Effect of pre-extrusion on microstructure and mechanical properties of caliber-rolled AZ80 alloy

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Abstract

In this paper, pre-extrusion treatment was applied to AZ80 alloy prior to caliber rolling, and microstructural evolution as well as mechanical properties of groove-rolled AZ80 alloy were studied. The results shown that the pre-deformation treatment can enhance the formability of AZ80 alloy and the cracking can be avoided. In addition, the pre-extrusion also resulted in fine grain structure after rolling process. Especially, 8-pass caliber rolling following by pre-extrusion of AZ80 alloy shown uniform and fine dynamic recrystallized (DRX) structure. Besides, the introduction of pre-deformation also promotes more second phases during caliber rolling. Therefore, the mechanical properties shown that the yield strength (TYS), tensile strength (UTS) and elongation after fracture (El.) of pre-extruded AZ80 caliber-rolled bars are 250 MPa, 440 MPa and 15%, respectively, which increased by 72%, 37%, and 88% compared with their as-cast counterparts. The improvement of mechanical properties is attributed to synergy effects of grain refinement, second precipitates strengthening and texture optimization.

1. Introduction

As the lightest structural metal, magnesium and its alloy has great potential for applications in automotive, aircraft, aerospace and 3 C fields, and it has become one of the fastest growing materials worldwide [1–5]. Especially, due to the threat of global warming, usage of lightweight materials become priority [6–8], but HCP structure of Mg alloy usually presented insufficient slip system, which in turn, poorer low-temperature formability compared with other structural materials, i.e. Al alloy. Generally, wrought products given better mechanical properties than as-cast magnesium alloys. In order to further expand the industrial application, more and more commercial wrought magnesium alloy systems have been developed, such as AZ, ZK, WE series, etc [9, 10]. Among various Mg-based alloys, Mg-Al systems are very popular and cost efficiency, for example, AZ80 alloy. However, large amount of alloying elements bearing lead to poor workability although the high strength can be obtained, and it was far behind the wrought Al alloys.

Studies have shown [11] that pre-deformation is an effective method to improve Mg alloy performance. Park et al [12–14], found that pre-cold forging before extrusion can introduce a large number of twins in Mg matrix, which can play significant role in promoting the recrystallization fraction and grain refinement through the twin induced recrystallization mechanism (TDRX). In addition, our previously work represented optimal extrusion parameters also given improvement of mechanical properties of Mg-Al-based alloy, for instance, low-temperature and low-speed extrusion shown ultrahigh yield strength over 400 MPa with better extrudability [15, 16]. Although such above studies can obtain fine microstructure, the strain rate is relatively low, which mean the production efficiency is not satisfied and not suitable for industrial mass production.
Recently, multi-pass caliber rolling as a novel approaching of large plastic deformation was applied for high-performance magnesium alloys [17–20]. The continuous accumulation of strain and deformation heat not only promoted the DRX with weaken texture, but also broken the second phases during deformation. Both particles fragment and dynamic precipitation can pin the dislocations and DRXed grain boundary [21]. Furthermore, the magnesium alloy prepared by caliber rolling has more small-angle grain boundaries than conventional extruded samples, which can inhibit the formation of microcracking, thereby improving the elongation and impact toughness of Mg alloys [22]. It is worth noting that more rolling pass will bring about failure of AZ80 alloy based on our foregone experiment. Therefore, in this study, the pre-extrusion was applied to AZ80 commercial Mg alloy subsequent by multi-pass caliber rolling. The microstructure and mechanical properties are investigated in detail.

2. Experimental

The chemical composition of the AZ80 magnesium alloy used in the experiment is shown in table 1. The extrusion of AZ80 alloy was carried out at extrusion temperature of 350 °C, extrusion speed of 3 mm s⁻¹ and extrusion ratio of 2.25. The pre-extruded rods were machined into Φ42 mm × 80 mm for caliber rolling. At the same time, the as-cast ingots with same size for comparison was also prepared.

The two kinds of machined test rods (as-cast: AC and pre-extruded: PE) were preheating at 350 °C for 0.5 h. Then the bar was caliber rolled at rolling temperature of 350 °C and the roll speed at 0.2 m s⁻¹ for 2, 4, 8 pass. During each pass, the bar was rotated 90°, and then the next pass of rolling is performed, as shown in figure 1. The last pass is rolled twice in order to straighten. When rolling was finished, all samples were cooling down at room temperature [23, 24]. The rolled bar produced from the as-cast sample is referred as ACR, and formed by pre-extrusion + rolling specimen is named as PER.

The samples used for microstructure analysis were cut from the center of bar, and after mechanical polishing, the picric acid etching solution (4.2 g picric acid + 10 ml acetic acid + 70 ml ethanol + 10 ml distilled water) was used for observation using optical microscope (OM: DM 2700) and scanning electron microscope (SEM: GSM IT500). The D8 Advance X-ray diffractometer was used to analyze the phases in different states of AZ80 and the diffraction intensity was calculated for texture analysis with the CuKα target and diffraction angle from 20° to 80°. Use transmission electron microscopy (TEM) and electron back-scatter diffraction (EBSD) to further characterize the microstructure of the samples. For TEM analysis, disc samples with a diameter of 3 mm were mechanically polished to a thickness less than 200 μm, and then were ion milled in a Precision Ion Polishing System (GATAN 691). In terms of EBSD specimen, the surface was first ground in the same way as the SEM sample preparation, then polished by colloidal silica for 30 min HKL Chanel 5 analysis software (OXFORD INSTRUMENTS, Shenyang, China) was used to analysis the EBSD data. In addition, tensile test of rolled samples was performed at room temperature using a SUNS-UTMS5105X universal tensile tester with deformation speed of 1.5 mm min⁻¹. The corresponding dog-bone tensile sample is shown in figure 2. The fracture morphology was observed using the SEM as well. All samples are tested three times to ensure consistency and repeatability of results.

| Alloy | Al     | Zn     | Mn     | Fe     | Si     | Cu     | Ca     | Mg     |
|-------|--------|--------|--------|--------|--------|--------|--------|--------|
| AZ80  | 8.587  | 0.530  | 0.216  | 0.002  | 0.012  | 0.003  | 0.001  | Bal.   |

Figure 1. Schematic drawing of the caliber rolling used in the present study.
3. Results and discussion

3.1. Workability enhancement

Figure 3 shows the macro morphology of the rolled AZ80 alloys with and without PE after different rolling passes, respectively. Obviously, the pre-extruded AZ80 alloy has more excellent surface finishing after caliber rolling. The surface quality of both alloys is intact after 2 passes. While after rolling 4 times, the edge of the alloy without PE began to crack. Further increasing the number of rolling passes, visible cracking occurred seriously along the rolling direction (RD), which means the maximum rolling pass of the as-cast AZ80 alloy was less than 5 passes. On the other hand, thanks to pre-extrusion treatment, the AZ80 alloy can be rolled for 8 passes without any cracks, which implied the PE deformation was significantly improved the plastic workability of AZ80 alloy. This may be due to grain refinement, uniform second phase and texture changing during the subsequent rolling process after the introduction of PE deformation and will be talked further in following sections.

3.2. Microstructural and texture evolution

As shown in figure 4, the microstructure of as-cast AZ80 alloy is mainly consist of α-Mg matrix and precipitated second phase distributed along the dendrite. It can be seen that the as-cast AZ80 alloy represented coarse dendritic morphology, The average grain size is measured about 120 μm, which consistent with the report of conventional Mg-Al series alloys. However, the existence of AlMn phase was not observed, which may be due to the lower Mn content in the alloy used in this study, which is different from other previous research [25, 26]. Table 2 shows the EDS results of second particles in both alloys. It can be found that, the continuous precipitated phases were identified as β-Mg17Al12. Further analysis shown network-like and lamellar morphology β-phase along the grain boundary or interior with similar concentration (see figure 4b).

The OM images of the pre-extruded AZ80 alloy is shown in figures 4(c), (d). Compared with figures 4(a) and (b), after pre-extrusion deformation, most of the second phases broken and distributed along the extrusion direction (ED). Table 2 also given the EDS results of Mg17Al12 phases, as shown in point 2 and 3, respectively. At the same time, the deformed grains are stretched along the extrusion direction somehow, as well as many fine equiaxed DRXed grains distributed around it. The size of the fine equiaxed grains is about 10–20 μm. The AZ80 alloy exhibits partial dynamic recrystallization characteristics after pre-extrusion deformation. Besides, the larger networked second phases from original as-cast structure is deformed and broken, and their shape changed into rod or granular. Such kinds of particles with size less than 10 μm usually given particle stimulate nucleation (PSN) effects. Moreover, the lamellar eutectic phase did not change significantly before and after pre-extrusion. Based on XRD profile as shown in figure 5, it can be found that the intensity of the β-Mg17Al12 diffraction peak
decreases after PE, which indicates that some the $\beta$-Mg$_{17}$Al$_{12}$ phase in AC condition is dissolved into the matrix during the pre-extrusion at 350 °C. According to the Mg-Al phase diagram, when the Al content is 8%, the corresponding temperature of the solid solubility is about 380 °C [27]. When the hot extrusion performed at 350 °C, both the friction between container and billet and the deformation heating generated would resulted in sharp increasing in the temperature inside the tool. Studies have also shown that the lamellar eutectic Mg$_{17}$Al$_{12}$ phase begins to coarsen at 290 °C, and re-dissolves into the matrix at about 370 °C [28]. Thus, the Mg$_{17}$Al$_{12}$ phase partial dissolved during extrusion at 350 °C.

Figures 6(a), (c) and (e) and (b), (d), (f) are microstructure of the as-cast AZ80 alloy after rolling for 2 and 4 passes, respectively. Obviously, there are large-sized unrecrystallized grains and recrystallized fine grains called bimodal structure, which shown partial DRX characteristics. After 4 passes, the size of non-recrystallized or deformed grains are further reduced, which in turn, the volume fraction of recrystallization increased significantly compared to the two-pass rolled alloys, which agreed well with other studies [29–31]. Besides, the second particles is also broken and distributed along the rolling direction (RD). In addition, there are lots of nano-scale second phases decorated around these broken second phases (figures 6(e), (f)), thus the second phases exhibited a micro-nano dual-size distribution. Studies have shown that the second phase with the micro-nano size distribution can greatly improve the mechanical properties of the alloys. In this experiment, it also shown some enhancement of mechanical properties but very limited due to their number is small.

Table 2. EDS results of corresponding precipitates marked in figures 4b and d.

| Point | Element | Atomic percentage (%) | Possible phase  |
|-------|---------|-----------------------|----------------|
| P1    | Mg      | 62.4                  | Mg$_{17}$Al$_{12}$ |
|       | Al      | 37.6                  |                |
| P2    | Mg      | 58.2                  | Mg$_{17}$Al$_{12}$ |
|       | Al      | 41.8                  |                |
| P3    | Mg      | 68.7                  | Mg$_{17}$Al$_{12}$ |
|       | Al      | 31.3                  |                |
Figure 7 shows the microstructure of the pre-extruded AZ80 alloy in different rolling passes. It can be found that with the increase of rolling passes, the alloy grains continue to refine. The volume fraction of recrystallization and the precipitation of the second phase gradually increased. In case of 8 passes of rolling, the alloy has completed DRXed structure. The second phase precipitated during the rolling process distributed homogeneously. As shown in figures 7(a), (b) with 2 passes of rolling, the microstructure showed partial dynamic recrystallization behavior. However, compared with the pre-extruded alloy structure (see figure 4(c)), the recrystallized volume fraction increased remarkably. When rolling up to 4 passes (see figures 7(c), (d)), most of the grains become equiaxed, and no large-sized deformed grains are observed, which means the DRX is basically completed. Meanwhile, the amount of the second phase is also dynamically precipitated simultaneously. In case of 8 passes (see figures 7(e), (f)), it is clear that the recrystallization of the alloy is totally complete and the grain size is much finer than that of the 4 passes specimen. Such finer grains can greatly increase the room temperature tensile strength and will be discussed in next section.

In order to further study the distribution and changes of the second phase during the rolling process of the PEed AZ80 alloy, the SEM observation also carried out, as shown in figure 8. Obviously, as the number of rolling passes increases, the second phase is continuously refined and extensively distributed along the RD. Furthermore, the number density of second phases precipitated in matrix increased a lot. This results also consisted with the increase in the diffraction peak intensity of the $\beta$-Mg$_{17}$Al$_{12}$ phase in figure 5(b). From figure 8(d), it can be seen that a large number of nano-scale second phases are dynamically precipitated during the rolling process. Generally, this nanometer-sized second phase can cooperate with the broken micron-level second phase during the rolling process to strengthen the matrix and greatly improve the mechanical properties of the alloy [32].
In addition, a large number of dislocations introduced by PE can be used as the nuclear sites for dynamic recrystallization. Therefore, the recrystallization volume fraction of the pre-extruded alloy during rolling is higher than that of the as-cast alloy under corresponding conditions. Similarly, the nano-scale precipitates occurred during the rolling of the PEed alloy is also higher than that of the as-cast counterpart, which is due to the large number of dislocations introduced in PE process, and the increase in the degree of supersaturation of the $\alpha$-Mg matrix after the PE process. Thus, the dynamic precipitation of nano-scale second phases were accelerated.

Another attention should be paid to the texture changes of as-cast and pre-extruded AZ80 alloys during rolling. Figure 5 shows the XRD patterns of AZ80 alloy under different circumstance. The cross-section plane was chosen for observation. Hereinafter, $I_{0002}/I_{1010}$ is used to represent (0002) basal plane diffraction intensity. The smaller the value, the stronger the basal plane texture.

As calculated in figure 5(a), the basal texture intensity ratio of the as-cast sample is 1.16, which implied that the basal plane texture is not obvious, that is, its grain orientation has no preferred orientation. When rolling 2 times, this value decreased sharply from 1.16 to 0.15. With rolling passes further increased to 4 passes, the (0002) basal plane diffraction intensity continued to decrease to 0.06, indicating that the basal texture strengthened. This above changes mean that the grains rotate to form a strong basal texture quickly, which is not conducive to the subsequent deformation. The above analysis explains the poor plastic workability of the as-cast alloy during direct rolling deformation.

Figure 6. OM (a)–(d) and SEM (e), (f) images of the as-cast alloy under different rolling passes: (a), (c), (e) ACR-2p, (b), (d), (f) ACR-4p.
Moreover, the basal plane diffraction intensity is reduced to 0.35 of as-cast AZ80 alloy after PE treatment (see figure 5(b)). The basal plane diffraction intensity is low, which means most of the crystal orientation of grains is parallel to the ED. During caliber rolling, the (0002) basal plane diffraction intensity increased after 2 passes and then decreased, representing the basal texture weakens firstly and then gradually intensive with the increase of rolling passes. This interesting trend of the basal surface texture in PE + Rolling alloy is different from that of AC + Rolling alloy. The reasons can be explained as follows:

In the initial stage of rolling, most grains’ orientation is parallel to the RD, because a strong basal fiber texture already formed during PE. So, the Schmid factor is approximately zero and hardly started to slip. It is easy to form additional internal stress among the grains to distort the local area near the grain boundary. Therefore, dynamic recrystallization can occur in the region with severe lattice distortion to form fine new grains. The newly generated DRXed grains are generally distributed in a ring shape around the grain boundaries of the original coarse grains, showing different grain orientation characteristics from their parent grains, thereby weakening the basal texture intensity and increasing the texture dispersion, which also makes the sliding easier. Thus, there is not difficult to understand the phenomenon in AC + Rolling alloy in figure 3(a) showing cracks when rolled up to 5 passes.

In order to further analyze the influence of pass rolling processing on the texture of pre-extruded AZ80 alloy, EBSD technology was used to characterize the microstructure and texture of AZ80 alloy after 4 and 8 passes, and

![Figure 7. OM images of the PE alloys with various rolling passes: (a), (b) 2p, (c), (d) 4p, (e), (f) 8p, respectively.](image)

Figure 7. OM images of the PE alloys with various rolling passes: (a), (b) 2p, (c), (d) 4p, (e), (f) 8p, respectively.
Figure 8. SEM images of the PE alloys upon different rolling passes: (a) 2p, (b) 4p, (c) 8p, (d) Enlarged images of the rectangular area in (c), respectively.

Figure 9. Inverse pole figure (IPF) maps of PE alloy rolled at (a) 4 passes and (b) 8 passes and (c) B-C cumulative misorientation in (b), volume fraction of DRX in (d), (f) 4-pass sample and in (e), (i) 8-pass specimen, IPFs of (g) 4-pass sample and (h) 8-pass samples, respectively.
the results are shown in figures 9(a)–(i). The grain orientation reconstruction results of both alloys show that after pre-extrusion pass rolling, with the increase of rolling passes, the grains are further refined and becomes more uniformly, which is consistent with previous OM observation. For example, after 4 passes of rolling, it was found that recrystallized grains with a grain size of about 1–3 μm appeared near the deformed coarse grains, forming a necklace-like bimodal structure. This phenomenon has also been reported in other research [33]. In addition, the recrystallization fraction and corresponding distribution of 4-pass and 8-pass specimen are shown in figures 9(d), (f) and figures 9(e), (i), respectively. It is clearly that from distribution histogram the volume fraction of DRX increases as the number of rolling passes increases. The recrystallization is almost complete after 8 passes rolling. In order to explore the recrystallization mechanism, figure 9(c) shows the results of the cumulative misorientation (Line B-C) from the inside of the grain to the vicinity of the grain boundary. It can be seen that the cumulative misorientation is greatly increasing, the difference in orientation from the center to the edge of the grain boundary region is continuously increasing, which means typical CDRX. Furthermore, there is also a phenomenon of grain boundary bowing in some areas (marked A and D) in figures 9(a), (b), which indicates that there is also a DDRX involved.

In order to characterize the texture changing of the as-extruded AZ80 alloy after various passes rolling, the inverse pole figure (IPF) results are shown in figures 9(g), (h). After extrusion and rolling, the texture configuration of AZ80 alloy is (0110) crystal orientation parallel to the rolling direction (RD). Further statistics of the texture intensity represented that with the increase of rolling passes, the texture intensity changed from 4.41 μd for 4 passes to 2.23 μd for 8 passes, respectively. According to the analysis of the change of the recrystallization fraction, it can be known that the weakening of the texture strength mainly comes from the increase in the DRX fraction, which agreed well with our previous research and other reported results in wrought magnesium alloys [15]. That is, the recrystallized grains tend to show more weaker basal texture than that of the deformed grains. This kind of features has also been confirmed by T. Mukai et al in caliber rolled AZ31 alloy [34]. Such texture variation also result in difference mechanical properties, which will be discussed in next section.

Figure 10 exhibited detailed microstructure characteristics of the AZ80 Mg alloy rolled by 8 passes using TEM. Generally, as shown in figures 10(a) and (b), two kinds of second particles with different morphologies are
According to the indexing results of the selected area electron diffraction pattern (SADP), combined with the XRD pattern, it is concluded that the second phases are profuse spherical Mg$_{17}$Al$_{12}$ phase and rare cubic Al$_8$Mn$_5$ phase, respectively, which are consistent with other AZ80 Mg alloy research [26]. In addition, tiny $\beta$-Mg$_{17}$Al$_{12}$ phases are mainly dynamic precipitated during thermal-mechanical processing. According to the Zener drag theory [35], these second phases can block the growth of recrystallized grains by pinning effect, thereby contributing to the refinement of the rolled alloy, which is consistent with previous researches [36]. Especially, more attention are paid to the sub-microstructure and interaction between these particles and dislocations, as can be found in figures 10(c) and (d), respectively. It can be found that there are a large number of dislocations after rolling (see figure 10(c)), and they are are mostly rearrange to substructures. Therefore, it can be concluded that during the rolling, deformation-induced dislocations and sub grains have an important contribution to the DRX nucleation. In addition, as can be seen from figure 10(d), the nano-scale Mg$_{17}$Al$_{12}$ phase is entangled with dislocations, which hinders the movement of dislocations and results in the enhancement of mechanical properties of rolled alloy.

3.3. Mechanical properties

Figure 11 shows the room temperature tensile properties of the pre-extruded AZ80 alloy under different rolling passes. It is easy to find that as the number of rolling passes increases, the mechanical properties of the alloys significantly improved. Especially, the alloy has the best mechanical properties after 8 passes of rolling (PER-8p). The yield strength (TYS), tensile strength (UTS), and elongation (El.) are 245 MPa, 398 MPa, and 21%, respectively. Compared with the pre-extruded AZ80 alloy, it has increased by 77.5%, 46.3%, and 109.3%, respectively. In addition, it should be note that the properties of the alloy after 4 rolling passes are relatively higher than that of other AZ80 alloys prepared by extrusion [32, 37].

The enhancement in strength of caliber rolled AZ80 alloy can be explained when considering following two main strengthening aspects:

Figure 11. Mechanical properties of the pre-extruded alloy under different rolling passes at room temperature: (a) engineering stress-strain curves, (b) TYS, UTS, El., respectively.
(i) Grain refinement. According to the Hall-Petch law, \( \sigma = \sigma_0 + kd^{-1/2} \). Where \( \sigma \) is the yield strength of the alloy material, \( \sigma_0 \) is the frictional stress, \( k \) is the Hall-Petch coefficient, and \( d \) is the grain size. From above mentioned microstructure analysis, the grain size of the rolled alloy is continuously refined with the increase of rolling passes. The Hall-Petch coefficient of magnesium alloys is relatively large [38, 39], so the refinement of grains has greatly improved the mechanical properties indeed.

(ii) Second phase and dynamic precipitation. It can be seen that the larger second particles broken during thermal-mechanical processing as well as number of nanoscale phases dynamically precipitated with the increase of rolling passes. The well-proportioned precipitated nano-level second phase can strengthen the matrix synergistically with the micron-level second phase broken during the rolling process. Besides, these nano-level second phases can inhibit the growth of recrystallized grains by pinning effect.

Besides, the texture evolution with rolling passes increasing also partially influence the strength response. According to XRD and EBSD analysis, the deformed grain with higher intensity and DRXed grain with random texture would give different effects on mechanical properties and will be discussed in near future.

The elongation of the alloys is significantly improved after rolling, which is attributed to grain refinement. On the one hand, because the original coarse particles were continuously broken and refined during the rolling process, the hard and brittle cracking is greatly weakened, thereby improving the toughness of the alloy. On the other hand, it can be found that the elongation of the 8p alloy is slightly lower than that of the 4p alloy, which may be due to the reduce of rolling temperature with increased rolling passes.

Figure 12 given the tensile fracture morphology of the PEed AZ80 alloy under different rolling passes. All alloy has the characteristics of tearing edges and dimples, indicating the fracture mode is ductile fracture. The number of dimples increased, and the tearing edges between the dimples became more obvious with rolling pass increased. It is generally believed that the generation of tear edges requires the participation of cross slip, and a certain degree of plastic deformation occurs during tearing, so the PE + Rolling alloy shows good balance of strength and toughness. In addition, a brittle fractured second phases can be found in the fracture of the PE + Rolling alloys, and a penetrating crack was observed, which presumed that the crack originated or expanded from the second phase. At the same time, the number of brittle fractured second phases gradually increases too, because with the increase of the number of rolling passes, the bonding between the matrix and the
second phase interface becomes stronger. These medium-brittle second phase is not easy to fall off, oppositely, when the stress is raised to a certain extent, brittle fracture occurs.

4. Conclusion

In summary, a high strength AZ80 alloy was successfully fabricated by pre-extrusion and caliber rolling process, and the effect of pre-extrusion deformation on the microstructure and mechanical properties is investigated in detail. Based on above results and discussion, the follow remarks about beneficial of Pre-extrusion (PE) can be drawn:

1. PE deformation can significantly improve the workability of AZ80 alloy. Introduction of PE promotes the occurrence of DRX behavior at the very beginning of rolling, which conduct easier movement of basal slip for subsequent deformation.

2. The grains are significantly refined and the basal texture along the RD is weakened after PE+caliber rolling. Besides, the precipitation of nano-scale second phases during rolling is also significantly promoted by PE.

3. PE combined with caliber rolling can remarkably enhanced the mechanical properties of AZ80 alloy for almost 400 MPa in UTS with reasonable elongation, which is mainly due to the grain refinement, precipitation strengthening and texture optimization.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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