Effect of Cyclic Extrusion on Microstructure Evolution and Mechanical Properties of Pure Magnesium

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Abstract

The evolution of microstructure and mechanical properties of pure magnesium subjected to reciprocating extrusion at 350 °C for 2, 4, and 8 passes, as well as at 250 °C, 350 °C, and 450 °C for 2 passes, was investigated. The results indicate that after reciprocating extrusion at 350 °C, the microstructure of pure magnesium was significantly refined. When the extrusion passes increased from 2 to 8, the grain size showed no obvious change, while the yield strength decreased and the elongation increased. Through electron backscatter diffraction (EBSD) analysis, it was found that after reciprocating extrusion at 350 °C for 2, 4, and 8 passes, a texture was formed where the {0001} basal plane was oriented at approximately 25°, 30°, and 40° angles to the extrusion direction, respectively, and the texture intensity increased, leading to an increase in the Schmid factor for basal slip systems. After reciprocating extrusion at 250 °C, 350 °C, and 450 °C for 2 passes, as the extrusion temperature decreased, the grain size decreased and the yield strength increased. The relationship between yield strength and grain size can be expressed as σ_s = 5.4 + 338.6d^{-1/2}.

Full Text

Microstructure and Mechanical Property of Pure Magnesium Processed by Cyclic Extrusion Compression

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Abstract

This study investigates the microstructure and tensile properties of pure magnesium processed by cyclic extrusion-compression (CEC) at 350°C for 2, 4, and 8 passes, as well as for 2 passes at 250°C, 350°C, and 450°C. The results demonstrate that CEC processing at 350°C significantly refines the microstructure of pure magnesium. While increasing the number of passes from 2 to 8 does not produce noticeable changes in grain size, the yield strength decreases and the elongation increases. Electron backscatter diffraction (EBSD) analysis reveals that after CEC processing at 350°C for 2, 4, and 8 passes, textures develop with basal planes inclined at approximately 25°, 30°, and 40° to the extrusion direction, respectively, with both texture intensity and Schmid factor for basal slip increasing with pass number. For samples processed for 2 passes at 250°C, 350°C, and 450°C, decreasing the processing temperature reduces grain size and increases yield strength. The relationship between yield strength and grain size can be expressed as $\sigma = 5.4 + 338.6\mathrm{d}^{-1}/^{2}$.

Keywords: metallic materials, pure magnesium, cyclic extrusion compression, microstructure, mechanical property, texture, grain size

Introduction

Magnesium alloys are the lightest structural materials in engineering applications, offering advantages including high specific strength, excellent machinability, abundant resources, and suitability for environmental protection and energy conservation requirements. Consequently, their applications in automotive, defense, and electronics industries have become increasingly widespread. However, their low strength and poor plastic deformability severely limit broader engineering applications, making the improvement of mechanical properties a major research focus. Due to its hexagonal close-packed (HCP) structure with limited active slip systems at room temperature, magnesium exhibits a significant grain size effect on strength [1].

Cyclic extrusion-compression (CEC), a severe plastic deformation technique developed in recent years, has attracted considerable attention for its exceptional grain refinement capability [2]. This process has achieved significant progress in enhancing the strength and ductility of magnesium alloys such as AZ31, ZK60, and GW102K [3-8]. While grain refinement is generally considered the primary reason for mechanical property improvement in CEC-processed magnesium alloys, other factors including texture, second phases, grain boundary characteristics, and alloying elements also play important roles [9]. Recent advances in magnesium alloy strengthening include the discovery of a new metastable precipitate phase βT in Mg-Gd-Y-Zr alloy after repeated upsetting [10] and nanoscale lamellar twin strengthening in Mg-Gd-Y-Ag-Zr during hot rolling [11]. However, most such studies focus on multi-component alloys where numerous interacting factors complicate the elucidation of strengthening and toughening mechanisms in CEC processing.

Although significant progress has been made in understanding microstructure and texture evolution in pure magnesium after severe plastic deformation techniques such as equal-channel angular pressing (ECAP) and high-pressure torsion (HPT) [12-14], few reports exist on microstructural evolution in pure magnesium after CEC processing. This work employs pure magnesium as a simplified model system to eliminate interference from second phases and alloying elements, investigating the effects of CEC deformation on microstructure and mechanical properties. The study aims to explore the influence mechanisms of grain refinement and texture evolution on strength and ductility to establish a strengthening and toughening model for CEC-processed magnesium alloys.

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1. Experimental Methods

Industrial pure magnesium billets were machined to Φ29.5 mm × 42 mm dimensions before CEC processing. The experiments were conducted on a 315 t double-cylinder hydraulic press at a ram speed of 6 mm/s, with immediate water quenching after processing. The CEC die configuration is schematically illustrated in [Figure 1: see original paper]. The process involves three stages: (1) the billet is placed in the upper chamber and extruded through a smaller intermediate channel into the lower chamber under Ram A; (2) the material in the lower chamber is upset to restore its original shape under Ram B, similar to upsetting deformation; and (3) Ram B reverse-extrudes the material back into the upper chamber, completing the first CEC cycle. Subsequent cycles alternate between upper and lower rams. Upon reaching the desired strain, one ram is removed to extrude the final product [5-6].

Both die and billet were held at the processing temperature for 1.5 hours before CEC, with graphite-oil lubrication applied. Processing temperatures were 250°C, 350°C, and 450°C, with pass numbers of 2 at 250°C and 450°C, and 2, 4, and 8 passes at 350°C. The cumulative strain can be calculated using the following formula [6]:

$$\varepsilon = n \cdot \ln\left(\frac{D^2}{d^2}\right)$$

where n is the number of cycles, D is the die cavity diameter, and d is the constriction zone diameter. In this study, $D = 30$ mm and $d = 20$ mm, yielding cumulative strains of 2.4, 5.7, and 12.2 for 2, 4, and 8 passes, respectively.

Samples for texture analysis and tensile testing were sectioned from the longitudinal plane of CEC-processed billets ([Figure 2: see original paper]). As-cast microstructures were examined using an Epson Perfection 4990 scanner. CEC-processed samples were etched with 4% nital for 15-20 seconds and observed under an Axio Observer A1 optical microscope, with grain sizes measured by linear intercept method. Texture characterization was performed using an EDAX-TSL EBSD system on SU-70 and Quanta FEG 250 scanning electron microscopes. Tensile tests were conducted on a Zwick Z100 testing machine at a crosshead speed of 1 mm/min using flat specimens with a gauge dimension of 10 mm × 2 mm × 1.5 mm.

2. Results and Discussion

2.1 Microstructure The as-cast microstructure of industrial pure magnesium exhibits coarse grains, primarily columnar crystals approximately 2 mm × 5 mm in size ([Figure 3: see original paper]). After CEC processing, the microstructure becomes significantly refined, relatively homogeneous, and dominated by equiaxed grains. Following 2 passes at 350°C, pure magnesium develops a fully recrystallized microstructure with an average grain size of approximately 50 m ([Figure 4a: see original paper]). Comparison of [Figure 4b: see original paper] and [Figure 4c: see original paper] reveals that further grain refinement is insignificant with increasing CEC passes, showing minimal microstructural variation. However, deformation temperature strongly influences recrystallized grain size. [Figure 5: see original paper] presents microstructures after 2 passes at 250°C and 450°C. The 250°C processing yields fine grains of only 15-25 m, while 450°C processing produces coarse grains averaging 85 m with evident twins. Larger grains facilitate greater dislocation slip distances and severe stress concentration near grain boundaries, promoting twin formation during deformation.

2.2 Texture Evolution EBSD analysis software was used to generate {0001} and {1010} pole figures for pure magnesium processed at 350°C. The results ([Figure 6: see original paper]) show pronounced preferred orientation. Unlike the typical basal texture observed after conventional extrusion of pure magnesium [15], CEC processing produces a texture where basal planes are inclined 20°-40° to the extrusion direction (ED), which can be approximated as a {1013}<3032> texture. With increasing CEC passes, the maximum texture intensity gradually increases, and the inclination angle between basal planes and ED shows a slight upward trend. After 2 passes at 350°C, the basal planes of textured grains are inclined approximately 25° to ED with a maximum pole density of 9.3. After 4 passes, the inclination angle increases to about 30° with a maximum pole density of 11.5. After 8 passes, the inclination angle further increases to approximately 40°, reaching a maximum pole density of 13.4—43% higher than that after 2 passes. This indicates continuous grain rotation under applied stress during CEC processing.

To investigate grain boundary characteristics, [Figure 7: see original paper] presents misorientation angle distributions after 2 and 8 passes at 350°C. The distributions show minimal differences, with average misorientation angles of approximately 40° for both conditions, suggesting that increasing pass number has limited effect on misorientation angles.

2.3 Mechanical Properties [Figure 8: see original paper] and [Figure 9: see original paper] show room-temperature tensile stress-strain curves of pure magnesium before and after CEC processing. After 2 passes at 350°C, the yield strength increases dramatically from 12 MPa in the as-cast condition to 60 MPa. With increasing passes at 350°C, the strength decreases slightly while ductility improves significantly. After 8 passes, the yield strength drops to 41 MPa, and elongation increases from 8% to 16.7%. As shown in [Figure 9: see original paper], decreasing processing temperature for 2-pass samples increases both yield strength and elongation. After 2 passes at 250°C, the yield strength reaches 75 MPa with 15% elongation. This is attributed to suppressed grain growth at lower temperatures, where fine-grain strengthening yields high strength and ductility.

2.4 Effect of Grain Size on Properties Grain refinement is a crucial approach for improving strength and ductility of magnesium and its alloys [16,17]. The relationship between yield strength (σ) and average grain size (d) follows the Hall-Petch equation [19]:

$$\sigma_s = \sigma_0 + Kd^{-1/2}$$

where σ_0 represents the yield strength of a single crystal and K is the Hall-Petch constant. For HCP magnesium and its alloys, the K value is large, making grain size effects on strength much more pronounced than in cubic materials [18]. However, inverse Hall-Petch relationships have been reported for CEC-processed AZ31 and ZK60 alloys [19,20] and equal-channel angular pressed AZ61 alloy [21], possibly related to texture softening effects.

Since CEC processing of pure magnesium at different temperatures has minimal effect on texture [20], this study investigated the relationship between yield strength and grain size after 2 passes at 250°C, 350°C, and 450°C to avoid texture interference. As shown in [Figure 10: see original paper], yield strength increases significantly with decreasing grain size, following the relationship:

$$\sigma_s = 5.4 + 338.6d^{-1/2}$$

Thus, the Hall-Petch relationship remains valid for CEC processing, though under certain conditions, texture softening may cause yield strength to decrease. The yield strength after 2 passes at 450°C is lower than predicted, likely due to numerous twins in the microstructure ([Figure 5b: see original paper]) that may act as crack initiation sites and adversely affect strength [22].

2.5 Effect of Texture on Mechanical Properties After CEC processing at 350°C, grain size shows minimal variation ([Figure 4: see original paper]) while yield strength decreases and elongation increases significantly ([Figure 8: see original paper]). [Figure 11: see original paper] illustrates the relationship between mechanical properties and maximum texture intensity for 350°C CEC-processed pure magnesium. Yield strength correlates negatively with maximum texture intensity, while elongation correlates positively.

As analyzed in Section 2.2, CEC processing at 350°C produces a texture with basal planes inclined 20°-40° to ED. After 2, 4, and 8 passes, the inclination angles are approximately 25°, 30°, and 40°, respectively, with maximum texture intensity increasing with pass number. In tensile tests, the loading direction is parallel to ED. According to crystallographic principles, as the angle between the applied stress direction and {0001} basal planes increases from 20° to 40°, the Schmid factor for {0001}<1120> basal slip increases accordingly. [Figure 12: see original paper] shows Schmid factor values for major slip systems after different passes at 350°C. With increasing passes, the texture strengthens, and the Schmid factor for basal slip increases from 0.3 after 2 passes to 0.41 after 8 passes, facilitating easier basal slip activation. Since non-basal slip is difficult at room temperature and basal slip dominates deformation [23], the texture effect on basal slip Schmid factor is a primary reason for the decreased strength and increased ductility after 8 passes at 350°C.

Both grain refinement strengthening and texture softening significantly affect mechanical properties of magnesium and its alloys, competing during CEC processing. When texture softening dominates, material strength decreases slightly while ductility improves. When grain refinement strengthening dominates, both strength and ductility increase substantially.

Conclusions

1. Pure magnesium processed by CEC at 350°C for 2 passes exhibits significant grain refinement from 2 mm × 5 mm columnar grains to approximately 50 m equiaxed grains, with a yield strength of 60 MPa. Increas-

ing passes from 2 to 8 produces minimal additional grain refinement but causes slight yield strength reduction and substantial elongation improvement, decreasing yield strength to 41 MPa and increasing elongation from 8% to 16.7% after 8 passes.

2. CEC processing at 350°C produces a texture with basal planes inclined 20°-40° to the extrusion direction. After 2, 4, and 8 passes, the basal plane inclination angles are approximately 25°, 30°, and 40°, respectively. Increasing pass number strengthens the texture and increases the Schmid factor for basal slip, making slip easier. This texture evolution is the main reason for decreased strength and increased ductility after 8 passes at 350°C.
3. After 2 passes at 250°C, 350°C, and 450°C, decreasing processing temperature refines the microstructure and improves mechanical properties. The 450°C condition yields an average grain size of 85 m and yield strength of 35 MPa, while the 250°C condition achieves a yield strength of 75 MPa—115% higher than at 450°C. The relationship between yield strength and grain size follows the Hall-Petch equation: $\sigma = 5.4 + 338.6\mathrm{d}^{-1}/^{2}$.

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