Grain-Size Strengthening in Equal-Channel-Angular-Pressing Processed AZ31 Mg Alloys with a Constant Texture

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The effects of ECAP temperature and post-ECAP annealing on grain size, texture and mechanical behavior have been examined. The softening of ECAPed Mg alloys despite the considerable grain size refinement has been ascribed to the texture change during ECAP. The strength of the ECAPed AZ31 Mg alloys, however, increased with decrease in grain size following the standard Hall-Petch relation when the similar texture could be retained. Based on the present analysis, it could be concluded that it was practically hard to improve the strength of the Mg alloys significantly by grain-size refinement when ECAP was used, because texture softening effect was often more dominant over the grain strengthening effect.

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1. Introduction

Recently, it has been demonstrated that grain-size refinement effectively proceeds with pass number in Mg alloys1-5 as in Al, Cu, and Fe alloys,6-10 during equal-channel angular pressing (ECAP) in which a bulk material can be repeatedly pressed without any change in its cross-sectional dimensions. However, unlike the ECAPed Al, Cu, and Fe alloys with BCC or FCC crystal structures, the ECAPed Mg alloys with HCP crystal structure often showed a negative slope in the plot of yield stress versus d-1/2 implying that strength decreases even though grain size is reduced effectively through ECAP process.1,2 To understand this unusual yield-stress behavior of the ECAPed Mg alloys, texture modification during ECAP of AZ61 alloy was examined and Schmid factors of three slip directions on the basal and prismatic planes of the ECAPed samples were calculated based upon the most dominant texture components assuming that the ECAPed material is a single crystal.1 According to the results, the original (1010) fiber texture of the extruded AZ61 alloy became disintegrated and a new texture progressively developed with increase in number of ECAP pressing. A completely new texture with high maximum intensity and developed with increase in number of ECAP pressing. A completely new texture with high maximum intensity and high Schmid factors was obtained after more than 4 passes,1 indicating that a large portion of basal planes in the as-extruded alloy had been rotated to the directions oriented more favorably for slip during ECAP process.

Texture modification has been reported to also occur in aluminum alloys during ECAP process.11,12 However, its effect on strength seems not to be so significant compared to the effect of grain refinement on strength, since a standard Hall-Petch relationship (a positive slope in the plot of yield stress vs. d-1/2) is valid even after ECAP.13,14 The effect of texture on strength in the Mg alloys may be important since Mg with hcp has limited number of slip systems. As slip is most prone to occur on basal planes in Mg at low temperatures, the rotation of high fraction of basal planes to the directions unfavorable for slip, for example, will increase the yield stress appreciably. By contrast, as aluminum with FCC structure has abundant slip systems, it is highly possible that alternative slip planes with high Schmid factor can be always found, even though some specific slip planes are rotated unfavorably for slip. For this reason, a relatively low sensitivity of yield stress to texture is expected.

In the present paper, the microstructure and texture development in the Mg alloy during ECAP process, and their relations with strength and ductility were further studied. We investigated the effects of lowering ECAP temperature during ECAP process and post-ECAP annealing on microstructure, texture and mechanical properties of the Mg alloys. Hall-Petch relationship among the ECAPed Mg alloys with different grain sizes but with a very similar texture was established to examine the grain size effect on the ECAPed AZ31 alloys and the result was compared with that for the ECAPed Mg alloys with different texture.

2. Experimental Method

The AZ31 alloy was extruded to a rod with a diameter of 14.5 mm and then cut to pieces with a length of 100 mm. ECA pressing was conducted on the as-extruded material through a die made of SKD 61 with an internal angle (Φ) of 90° between the vertical and horizontal channels and a curvature angle (Ψ) of 30° (Fig. 1). Molybdenum disulphide (MoS2) was used as a lubricant. Three ECAP temperatures were chosen: 553 K, 523 K and 493 K. The rod was held at each temperature for 1.8 ks. and then pressed through the die preheated to the given temperature, with a speed of 4 mm/s. Repetitive pressings of the same sample were performed to a maximum of 6 passes by rotating each sample about the longitudinal axis by 90° in the same direction between consecutive passes (designated as route B15). Except at 553 K, however, visible surface cracking occurred at the beginning of 2 or 3 passes. To suppress the surface cracking at the low ECAP temperatures, the as-extruded materials were initially processed to 2 passes at 553 K and then ECAP temperature was lowered to 523 K or 493 K for subsequent pressing from 3 to 6 passes. When this method of lowering

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ECAP temperature was used, the rods could be successfully deformed up to 6 passes without surface cracking. The enhancement of high-temperature ductility by grain refinement during ECAP process at 553 K might have helped the alloys to endure further straining at lower ECAP temperatures where slip was more difficult since non-basal slip systems would be less active.

Tensile specimens of dog-bone geometry with the 5 mm gauge length, 4 mm width, 2 mm thickness, and 2 mm shoulder radius, having the gauge length parallel to the longitudinal axis and the width contained in the y plane (Fig. 1), were extracted from the center portion of the ECAP processed materials by using electro-discharge machining. Microstructures of the tensile samples were examined by using light optical microscopy (LOM) after mechanical and electrolytic polishing and then etching at room temperature in a solution of 1% HNO$_3$, 24% C$_2$H$_6$O$_2$ and 75% water. An image analysis program of Matrox Inspector 2.2 was used to determine the grain size distribution and average grain size from optical micrographs.

The evolution of textures during ECAP processes was studied on the z plane (Fig. 1) by performing X-ray texture analysis. The [0002] and [1011] pole figures were measured up to a tilt angle of 70 degrees using the Schultz reflection method and constructed using commercial software of LaboTex 2.1.

3. Results

Figure 2 shows the photographs of the materials before ECAP (a) and those of the materials ECAPed at 553 K by (b) 1-pass and (c) 2-passes. Their mean grain sizes determined by the image analysis result were 24 $\mu$m, 6.4 $\mu$m and 5.8 $\mu$m, respectively (Fig. 3). As seen from these figures, the microstructure of the as-extruded material was quite inhomogeneous (standard deviation, std.d = 38.6), having a bimodal distribution of fine grains of 10–20 $\mu$m and coarse grains of 50–200 $\mu$m in size. This coarse microstructure was refined after 1 pass and further refined after 2 passes. Degree of homogeneity in grain size distribution was also increased (std.d = 4.8). Figure 4 shows the photographs of the 6-passed AZ31 alloys processed at different three temperatures of (a) 553 K, (b) 523 K and (c) 493 K after 2 passes at the common temperature of 553 K. Top half part of each picture was replaced by the image drawing to show grain boundaries more clearly. Marked refinement of microstructure is evident at all the three temperatures. The mean grain size (=$d$) of these materials are determined to be 4.8 $\mu$m, 3.2 $\mu$m, and 2.2 $\mu$m, respectively, according to the image analysis shown in Fig. 5. This result indicates that finer grains are obtained when the applied ECAP temperature is lower. Furthermore, the alloy processed at the lower temperature reveals more homogeneous microstructure (std.d = 3.01, 1.85 and 1.20 in order with decreasing ECAP temperature). These observations clearly indicate that lower ECAP temperature is more effective in refining the microstructure, probably because dynamic grain growth or dynamic recrystallization can be
more suppressed at lower temperatures.

Figures 6(a)–(c) show the engineering stress-engineering strain curves of the unECAPed and ECAPed AZ31 alloys processed at 553 K after 1, 2 and 6 passes (Fig. 6(a)), the ECAPed AZ31 alloys after 6 passes processed at different temperatures: 493–553 K (Fig. 6(b)), and the ECAPed AZ31 alloys (after 6 passes at 523 K) annealed at 573 K for different time (Fig. 6(c)), respectively. Comparison of the unECAPed alloy with the ECAPed alloys indicates that uniform tensile elongation as well as total tensile elongation has been considerably improved after 6 passes (Figs. 6(a) and (b)). The increase is about two or three times. In fact, the improvement is pronounced even after 1 pass. Another important observation is that yield strength decreases with ECAP pass number, most appreciably after 6 passes, though grain size has been effectively reduced during ECAP. These results are in a sharp contrast to the case when grain size is reduced by direct extrusion process. The alloy with finer grains \(d = 7.2 \mu m, \text{std.d} = 3.7\) exhibits higher yield stress without improvement of tensile ductility (Fig. 6(a)). The decrease in strength and increase in tensile elongation after ECAP agree well with the reports by Mukai et al.\(^3\) on the ECAPed AZ31 alloy and by the present authors on the ECAPed AZ61 alloy.\(^1,2\) When the three ECAPed alloys after
6 passes at three different ECAP temperatures are compared (Fig. 6(b)), however, it is realized that the alloy ECAPed at the lower temperature, thereby having the smaller grain size, is stronger. Tensile elongations, on the other hand, are similar to one another. Figure 6(c) shows that the strength of the ECAPed alloys decreases with increasing annealing time. Correspondingly the mean grain size of the ECAPed alloy is increased from 4.8 μm to 5.5 μm and 8 μm, after static annealing of 1.2 ks. and 4.8 ks, respectively. Tensile elongation, on the other hand, tends to increase with increasing annealing time. Total elongation as large as 80% is obtained after the 4.8 ks annealing, which is about four times larger than that of the unECAPed alloy.

Difference in texture and grain size is thought to be responsible for the strength differential between the materials in Fig. 6. To understand the origin of the strength differential, their textures were analyzed. The \{0002\} and \{1011\} poles figures of the alloys after 6 passes at (a) 553 K, (b) 523 K, (c) 493 K and (d) the alloy ECAPed at 523 K and then annealed at 573 K for 1.2 ks and 4.8 ks. are shown in Fig. 7. The \{0002\} and \{1011\} poles of (0223)|[4154] orientation are marked on
corresponding pole figures using the ‘+’ symbol. The texture analysis is summarized in Table 1. The ED || (1010) fiber texture of as-extruded material where (0001) basal planes and (1010) directions in most grains are distributed parallel to the extrusion direction (ED) has been completely changed after 6 passes. The dominant textures are (0112)[2243], (0223)[4154] and (0223)[4154] in the alloys after 6 passes at 553 K, 523 K and 493 K, respectively. Though it is obvious that the crystallographic plane parallel to z plane rotates from (0112) to (0223) as the ECAP temperature is decreased from 553 to 493 K, the angular distance between preferred orientations of the three specimens is very small. The maximum texture intensity of (0002) pole figures is high (9~12) at all the three temperatures, indicating that strong texture has been developed after 6 passes. When the alloys ECAPed at 523 K were annealed at 573 K for 1.2 ks. The same texture was retained after further annealing up to 4.8 ks. As the angular distance between preferred directions before and after annealing is very small, it can be considered that texture does not change during the annealing. The maximum texture intensity of (0002) pole figures, on the other hand, decreased from ~10 to ~7 after the annealing treatment.

Table 1: Textures and Schmid factor values for the ECAPed AZ31 alloys.

<table>
<thead>
<tr>
<th>ECAP pass no.</th>
<th>ECAP temp.</th>
<th>Texture</th>
<th>Schmid factor</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>basal</td>
</tr>
<tr>
<td>unECAPed (0)</td>
<td>—</td>
<td>ED</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>553 K</td>
<td>(0112)[2243]</td>
<td>0; 0.432; 0.432</td>
</tr>
<tr>
<td>6</td>
<td>523 K*</td>
<td>(0223)[4154]</td>
<td>0; 0.423; 0.423</td>
</tr>
<tr>
<td>6</td>
<td>493 K*</td>
<td>(0223)[4154]</td>
<td>0; 0.423; 0.423</td>
</tr>
<tr>
<td>6</td>
<td>523 K*</td>
<td>+annealing at 573 K for 20 min.</td>
<td>(0112)[2243]</td>
</tr>
<tr>
<td>6</td>
<td>523 K*</td>
<td>+annealing at 573 K for 80 min.</td>
<td>(0112)[2243]</td>
</tr>
</tbody>
</table>

*ECAPed to 2 passes at 553 K

Fig. 7: The [0002] and [1011] pole figures of the materials after 6 passes at (a) 553 K, (b) 523 K, (c) 493 K, and (d) the material ECAPed at 523 K and then annealed at 573 K for 1.2 ks. (The [0002] and [1011] poles of (0223)[4154] orientation are marked on the corresponding pole figures using the ‘+’ symbol.)
4. Discussion

Schmid factors of three slip directions on the basal and prismatic planes were computed based on the most dominant texture components in the pole figures and the result was summarized in Table 1. For all the alloys after 6 passes, the Schmid factors on both basal and prismatic slip systems are very similar to one another as their textures are similar. The basal planes have been more favorably oriented for slip after ECAP as proved by the increased Schmid factors, while the prismatic planes have been less favorably oriented as proved by the decreased Schmid factors. Slip on non-basal slip systems with high critically resolved shear stress, \( \tau_{\text{CRSS}} \), may dominate the yielding in the as-extruded Mg alloy whereas slip on basal slip systems with low \( \tau_{\text{CRSS}} \) may govern the yielding in the ECAP Mg alloys. This result suggests that activation of the basal slip systems by rotation of the basal planes has caused the significant drop in yield stress after 6 passes in ECAP. As the subsequent annealing after ECAP does not alter the texture, their Schmid factors are similar to those of the ECAPed alloys before annealing. Therefore, it is highly possible that the strength differential between three 6-passed alloys and two annealed alloys is primarily due to the difference in grain size.

In Fig. 8, the 0.2% proof stress is plotted against \( d^{-1/2} \) for the unECAPed and ECAPed AZ31 alloys along with the data for the unECAPed and ECAPed AZ61 alloys studied previously, based on the Hall-Petch relation given as

\[
\sigma_y = \sigma_0 + k_y d^{-1/2},
\]

where \( \sigma_y \) is the yield stress, \( \sigma_0 \) is the friction stress, \( d \) is the grain size, and \( k_y \) is the characteristic constant representing the resistance to dislocation motion provided by the grain boundaries. The standard Hall-Petch relationship (positive slope) is valid among the data points for the unECAPed Mg alloys with different grain sizes but with a constant texture. Though only two data points are available for the unECAPed AZ31 alloys with different grain sizes, as they are onto the line for the unECAPed AZ61 alloys it can be accepted that the strength differential between the unECAPed (extruded) AZ61 and AZ31 alloys at a given grain size is small. When the data of the ECAPed AZ61 Mg alloys are considered with that of the same alloy prior to ECAP the sign of yield stress versus \( d^{-1/2} \), however, is macroscopically negative. This is also true for the AZ31 alloys after 0 (unECAPed) to 2 passes. On the other hand, three ECAPed AZ31 alloys after 6 passes and two alloys additionally annealed after 6 passes are well correlated on a single line and exhibit the positive slope like the unECAPed alloys. This result suggests that a standard Hall-Petch relation is expected even in the ECAPed alloys when texture is constant. This provides a direct evidence that the negative slope in the AZ61 and AZ31 alloys with different pass number is due to the texture difference. Measurement of \( k_y \) and \( \sigma_0 \) values for the unECAPed AZ61 alloys and ECAPed AZ31 alloys after 6 passes in Fig. 8 indicates that after ECAP, both values drop from 350 to 180 MPa \( \mu \)m \( ^{-1/2} \) and from 140 to 30 MPa, respectively. The definition of \( k_y \) in eq. (1) is as follows according to Armstrong et al.,

\[
k_y = m^2 \tau_{\text{CRSS}}^{-1/2},
\]

where \( \tau_{\text{CRSS}} \) is the critically resolved shear stress for slip, \( m \) is the orientation factor related to the number of activated slip systems and \( r \) is the distance from the nearest dislocation piled-up to the dislocation source in the adjacent grain. This relation lets us to conjecture that the decrease of \( k_y \) after ECAP is due to the substantially increased easiness of basal-slip operation by texture alternation during ECAP. The decrease of friction stress \( \sigma_0 \) after ECAP, which is the yield stress of a single crystal AZ31 alloy, can be also explained in terms of rotation of basal planes to directions favorable for slip, since the yield stress is proportional to \( m \), which is inversely proportional to Schmid factor.

Extrapolation of the line for the ECAPed and annealed AZ31 alloys to the smaller grain sizes in Fig. 8 shows that the ECAPed AZ31 alloy will have the same strength as the unECAPed alloy when its grain size is as small as \( \sim 1.5 \mu \)m. This demonstrates how much the effect of texture softening is dominant over the grain-strengthening effect. When \( d \) is reduced to \( \sim 0.7 \mu \)m which is the size typically obtained for the Al alloys after ECAP, the yield stress is 250 MPa. Based on this work, therefore, it may be concluded that it is practically hard to increase the strength of Mg alloys greatly using ECAP method unless grain size is significantly reduced by using the ECAP temperature lower than 493 K. As Mg alloy has hcp structure, however, it may be difficult to apply the ECAP temperature similar to that for Al alloys in typical ECAP practice (below 423 K). If one finds the method to restore the original state before ECAP, yield stress as high as 500 MPa can be obtained at \( d = 1 \mu \)m. Application of direct extrusion to the ECAPed Mg alloys may be one of the methods to restore the original fiber texture.

The strain hardening behavior of the ECAPed AZ31 alloys is analyzed using the Hollomon relation assuming that the stress–strain curve is described as

\[
\sigma = Ke^n,
\]

where \( \sigma \) is the true flow stress, \( \varepsilon \) the true plastic strain, \( n \) the strain hardening exponent and \( K \) the strength coefficient. The strain hardening exponent measures the uniform strain in tension obtained prior to initiation of neck instability.
Therefore, it is the maximum elongation in sense of engineering application. To determine $n$ value, the engineering curves in Fig. 6(a) have been converted to true stress–true strain curves and then plotted in log-log format as shown in Fig. 9(a). The strain hardening exponents are initially very low (at $\varepsilon$ less than 0.03) and essentially same for all the alloys with different deformation history, but increase rapidly with further straining and become constant over the wide range of strain. The strain hardening exponents in the high strain range for the various alloys studied in the present work measured are listed in Table 2. Figure 9(b) shows the relation between the strain hardening exponent and uniform strain in the unECAPed and ECAPed AZ31 alloys. As can be expected theoretically, a good linear correlation is observed, where the uniform strain increases linearly with the strain hardening exponent, indicating that increase in strain hardening directly leads to improvement of tensile ductility. The improvement is most likely related to the activation of the basal planes but this is not sufficient to explain the significant increase of the ductility since the von Mises criterion cannot be satisfied. As summarized in Table 1, two slip systems on the prismatic plane still have a relatively high Schmid factor (0.23) after ECAP. Thus, it is possible that these operate together with the basal slip systems during tensile deformation. Twinning may be induced more favorably owing to the texture modification by ECAP. This is because mechanical twinning in magnesium $\langle 1011 \rangle / c $ is known to hardly occur in tension on the extruded rods with the basal planes of the grains strongly aligned parallel to the axis of the rod. Therefore, operation of two slip planes and mechanical twinning may lead to the large hardening. The further increase of tensile ductility after annealing (Table 2), on the other hand, should be attributed to the change of microstructure since the texture remains essentially unchanged during annealing. As dislocation density is reduced by recovery or recrystallization during annealing, the strain hardening capability is anticipated to increase. This is because probability of dynamic recovery by cross slip would decrease with increase in distance between dislocations, which occurs when dislocation density is lowered.

![Fig. 9](image_url)

**Fig. 9** (a) Log true stress vs. log true strain for the unECAPed and ECAPed AZ31 alloys processed at 553 K. (b) Relation between strain hardening exponent and uniform tensile elongation.

<table>
<thead>
<tr>
<th>ECAP pass no.</th>
<th>ECAP temp., $T/K$</th>
<th>Grain size, $d/\mu m$</th>
<th>Yield stress, $YS/MPa$</th>
<th>Ultimate tensile stress, $UTS/MPa$</th>
<th>Stress hardening exponent $n/MPa$</th>
<th>Uniform elongation %</th>
<th>Total elongation %</th>
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<tr>
<td>unECAPed (0)</td>
<td>—</td>
<td>24</td>
<td>202</td>
<td>271</td>
<td>0.17</td>
<td>16</td>
<td>21</td>
</tr>
<tr>
<td>1</td>
<td>553 K</td>
<td>6.4</td>
<td>142</td>
<td>260</td>
<td>0.38</td>
<td>34</td>
<td>36</td>
</tr>
<tr>
<td>2</td>
<td>553 K</td>
<td>5.8</td>
<td>132</td>
<td>258</td>
<td>0.43</td>
<td>42</td>
<td>50</td>
</tr>
<tr>
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<td>110</td>
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<td>3.2</td>
<td>130</td>
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<td>8</td>
<td>88</td>
<td>221</td>
<td>0.64</td>
<td>66</td>
<td>80</td>
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*ECAPed to 2 passes at 553 K
5. Summary and Conclusion

The effects of lowering ECAP temperature during ECAP process and Post-ECAP annealing on microstructure, texture and mechanical properties of the AZ31 alloys have been investigated in the present study. The as-extruded materials were ECAP processed to 2 passes at 553 K prior to subsequent pressing up to 6 passes at 523 K or 493 K. When this method of lowering ECAP temperature during ECAP was used, the rods could be successfully deformed up to 6 passes without any surface cracking. Grain refinement during ECAP process at 553 K might have helped the material to endure further straining at lower deformation temperatures probably by increasing the strain accommodation effect by grain boundary sliding, causing stress relaxation. Texture modification during ECAP has a great influence on the strength of Mg alloys because HCP metals have limited number of slip systems. As slip is most prone to take place on basal planes in Mg at room temperature, the rotation of high fraction of basal planes to the directions favorable for slip during ECAP decreases the yield stress appreciably. The strength of AZ31 Mg alloys increases with decrease of grain size if the texture remains constant though ECAP deformation history is different. The standard positive strength dependence on the grain size for Mg alloys with the similar texture remains constant though ECAP deformation history is different. The standard positive strength dependence on the grain size for Mg alloys with the similar
texture supports that the softening of ECAPed Mg alloys (a negative slope) typically observed despite the significant grain refinement is due to the texture modification where the rotation of basal planes occurs towards the orientation for easier slip. It could be predicted that if the original fiber texture of the extruded state is restored after ECAP treatment that yields marked grain refinement, yield stress as high as 500 MPa will be obtained at the grain size of ~1 μm.

Acknowledgments

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