The improvement of grain refinement, texture modification and mechanical properties of pure Mg prepared by cyclic expansion extrusion with an asymmetric extrusion cavity

Yusha Shi, Jie Zheng, Jinsheng Ji, Heng Zhang, Zhimin Zhang, Qiang Wang and Yong Xue©
School of Materials Science and Engineering, North University of China, Taiyuan, 030051, People’s Republic of China
E-mail: yongxue395@163.com

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Abstract
In this article, a new type of severe plastic deformation process named cyclic expansion extrusion with an asymmetric extrusion cavity (CEE-AEC), was used to prepare pure magnesium with three passes repetitive deformation at 250 °C. This article mainly studies the microstructure evolution, texture analysis and mechanical properties of pure magnesium. The results display that the grain size is refined, from 74 um to 19 um, which is attributed to continuous dynamic recrystallization (CDRX) and discontinuous dynamic recrystallization (DDRX). With the increasing CEE-AEC passes, the texture is weakened, and the (0001) plane of a large number of grains are inclined from the ED direction. The Schmid factor that activates the basal slip system gradually increases from 0.29 to 0.3 after three passes. After three passes of CEE-AEC, from the tensile tests of the deformed samples at room temperature, it can be seen the samples have excellent comprehensive mechanical properties, the tensile yield strength (TYS) is 54 MPa, ultimate tensile strength (UTS) is 110 MPa, and fracture elongation is 19%.

1. Introduction
Magnesium (Mg) and its alloys are characterized by low density, high specific strength and high rigidity, have a wide range of applications such as automotive, transportation, aerospace, electrical communication and other fields [1, 2]. Nevertheless, they are restricted in these fields due to their limited strength, poor plasticity and low formability. Because the crystal structure of magnesium alloy is hexagonal close-packed (HCP), and at room temperature, the activation of non-basal slip is not easy, the mechanical properties of magnesium alloy are decreased [3].

In recent years, severe plastic deformation (SPD) has important value for the preparation of ultrafine-grained (UFG) materials with high strength and toughness, and has attracted the attention of researchers [4–6]. There are many methods of SPD, such as equal channel angular pressing (ECAP) [7], high pressure torsion (HPT) [8], cyclic extrusion compression (CEC) [9] and cyclic expansion extrusion (CEE) [10], which have attracted great interest in the scientific community in recent years. The initial grain size is refined under the application of large plastic strain, and the ultra-fine materials with excellent mechanical properties are prepared. Among these methods, as studied by Lei et al [11], with the increasing ECAP passes, due to the accumulated large strain, the crystal grains of pure magnesium samples are further refined and the microstructure becomes homogeneous. The CEC process activates the dynamic recrystallization of twins in deformed magnesium. As reported by Sulkowski et al [9], Severe plastic deformation has a certain impact on the grain refinement and recovery mechanism of magnesium. CEE has not complicated die mold settings and back pressure system. Therefore, when process the sample of the required passes without removing it from the mold until all passes is completed. However, the size of CEE samples is small and there is no shear strain, which limits the improvement of the comprehensive mechanical properties of the alloy [12, 13]. The current research introduces shear strain by...
adding asymmetric die geometries, and retains the advantages of the combination of CEE compression and
tensile stresses, which solves the limitations of CEE, as reported by Yan et al [14]. Therefore, a new method of
SPD is proposed based on CEE. The new SPD method is called CEE-AEC. It’s expansion cavity of the die is provided with differential angles
on both sides, and the heights of the differential angles on the two sides are different. In this method, the billet is
upset and then extruded in multiple passes, and the metal flow state is changed by changing the structure of the
concave mold cavity. Introducing shear deformation to achieve strain growth can not only reduce the number of
deformations, but also increase the strain effect, improve the production efficiency, refine the grain structure,
optimize the second phase structure [15, 16].

CEE-AEC has the advantage of increasing differential angle and introducing shear deformation. Generally, CEE deformation is a combination of upsetting and extrusion deformation. Although severe plastic
deformation can be achieved, there are many passes and the test takes a long time. CEE-AEC increases the
differential deformation of the concave model cavity, introduces shear deformation force, increases the strain
effect, shortens the test process to reach the target strain, and improves efficiency.

As a new type of SPD process, we have studied the effect of CEE-AEC on AZ31 and Mg–Gd–Y–Zn–Zr [6, 14–16]. However, CEE-AEC has not been studied on pure magnesium so the influence on the microstructure and mechanical properties of pure magnesium has not yet been confirmed. Thus, in this paper, we analyzed the
microstructure evolution and mechanical properties for the edge of pure Mg with three passes repetitive deformation CEE-AEC at 250 °C.

2. Process of CEE-AEC

The CEE-AEC process is divided into five steps, the specific steps of the process are shown in figure 1. First of all, put the bulk-shaped block at the bottom of the mold for expansion and deformation, and then put the billet into
the mold. As the punch pressed down, the billet expanded to fill the cavity. Secondly, removed the previous block and put in another billet that was exactly the same as the first billet. As shown in figure 1(a), as the punch pressed down, the second billet filled the cavity and replaced the first billet. Finally, the billet 2 extruded the billet 1 out of the cavity and filled the expansion area. Among them, the initial cavity (L1) and the extrusion cavity (L3)
had the same size. In addition, the billet after one pass was rotated 180° in the extrusion direction, and then
repeatedly extruded until the required pass was satisfied.

3. Experimental procedure

The material used in this experiment was high purity magnesium (pure Mg; Mg ≥ 99.99%, other alloying elements
≤ 0.01%) rectangular block with a dimension of 200 mm × 100 mm × 50 mm. Firstly, the as-cast billet was
homogenized for 12 h at 250 °C. The size of the sample from edge of billet was 10 mm × 10 mm × 10 mm.
Observed and analyzed the surface parallel to the extrusion direction, grinded and polished it, then electropolished
the sample at minus 15 °C, the electrolyte was a mixed solution of perchloric acid and ethanol.
Cut a sample parallel to the extrusion direction from the billet, and then performed an electron backscatter diffraction (EBSD) test on the sample. Before the test, ground with sandpaper and mechanically polished the observation plane, which was the extrusion direction (ED) - transverse direction (TD) plane.

In addition, a scanning electron microscope (SEM; SU5000, Hitachi, Tokyo, Japan) equipped with EDAX/TSL electron backscatter diffraction (EBSD, EDAX Inc., Mahwah, NJ, USA) was used to observe and analyze the above plane in depth.

Electrolytic polishing of samples could get more accurate EBSD results. More detailed EBSD data was analyzed by orientation imaging microscopy (OIM) software (EDAX Inc., Mahwah, NJ, USA). In order to ensure the accuracy of the data, approximately 300,000 grains were selected for analysis in each sample.

Used the Instron 3382 universal test machine to perform tensile tests at room temperature, with a strain rate of 0.001 mm s$^{-1}$. The length of the tensile specimen was 15 mm, the width was 4 mm, the thickness was 2 mm. All the tensile specimens were selected to be parallel to the extrusion direction of the CEE-AEC billet. The mechanical properties of extruded direction were tested.

4. Results and discussion

4.1. Microstructures

The EBSD results of the edge of pure Mg alloy sample prepared at 250 °C after CEE-AEC are shown in figure 2. The microstructure is significantly inhomogeneous because of relatively low strain during CEE-AEC. After one-pass of CEE-AEC, there are both coarse grains and fine grains. Observing the microstructure of the coarse grains, it is found that they are all elongated and surrounded by fine dynamic recrystallization grains. The average grain size is 74 $\mu$m [11].

After two-pass of CEE-AEC, the grains are obviously refined, and the average grain size is 30 $\mu$m, the distribution of grains is more homogeneous, which can be resulted from dynamic recrystallization (DRX) caused by the recovery of driving energy in the CEE-AEC process, as reported by Biswas et al [17]. After three-pass of CEE-AEC, the grain size is refined from 30 $\mu$m to 19 $\mu$m. The microstructure becomes more homogeneous, with almost no coarse grains and a lot of refined grains and equiaxed grains. It can be seen from the figure 2 that the increasing in the number of CEE-AEC passes leads to finer grains and a relatively more uniform structure. In this article, CEE-AEC has an asymmetrical extrusion cavity, and high accumulative strain is gradually accumulated and imposed on pure Mg [18]. Therefore, the grains in this area are relatively fine. With the increasing CEE-AEC passes, the coarse grains first converted to a large number of sub-grains and then to dynamic recrystallization (DRX), as shown in figures 2(b), (c).

We can draw a conclusion from figures 2(d)–(f) that after one-pass, the average grain size was about 74 $\mu$m, but it reduced from 30 $\mu$m after two-pass to 19 $\mu$m after three-pass, indicating that the reduction of grains from one-pass to two-pass is more quickly.
Figure 3 displays the misorientation distribution of the edge of pure Mg after CEE-AEC. Figure 3 (a) displays that after one pass of CEE-AEC deformation, there are many high-angle grain boundaries distributed at the grain boundaries of the coarse grains, and there are some large-angle grain boundaries surrounding the newly formed DRX grain boundaries and some low-angle grain boundaries can be seen around the coarse grain boundaries. The amount of deformation after one pass is small, and the main microstructure characteristics of the alloy are a lot of coarse primary crystallization and a few DRX. DDRX is the main feature in this period. From one to three passes of CEE-AEC, the number fraction of low-angle grain boundaries (LAGBs) absorb dislocations and transform into recrystallized high-angle grain boundaries (HAGBs). New grains began to form and this process was classified as the formation of continuous dynamic recrystallization (CDRX). After three passes CEE-AEC deformation (figure 3(c)), in the alloy, most areas are filled with fine DRX grains. However, in the recrystallization grain boundary distribution, plenty of grain boundaries with small angles still exist, indicating that DRX grains are also gradually refined. The results show that recrystallization and equiaxial grains occur due to the increase in cumulative strain, as shown in figure 2.

4.2. Texture

Figure 4 shows the pole figures on the (0001) basal plane and (10–10) prismatic planes of the pure Mg sample processed by CEE-AEC. In order to better observe the extrusion surface, the experimental pole figures are displayed on the transverse direction (ED)-transverse direction (TD) plane.

After one pass of CEE-AEC, the basal texture has a strong pole intensity of 13.23. As shown in figure 4(a) that the basal plane of the pure magnesium sample after extrusion is parallel to the extrusion direction. The study shows that this is a typical basal fiber texture in magnesium alloys. The c-axis of the grain was not always on the
TD, some of them deviated from TD in a certain angle. With the increase of CEE-AEC passes, due to deformation, the grain size decreases and the volume fraction of dynamic recrystallization (DRX) grain increases, which offsets the strong deformation grain texture \cite{14,15,19,20}. As shown in figure 4(b), the basal texture weakened to 11.21 and split significantly compared with the texture after one pass of CEE-AEC, in addition, the increase of dynamic recrystallization volume fraction makes the texture more random. It is recognized that in addition to the influence of DRX, the activation of the non-basal slip system also has a great influence on the texture softening. The increase of DRX volume fraction and the texture softening are both affected by accumulation of strain, as reported by Zhang et al \cite{21}. According to figure 4, it can be found that the (0001) planes of a large number of grains are inclined to the ED direction. The study of Chang et al showed that this phenomenon was related to the shear strain, which was introduced by the asymmetric cavity. After three-pass of CEE-AEC process, it can be seen from figure 4(c), the texture of the basal plane was slightly enhanced, which is 11.96. This is because the decrease of the DRX volume fraction hinders the further weakening of the texture to some extent. From one to three passes of CEE-AEC, the non-basal texture of the prismatic texture
gradually increases, indicating that the compatible stress near the grain boundary activates more slip systems besides basal slip [22].

The Schmid factor of (0001) (11−20) basal slip of the edge of pure magnesium after three-pass CEE-AEC process loading along ED is shown in figure 5. It can be seen that with the increase of CEE-AEC process, the value of Schmid factor gradually improves, from 0.29 after one-pass to 0.3 after two-pass. Tensile yield strength (TYS) is related to the Schmid factor value in (0001) (11−20). The relationship between Schmidt factor and yield strength is that a higher Schmid factor determines a lower yield strength [14, 23, 24]. The specific conditions are discussed in detail in section 4.3.

### 4.3. Tensile properties

Figure 6 shows the mechanical properties of the edge of Mg samples after CEE-AEC process. Table 1 shows the values of tensile yield strength (TYS), ultimate tensile strength (UTS) and fracture elongation (%) of samples after one to three-pass. It can be seen that after two passes of CEE-AEC, UTS increased from 95 MPa to 103 MPa. Besides, the elongation also increases from 6.9% to 17%. Taking into account the microstructure and texture discussed above, changes in strength and ductility during CEE-AEC may be caused by grain refinement and weakened texture [25, 26]. The volume fraction of DRX increases as the cumulative strain increases, and the high-density dislocation hinders the movement of dislocations. As a result, the strength increased significantly.

| Passes | Ultimate yield strength /MPa | Tensile yield strength /MPa | Fracture elongation /% |
|--------|-----------------------------|---------------------------|-----------------------|
| One    | 95                          | 48                        | 6.9                   |
| Two    | 103                         | 41                        | 17                    |
| Three  | 110                         | 54                        | 19                    |

In general, according the Hall-Petch equation, the TYS increases with the decrease of grain size. As shown in figure 2, after two-pass of CEE-AEC, grain size decreased significantly. However, the TYS decreased from 48 MPa to 41 MPa. There are many factors that affect the mechanical properties of the alloy. Obviously, the grain size is not the only factor [27]. At room temperature, the mechanical properties of pure magnesium processed by CEE-AEC are also related to the changes in crystal texture during the CEE-AEC process. This is because the strong anisotropy of the magnesium slip system with the HCP structure, as reported by Lei et al [11]. According to previous studies, the TYS of magnesium alloys at room temperature has a great relationship with the activation of basal dislocation slip. As shown in figure 4 that the grain orientation is tilted, which means that with the inclination of basal plane toward ED direction, a new texture is formed, which leads to decrease in yield strength. The Schmid factor of (0001) (11−20) basal slip of CEE-AEC samples along the ED direction is shown in figure 5. It can be seen that the Schmid factor gradually increases with the increase of CEE-AEC process. After two-pass of CEE-AEC, SF increased from 0.29 to 0.3, resulting in a decrease in the tensile yield stress of the (0001) (1120) slip system when it was activated, from 48 MPa to 41 MPa. This indicates that the slip of the basal plane can run more easily during the tensile test along the ED direction. Therefore, after the CEE-AEC process, the grains of pure magnesium have been refined and the softening effect of the texture offsets the grain-refining strengthening effect. In addition, more slip systems will participate in the deformation process because of the activation of the non-basal slip system. After three-pass of CEE-AEC, as the accumulated strain increases, the yield strength and tensile strength also increased slightly. In addition, the accumulation of dislocations leads to the occurrence of dislocation slippage, which is beneficial to increase the elongation of the pure Mg.

Figure 7 shows tensile fracture images for the edge of pure Mg samples processed by CEE-AEC. Figure 7(a) shows the fracture morphology of tensile samples after one pass of CEE-AEC. It can be seen that the tensile sample has typical cleavage fracture characteristics in the scanning area, that is, complete cleavage steps and tearing edges. There are few dimples.

It is well known that cumulative strain and high-density dislocations cause stress concentration at the interface between coarsely deformed particles and DRX grains, then the stress concentration leads to the damage of fracture elongation. Figure 7(b) shows the fracture morphology of samples after two-pass of CEE-AEC. Observing the fracture morphology, it can be seen that after two passes of the CEE-AEC process, there are a good deal of dimples and limited cleavage. The increase in the volume fraction of DRX grains releases the stress concentration. The finer the grains, the more grain boundaries, which prevents the development of cracks. The fracture morphology after three passes of CEE-AEC are shown in figure 7(c). As can be seen from the figure, with the increase of accumulated strain, the sample has completely recrystallized, no obvious tear cracks are found on the fracture surface, and the whole sample is covered by a large number of dimples, indicating better ductility [14, 18].
5. Conclusion

In this study, a new type of SPD process was performed on pure magnesium. The advantage of this process is to use the shear strain generated by the asymmetric cavity to refine the grain and improve the mechanical properties. The pure magnesium after being homogenized at 300 °C as an as-cast for 16 h was pressed by multi-pass CEE-AEC at 250 °C. The effects of CEE-AEC on the microstructure and mechanical properties of magnesium samples after one to three-pass are studied, and the conclusions are as follows.

(1) Through the CEE-AEC process, dynamic recrystallization of pure magnesium has been developed and the grains are obviously refined. After three passes, the grain structure becomes homogeneous, and the average grain size is refined from 74 μm to 19 μm.

(2) With the increase of CEE-AEC process, a weakened texture component was obtained due to the grain refinement. In addition, it can be seen that the shear strain introduced by the asymmetric cavity makes the basal texture of the alloy incline or spread to different degrees in each pass.

(3) The tensile test at room temperature showed that the mechanical properties of pure magnesium have improved after three-pass of the CEE-AEC process. During the CEE-AEC process, dynamic recrystallization occurred, and the non-basal surface slipping started. After the second pass, the yield strength of the alloy decreased because of the new texture and non-basal surface slip. In addition, the increase of the Schmid factor in the yield strength also has an effect on the decrease of the yield strength. After three passes of CEE-AEC process, the grains of the sample are significantly refined, the elongation is increased to 19%, and the mechanical properties are improved, but the strength is not reduced, which is mainly due to the significantly refined grain size.

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ORCID iDs

Yong Xue @ https://orcid.org/0000-0002-1516-3035

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