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Effect of twins and non-basal planes activated by equal channel angular rolling process on properties of AZ31 magnesium alloy

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Abstract Ultrafine-grained (UFG) metallic materials because of their superior properties have received considerable research interest. Recently, severe plastic deformation (SPD) processes are widely used for refining the grain size in magnesium alloys. Equal channel angular rolling (ECAR) is a SPD process based on equal channel angular pressing (ECAP) which is carried out on large, thin sheets. After doing this process, no significant change is occurred in cross-sectional area of specimen. In this research, an AZ31 magnesium alloy was subjected to ECAR. After completing eight passes of process, significant grain refinement was occurred, and the average grain size of about 3.9 μm was achieved. The distribution of grain size becomes more limited by increasing number of passes. Rotation of basal plane and activation of non-basal and twin planes were clearly observed in X-ray diffraction (XRD) pattern results. Mechanical properties were studied via tensile and hardness tests at room temperature. Tension tests indicated that better ductility due to the rotation of basal plane was achieved. Elongation-to-failure was increased from 8% of as-received material to 19% after two passes of process. Hardness values showed an increase of about 53% at eighth pass.

Introduction

Magnesium alloys because of their low density ($\rho\text{Mg} = 1.74\ \text{g/cm}^3$) and high specific strength have a great potential as structural materials for aerospace, automotive, and electronic applications [1, 2]. Usage of magnesium alloys in automotive industries could reduce vehicle weight and fuel consumption [3]. Moreover, they are the lightest metals which are used for structural applications [4]. Despite these advantages, it is well known that magnesium alloys sheet exhibit very limited drawability at ambient temperature via conventional forming operations, such as symmetric rolling, and this limits their industrial applications [5, 6]. But magnesium alloys can be extremely ductile at room temperature by controlled texture through equal channel angular pressing (ECAP) which involves a simple large shear deformation via two intersecting channels [6]. Indeed, the ductility enhancement in magnesium alloys could be achieved by refining their grain structure [7–9]. Although, the ECAP process could significantly control the texture of magnesium alloys [6, 8], it is impossible for ECAP to fabricate sheet [6]. A novel severe plastic deformation (SPD) technique, equal channel angular rolling (ECAR), based on ECAP can apply the shear deformation to long and thin metallic sheets whereas no significant changes occur in their cross-sectional area [10, 11]. Moreover, this technique could change the texture. So, better ductility is achieved in magnesium sheets processed by this technique [12].

Soft materials such as Cu, Al alloys, Fe, and low-carbon steels could be processed by SPD at room temperature [13]. However, hard materials such as magnesium alloys are processed at high temperatures and this is due to their poorer ductility at low temperatures [13, 14]. Therefore, the processing temperature is an important parameter which changes microstructural and mechanical properties of hard metals [13]. In spite of processing temperature, twins are important factors affecting properties of materials. Lots of twins were observed in ECARed AZ31 magnesium alloy

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[15]. Also, occurrence of deformation twinning in austenitic stainless steels (SSs) infers the profit of twins to the microstructure/grain refinement of this metal [13].

So far not many researches have been reported about the effect of processing temperature and activated twins on microstructural and mechanical properties of AZ31 magnesium alloy after ECAR process. In our recent investigation, ECAR process was used to deform the sheets of AZ31 magnesium alloy. The effect of the number of passes on the grain refinement, distribution of grains and rotation of basal plane during process were investigated. The influence of twins and non-basal slip systems on properties of alloy is discussed.

Experimental procedure

The AZ31 magnesium alloy of 250 × 20 × 1.8 mm^3 (length × width, thickness) was used. The composition of studied material is summarized in Table 1. The sheets were heated at desired temperatures for 3 min, and then ECAR was applied via a mold having the oblique angle (θ) of 120°. The thickness of the inlet and outlet channel was 1.7 and 1.8 mm, respectively. The schematic of ECAR device is illustrated in Fig. 1.

The ECAR process was started at 633 K in the first pass, and the temperature of process was gradually decreased in the sequent passes. The final pass (8-pass) was carried out at 473 K.

Table 1 Chemical composition of the studied AZ31 magnesium alloy (wt%)

Al	Zn	Mn	S	Cl	Fe	Mg
3.20	1.05	0.46	0.012	0.01	0.0018	Rem

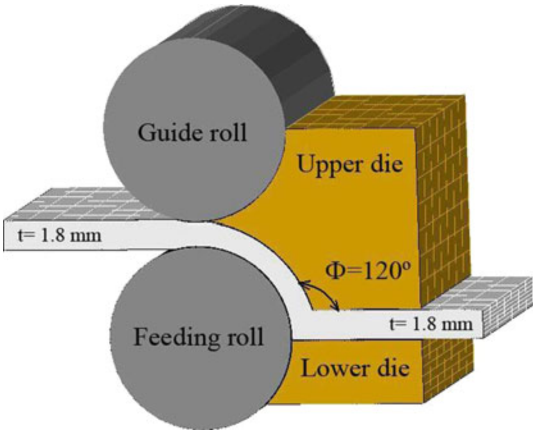

Fig. 1 A schematic illustration of the ECAR device

Before doing each pass, the sheets were rotated around the longitudinal sections by 180° in the same rolling direction. This means of rotation had been known as route “C” in ECAP. Route “C” is led to homogeneous structure [16].

Eight passes ECAR was applied to AZ31 magnesium alloy. The effect of number of passes on the microstructural changes and grain distribution of the sheets were examined by optical microscopy and image tool software. Abrasive papers were used for polishing, and then the sheets were etched at room temperature by a solution of 10 mL acetic acid, 4.2 g picric acid, 10 mL H_2O, and 70 mL ethanol [17]. The X-ray diffraction (XRD) spectra were used to examine the change of texture and formation of twins during ECAR. Tensile tests were performed at room temperature under a constant cross-head speed condition (0.4 mm/min). Microhardness tests were carried out on the sheets using a Vickers hardness tester with pyramidal diamond indentor by imposing a load of 100 g for 5 s.

Results and discussion

Microstructures

Figure 2a shows the optical microstructure of the (a) as-received; (b) 2-pass ECARed; (c) 6-pass ECARed, and (d) 8-pass ECARed specimens. As shown in Fig. 1a, the grain structure of the as-received AZ31 magnesium alloy was not homogeneous before ECAR process. This material had coarse grains adjacent of fine ones, and an average grain size of about 21 μm. The initial non-uniform microstructure is changed during ECAR process. As is shown, many grains were refined after 8-pass, and the microstructure exhibited more uniformity and smaller grain size. This indicates that severe plastic deformation could significantly refine grains. These results are in agreement with those previously reported for equal channel angular extrusion (ECAE) of magnesium alloy [18]. These refinement and homogeneity are due to the large accumulated shear strain imposed by ECAR.

Figure 3 shows the distribution of grain size through multi-pass ECAR, determined by the image tool software. This figure clearly shows that the ECARed microstructure following several passes had been converted to reasonably homogeneous microstructure with more limited distribution of grain size.

According to Fig. 3, the initial as-received specimen had grains ranging from 5 to 60 μm with an average grain size of about 21 μm. The average grain size was gradually decreased with pass number, and after 8-pass it had been decreased significantly to about 3.9 μm. It seems that

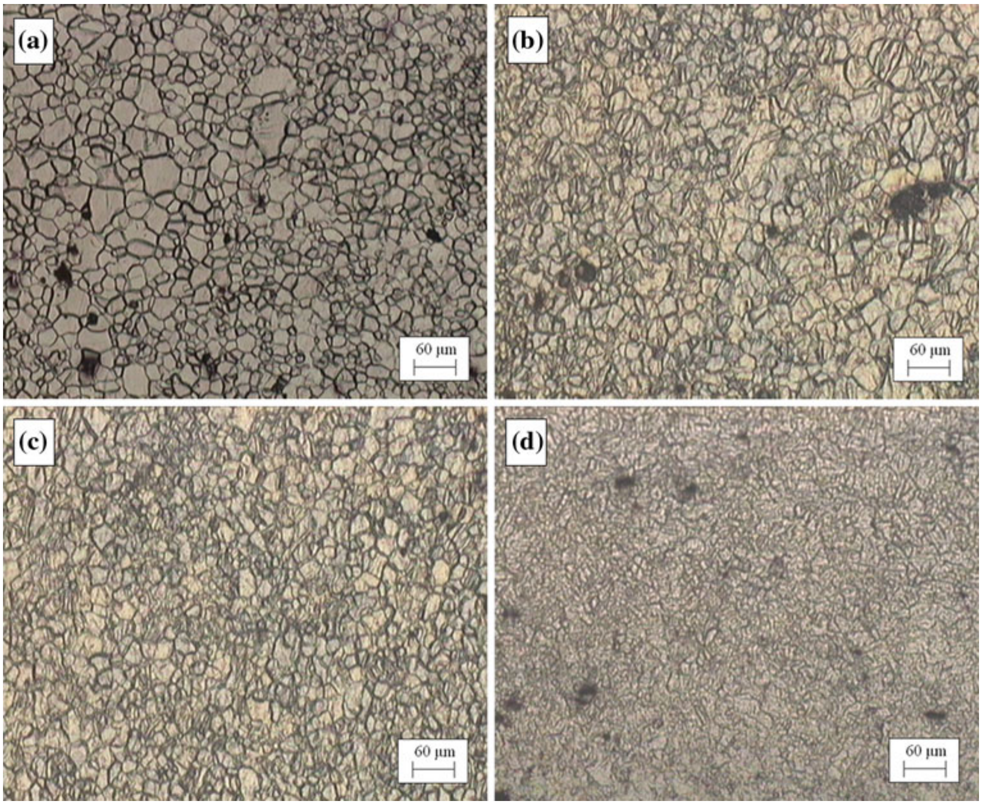

Fig. 2 Optical microstructure of **a** as-received; **b** 2-pass ECARed; **c** 6-pass ECARed, and **d** 8-pass specimens

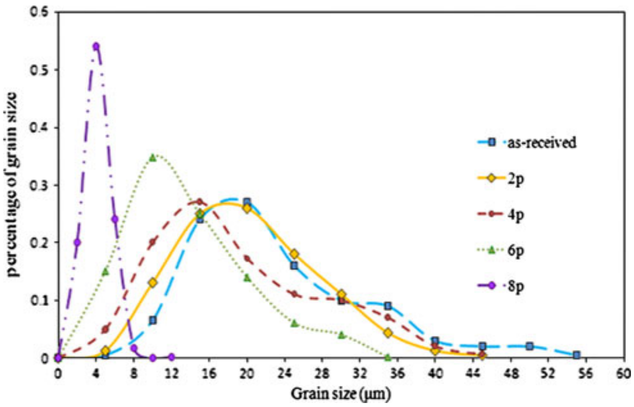

Fig. 3 Distribution of grain size of AZ31 magnesium alloy for as-received and ECARed specimens

increasing the pass number significantly caused reducing distribution of grain sizes. It means that after several passes, the range of grain size is limited. The 8-pass specimen had grains in range of 1–12 μm which had been clearly decreased compared to as-received specimen.

The mechanism of grain refinement varies with materials. Different mechanisms have put forward for the grain refinement of Mg alloys during the SPD process. Twining and dynamic recrystallization (DRX) can easily occur during different process upon magnesium alloys resulting in refined grains and improvement of mechanical properties [19].

At the initial stage of ECAR a lot of dislocations are produced. These dislocations accumulate in the large grains, and at the latter passes of process the energy due to existence of dislocation is high enough for initiation of recrystallization. Moreover, higher degree of deformation due to the increase in pass number is caused to greater amount of recrystallization [20]. The best regions for occurrence of DRX are grain boundaries and twin boundaries *and free surface* where the local degree of deformation is highest [21].

The grain size remains nearly stable before four passes, and then it changes significantly with the number of ECAR passes. The reason for this phenomenon is that the grains were grown up during the repeated heating associated with the multiple rolling passes. Thus, it appears that, after four passes the grain refinement due to the number of ECAR passes is more significant compared to the grain growth due to the repeated heating. Moreover, the grain size was not so refined before four passes indicating that the recrystallization did not significantly take place. As Zhu et al. [19] reported, DRX is more difficult to initiate in the regions without twins, so the lower amount of DRX was occurred could be attributed to lower twins existed in structure before 4-pass in compare to those were in higher passes. As it is well known, pyramid twinning $(10\bar{1}1)$ can take place when the deforming temperature is below 498 K [12]. Moreover, existence of twins during ECAR process could be attributed to the fast dissipation of heat from sheet to ECAR mold [22]. In fact before 4-pass twinning were observed in some grains, but twinning traces were observed in most grains of sixth and eighth passes of specimens in the sections perpendicular to normal direction. The twinning traces which were observed in sixth pass specimen are shown in Fig. 4. Twins and grain boundaries can be the nucleation sites for DRX [19]. Therefore, after 4-pass, DRX along with the strain which is imposed due to high number of passes could significantly refine the grains of specimen.

An important point which was observed in optical microstructure of specimens is that after several passes of ECAR processing, two distinct regions were distinguished. (i) The top surface layer of sheet, which was the contact surface of roller and sheet and (ii) the region which was standed in the center of the specimens in longitudinal sections. This means that the grain size is increased with increasing distance from the top surface. The micrograph of the longitudinal section of 4-pass ECAR specimen has been shown in Fig. 5. These two regions are clearly visible in this figure, the upper section of picture is the (i) region and the lower section of picture is the (ii) region. After sufficiently high number of ECAR processing was accomplished, (i) region is extended to reach to the (ii) region and the distribution of grains becomes more homogeneous, and the difference between these two zones would be reduced. In fact, this inhomogeneity would be disappeared after several passes.

X-ray results

XRD spectra of the primary and ECARed specimens for planes perpendicular to normal direction are shown in Fig. 6. High intensity of (0002) basal plane is observed in the primary, before ECAR processing, specimen. While, for specimen after 4-pass ECAR processing (Fig. 6b) highest intensity value is observed for $(10\bar{1}1)$ pyramid plane. This is due to rotation of basal plane after process. Cheng et al. [6], reported that after ECAR processing the basal plane in most grains incline 45° against the rolling direction. This trend is due to the shear strain applied by ECAR processing [5].

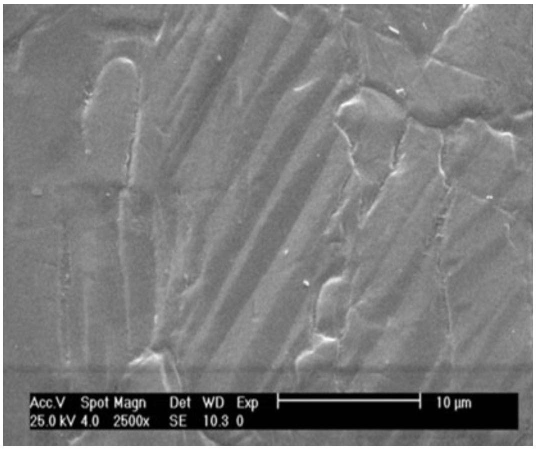

Fig. 4 Twinning trace inside the grains of sixth pass specimen

Furthermore, Fig. 6 shows that the intensity of $(10\bar{1}0)$ prismatic plane is increased after four passes of ECAR processing. The higher intensity of two non-basal planes which are observed in XRD results indicated the activation of non-basal slip systems in AZ31 magnesium alloy after ECAR. Activation of these systems confirms rotation of basal plane.

The XRD results also shows the higher intensity of $(10\bar{1}2)$ plane for 4-pass ECARed specimen compare to primary specimen. This plane and $(10\bar{1}1)$ plane are related to twin planes in hexagonal closed packed (HCP) structure materials. So ECAR processing could increase the intensity of twin planes along planes perpendicular to normal direction. This indicates that twins are also the important deformation mechanism for AZ31 magnesium alloy after ECAR [15]. Existence of twins also confirms the activation of other slip systems [19], and cause to better ductility after process.

Mechanical behavior

Tensile properties

Results of tensile tests of the as-received and ECARed specimens for AZ31 magnesium alloy sheets at room temperature are shown in Fig. 7. The yield strength (YS) of

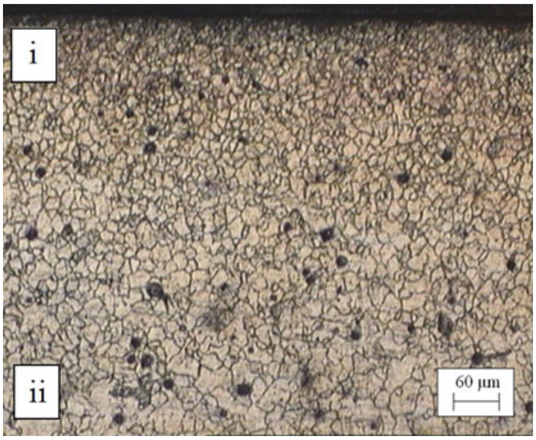

Fig. 5 Optical microstructure of 4-pass ECARed specimen in longitudinal section with two distinct regions of (*i*) contact surface of roller and sheet and (*ii*) center of longitudinal section

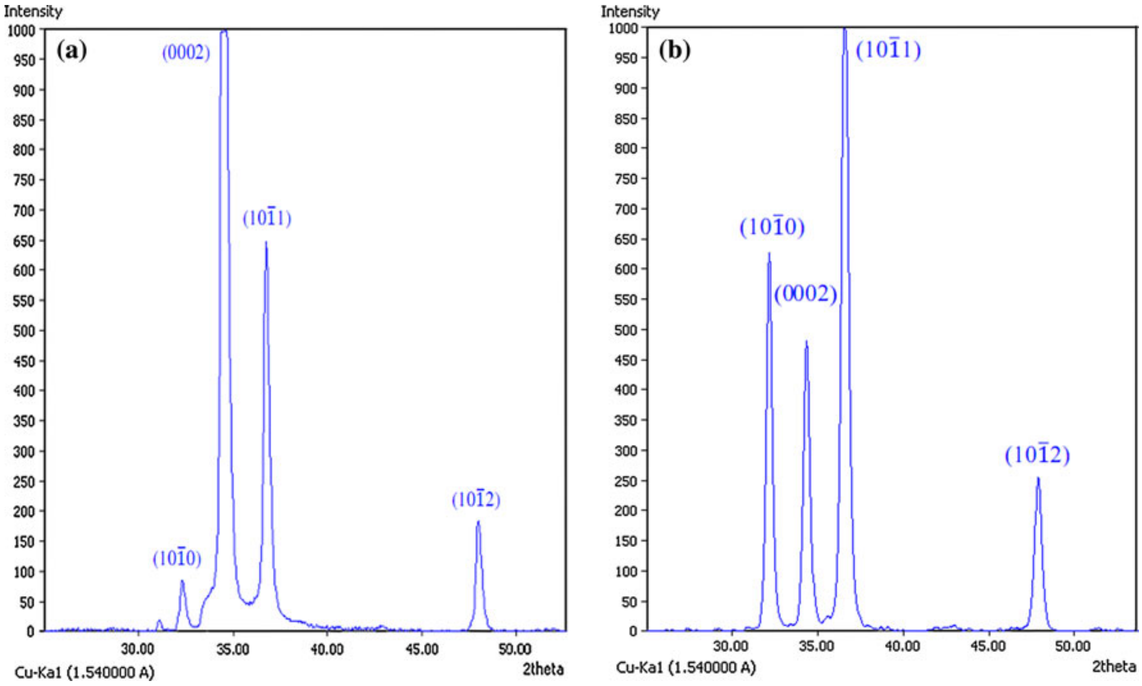

Fig. 6 X-ray diffraction patterns of AZ31 magnesium alloy for **a** as-received condition and **b** four passes ECAR specimen

specimens is reduced after process and higher strain hardening and better ductility is observed after ECAR. Temperature, texture of specimens, twins, and number of passes are important factors affecting the changes of strength and ductility of specimens after ECAR. Results showed that in spite of considerable grain refinement was occurred in Mg alloy, this sample with relatively small grain size exhibited a lower yield and ultimate tensile stress than unECARed sample, but the tensile ductility is improved after ECAR. These trends could be attributed to texture modification during ECAR, primary slip occurs on basal plane in Mg alloys at room temperature with limited non-basal slip activities. Slip on the basal plane would be unfeasible, resulting in an increase of yield strength, even with the larger grain size in the unECARed samples. Texture modification during ECAR is led to the rotation of the basal planes to the orientations favored for slip (Fig. 6), and consequently, it is caused to lower yield stress and higher elongation after ECAR. Large strain hardening after yielding which is clearly shown in the stress–strain curve of ECARed specimens confirms the activation of two or more slip planes as the consequence of rotation of slip planes.

The elongation-to-failure is increased from 8% in as-received material to 19% after two passes of process. The improvement of ductility could be related to the high temperature of ECAR process and the texture of the specimen. Increasing the temperature of process provides an effect similar to annealing. Therefore, the dislocation density and internal stress would be decreased by increasing the processing temperature and then strength would be decreased slightly, but the ductility is increased [13]. In addition, after two passes many portions of basal planes are oriented more favorably for slipping, and they caused higher ductility and lower strength.

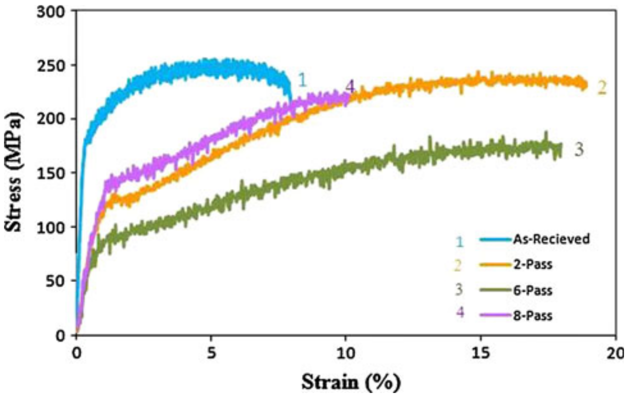

Fig. 7 Stress–strain curves for AZ31 magnesium alloy for as-received and ECARed specimens

As indicated in Fig. 6, intensity of twins is increased after ECAR. The lower strength of 6-pass ECAR specimen compared to that in second pass could be attributed to the presence of plenty of twins. In fact, twins could activate non-basal slip systems, and are caused to lower strength of sixth pass specimen. The lower ductility and higher strength of eighth pass specimen could be attributed to the lower temperature of eighth pass compared to sixth pass. Moreover, the smaller grain size achieved after several pass according to Hall–Petch relationship is another reason for these behaviors.

It could be concluded that texture softening due to existence of twins and grain refinements are the most important factors competing to one another during ECAR processing of AZ31 magnesium alloy. Therefore, at the initial passes of ECARed specimen's texture softening because of rotation of basal plane was more significant compare to grain refinement strengthening, and was caused to better ductility and lower strength. Later, the grain refining was dominated, so the strength slightly was increased at eighth pass.

The strain hardening exponent (n) was derived for samples using Hollomom equation ($\sigma = K\varepsilon^n$) for uniform deformation section of stress–strain curves. Results had been shown in Fig. 8. According to Fig. 8, higher strain hardening exponent is observed for specimen after ECAR than that before it. The (n) values are about 0.11, 0.33, 0.32, and 0.27, respectively, for the as-received, 2-pass, 6-pass, and 8-pass specimens. The high strain hardening observed after yield point for ECARed specimens confirms the activation of more slip planes due to the rotation of basal plane [23]. Also, higher strain hardening after ECAR could be the result of piles-up of dislocation on grain and twin boundaries of specimen, and interaction occurred between them.

Microhardness

The value of the Vickers microhardness, Hv, was determined for each sheet after process. The results are plotted in Fig. 9 against the number of passes. Apparently, the microhardness increases significantly after two passes of ECAR by a factor of 1.25. Thereafter, it is continued to increase each additional pass up to maximum value of Hv ≈ 70 after eight passes. Shaarbaf et al. [24] reported that rapid increase of hardness at low passes of process is due to strain hardening. According to Fig. 8, higher strain hardening was achieved in 2-pass ECARed AZ31 alloy, and this verified rapid increase of hardness at initial passes of process. Moreover, density of dislocations, grain size, and twins are important factors affecting the hardness values [25]. Dislocation density is increased by ECAR as a severe plastic deformation [11]. A lot of twins are observed after ECAR and more grain refinement is occurred by increasing number of passes. Therefore, as expectation; hardness values are increased up to 8-pass. The most important factor affecting hardness values in ECAR of AZ31 is grain size. It is verified that the hardness versus grain size obey Hall–Petch relationship [26, 27]. As it is well known, fine grain material which is obtained after several passes of ECAR has the grater total grain boundary area to prevent dislocation motion. Therefore, more number of passes cause to more refinement of grains, and cause high values of hardness.

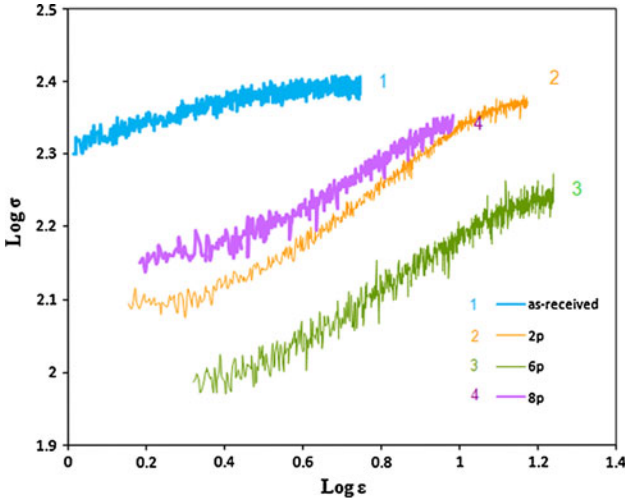

Fig. 8 Strain-hardening determined from Hollomon equation

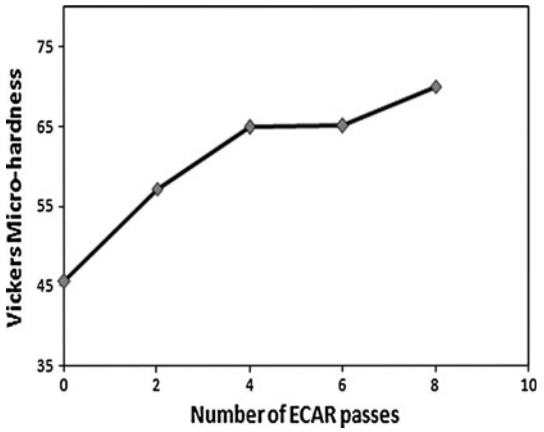

Fig. 9 Variation of microhardness with number of ECAR passes

Conclusions

Equal channel angular rolling process as a severe plastic deformation was carried out on thin sheets of AZ31 magnesium alloy up to eight passes. The main points resulted from this study are as follows:

- The grain size was reduced from 21 μm to about 3.9 μm after 8-pass of ECAR process, and distribution of grain size becomes more limited by rising the number of passes.
- Twin planes which are activated after ECAR process caused to activation of other slip systems rather than

basal slip, and due to this reason ductility of AZ31 alloy is significantly improved after process.

- Results of tensile tests showed that better ductility and strain hardening observed after ECAR could be attributed to the two most important factors (texture softening and grain refinement) competing to each other during the process. At initial passes of process texture softening was more effective, and after that, grain refinement was dominated.
- Low microhardness values of initial specimen showed an increase of about 25% at the first two passes of process, and this followed up to eighth pass. This increase in microhardness was attributed to several factors include: more number of twins, grains, and density of dislocations which were observed after ECAR.

References

1. Janeček M, Popov M, Krieger MG, Hellmig RJ, Estrin Y (2007) Mater Sci Eng A 462:116
2. Lapovok R, Estrin Y, Popov MV, Rundell S, Williams T (2008) J Mater Sci 43:7372. doi:10.1007/s10853-008-2685-z
3. Masoudpanah SM, Mahmudi R (2010) Mater Des 31:3512
4. Lin HK, Huang JC, Langdon TG (2005) Mater Sci Eng A 402:250
5. Zhu SQ, Yan HG, Xia WJ, Liu JZ, Jiang JF (2009) J Mater Sci 44:3800. doi:10.1007/s10853-009-3513-9
6. Cheng YQ, Chen ZH, Xia WJ (2007) Mater Character 58:617
7. Figueiredoa RB, Langdon TG (2009) Mater Sci Eng A 501:105
8. Barnett MR (2007) Mater Sci Eng A 464:1
9. Yunbin H, Qinglin P, Yinjiang Q, Xiaoyan L, Wenbin L (2010) J Mater Sci 45:1655. doi:10.1007/s10853-009-4143-y
10. Han JH, Oh KH, Lee JC (2004) Mater Sci Eng A 387–389:240
11. Nam CY, Han JH, Chung YH, Shin MC (2003) Mater Sci Eng A 347:253
12. Cheng YQ, Chen ZH, Xia WJ, Zhou T (2007) J Mater Process Technol 184:97
13. Huang CX, Yang G, Gao YL, Wu SD, Zhang ZF (2008) Mater Sci Eng A 485:643
14. Yi SB, Zaefferer S, Brokmeier HG (2006) Mater Sci Eng A 424:275
15. Cheng YQ, Chen ZH, Xia WJ (2007) J Mater Sci 42:3552. doi:10.1007/s10853-007-1559-0
16. Valiev RZ, Langdon TG (2006) Prog Mater Sci 51:881
17. Xia K, Wang JT, Wu X, Chen G, Gurvan M (2005) Mater Sci Eng A 410–411:324
18. Jin L, Lin D, Mao D, Zeng X, Chen B, Ding W (2006) Mater Sci Eng A 423:247
19. Zhu SQ, Yan HG, Chen JH, Wu YZ, Liu JZ, Tian J (2010) Scr Mater 63:985
20. Ma A, Jiang J, Saito N, Shigematsu I, Yuan Y, Yang D, Nishid D, Nishida Y (2009) Mater Sci Eng A 513–514:122
21. Chang CI, Du XH, Huang JC (2008) Scr Mater 59:356
22. Kim WJ, Yoo SJ, Chenb ZH, Jeongc HT (2009) Scr Mater 60:897
23. Agnew SR, Duygulu O (2005) Int J Plast 21:1161
24. Shaarbaf M, Toroghinejad MR (2008) Mater Sci Eng A 473:28
25. Hamu GB, Eliezer D, Wagner L (2009) J Alloy Compd 468:222
26. Hakamada M, Nakamoto Y, Matsumoto H, Iwasaki H, Chen Y, Kusuda H, Mabuchi M (2007) Mater Sci Eng A 457:120
27. Kim HK, Kim WJ (2004) Mater Sci Eng A 385:300