Victoria-Hernandez, J.; Kurz, G.; Suh, J.S.; Letzig, D.:
Microstructure development and related mechanical behavior of
the ZEW200 Mg alloy processed by differential speed rolling and
equal channel angular pressing

DOI: 10.4028/www.scientific.net/MSF.941.931
Microstructure Development and Related Mechanical Behavior of the ZEW200 Mg Alloy Processed by Differential Speed Rolling and Equal Channel Angular Pressing

José Victoria-Hernández1,a*, Gerrit Kurz 1,b, Joungsik Suh2,c and Dietmar Letzig1,d

1Helmholtz-Zentrum Geesthacht, Max-Plank Strasse 1, 21502, Geesthacht, Germany
2Materials Implementation Center, Korean Institute of Materials Science, Changwondaero 797, Changwon, Gyeongnam, 51508, South Korea

a jose.victoria-hernandez@hzg.de, bgerrit.kurz@hzg.de, cjssuh@kims.re.kr, dietmar.letzig@hzg.de

Keywords: Crystallographic texture, Differential speed rolling, ECAP, deformation mechanisms, anisotropy

Abstract. The present work investigates the influence of shear deformation on microstructural-texture and mechanical behavior of ZEW200 Mg alloy sheets. For the introduction of extra shear deformation during thermomechanical processing, the separate effect of differential speed rolling (DSR) and equal channel angular pressing (ECAP) were analyzed. The results were compared with the microstructure and mechanical behavior of equal speed rolled (ESR) ZEW200 sheets. No significant texture changes were observed after the utilization of DSR, while ECAP processing was effective in changing the character of the texture and reducing the texture intensity. The large yield stress asymmetry observed in the rolled sheet is strongly reduced in the ECAP processed sheets. Results showed the potential to use shear deformation to modify the crystallographic texture via the profuse activation of \( \{10-12\}<10-11> \) extension twins. The presence of a large twin fraction of the microstructure modified the work hardening behavior of the processed sheets due to the further activation of basal \(<a>\) slip. The application of extra shear deformation to tailor the texture during processing is, therefore, an alternative to optimize the deformation behavior of already formable Mg alloys.

Introduction

In order to overcome the lack of formability, the microstructure and texture of Mg alloys can be modified via alloying. The use of rare earth elements (RE) is known to be effective to modify the microstructure and weaken the crystallographic texture. As a result, the formability of Mg-RE alloys is significantly improved [1-3]. However, the mechanical and forming behavior of Mg-RE alloys shows an anisotropic performance. Consequently, alternative processing technologies are necessary to optimize the performance of already formable Mg alloys.

Intelligent processing of the alloys could definitely alleviate the anisotropy of these formable alloys. Weakening or controlled tilting of the basal planes of conventional alloys via thermomechanical treatments can significantly improve the formability of Mg alloys. In this regard, the application of extra shear deformation during thermomechanical processing to tailor the microstructure-texture is an interesting approach. It is especially attractive to introduce shear strain during rolling to improve the efficiency of the process. DSR is expected to have an important influence. Because, it can modify the texture of the conventional AZ31 Mg alloy improving its ductility and formability [4, 5]. In addition to this thermomechanical processing, recently, the utilization of ECAP to deform Mg sheets has been prove to be feasible [6-8]. Therefore, in the present investigation, the effect of introducing extra-shear strain to deform sheets of a recently develop ZEW200 Mg alloy [9] will be described. It is important to highlight that, this alloy is already good formable (Erichsen index of 6.8 mm at room temperature) but it still shows a markedly anisotropic behavior in mechanical and forming properties.
Experimental Procedure

ZEW200 (1.81Zn – (0.1Nd- 0.1Ce - 0.05La)- 0.2 Y in wt.%) twin roll cast (TRC) strips with thickness of 5.3 mm were rolled at 400 °C to a final gauge thickness of 1.8 mm. The rolling schedule consisted in four rolling passes with the first one applying a degree of deformation of φ= 0.1 (true strain) and φ= 0.3 for the last three passes. The rolling speed was set to 10 m/min. The diameter of the rolls was 360 mm. DSR tests were performed using in principle the same rolling schedule, yet the main difference was the change of the rotation speeds of the rolls. Differences of 1:1.1, 1:1.5 and 1:2 were employed. Before the first rolling pass and at intermediate steps the sheets were heated at rolling temperature for 15 min. After the final rolling pass, the samples were air-cooled. In order to recrystallize the microstructure, samples were annealed at 350 °C for 30 min.

ECAP processing was carried out at 225 °C using a channel angle of 110° to deform samples with dimensions of 200 x 200 x 1.8 mm³. Samples were processed with the RD maintained parallel to the pressing direction (PD). The pressing speed was set to 5 mm/s and oil was used as lubricant. Due to slight bending of the samples after the ECAP process, the samples were hot leveled at 200 °C for 30 min applying a pressure of 7.3 kN. More details about the ECAP tool can be found in [6-8].

Microstructural examination was performed by optical microscopy and electron backscatter diffraction (EBSD) on the normal direction (ND) and RD plane. Samples were ground with sand paper grit 800-2500 and polished with a water free suspension of silicon oxide (OPS 0.5 µm). After mechanical polishing, samples were electropolished using a Struers® AC2 solution at 16 V for 70 s at -25 °C. EBSD measurements were performed in a field emission gun scanning electron microscope Zeiss® Ultra 55 equipped with an EDAX-TSL OIM™ system. Measurements were carried out on different sections through the sheet thickness to characterize the local microstructure-texture. An acceleration voltage of 15 kV and a step site of 0.3 µm were used. Energy dispersive X-ray spectroscopy (EDS) was used to determine the chemical composition of secondary phase particles.

Quantitative texture measurements were carried out with a Panalytical™ X-ray diffractometer in reflection geometry using Cu-Kα radiation. Six pole figures (0001), (10-10), (10-11), (10-12), (10-13) and (11-20) were measured up to a tilt angle of 70°. The orientation distribution function (ODF) was calculated using the MTEX toolbox [10].

Tensile samples were machined according to the standard DIN 50125-H and were tested in an universal testing machine Zwick™ 050. Tension test up to fracture were performed along the RD, 45° and TD at room temperature. Tensile tests were performed at constant strain rate of 10⁻³ s⁻¹. This was ensured by employing a clip-on extensometer that allowed the continuous calibration of the strain rate during the experiment.

Results and Discussion

Figs 1a and 1b, show the microstructure of the conventionally rolled sheets in as-rolled and after recrystallization annealing respectively. The microstructure in as-rolled condition shows a relative homogeneous microstructure with deformed grains and the presence of several secondary phase particles. Coarse particles contain Mg-Zn-RE-Y elements, while small-rounded particles distributed homogeneously on the microstructure only contain Mg-Zn-Y. Besides, in the deformed microstructure several deformation twins are observed. The microstructure after recrystallization annealing shows a homogeneous microstructure with equiaxed grains. The average grain size (G.S.) estimated with the linear intercept method is 10.2±2 µm. DSR samples rolled using different rotation ratios of 1:1.1, 1:1.5 and 1:2 are presented in Figs. 1c-d, 1e-f and 1g-h for the as-rolled and annealed condition respectively. In the case of the as-rolled condition, they show a homogeneous microstructure with deformed grains. Samples after annealing present fine-grained microstructures with equiaxed grains. Although, differences on grain sizes are marginal with respect to the conventionally rolled samples, there is a tendency of reducing the grain size by using DSR scheme. Moreover, smaller particles and well-distributed secondary phase particles are observed in
To understand the reason of the difference in the grain size evolution, an analysis of the stored strain of the as-rolled sheets was carried out using kernel average misorientation (KAM). Since KAM is based on the degree of misorientation between a kernel and its neighbors, the estimation of stored strain is underestimated. Because the KAM method only considers geometrically necessary dislocations and omits statistically stored dislocations [11]. Yet, this analysis has been used to show the distribution of the stored strain in ECAP processed AZ31 sheets [12]. Moreover, it allows a graphical evaluation of the stored strain in the ESR and DSR processed sheets. Fig. 2 exhibits the KAM and respective image quality (IQ) maps of samples in as-rolled condition. Qualitatively, the stored strain in the ESR sheet (Fig. 2a) is significantly lower than the DSR sheets (Figs. 2b-c for 1:1.1, 1:1.5 and 1:2 respectively). Especially, the finer microstructure of the 1:1.1 DSR sheet, in comparison with the other rolled samples, is related with the higher stored strain. In such case, the strain is homogeneously distributed, not only near the grain boundaries, but also to the center of the grains. As the DSR ratio is increases, the stored strain is no longer homogeneously distributed. The IQ maps also reveal interesting features on the microstructure development. Because of the shear strain introduced by the DSR process, the amount of twins increases in comparison to the ESR sheet. Especially the content of \{10-11\}/\{10-12\} secondary twins is increases with respect to the ~2% in the ESR sheet. The fraction of the microstructure covered by secondary twins accounts to 4, 5 and 7% for the 1:1.1, 1:1.5 and 1:2, respectively. The amount of \{10-12\} extension twins and \{10-11\} contraction twins is low and they account to 1-2 % in all sheets.
Fig. 3 presents the optical microstructure and, the KAM and IQ maps of the ECAP ZEW200 sheet. The microstructure in comparison to the initial state, i.e. ESR annealed microstructure, is slightly refined. The average grain size is about 7±4 µm. There is an inhomogeneous distribution of the shear strain, as it is visible on the KAM map (see Fig.3b). In direction to the surface, the sheet contains significantly more deformation with respect to the center of the sheet (see the arrow in Fig. 3b). The IQ maps, presented in Fig. 3c, shows a change in the balance of the activation of twinning. In this regard, the fraction of the microstructure covered by {10-12} extension twins is significantly increased to almost 8%, while the amount of {10-11} contraction twins and {10-11}/{10-12} secondary twins remain very low (<1%). It is important to highlight that, after ECAP processing and leveling, no changes in the microstructure-texture were observed. Therefore, this condition of the microstructure (deformed-twinned) was subsequently used for mechanical tests.

Figure 3. a) Optical microstructure, b) KAM map and c) IQ map of the ZEW200 sheet after leveling.

The resulting textures after annealing for rolled sheets and after leveling for the ECAP sample are presented in Fig. 4. The textures are represented in terms of the basal {0001} and prismatic {10-10} recalculated pole figures. The texture of the ESR sheet, shown in Fig. 4a, is weak with a maximum intensity of 3.9 multiple random distribution (m.r.d). It shows two main peaks with a broad distribution of the basal planes towards TD and the prismatic {10-10} poles aligned parallel to RD. The textures of the DSR sheets Fig. 4b) 1:1.1, 4c) 1:1.5 and 4d) 1:2 show virtually no significant change with respect to the counterpart ESR sheet. In all cases, there is an increase in the texture intensity and some inclination of the basal planes towards RD (especially in the case of the DSR 1:1.5 sheet). In the case of the texture of the ECAP sheet, the measurement was carried out in the middle section of the sheet. The texture after ECAP processing is the weakest among all measured materials. The two main components from the rolled condition persisted through the ECAP process, yet they are weaker (see Fig. 4e). Another important feature of this texture is the development of an additional peak on the (0001) pole tilted around 60-70° from ND to pressing direction (RD//PD). The origin of such tilted component is related with the activation of {10-12} extension twins. Most of the prismatic {10-10} poles are still aligned parallel to RD//PD with some of them also tilted due to the rotation of the basal planes towards RD//PD as a result of the shearing process. More details are found in [13]

Figure 4. Crystallographic texture presented in terms of {0001} and {10-10} pole figures for the a) ESR, b) DSR 1:1.1, c) DSR 1:1.5, d) DSR 1:2 and e) ECAP sheet.
In Fig. 4, the engineering stress-strain curves at room temperature and constant strain rate for the ESR, DSR and ECAP sheets are displayed. Based on the similarities in grain sizes and similar textures, the stress-strain curves of only the DSR 1:1.5 is plotted. The mechanical properties are listed in Table 1. High yield asymmetry is found in the ESR sheet, where the yield stress along RD is significantly higher than the yield stress along 45° and TD. With respect to ductility, the sheet also shows an anisotropic behavior, where the 45° and TD exhibit higher ductility in comparison to RD. The difference in ductility can be related with the large difference in the n-values as observed in Table 1. Such difference in the hardening behaviors can be related with the different activation of slip systems (mainly basal \(<a>\), prismatic \(<a>\) and pyramidal \(<c+a>\) slip) and twinning due to texture. More information about the analysis of the activation of deformation mechanisms can be found in [13]. Owing to the similar microstructure-texture, the DSR sheet showed a rather similar tendency in the yield asymmetry and ductility. There is a minor change, yet important, as a result of the slight inclination of basal planes. As a result of the introduction of shear deformation, the uniform elongation and n-value along RD are improved in comparison to the ESR sheet. As expected, the most striking effect comes from the ECAP process. There is a strong reduction of the yield stress asymmetry, similar n-values in all directions tested and a significant improvement of the uniform elongation along RD. Analysis shows that further activation of basal \(<a>\) slip, prismatic \(<a>\) slip and \{10-12\} extension twins are behind the improvement of the deformation behavior along RD. Although, the fracture strain along 45° and TD are reduced, the most important aspects to consider for further forming steps are high uniform elongation, well balanced work hardening and, actually, low yield stresses. It is important to highlight that the effective introduction of extra shear strain during processing can influence all of these important parameters via texture engineering.

![Figure 5. Engineering stress-strain curves of a) ESR, b) DSR 1:1.5 and c) ECAP sheets.](image)

Table 1. Tensile properties at room temperature of the ESR, DSR and ECAPed sheets.

<table>
<thead>
<tr>
<th>Processing</th>
<th>Direction</th>
<th>YS&lt;sub&gt;0.2&lt;/sub&gt; (MPa)</th>
<th>UTS (MPa)</th>
<th>ε&lt;sub&gt;u&lt;/sub&gt; (%)</th>
<th>ε&lt;sub&gt;f&lt;/sub&gt; (%)</th>
<th>n (-)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ESR</td>
<td>RD</td>
<td>186</td>
<td>248</td>
<td>11</td>
<td>24</td>
<td>0.16</td>
</tr>
<tr>
<td></td>
<td>45°</td>
<td>122</td>
<td>224</td>
<td>23</td>
<td>42</td>
<td>0.31</td>
</tr>
<tr>
<td></td>
<td>TD</td>
<td>108</td>
<td>223</td>
<td>23</td>
<td>34</td>
<td>0.45</td>
</tr>
<tr>
<td>DSR (1:1.5)</td>
<td>RD</td>
<td>182</td>
<td>259</td>
<td>14</td>
<td>22</td>
<td>0.18</td>
</tr>
<tr>
<td></td>
<td>45°</td>
<td>121</td>
<td>229</td>
<td>23</td>
<td>38</td>
<td>0.29</td>
</tr>
<tr>
<td></td>
<td>TD</td>
<td>109</td>
<td>229</td>
<td>25</td>
<td>32</td>
<td>0.39</td>
</tr>
<tr>
<td>ECAP</td>
<td>45°</td>
<td>171</td>
<td>246</td>
<td>18</td>
<td>26</td>
<td>0.23</td>
</tr>
<tr>
<td></td>
<td>TD</td>
<td>165</td>
<td>258</td>
<td>20</td>
<td>25</td>
<td>0.25</td>
</tr>
</tbody>
</table>

Summary

The effect of the introduction of shear deformation, as a way, to optimize the microstructure-texture and related mechanical behavior of a formable ZEW200 Mg alloy was investigated. The introduction of shear strain via the use of differential speed rolling and ECAP was evaluated. Results showed that microstructure-texture can be only marginally be changed via DSR, while the
character and intensity of the texture is significantly modified by the use of ECAP. As a result of this texture modification, the mechanical behavior was substantially enhanced in the ECAP processed sheet. The most important improvements are the increase in the uniform elongation along RD, the material shows similar n-values in all direction tested and there is a strong reduction of the yield asymmetry. All of this as a result in the balance of active deformation mechanisms i.e. enhance activation of mainly basal <a>, prismatic <a> slip and {10-12} extension twinning.

Acknowledgments

The results in this study were performed under the joint research project HO 2165/47-1 and LE 1395/6-1 sponsored by Deutsche Forschungsgemeinschaft (DFG). The technical assistance during EBSD measurements at HZG of Petra Fischer and Yukyung Shin are gratefully acknowledged.

References

[2] Chino Y., Kado M., Mabuchi M., Enhancement of tensile ductility and stretch formability of magnesium by addition of 0.2 wt% (0.035 at%) Ce, Mater. Sci. Eng. A 494, 2008, 343-349