Reducing the tension-compression yield asymmetry in an extruded ZK60 alloy by ultrafine grains

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Abstract

Ultrafine-grained ZK60 alloys were successfully fabricated by powder metallurgy followed by hot extrusion at different temperatures. The effects of the texture changes of ultrafine-grained ZK60 magnesium alloy on the asymmetry of tension-compression yield under different extrusion temperatures, and the relation between texture evolution and mechanical properties of the alloys during axial tension-compression deformation were studied. The results show that, in the ultrafine-grained ZK60 alloy, the initial texture of the alloy is a weak (0002) basal fiber texture. As the extrusion temperature increases from 523 to 623 K, the fibrous texture of the base material is weakened, and the tension-compression yield asymmetry is depressed from 1.1 to 1.0. During axial tensile deformation, twinning is not activated. With increasing tensile strain, no significant rotation of crystal grains occurs, and the stress remains stable until tensile fracture occurs. In the early stage of axial compression yield, no significant rotation of crystal grains occurs. As the compressive strain increases until the end of the compressive strain, the (0002) basal plane of the crystal grains rotates in a direction approximately perpendicular to the compression axis. At this point, the grain orientation factor is low, and the slip system is still in a hard orientation and is inhibited by the ultrafine grains. Twinning is difficult to start, so that the strain hardening rate rises rapidly until compression fracture occurs. Therefore, the weak extruding fiber texture of the basal plane and the ultrafine-grained structure both determine the deformation mechanism of ZK60 alloys at room temperature during axial tension and compression deformation, thereby significantly depressing the axial tension-compression asymmetry of ZK60 alloys.

1. Introduction

Magnesium (Mg) alloy, the lightest structural metallic material at present, has several advantages, such as low density, high specific strength ratio, good damping, and good thermal conductivity. It has wide application in the automotive industry, aerospace, aviation, electronic products, and other fields. Nonetheless, because of its special hexagonal-close-packed crystal structure, wrought Mg alloys are prone to multiple deformation textures during plastic-forming processes [1, 2]. For example, basal fiber textures tend to form in extruded Mg alloys [3] and lamellar textures tend to form in Mg alloys during rolling [4]. Deformation textures in Mg alloys result in tensile-compressive yield asymmetry. Under the loading modes of axial compression and tension, the compression yield stress is lower than that of tension. For some structural applications, such as supporting beams, the materials will bear both tensile and compressive stresses. This will therefore limit the applications of Mg alloys in some structural parts. It is significant to develop magnesium alloys with high performance, and to expand their industrial applications by study the micro-mechanism of tensile-compressive asymmetry in magnesium alloy.
Related studies have shown that twinning, second phase, and basal texture are the main factors affecting the tension-compression asymmetry in deformed Mg alloys [5–7]. Deformed Mg alloys all have intense basal textures; that is, the basal plane is parallel to the extrusion or rolling direction. In general, deformed Mg alloys have an axial ratio \( c/a < 1.624 \). When the Mg alloy is strained in the extrusion or rolling direction under load, twinning is prone to occur. The content of twinned crystals plays an important role in the tensile-compressive yield asymmetry [8]. Lin et al. [9] studied the axial tension-compression asymmetry of extruded ZK60 alloys and found that the extruded (0002) basal fiber textures and a large number of activations of \{101̅2\}, \{101̅1\} tensile twinning are the main factors leading to the tension-compression asymmetry of the alloy. However, twinning activation depends on the grain size [10], so a fine-grained structure helps reduce the tension-compression asymmetry. In addition, a fine second phase also helps to reduce the asymmetry of Mg alloys. For example, Jain et al. [11] reported that the Mg17Al12 phase in aged Mg–8Al–0.5Zn alloy weakened the tension-compression asymmetry by suppressing the growth of twinned crystals. In traditional thermal processing, grain refinement, precipitation of the second phase, and cutting of the basal texture are achieved by means of rolling, equal-path angular pressing, addition of rare-earth elements, and heat treatment to weaken the tension-compression asymmetry [12–15]. However, in the plastic-forming process of Mg alloys, texture, grain size, and second phase are all generally dependent on plastic-forming parameters. For example, extrusion ratio and extrusion temperature have a great influence on texture and grain size during extrusion. However, there have been few studies on this aspect, and the influence of texture evolution in ultrafine-grained Mg alloys on the tension-compression asymmetry has not yet been studied.

Therefore, in this paper, ultrafine-grained ZK60 alloys were prepared using the powder metallurgy process. Texture evolution at different extrusion temperatures and the relationship between texture and tension-compression mechanical properties during tension and compression deformation were investigated.

2. Materials and methods

The ZK60 alloy powder used in the experiment was provided by Tangshan Weihao Magnesium Powder Co., Ltd. Its main chemical composition (mass fraction%) was Zn 5.1 and Zr 0.18, and the particle size was approximately 50 \( \mu \text{m} \). The alloy was prepared by powder metallurgy. First, ball milling was performed using a DYXQM-12L planetary ball mill. The ZK60 alloy powder was placed in a stainless-steel ball-mill jar, 2–3 drops of alcohol were added, and the jar was protected by Ar gas. The ratio of ball: material was 20:1, the rotation speed was 200 rpm, the forward rotation was 15 min, and the reverse rotation was also 15 min, with an interval of 20 min. The effective ball milling time was 1.5 h. The ball-milled powder was then placed in a mold and pressed under vacuum in a hot-pressing sintering furnace (temperature, 180 °C; pressure, 100–150 MPa; time, 1 h; vacuum degree, \( 10^{-1} \) Pa). The mold was then removed from the furnace, placed on a four-post hydraulic press, and heated to the extrusion temperatures (523, 573, and 623 K) in a heating furnace, where it was kept warm for 1 h, and then compacted with a pressure of 500–600 MPa (5–10 min). Finally, a 10 mm-diameter alloy rod was directly extruded at an extrusion ratio of 20:1.

An Instron-5982 high-temperature electronic universal material testing machine was used for room-temperature tensile and compression testing. The stretching rate was 0.2 mm min \(^{-1}\) and the compression rate was 0.3 mm min \(^{-1}\); Yield strength reported here corresponds to a 0.2% offset. The asymmetry of tension-compression yield was described by the ratio of tensile yield strength to compressive yield strength (TYS/CYS).

The sampling position and size of the samples are shown in figure 1. A Bruker XD8 ADVANCE-A25 x-ray diffractometer was used to conduct x-ray diffraction (XRD) measurements and analyze the macro-texture of the samples. The macro-texture measurement parameters were as follows: zero-dimensional mode, copper target \( K_\alpha \) line (\( \lambda = 0.1541 \) nm), voltage 40 kV, current 40 mA, \( \Phi \) (0°–360°), and \( \Psi \) (0°–80°). The five groups of crystal planes measured were the \{10\( \overline{1} \)0\}, {0002}, {10\( \overline{1} \)1}, {10\( \overline{1} \)2}, and {11\( \overline{2} \)0} crystal planes. The microstructure of the samples was observed by scanning electron microscopy (SEM) with a ZEISS-6035 field-emission scanning electron microscope. The specimens for SEM observation were polished and etched with a solution consisting of 2 g picric acid, 5 ml distilled water, 5 ml acetic acid and 33 ml alcohol. The average grain size of samples was measured by Image-Pro software. The surface of samples for EBSD analysis was parallel to the extrusion direction, and the specimens were mechanical polishing and subsequently argon ion polishing. The EBSD calibration was performed on the characteristic region of the samples using an Oxford HKL Channel 5 EBSD system. The accelerating voltage was 20 kV, the probe distance was 168 mm, the sample was tilted by 70°, and the sample calibration EBSD had a step length of 0.08 \( \mu \text{m} \). HKL Channel 5 software was used to process the EBSD data.
3. Results and discussion

3.1. Microstructure of ZK60 at different extruded temperatures

Figure 2 shows the optical microscope (OM) microstructure of original powder and hot-pressing sintered sample, and figure 3 shows the SEM microstructure of ZK60 alloys at different hot extrusion temperatures (523, 573, and 623 K). As shown in the figure 2(a), the grain size of raw powder is about 7 μm. As can be seen from figure 2(b), grain size in the sintered sample grows to about 15 μm. After extrusion, as can be seen from the figure 3, fine white MgZn2 phase particles were uniformly distributed at the grain boundaries and the grains were broken. The extrusion temperature has a significant effect on the dynamic recrystallization of the alloy. As shown in figures 3(a) and (b), the ZK60 alloy extruded at 523 K exhibited an ultrafine-grained structure. Both the grains with irregular shape and the grains with equiaxial shape coexist with an average grain size of approximately 0.7 μm. As shown in figures 3(c) and (d), the grain size in the ZK60 alloy extruded at 573 K dramatically increased and the average grain size reached approximately 1.1 μm. As the extrusion temperature was further increased to 623 K, the grain size increased more slowly, and the average grain size reached approximately 1.3 μm, as shown in figures 3(e) and (f). The results indicate that, as the extrusion temperature increases, the dynamic recrystallization structure gradually increases, and recrystallized grains grow.

3.2. Tensile-compressive yield asymmetry

Figure 4 is the stress–strain curve of an as-extruded ZK60 alloy. As shown in figure 4(a), the tensile stress–strain curve of the as-extruded ZK60 alloy at 523 K showed an obvious work-softening behavior. With increasing extrusion temperature, softening of the extruded ZK60 alloy was not observed at 573 and 623 K. Wei et al [16] proposed that work softening of the nanocrystalline Mg alloy during tensile deformation at room temperature was mainly caused by grain-boundary damage inside the metal material, and was almost not affected by texture changes. In this study, the average grain size of extruded ZK60 alloy at 523 K was approximately 0.7 μm, and the average grain size increased to about 1.3 μm at 623 K, which is consistent with the results in the literature [16]. It can be seen from figure 4(b) that the as-extruded ZK60 alloy prepared by the powder metallurgy method did not have similar work-softening behavior as other nanocrystals or ultrafine-grain metals during the compression process [17, 18]. On the contrary, with the strain increasing, the work-hardening of ZK60 alloy during the compression process was increased. This is mainly due to the insensitive to defects caused by grain-boundary
damage of the material during the compression process; however, the significant texture changes of the material have a greater effect on work hardening. As shown in Table 1, with extrusion temperature increasing, the tensile and compressive yield strengths of the ZK60 alloy gradually decreased, and the tensile-compressive yield strength...
asymmetry gradually decreased from 1.1 to 1.0. These phenomena are all related to the evolution of the corresponding microstructure and texture.

3.3. Textures of ZK60 Mg alloys at different extrusion temperatures

Figures 5(a)–(c) are the pole figures of the extruded ZK60 alloy at different extrusion temperatures, ED is the extrusion direction, and TD is the transverse direction. It can be seen from figure 5(a) that the textures of ZK60 alloy extruded at 523 K was a weak basal fiber texture with a maximum intensity of 4.04. It can be seen from the (0002) pole figure in figure 5(a) that the pole density points are distributed at the two ends of the TD. It is indicated that the (0002) base of the grain was inclined parallel to the extrusion direction (ED). It can be seen from the (1010) pole in figure 5(a) that the pole density points are at both ends of the ED direction, indicating that the distribution of the a axis had an orientation; that is, the (1010) plane of the cylinder was approximately perpendicular to the ED direction. Thus, the weak texture component in the as-extruded material was (0002) [1010]. From figures 5(b) and (c), it can be seen that the (0002) and (1010) planes of the extruded ZK60 at 573 K and 623 K still have the same pole density points as that of the extruded ZK60 at 523 K. However, both of them exhibited weak basal fiber textures. The texture intensity value was 2.65 when the ZK60 alloy was extruded at 623 K. It is shown that the basal texture weakens when the extrusion temperature increases. This is mainly due to the fact that the dynamically recrystallized grains occupy a larger amount than those of deformed microstructures. From figures 5(b) and (c), the pole figure of the (0002) plane shows a weak pole density point in the ED direction, which confirms that some of the grains rotate and the (0002) plane is perpendicular to the ED direction. Therefore, the weak basal texture occurs in extruded ZK60. This weak basal texture can effectively weaken the tension-compression asymmetry [19].

| Materials | T(K) | TYS(MPa) | UTS(MPa) | TFS(%) | CYS(MPa) | UCS(MPa) | CFS(%) | TYS/CYS |
|-----------|------|----------|----------|--------|----------|----------|--------|---------|
| ZK60 523 K | 386.9 | 421.6 | 6.1 | 359 | 523 | 14.2 | 1.1 |
| ZK60 573 K | 336.4 | 365.7 | 6.8 | 300 | 527 | 16.8 | 1.1 |
| ZK60 623 K | 287.8 | 326.5 | 8.5 | 283 | 505 | 15.4 | 1.0 |

Figure 5. The textures of ZK60 at various extrusion temperatures (a) 523 K, (b) 573 K and (c) 623 K.

Table 1. Mechanical properties of ZK60 alloys at various temperature.
3.4. Texture evolution of ZK60 alloy during tensile and compressive deformation

Figure 6 shows the textures' evolution during the tensile and compression of ZK60 alloy extruded at 573 K. From figure 5(b), the textures in as-extruded ZK60 of the (0002) and (10\(\bar{1}0\)) planes was a typical basal fiber texture, namely, the (0002) planes of most grains in the ZK60 alloy were parallel to the extrusion direction, and the (10\(\bar{1}0\)) plane was perpendicular to the extrusion direction, showing an intensity value of 3.39. From figure 6(a), it can be seen that the (0002) basal plane had a very small increase in its polar density value before tensile fracture occurred as the tensile deformation increased, and its intensity value was 3.75. This indicates that although the (0002) plane of a small portion of the grains rotated to be parallel to the extrusion axis, the change of the textures between figures 5(b) and 6(a) in the as-extruded ZK60 under varied elongation is small. It shows that the pole figure type and pole density of the (0002) and (10\(\bar{1}0\)) planes during the tensile process have almost not been changed, and they still are weak basal fiber textures. The change in the inverse pole figures as shown in figures 6(a) and 5(b) also supports above-mentioned conclusion. In the axial compression process, before the yield of the material, namely the compressive strain reached 3%, the textures' intensities of the (0002) and (10\(\bar{1}0\)) planes slightly increased, and the grain orientation did not change significantly between figures 6(b) and 5(b). This indicates that no significant rotation of the grains occurred at the beginning of compression. As shown in figure 6(c), when the compressive strain reached 5.5%, the pole density point in the (0002) pole figure started to be distributed along the ED direction, the texture intensity increased significantly, the (10\(\bar{1}0\)) pole figure became scattered, and the texture became weakened. This shows that part of the (0002) planes of the grains gradually turned to the direction perpendicular to ED. As shown in figure 6(d), when the plastic strain reached 11%, the most of the (0002) planes of crystal grains was perpendicular to the ED direction. This is also consistent with the results in the inverse pole figure. In short, during compression, the (0002) basal plane of the crystal rotates from a direction parallel to ED to a direction perpendicular to ED. In this process, the corresponding texture changes affect the mechanical behavior of compression deformation.

Figure 6. Textures of extruded ZK60 alloy at 573 K (a) near the fracture surface of tension, (b) 3% strain of compression, (c) 5.5% strain of compression and (d) 11% strain of compression.
3.5. Deformation microstructures

Figure 7 shows the EBSD graphs of as-extruded ZK60 alloy after tension and compression deformations. Figures 7(a) and (c) are the EBSD graphs in the vicinity of tensile fracture and 11% strain compression, respectively; it can be seen that the grain boundaries of both of them are clear, and the grain orientations were apparent along the longitudinal section; however, no obvious twinnings were observed. From the pole figure in figure 7(b), it can be seen that the weak basal fiber texture remained after tensile deformation with an intensity of 5.69. The inverse pole figure from the ED direction also clearly shows that the pole density point of (1010) was the largest among the pole density points, which means that [1010] was parallel to ED. Therefore, the grain orientation did not change significantly during the tensile deformation process, which is in accordance with the macro-texture results measured by XRD. It can be seen from the pole figure in figure 7(d) that, after the compression deformation, the pole density points of the (0001) pole figure were distributed along the ED direction, and the pole density points of the (1010) pole figure were distributed along the TD direction. The inverse pole figure of the ED direction also shows that the pole density point of (0001) was the largest. This shows that, during the compression process, the crystal grain has undergone a significant rotation, and the (0001) base surface of the crystal grain has been rotated approximately perpendicular to the extrusion direction (ED), which is consistent with the results shown in figure 6.

3.6. Deformation mechanism

According to Reference [20], in the process of axial tensile of Mg alloys, the initial deformation stage is dominated by basal slip and supplemented by prismatic slip. Some previous studies have also shown that when Mg alloy is tensile deformed at room temperature, basal slip of the alloy is very easy to activate, while (a) prismatic slip and (c + a) pyramidal slip are difficult to activate [21–24]. During the tension and compression deformation processes, whether or not the slip plane can be activated depends on whether or not the shear stress \( \tau \) in the slip direction of the slip plane reaches the corresponding critical resolved shear stress (CRSS). The resolved shear stress is calculated as follows:

\[
\tau = \left( \frac{F}{A} \right) \cos \phi \cos \lambda \cos \phi \lambda
\]

where \( F/A \) is the yield strength, \( \cos \phi \) is Schmid factor, \( \phi \) is the angle between the normal direction of the slip plane and stress vector, and \( \lambda \) is the angle between the slip direction and resolved stress vector on the slip plane. From figure 8(a), the (0002) basal plane of the grains was approximately parallel to the ED direction in the samples of the as-extruded ZK60, while the (1010) prism plane was approximately vertical to the ED direction. In this situation, the Schmid factor of the basal slip was close to zero, indicating that the basal slip is difficult to activate. Therefore, the basal slip can only be activated when the external force is large enough in order for the resolved shear stress to reach the CRSS. Before the tension and compression samples of as-extruded ZK60 alloy yielding, the grains did not rotate, and the weak texture did not change obviously. Therefore, both axial tension and compression have high-yield strengths, and the weak textures also weaken the tension-compression asymmetry [19]. When samples of the as-extruded ZK60 alloy yielded under tensile deformation as shown in figure 8(b), the orientations of the grains were hardly changed, and the deformation mechanism was dominated by the slip type. In general, for Mg alloy having a \( c/a = 1.624 \), the \( c \) axis is in a compressively stressed state when...
it is tensile deformed in the direction of the extrusion axis. In this situation, the tensile twinning \{1012\} \{101\} cannot be activated, while the compression twinning \{1011\} \{101\} is difficult to activate \[25\]. Therefore, after the sample of the as-extruded ZK60 yielded, with tensile strain increasing, the stress remains stable. Then, the grains mainly slip on the non-basal plane, and the work hardening rate gradually decreases, resulting in the cracks initiating at the grain boundaries and then propagating until the fracture of the tensile sample.

The c axis is in a tensile stress state during axial compression. Although the CRSS of the \{1012\} \{101\} is small and the tensile twinning is easy to activate, twinning is difficult to activate because of the ultrafine grains \[7\]. It has been reported that the twinning was more difficult to activate than dislocation slip \[26–28\]; with decreasing grain size, the rate of increase of CRSS for twinning was much higher than that of dislocation slip. Therefore, twinning is more difficult to activate than dislocation slip in the ultrafine-grained Mg alloys. Thus, \{1012\} \{101\} tensile twinning is suppressed at room temperature. As a result, the axial compression yield strength increases significantly and is slightly lower than the tensile yield strength. When the strain reached 5.5%, the (0002) basal plane of some grains rotated to a direction approximately perpendicular to the extrusion axis, as shown in figure 8(c). Therefore, the tensile twinning of the grains was suppressed and the slip system difficult to activate. The CRSS of compression twinning is very high at room temperature, and it is suppressed due to ultrafine-grained metals. Therefore, compression twinning is difficult to activate at room temperature, which makes the strain-hardening rate increase rapidly. Similar results have also been reported in the literature \[27, 29\]. When the compression strain reached 11%, the (0002) basal plane of most of the grains rotated to the direction perpendicular to the extrusion axis. As a result, the tensile twinning and the slip system were suppressed, and the strain-hardening rate increased until compression fracture occurred.

4. Conclusions

(1) With extrusion temperature increasing from 523 K to 623 K, and the (0002) basal texture of ultrafine-grained ZK60 alloys gradually weakening, the tension-compression asymmetry decreased from 1.1 to 1.0.

(2) Under axial tensile stress, there is no obvious change in the texture of the ultrafine-grained ZK60 alloy, the grains do not rotate obviously. Under axial compressive stress, Twinning is inhibited by ultrafine-grained, and significant texture changes resulted in the rapid increase of strain-hardening rate until compression fracture occurs.

(3) The high tensile and compressive yield strengths and weak tension-compression asymmetry of the as-extruded ZK60 are attributed to the combination of weak (0002) basal fiber texture and ultrafine-grained microstructure.

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