Reinforcing effects of cyclic expansion extrusion with an asymmetrical extrusion cavity (CEE-AEC) on pure magnesium

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Abstract
In this paper, a relatively novel severe plastic deformation method cyclic expansion extrusion with an asymmetrical extrusion cavity (CEE-AEC) was carried out to prepare large-sized pure magnesium (Mg) with high comprehensive performance. Finite element analysis (FEM) was used to study the plastic deformation process and Electron Back-Scattered Diffraction (EBSD) was aimed to research the microstructural evolution. Three passes and isothermal deformation at 250 °C were chosen to satisfy the research. The central part of the billet was cut as the research area, and the resultant microstructure and mechanical properties were analyzed systematically. The results showed that the grain size was remarkably refined due to continuous dynamic recrystallization (CDRX) and discontinuous dynamic recrystallization (DDRX). Twins participated in the progress of deformation. The shear strain introduced by the asymmetrical extrusion cavity determined the formation of inclined basal texture, leading to the increasing value of basal $\langle a \rangle$ slip system Schmid factor. The best comprehensive tensile properties were obtained after three passes of deformation, and the contributions came from grain refinement strengthening and texture modification, respectively.

1. Introduction
Magnesium (Mg) alloy attracted more and more attention for its lightweight and specific strength [1–3]. Moreover, specific performance in some fields such as damping, stability, and recovery, reflect the huge economic and application value in the manufacturing industry [4–6]. At present, because Mg alloy has smaller atomic masses than other metals, it has been applied widely in aviation and other high-tech industry [7]. Compared to other alloys, however, it’s also limited by its poor ductility in wide production and application, and the important cause is the existence of strong basal texture or sheet texture limits the slipping deformation [8–11]. How to achieve the weaker and non-basal texture has been the main research content in the application field of Mg alloy.

Aimed to handle the hard plasticity in Mg alloy, Severe plastic deformation (SPD), works as a relatively mature technology and obtains the amounts of efforts in the field of material processing [12–15]. During the progress of the SPD methods, researchers have concluded some representative techniques like equal channel angle extrusion (ECAE) [12], accumulative rolling bonding (ARB) [13], and cyclic extrusion and compression (CEC) [14]. By the way of SPD, materials obtain the deformation with the large ratios for the excellent superplastic properties. Plenty of studies have proved it. B. Sulkowski et al [14] made the conclusion that pure Mg after 4 passes of CEC gets the smaller size and better performance. W. Lei [15] studied the microstructure of pure Mg become homogeneous for the accumulation of large strain.

People came to the conclusion that the evolution in microstructure made a close link to the changes of properties, so many studies in deformation mechanism were done [16–18]. Slip and twins play the important role in deformation [19, 20]. At room temperature, the basal slip is easy to be activated, due to the lower critical resolved shear stress (CRSS) than others [21]. According to Mises criterion, twins frequently work as the supplementary deformation modes to satisfy the condition of homogeneous deformation [22]. And the
coordination function of twins is due to different orientations from the matrix [23, 24]. Zhang et al [25] found that twin-twin interactions accommodate the recrystallization nucleation position for the homogenous microstructure with finer grains. Twins contribute to the process of further deformation mode that slip and recrystallization.

However, many methods to improve performance were only suitable for billets with small sizes. Cyclic expansion extrusion (CEE) [26] endows materials with better ductility and strength by extruding through a mold cavity where materials are expanded in the cavity and extruded with large size. In this study, on the basis of the former technique, the introduction of extra sheer stress from the asymmetric cavity creates more possibilities for improvement in product performance. To investigate clearly the deformation mechanism, pure Mg was chosen as the experimental object. Subsequently, aimed to figure out the advantages brought from cyclic expansion extrusion with an asymmetrical extrusion cavity (CEE-AEC) and relate the properties with the microstructure, a series of researches were carried out.

2. Experimental procedure

As-cast pure magnesium was chosen as materials in this experiment. The billet was 200 mm × 100 mm × 50 mm and homogenized for 18 h before extrusion. As shown in figure 1(a), it was extruded by CEE-AEC with three passes at 250 °C, and the sample was cut from the center of billets. During the process of CEE-AEC, the punch moved down at 1 mm s⁻¹. Under the press, the billet filled the cavity. Then the block at the bottom was taken away and the second billet of the same size was put into the cavity from the above. Finally, the first billet was squeezed out of the cavity. A full process operated in a cycle. The parameters of the die cavity are \( L_1 = L_3 = 50 \) mm, \( L_2 = 100 \) mm, \( \alpha_1 = \alpha_2 = 138^\circ \), and \( H = 5 \) mm as shown in figure 1(b).

Finite element analysis (FEM), an effective method to simulate the process of reality, is put forward to verifying the condition of deformation in the cavity during the extrusion. Designed models with origin sizes were made as shown in figure 1(a) and introduced into DEFORM-3DV11.2 for further simulation. In pre-processing, the billet was defined by a plastic body with a mesh number of 50000 at the temperature of 250 °C and others were rigid objects. The punch with a falling speed of 1 millimeter per hour. After the parameters finishing designed, the database was generated to run for the resolved results. Finally, relevant data was output in postprocessing.

As shown in figure 1(c), the samples were cut from the center of the billets, and the red face was the observed surface in the experiments. The microstructures of samples extruded by single and multi-pass CEE-AEC were observed by Hitachi SU5000 scanning electron microscopy (SEM) and Axio Observer A2m Carl Zeiss optical microscope (OM). Microstructure evolution in each pass was researched by Electron Back-Scattered Diffraction (EBSD). The data collected by EBSD was handled and analytical processing was developed by the TSL OIM™ software. The tensile specimens in the center of extrusion from each pass were cut along the ED, and extension tests of room-temperature tensile property were developed on 3382 Instron universal material experiment machine at a strain rate of \( 1 \times 10^{-3} \) mm s⁻¹. The tensile yield strength and other data of the extension test were processed by the extensometer of universal material and analyzed by the Blue Hill software.

3. Results and discussions

3.1. FEM analysis

As shown in figure 2(a), the finite element analysis reveals the process of the CEE-AEC. The whole deformation process can be broadly divided into three stages: the initial stage; the intermediate stage and the ultimate stage.
A variation tendency is present from figure 2(b), in the process of CEE-AEC, which simulates the changes of strain located in the asymmetric extrusion outlet from left to right. During the extrusion, the black line presents a bimodal structure and the second peak gradually increases to the approximation of the first peak value with the punch down. After squeezing 30 millimeters, one-seventh of the whole billet, the state of strain is shown by the red curve. The distribution of strain is high in the middle and low on both sides which is a single peak curve and it lasts for a long time. The intermediate phase takes up a large part of the whole deformation. The strong and long-time strain occurs in the middle of the billets. Therefore, it’s of great value to do the research on the middle part. Ultimately, the rest are squeezed out where a bimodal structure appears and the figure of the middle part has been decreasing. In addition, the difference that strains on the left is higher than the right one brought by the asymmetric cavity.

3.2. Microstructures under different passes

Figure 3 displays the inverse pole figure (IPF) maps of the CEE-AEC in each pass. As shown in figure 3(a), the relationship between color and orientation is in the upper right corner. An obvious shear band through the large grain along the ED can be observed in the first pass, due to the shear stress caused by the asymmetric cavity, and the shear band provides the nucleation places [27]. A large amount of the low-angle grain boundaries (LAGBs) which are signed by the white line. Continuous dynamic recrystallization (CDRX) which fine grains with different orientations occur within the large grains and discontinuous dynamic recrystallization (DDRX) which fine grains appear at grain boundaries exist together during deformation progress. The initial grains are broken and formed into new grains with small sizes within the shear bands, and most new grains hardly rotate due to inadequate deformation which brought from the first extrusion. A typical bimodal structure that large-size grains are surrounded by small-size ones is generated. Besides, extreme micro-grains with non-basal orientation appear at the inner part of few large grains.

After two passes, further refining and more uniform distribution occur that fine grains surround the middle ones which is also a bimodal structure. Compared to the first deformation, the obvious changes of microstructure can be seen that DRX and twinning. Besides the grain boundaries, the interior of the grains is also an important area for dynamic recrystallization (DRX). The grains generated by the way of DDRX, compared to
the one-pass CEE-AEC, the increment of them in size can be seen. A large number of twins through the whole grains can be seen and equally distributed which shows the different grain orientation from basal plane orientation. Twins occur in the large grains with basal plane orientation which provides enough sites for twins and the growth of twins is hindered by the grain boundary. More grains with finer size occur after the triple CEE-AEC and the major size concentrates relatively. The same phenomenon occurs that the large grains break up into smaller sizes. The further deformation leads to twins nucleating and growing up, and twins become short and small which exist within grains. Compared to the previous deformation, the orientation of grains shows a trend that major grains are with basal orientation which is signed by red in figure 3. During the multi-pass CEE-AEC, the white lines which represent LAGBs make the declining trend, especially the transform from one-pass CEE-AEC to two-passes one. The bimodal distribution of grains exists persistently though grains have been refining with CEE-AEC increasing, and the orientation of grains becomes richer. According to the former description, an obvious microstructural evolution exists that microstructure becomes more homogeneous. A nonuniform deformation brought by the first CEE-AEC, the initial grains break up and recrystallization begins in this process. Under the press, large grains are separated and the fine grains are formed at stress concentration. In the second CEE-AEC, twins occur in large grains. With the following deformation, more power contributes to the growth of fine grains and further refinement of coarse grains. After the third CEE-AEC, there are no obvious large grains left, and a more homogeneous microstructure was obtained.

It can be seen from figure 4, mostly grains in the first pass with the large size is up to about 200 μm. The deformation of pure Mg after the first pass is not uniform. After the second deformation, the size of grains between 120 μm and 250 μm is none and others increase at a little level. The trend concentrates around the middle part which counts from 10 to 50. After the third CEE-AEC, no large grains are left and deformation is more homogeneous. Then multi-pass CEE-AEC is completed, the strongly unimodal trend emerges apparently and the final average is about 26.82 μm. Compared to the former passes, the distribution of size presents the central tendency. Besides, the average size is decreasing as the number of passes increasing, which indicates the outstanding transform, and the range reduces from one pass to three passes by CEE-AEC.

According to the pole figures, a transform that distribution of strong texture gets dispersive is concluded from them. It can be concluded from figures 5(a) and (d) that the two strong poles are around the basal plane along with TD, and the angle of difference between the strong and basal pole is low, which is consistent with the IPF. Combined with the IPF, many cylindrical oriented grains occur in the shear band. According to it, the cylindrical oriented grains make a tendency that the distribution of whole pole figure is along with ED. Besides, the low fraction of cylindrical oriented grain shows a weak texture. The strong points exist near the focus of the IPF for a large amount of basal body with an orientation close to (0001). DDRX grains with different orientations from the original grains are generated during hot deformation, which can weaken texture [28]. The interaction between tension twins and DRX grains makes the same function [29]. Twins contribute to the changes in the pole figure. As shown in figures 5(b) and (e), with the process of CEE-AEC, partial cylindrical oriented grains with large sizes occur and weaken the texture. Then a mind is put forward that recrystallized grain growth may happen, however, average size decreases for the reason that the majority of grains are broken by stress. With the further grain refinement after three passes, the texture spreads along with ED evidently as shown in figure 5(c), though texture strength increases slightly which may be caused by the growth of DRX grains. On the whole, the texture has a consistent distribution along with ED and strength makes a declining trend. The changes of texture and grains in CEE-AEC indicate that grain refinement and texture adjustment are useful methods of improving properties [30].

As shown in figure 6, a change of misorientation angle exists with passes increasing. After one-pass CEE-AEC, for the reason that inadequate deformation, many large grains aren’t broken and keep the relatively...
consistent orientation. Related to the IPF and loading features, it’s acknowledged that most grains are compressed vertically to C-axis. When it’s stretched along or compressed perpendicular to the C-axis, tensile twins are prone to occur. During the progress of extension in one-pass CEE-AEC, many large grains with basal fiber texture exist which are broken up from the initial structure and LAGBs reach up to nearly 70 percent. Excessive LAGBs express the message that huge grains are bad for the rotation of broken grains. In the subsequent processing, large grains are broken into the finer ones and rotated which contribute to more well-distributed data of ma. Especially, the increasing distribution of angles around 30° and 85° is significant which expresses a message that tension twinning gets more. According to the IPF, the obvious twins within grains are hardly left and the cause is that the growth of twins occurs which is also beneficial to grain refinement.

### 3.3. Mechanical properties and discussion

Figure 7 shows the curve diagram about mechanical properties at room temperature. According to it, the curves all increase steadily in the early stage and decrease suddenly when getting the peak value. The values of mechanical properties are summarized in table 1, the obvious tendency can be concluded from it. After one-pass CEE-AEC, a fracture occurs when the elongation reaches about 3.4%, for the reason that existence of large grains brought by the inhomogeneous deformation. High performances aren’t achieved in elongation and strength at
the same time [31]. With passes increasing, the tensile yield strength (TYS) presents the stable decline trend, ultimate tensile strength (UTS), and elongation (EL) present the rising trend. Judging from the trend, it can be concluded that the advantages brought by multiple extensions are more likely to generate plastic deformation with better results. In the aspect of per-pass slope, with passes increasing, the primary values of slope decrease, and the process of extension last longer. After three-passes CEE-AEC, it can be noticed that the mechanical properties get a great improvement, UTS, TYS, and EL are respectively measured to \(\sim 103.51\) MPa, \(\sim 43.41\) MPa, and \(\sim 21.5\%\).

The properties have a close relationship with microstructure, it’s obvious that the grain refinement as the common optimizing way greatly improves the capability of deformation. From the perspective of the fracture appearance, dimples and tear ridges are the typical features in the analysis. Figure 8 shows the fracture appearance of each pass. The number and depth of dimples have been adding with the more extrusion passes. In one-pass CEE-AEC, few tear ridges are observed in the SEM micrograph. Few small dimples are formed. Figure 8(b) shows a large number of dimples and cleavage planes, these characters of fracture are observed clearly. Besides the phenomena of former passes, it’s noticed that a huge dimple was formed which was made up of many dimples with small size. According to SEM of the fractures, the number of dimples and torn edges

Figure 7. Stress-strain curve of the tensile property after each pass at room temperature.

Figure 8. The fracture morphologies of samples after different CEE-AEC passes: (a) one-pass, (b) two-passes and (c) three-passes.

Table 1. The mechanical properties (TYS, UTS, EL) of CEE-AEC processed samples with per pass.

| Passes | TYS/MPa | UTS/MPa | EL/A% |
|--------|---------|---------|-------|
| One    | \(\sim 54.28\) | \(\sim 74.91\) | \(\sim 3.4\) |
| Two    | \(\sim 46.42\) | \(\sim 86.42\) | \(\sim 8.7\) |
| Three  | \(\sim 43.31\) | \(\sim 103.51\) | \(\sim 21.5\) |

Mater. Res. Express 8 (2021) 056502 Ji et al
increases significantly, which shows better ductility. It’s consistent with the values change in grain size and mechanical properties. It’s summarized that grain refinement improves the plasticity in pure Mg and materials with multi-pass deformation have excellent performance.

The major mode of Mg alloy during deformation is the interaction between slip and twinning [32]. Combined with the pole figure, Schmid Factors of basal slip per CEE-AEC were investigated. Figure 9 shows the Schmid factor of (0001) \(\langle 11\overline{2}0\rangle\) after different passes. After one-pass CEE-AEC, a strong basal fiber texture (12.2) was formed which makes the hard orientation and acquisition of much distribution in low Schmid factor, due to inadequate deformation resulting in large grains and a small number of grains rotating. It’s acknowledged that the basal \(\langle a\rangle\) as the main pattern is triggered when the strain perpendicular to the \(c\)-axis in Mg and its alloy [33]. In terms of the data about the Schmid Factor in each pass, the distribution is different but the average is about 0.3, which indicates the high possibility of slip in (0001) \(\langle 11\overline{2}0\rangle\). For this reason, the TYS decreases with the increment of passes. As we all know, monocrystal magnesium shows better extensibility in the \(c\)-axis than the \(a\)-axis. With the increased passes, the basal plane keeps a stable quantity and basal slip occurs easily. During the process of extension test, the basal plane parallel to the stretching direction, for which the basal slip is easily actuated. It’s reported that extension twinning makes more contribution with strain increasing [34]. The change rules of property conclude that the complexity and ratio of the plastic deformation make an excellent performance. It’s summarized that grain refinement mechanism and basal slip make a major effect on properties.

4. Conclusion

CEE-AEC, a novel method to refine grains and improve properties, is investigated in this paper. Its core advantage is the shear strain brought by the asymmetrical cavity and the practicability of preparing the large billet. The pure Mg was chosen as the experimental object. After three-passes CEE-AEC, obvious variation trends were shown and some conclusions were made as follows:

(1) During the CEE-AEC, the typical bimodal microstructure exists that coarse grains are surrounded by finer ones. Obvious deformation bands are formed through the one-pass CEE-AEC. After multi-pass CEE-AEC, the grains become more well-distributed and finer, the final average size is up to \(~26.82 \mu m\).

(2) With the grain refinement, LAGBs make a declining trend and the tension twinning makes a continuous enhancement, which indicates the formation of a homogeneous microstructure.

(3) The mechanical properties make an excellent performance and variation tendency is obvious in each pass. With the increasing passes, the texture intensity gets weaker and more grains with non-basal orientation are generated, the orientation of major grains transforms from TD to ED. Besides, the causes of improvement in ductility are grain refinement after CEE-AEC and basal slip during tension test.

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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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