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Effect of equal channel angular pressing on microstructure, texture, and high-cycle fatigue performance of wrought magnesium alloys

The magnesium alloys AZ80 und ZK60 received from Dead Sea Magnesium in as-cast conditions were extruded at $T = 350\,^{\circ}C$ using an extrusion ratio of $ER = 22$. The extruded bars were severely plastically deformed by equal channel angular pressing (ECAP). Multiple ECAP processing up to 8 passes was done. The ECAP-induced changes in grain size and grain size distribution were measured by transmission electron microscopy while changes in dislocation density and crystallographic textures were determined by positron annihilation spectroscopy and X-ray diffraction analysis, respectively. The strain induced by ECAP was found to influence the microstructural characteristics, in particular the grain size, the dislocation density, and the crystallographic texture, which in turn enhance (or deteriorate) the mechanical or fatigue response of both alloys.

Keywords: Magnesium alloys; AZ80; ZK60; HCF; Positron lifetime spectroscopy

1. Introduction

Among the various methods of severe plastic deformation, equal channel angular pressing (ECAP) [1, 2] is particularly attractive since very large strains can be achieved by repeatedly pressing the material through a die having an angular channel. Ultrafine-grained bulk materials can be produced without changing the cross-sectional dimensions of the billet [1, 2]. While ECAP-induced ultrafine-grained microstructures are often reported to possess superior mechanical properties such as enhanced yield stress, tensile elongation, and superplasticity [3–5] less information is available regarding high-cycle fatigue (HCF) performance. It has been shown that the massive shear deformation during the ECAP process may result in unfavourable crystallographic textures in AZ80 [6]. The objective of the present work is to correlate the HCF performance with microstructure and texture development in two commercial Mg alloys processed by ECAP.

2. Experimental procedure

The materials used in this work were wrought magnesium alloys AZ80 (Mg-8.6% Al-0.5% Mn-0.21% Mn) and ZK60 (Mg-5.9% Zn-0.5% Zr) which were received as chill-cast material from Dead Sea Magnesium, Israel. The material was extruded at $T = 350\,^{\circ}C$ to an extrusion ratio $ER = 22$ using a 630 t direct extrusion press. ECAP was conducted up to 8 passes via route B_C using a die whose design is described elsewhere [7]. Pressing was done starting at $T = 250\,^{\circ}C$ for the first pass and decreasing the temperature by 10 K for each of the following passes.

Specimens for transmission electron microscopy (TEM) observation of the ECAP-generated microstructure were taken from the middle part of the billet perpendicular to the pressing direction. TEM foils were prepared by ion milling using a Gatan PIPS™ ion mill at 4 kV and an incidence angle of 4°. TEM investigations were performed with a Philips CM 200 electron microscope operated at 200 kV.

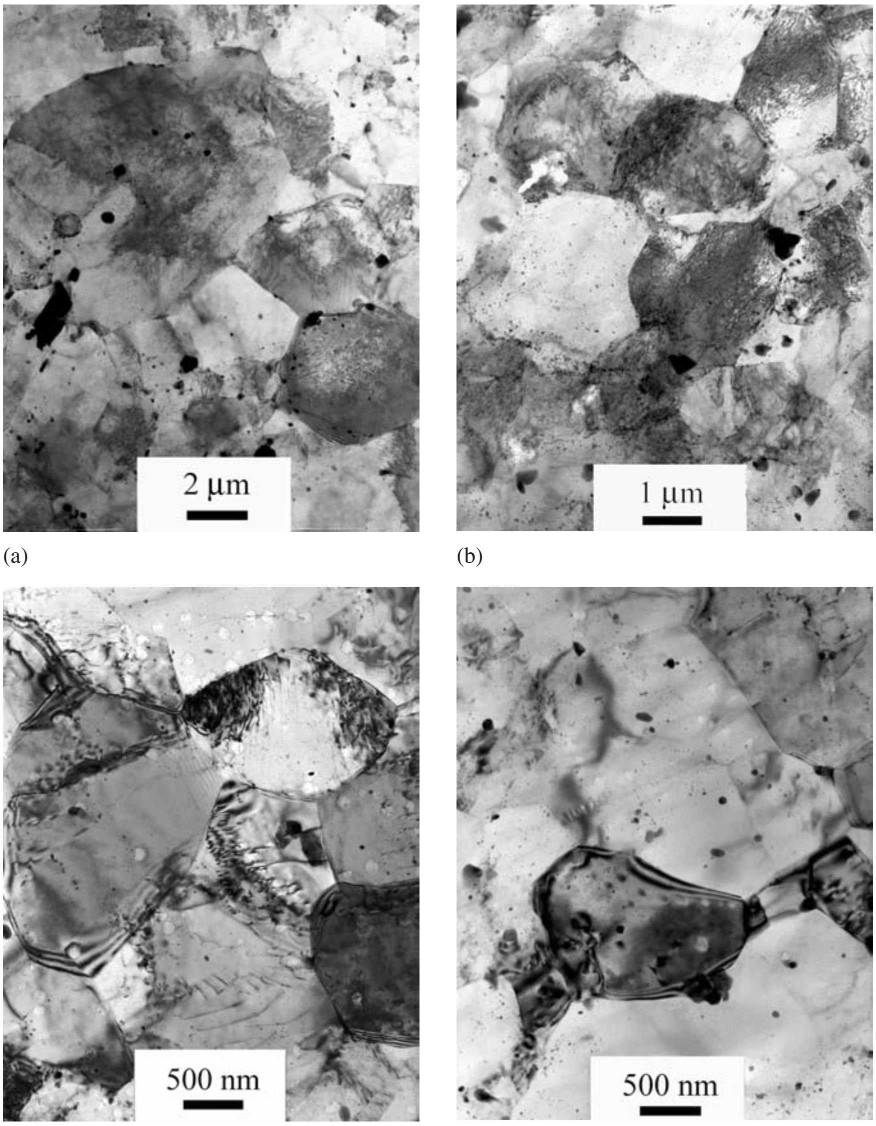
Macroscopic crystallographic textures of the various conditions were determined by X-ray diffraction using Co-K_{α} radiation and the results will be presented in terms of (0002) pole figures.

For the positron lifetime (annihilation) spectroscopy (PAS) a $^{22}Na_2CO_3$ positron source ($\sim$1.5 MBq) deposited on a 2 μm thick Mylar foil was used. This source always formed a sandwich with two identically treated specimens. Positron lifetime (PL) measurements were performed using a fast-fast spectrometer [8] with a timing resolution of 160 ps (FWHM ^{22}Na). At least 10^7 positron annihilation events were accumulated at each PL spectrum which was subsequently decomposed using a maximum likelihood procedure [9].

3. Results and discussion

3.1. Microstructure development

Hot extrusion of both alloys resulted in significant grain refinement (factor of 10 approximately). The average grain size after extrusion was approximately 10 μm with slightly larger grains in AZ80 than in ZK60 (cf. Fig. 1a) (the microstructure of ZK60 alloy is shown here only). ECAP pressing caused further grain fragmentation with gradual reduction in grain size and a transition from the nonequilibrium grain-boundary structure consisting predominantly of boundaries with typical fuzzy contrast with a high density of dislocations as in the specimen after 2 passes (2 P) (cf. Fig. 1b) to rather equilibrium high-angle boundaries with a typical fringe contrast in specimens after 4 and 8 passes



(a) (b) (c) (d)

Fig. 1. TEM micrographs of the ZK60 alloy (a) EX, b) 2 P, (c) 4 P, (d) 8 P.

(Fig. 1c–d). Moreover, the density of dislocations within the grains gradually decreased with increasing strain due to ECAP. Many new dislocation-free recrystallized grains were observed in the 8 P-specimen (cf. Fig. 1d). The average grain size in the AZ80 alloy after 2, 4 and 8 passes was 5 μm, 2 μm, and 1 μm, respectively. Slightly smaller grains were observed in the ZK60 alloy resulting in grain sizes already in the submicrometer range in the 8 P-specimen.

3.2. Texture evolution

The texture evolution in terms of (0002) pole figures is displayed in Figs. 2 and 3 for AZ80 and ZK60, respectively. Starting with the as-extruded conditions, the (0002) pole figures of both AZ80 (Fig. 2) and ZK60 (Fig. 3) are characteristically affected by the various passes of ECAP. In the as-extruded conditions (cf. Figs. 2a, 3a), most grains are oriented with their basal planes parallel to the extrusion direction, marked by 'L'.

The as-extruded texture in AZ80 (Fig. 2a) is much weaker than that of ZK60 (Fig. 3a). This originates from the difference in dynamic recrystallization of the two alloys. Extrusion of AZ80 leads to an almost fully recrystallized microstructure while ZK 60 only partially recrystallizes. After 2 passes of ECAP, the texture component corresponding to the extrusion process is still observed (cf. Figs. 2b and 3b). More importantly, a new texture component is formed by shear-induced rotation of grains in such a way that their basal planes become aligned at 45° to the pressing direction, marked by 'x'. As the number of ECAP passes increases from 2 to 8, the intensity of this texture component is clearly becoming more intense in both AZ80 (Fig. 2) and ZK60 (Fig. 3). However, this marked orientation change induced by ECAP-shear requires a higher number of passes in ZK60 than in AZ80. After 8 passes, almost all grains of AZ80 have their *c*-axis 45° tilted away from the pressing direction (Fig. 2d) while in ZK60 the initial (EX) texture component is still clearly visible (Fig. 3d). Besides the difference in the initial texture intensities between the two alloys, it is presumably the different amounts of secondary-phase particles mostly located at grain boundaries that are responsible for the differences in texture development. More secondary-phase particles are present in ZK60 which can hinder grain rotation by plastic deformation.

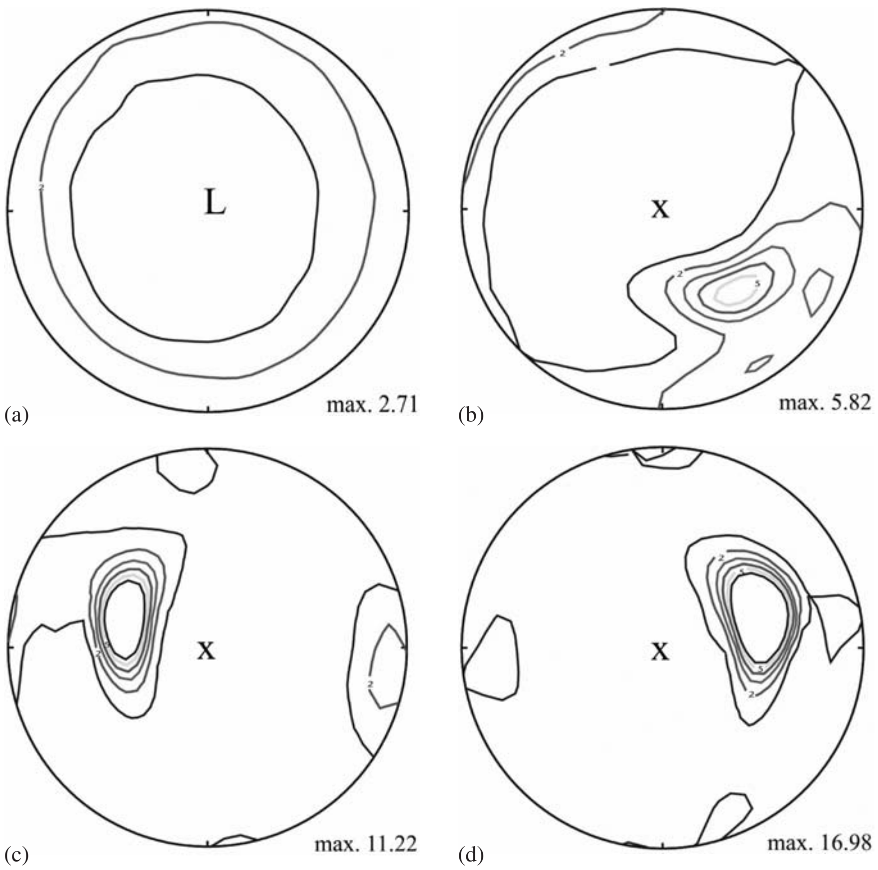


Fig. 2. (0002) pole figures of the various conditions for AZ80 (L = ED and x = ECAP-pressing direction) (a) EX, (b) 2 P, (c) 4 P, (d) 8 P.

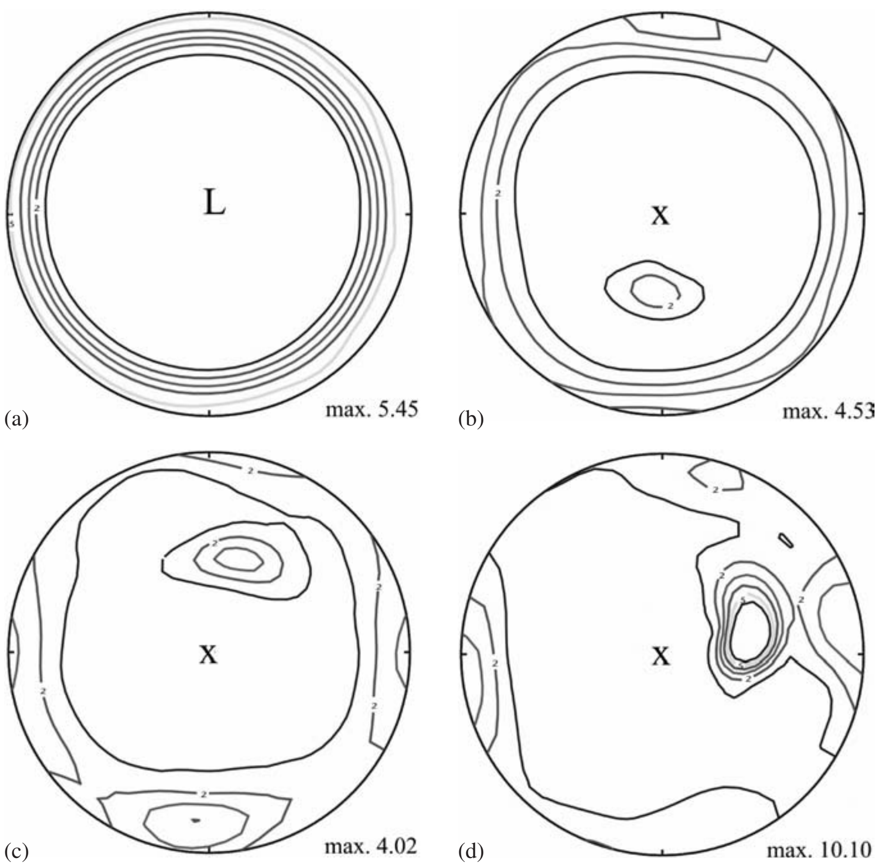
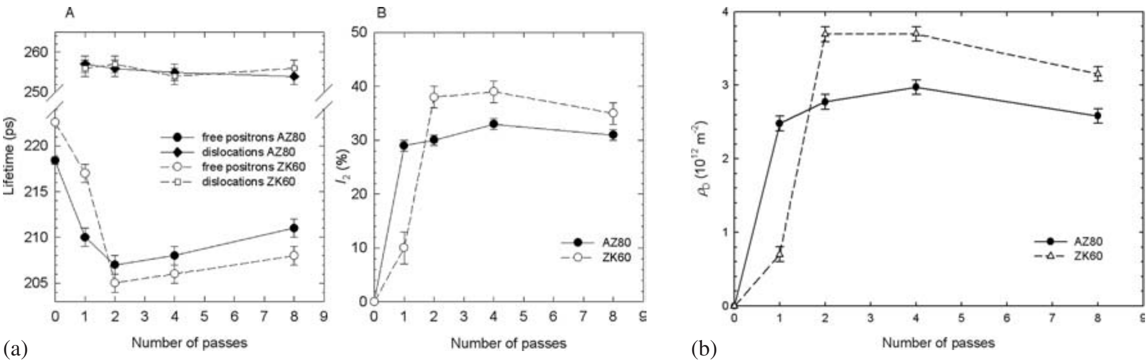


Fig. 3. (0002) pole figures of the various conditions for ZK60 (L = ED and x = ECAP-pressing direction) (a) EX, (b) 2 P, (c) 4 P, (d) 8 P.

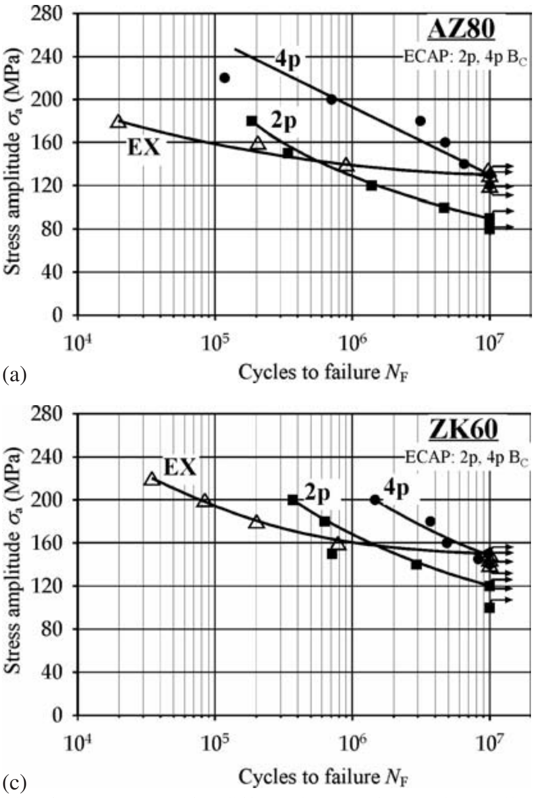


(a) (b)

Fig. 4. (a) PL results for AZ80 and ZK60 subjected to various numbers of ECAP passes: (A) lifetimes τ_1 and τ_2 of the free positron and dislocation component, (B) intensity I_2 of the components arising from positrons trapped at dislocations; (b) Development of dislocation density ρ_D with increasing number of ECAP passes.

3.3. Positron annihilation spectroscopy

Virgin coarse-grained AZ80 and ZK60 alloys exhibit a single component PL spectrum with lifetimes (218.4 ± 0.4) ps and (222.6 ± 0.2) ps, respectively. These lifetimes are comparable to the free positron lifetime in well annealed Mg [9]. Thus, the coarse-grained alloys contain a very low density of defects, i. e., below the lower sensitivity limit of PL spectroscopy. Virtually all positrons are delocalized in the lattice and annihilate from the free state. On the other hand, PL spectra of the alloys subjected to various numbers of ECAP passes exhibit two-component spectra. In addition to the short-lived free positron component with lifetime τ_1, a new component with lifetime $\tau_2 \approx 260$ ps appeared in PL spectra. This component arises from positrons trapped at dislocations introduced by severe plastic deformation during ECAP processing. Figure 4a shows the development of lifetimes τ_1, τ_2 (A) and of the relative intensity I_2 (B) of the dislocation component with increasing number of ECAP passes. It is clear from the figure that the lifetime of positrons trapped at dislocations remains approximately constant documenting that the nature of positron traps does not change during ECAP processing. On the other hand, the intensity I_2 of positrons trapped at dislocations increases in specimens subjected to 1 and 2 ECAP passes reflecting an increasing number of dislocations. The intensity I_2 saturates at 4 ECAP passes and slightly decreases in the specimens subjected to 8 ECAP passes. The alloys deformed by ECAP contain indeed a single type of defects, namely dislocations which are distributed homogeneously in the specimens. The dislocation density, ρ_D, is plotted in Fig. 4b as a function of the number of ECAP passes. Obviously, the main increase in the dislocation density occurs after 1 and 2 ECAP passes. Note that ρ_D in alloys subjected to 2 ECAP passes is comparable with that measured in heavily cold-rolled Mg (thickness reduction of about 40 %) [10]. Further ECAP processing does not result in an additional increase in ρ_D and the density of dislocations even moderately decreases when the number of ECAP passes exceeds 4. This indicates dynamic recovery of dislocations during ECAP processing. One can see in Fig. 4b that the overall behaviour of ρ_D in both alloys deformed by ECAP is similar, but ZK60 exhibits a higher dislocation density than AZ80.



(a) (c)

Fig. 5. S–N-curves for differently manufactured Mg-alloys.

3.4. The HCF performance

Two passes of ECAP clearly reduce the HCF strengths of the as-extruded references in both AZ80 (Fig. 5a) and ZK60 (Fig. 5b). This result may be explained by the detri-

Table 1. The summary of the results of monotonic testing.

	Grain size (μm)	$\sigma_{0.2}$ (MPa)	UTS (MPa)	El (%)
		AZ80		
EX	10	225	310	15
2× ECAP	5	220	305	15
4× ECAP	2	280	335	10
8× ECAP	1	165	255	12
		ZK60		
EX	9	295	340	15
2× ECAP	3	285	335	16
4× ECAP	1	310	340	18
8× ECAP	0.8	220	270	17

mental effect of the ECAP-induced texture component. Obviously, this detrimental effect is not fully compensated by the beneficial effect of grain-size refinement (cf. Fig. 5a–b). The HCF performances after 4 passes of ECAP are much superior to those observed after only 2 passes. This result is due to the further decrease in grain size and concomitant increase in yield stress and agrees well with the observations of other authors [11–13]. For comparison of the HCF performance with that of monotonic testing the summary of results of mechanical testing is given in Table 1. The detail analysis of mechanical behaviour is given elsewhere [7].

4. Conclusions

The development of an ultra-fine grained microstructure in the wrought Mg alloys AZ80 and ZK60 processed by severe plastic deformation using ECAP were determined by TEM, texture measurements, and PAS. Tensile and fatigue tests were conducted to analyze the mechanical performance.

The following conclusions may be drawn from this investigation:

1. Significant refinements in grain size (by a factor of 5–10) relative to the as-extruded condition were achieved by ECAP.
2. ECAP results in the development of a shear-induced unfavourable texture component.
3. PAS revealed a moderate dislocation density in ECAP specimens. After a sharp increase the dislocation density tends to saturate even showing a slight decline with further ECAP straining.
4. The highest HCF strength values were observed in the as-extruded material and after 4 ECAP passes.

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References

[1] R.Z. Valiev, R.K. Islamgaliev, I.V. Alexandrov: Prog. Mater. Sci. 45, No. 2 (2006) 1.
[2] R.Z. Valiev, A.V. Korzikov, R.R. Mulyukov: Mater. Sci. Eng. A 168 (1993) 141.
[3] J. Jiang, A. Godfrey, W. Liu, Q. Liu: Scripta Mater. 58 (2008) 122.
[4] M.D. Nave, M.R. Barnett: Scripta Mater. 50 (2004) 881.
[5] L. Jiang, J.J. Jonas, A.A. Luo, A.K. Sachdev, S. Godet: Scripta Mater. 54 (2006) 771.
[6] S.R. Agnew, P. Mehrotra, T.M. Lillo, G.M. Stoica, P.K. Liaw: Mater. Sci. Eng. A 408 (2005) 72.
[7] J. Müller, M. Janeček, L. Wagner: Mater. Sci. Forum Vols. 584–586 (2008) 858.
[8] F. Becvář, J. Čížek, L. Lešták, I. Novotný, I. Procházka, F. Šebesta: Nucl. Instr. Meth. A 443 (2000) 557.
[9] I. Procházka, I. Novotný, F. Bečvář: Mater. Sci. Forum 255–257 (1997) 772.
[10] J. Čížek, I. Procházka, B. Smola, I. Stulíková, V. Ocenášek: J. Alloys Comp. 430 (2007) 92.
[11] Hilpert, A. Styczynski, J. Kiese, L. Wagner, in: B.L. Mordike, K.U. Kainer (Eds.), Magnesium Alloys and their Applications, MATINFO (1998) 319.
[12] M. Hilpert, L. Wagner, in: H.I. Kaplan, J.N. Hryn, B.B. Clow, (Eds.), Magnesium Technology 2000, TMS (2000) 375.
[13] H.-K. Kim, Y.-I. Lee, C.-S. Chung: Scripta Materialia 52 (2005) 473.

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Bibliography

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