treatment was introduced as the “pre-aging treatment” in APE for ZK60, more and finer Mg–Zn precipitates may be formed, so that the microstructure and mechanical properties of the extruded alloy could be also improved. In this paper, the cryogenic treatment prior to extrusion on the microstructure and mechanical properties of the extruded ZK60 alloy was investigated. Semi-continuous as-cast billets were put in liquid nitrogen for cryogenic treatment, and then, they were hot extruded at 250°C. The microstructure and mechanical properties of the extruded alloy by the CT treatment above were compared with that by conventional extrusion.

2 Experiments

In this work, the commercial Mg–Zn–Zr as-cast billet was prepared for cryogenic treatment and extrusion. The composition of the Mg–Zn–Zr alloy is 5.20 Zn–0.58 Zr–balance Mg (wt%). The billets were 96 mm in diameter and 100 mm in length. Two sets of experiments were conducted. In the first set, the billets were put in liquid nitrogen for 16 h for the cryogenic treatment. Then, they were heated in a furnace at 250°C for 2 h and hot extruded by an 800-ton horizontal extruder at 250°C, with the extrusion ratio of 56 and 0.3 mm/s RAM speed, followed by air cooling to room temperature. For comparison, the billets went through the same procedures as the first set, but without cryogenic treatment.

Tensile tests were conducted on a Shijin materials testing machine, using displacement control at a speed of 2 mm/min at room temperature. Smooth dog-bone-shape specimens were used with $\Phi 5 \times 25$ mm. All three tested specimens were machined along the extrusion direction (ED) of each sample. The load direction was

parallel to ED. The yield stress was determined as the 0.2% offset.

The microstructure observation of the as-cast and extruded samples was performed by scanning electron microscope (SEM; Zeiss EVO MA10) with EDS microanalysis. The samples were polished mechanically and etched in a corrosive solution of 4.2 g picric acid, 70 mL ethanol, 10 mL glacier acetic, and 10 mL deionized water. Electron backscatter diffraction (EBSD) was done using SEM equipped with a Nordlys-S detector, a CCD camera, the HKL data acquisition software, and the Channel 5.0 post-processing analysis software package. For EBSD mapping analysis, EBSD measurements were performed on the conditions of 20 kV electron beam, 10 nA probe current, 17 mm working distance, 20 ms integration time, and 0.5 μ m step size. The EBSD specimens were mechanically polished, then electrolytic polished in commercial AC2 solution at 16 V for 80 s at –15°C. The misorientation angles between the adjacent grains were used to identify the low-angle grain boundary (LAGB, 2°–15°) and the high-angle grain boundary (HAGB, >15°). The fraction of recrystallized grains was evaluated by the following method. If the boundary angles exceed 15°, the grains were considered as recrystallized regions. The remaining grains with boundary angles lower than 15° were considered as the un-recrystallized regions. The average grain size was estimated by employing the linear intercept method on SEM images.

X-ray diffraction (XRD) tests were conducted. The XRD specimens, mechanically polished, were investigated using a Rigaku D/Max 2500 PC X-ray diffractometer (Rigaku Corporation, Tokyo, Japan) with Cu Ka radiation at 50 kV and 300 mA. The scanning range was from 20° to 100°, and the scanning step is 0.02°.

Detailed microstructure observations were carried out using a transmission electron microscope (TEM; JEM-2100), with an accelerating voltage of 200 kV. The TEM specimens were prepared by mechanical polishing to a thickness of ~30 μ m, followed by ion beam thinning (GATAN 695).

3 Results and discussion

Figures 1 and 2 show the microstructure of the as-cast ZK60 alloy. Figure 1 represents the SEM microstructure, showing that the average size of the equiaxed α -Mg grains was measured at ~60 μ m because of Zr addition [5]. Many bulk laminar and strip-like β phases were observed at the grain boundaries, which were identified

Figure 1: SEM images of the as-cast ZK60 alloy with (a) low magnification and (b) high magnification.

Figure 2: TEM image of the as-cast ZK60 alloy.

as MgZn_2 by EDS analysis [36]. By TEM results shown in Figure 2, there are few dot-like β -phases in the matrix, which are composed of Mg, Zn, and Zr [37].

First, it can be seen that the grain size is similar in the alloy with and without cryogenic treatment. It was reported that the grain size in the as-cast ZK60 alloy was significantly reduced from 70–100 to 30–50 μm after the cryogenic treatment, due to the sub-grain generated by internal stress [31]. In contrast, it was demonstrated that, in the paper [38,39], the grains in the Mg–1.5Zn–0.15Gd alloy and AZ91 alloy were refined little by the cryogenic treating. In this work, the grains in the as-cast ZK60 alloy were not observed to be refined by the treatment, neither.

Then, the SEM results show that the volume fraction and morphologies of the bulk β -phases changed little after the cryogenic treatment. The matrix in the treated alloy still consisted of α -Mg (dark regions) and light trips

of β -phase (MgZn_2). The volume fraction of the phases changed from $1.48 \pm 0.30\%$ to $1.42 \pm 0.29\%$, measured from the SEM images (Figure 3a and b) by Image Pro-plus software. The reduction or disappearance of the bulk β -phases was no significant, and their morphologies were kept in laminar and strip-like.

So, according to the SEM analysis, the obvious variations in the morphology, size, and fraction of the grains and β -phases were not observed after the cryogenic treatment [29].

Figure 4 shows the XRD patterns of the as-cast alloy with and without cryogenic treatment. The results indicate that the as-cast alloy mainly contains α -Mg and MgZn_2 phases, which are confirmed by the SEM and TEM results above. After the cryogenic treatment, the peak of the MgZn_2 phases was raised, meaning that more particles were precipitated in the alloy. While there were few MgZn phases formed.

Figure 5 illustrates the typical TEM microstructure in the as-cast alloy with the cryogenic treatment. It can be seen that a high number of fine dot-shaped particles are homogeneously distributed in the matrix after the cryogenic treatment. The particles are G.P. zones for the MgZn_2 phase whose average size is less than 10 nm [40]. These particles were (semi-)coherent with the matrix, with its lower total interfacial energy including strain energy [41]. The rod-shaped phase and disk-shaped phase were not observed.

During the cryogenic treatment, volume contraction in the matrix was generated due to shrinkage. The contraction caused internal stresses and defects such as dislocations and vacancies so that precipitation was promoted. In the Mg–Zn–Gd alloy [28], W phases were easily precipitated because of additional space provided by the volume contraction during the cryogenic

Figure 3: SEM images of the as-cast ZK60 alloy: (a) without cryogenic treatment and (b) with cryogenic treatment for 16 h.

Figure 4: XRD patterns of the as-cast ZK60 alloy with and without cryogenic treatment.

Figure 5: TEM image of as-cast ZK60 alloy with cryogenic treatment for 16 h.

treatment. And the precipitation would be encouraged if the precipitates had a lower atom density than the Mg matrix. For Mg–Zn alloy, the atom density of MgZn_2 is

13.1 nm^{-3} , lower than that of the Mg matrix, 57.1 nm^{-3} . As a result, the MgZn_2 phases were readily formed in the α -Mg matrix during the cryogenic treatment [28].

Before extrusion, the as-cast alloy with and without cryogenic treatment were both heated at 250°C for 2 h. Through TEM observation in Figure 6, it can be seen that there are many precipitates distributed in the heated as-cast alloy with and without cryogenic treatment, but with different morphologies. Typical aging precipitation in Mg–Zn alloy is shown in the heated alloy without cryogenic treatment (Figure 6a). The thin and long precipitates are rod-shaped β'_1 phase, the thick precipitates are plate-shaped β'_2 . β'_1 rods were formed along the c -axis and high coherency with the matrix, while the β'_2 plates kept an incoherent lattice relationship with the matrix [42]. The orientation relationships of them are distinguished. The orientations were $[0001]_{\beta'_1}/[11\bar{2}0]_\alpha$ and $(11\bar{2}0)_{\beta'_1}/(0001)_\alpha$, $[11\bar{2}0]_{\beta'_2}/[10\bar{1}0]_\alpha$, and $(0001)_{\beta'_2}/(0001)_\alpha$, respectively [35,42]. The size of the β'_1 phases is $\sim 120 \text{ nm}$ in length and $\sim 20 \text{ nm}$ in thickness. The β'_1 phases, perpendicular to the rod-like phases, are $\sim 70 \text{ nm}$ in length and $\sim 30 \text{ nm}$ in thickness.

In contrast, a different morphology of precipitates was observed in the heated alloy with cryogenic treatment (Figure 6b). Some precipitates are still in dot-like shape, others grew up in a short and thick rod-like. These precipitates are typical β'_1 phases in the ZK60 alloy [40]. The average size of them is smaller, about 15 nm in length and 10 nm in thickness.

The difference in the morphologies of the precipitates in the heated alloy with and without cryogenic treatment demonstrated that the cryogenic treatment could promote nucleation of the β'_1 phase and then high-density finer β'_1 phases were precipitated or grew during

Figure 6: TEM images of the as-cast ZK60 alloy heated at 250°C for 2 h before extrusion: (a) without cryogenic treatment and (b) with cryogenic treatment.

the heating at 250°C for 2 h. By the treatment, densely G.P. zones were formed which provided many nucleation sites for the β'_1 phases. The G.P. zones would grow uniformly during the heating as Zn elements in the Mg matrix were likely attached to these dispersed sites and grew up [35]. In contrast, in the alloy without cryogenic treatment, the nucleation sites were fewer, and the size of the β'_1 or β'_2 phases was rash to grow up to a larger size. Thus, much more and smaller rod-like precipitates were observed in the as-cast alloy with cryogenic treatment after the heating.

After the heating, the alloys with and without cryogenic treatment were both extruded at 250°C with a RAM speed of 0.3 mm/s. Figure 7 shows the SEM images of the hot extruded ZK60 alloy without and with cryogenic treatment. It is shown that the microstructure of them is different. The average grain size is smaller in the hot extruded cryogenically treated alloy and there

are more and finer precipitates, although both of them contain large broken particles and randomly distributed fine particles in the grain boundaries and grain interior. The dynamically recrystallized grains are more and smaller due to the effective pinning of grain boundaries by the very fine precipitates in the extruded treated alloy [43]. From EBSD analysis in Figure 8, the recrystallized grains are about $\sim 2\mu\text{m}$, smaller than that in the traditional extruded alloy ($\sim 4\mu\text{m}$). Figure 9 shows the TEM images of the hot extruded ZK60 alloy with cryogenic treatment. It can be seen that a lot of extreme fine spherical precipitated particles, with several decade nanometers, uniformly distribute, leading to the finer grain size in the extruded cryogenic-treated alloy.

The tensile mechanical properties of the extruded ZK60 alloy with and without cryogenic treatment at room temperature including tensile yield strength (TYS), ultimate tensile strength (UTS), and elongation after

Figure 7: SEM images of the ZK60 alloy extruded at 250°C with a RAM speed of 0.3 mm/s: (a) without cryogenic treatment and (b) with cryogenic treatment.

Figure 8: Inverse pole figure maps of the ZK60 alloy extruded at 250°C with a RAM speed of 0.3 mm/s: (a) without cryogenic treatment and (b) with cryogenic treatment.

Figure 9: TEM image of the hot extruded ZK60 alloy with cryogenic treatment.

fracture (EI) are listed in Table 1. And the typical tensile stress–strain curves are shown in Figure 10. By comparison, the mechanical properties of the extruded alloy were improved when the cryogenic treatment was applied. Strength and ductility were both increased. The yield strength and the tensile strength were increased from 312 and 378 MPa to 324 and 386 MPa, respectively. The elongation was increased from 20.4% to 26.4%, improved by about 29%. The improvement was mainly attributed to grain refinement and enhanced precipitate strengthening.

Figure 10: Engineering stress–strain curves of the extruded ZK60 alloy with and without cryogenic treatment.

Finer grains had a better effect on preventing the formation of deformation twins and activating dislocations on non-basal planes, which caused the enhancement of both strength and ductility [32,33].

Figure 11 shows SEM images of the tensile fracture surfaces of the extruded ZK60 alloy without and with cryogenic treatment. Both the alloys exhibit typical ductile fracture features comprising a lot of deep dimples and tear edges. In contrast, more and finer dimples were observed

Table 1: Mechanical properties of the extruded ZK60 alloy without and with cryogenic treatment at room temperature

	TYS (MPa)	UTS (MPa)	EI (%)
Without cryogenic treatment	312 (+4/−1)	378 (+4/−8)	20.4 (−0.4/+2.2)
With cryogenic treatment	324 (+8/−2)	386 (+8/−7)	26.4 (−3.9/+3.0)

TYS, tensile yield strength; UTS, ultimate tensile strength; EI, elongation after fracture. Values in brackets indicate the uncertainty.

Figure 11: Tensile fracture surface images of the extruded ZK60 alloy (a) without cryogenic treatment and (b) with cryogenic treatment.

in the cryogenically treated extruded alloy. Some dimples are larger and deeper, containing many fine dimples. Also, the tear edges in the alloy are fewer and smaller than that in the conventionally extruded alloy. This difference in the fracture morphologies indicates that the ductility of the extruded ZK60 alloy was improved when the cryogenic treatment was used, which is in line with the tensile mechanical properties results.

By the above analysis, it can be illustrated that the precipitates in the hot extruded cryogenic-treated alloy were finer and well-distributed at the grain boundaries and inside the grains. First, in the cryogenic treatment, a large number of finer G.P. zones with several nanometers were formed, which grew to smaller dispersed β_1' precipitates during the heating before extrusion. Then, in the extrusion, these much finer precipitates had a better pinning effect on the grain refinement than that in the traditional extruded alloy in which the precipitated phases were larger and the grain size was bigger. According to the Hall–Petch equation, the increment of yield strength was due to grain refinement that also led to an increase of elongation [43]. Moreover, according to the Orowan strengthening mechanism, a greatly increased number of precipitates reduced the particle spacing, which also contributed to the enhancement of the strength. Hence, the cryogenic treatment is comparable to the APE on promoting precipitation and grain refinement. Consequently, the comprehensive mechanical properties of the hot deformed Mg alloy were enhanced.

In summary, cryogenic treatment mainly promoted the formation of a large number of dispersed fine β_1' phases formed before extrusion. The phases act as pinning dynamically recrystallized grain boundaries during extrusion, thereby refining the grain size and strengthening the Mg-matrix, hence, the strength and ductility of the extruded ZK60 alloy were both improved.

4 Conclusions

The influences of cryogenic treatment on microstructure and mechanical properties of the hot extruded Zk60 alloy were investigated. The following results were obtained.

- (1) The introduction of a cryogenic treatment prior to extrusion for ZK60 alloy has shown a beneficial effect, improvements in elongation and strength. The yield strength and tensile strength slightly are increased while the elongation is improved by 29%, increased from 20.4% to 26.4%.
- (2) The improvement of strength and ductility is due to the combination of fine grains and precipitation strengthening. Fine grains are obtained because of the pinning effect on the dynamically recrystallized grain boundaries by a great number of β_1' precipitates formation by utilizing the cryogenic treatment.

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