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Effect of the second phase on the microstructure of magnesium alloys during cyclic extrusion compression

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Abstract: Cyclic extrusion compression (CEC) is an effective severe plastic deformation (SPD) process which can be used for fabricating ultrafine grained light materials such as magnesium alloys. This method introduces three-dimensional compression and shear stresses and the process can be repeated for a certain number of passes until the desired accumulated strain has been introduced. In order to reveal the effect of second phases on the microstructure developed in magnesium alloys during CEC, three different alloys (AZ31, AZ31-1wt.%Si and AZ91) were investigated after CEC 7 passes performed at 225˚C. The experimental results show that the CEC process can effectively refine the microstructures of these alloys and the mean grain size achieved is 1.3μm, 1.5μm and 1.4μm, respectively. It is revealed that the grain size, grain shape and grain boundary structures are little affected by coarse phase Mg_2Si but strongly affected by the fine phase $Mg_{17}Al_{12}$. The fine phase $Mg_{17}Al_{12}$ seems to increase the relative grain misorientations, hence enhancing the formation of high angle grain boundaries (HAGBs). It is expected that such changes are improving mechanical properties, subsequent forming behavior and surface quality.

Introduction

In recent years, severe plastic deformation (SPD) such as equal channel angular pressing (ECAP) [1], CEC [2] and accumulative roll bonding (ARB) [3] has been applied to magnesium and its alloys in order to obtain grain refinement and to improve ductility and strength at room temperature. However, some researches [4] have reported that strength increases with the accumulated strain while other results [2] show the opposite trend. There are therefore discussions in the literature about the effects of microstructure including grain size, grain-boundary structure and crystallographic texture in regard to strength of fine grained magnesium alloys after SPD [1, 2]. Further, the characteristics of second phase particles could play an important role upon microstructure development during SPD – especially when performed at elevated temperatures.

The present work investigates the effects of both particle numbers and the type of particles on the microstructure development. In order to compare the effect of particle quantity, the AZ31 alloy characterized by a small amount of fine $Mg_{17}Al_{12}$ particles and the AZ91 alloy having a large population of fine $Mg_{17}Al_{12}$ particles were employed. In addition, the AZ31-1Si alloy was selected to investigate and compare the effect Mg_2Si particles on the microstructure development.

Experimental procedure

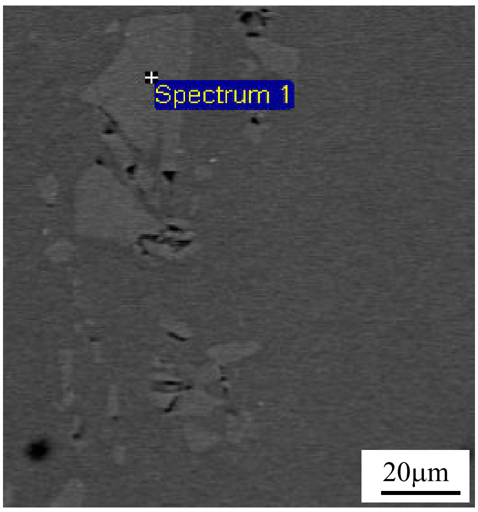
The actual chemical compositions of the employed alloys were determined in an inductively coupled plasma (ICP) analyzer, i.e. see Table 1. The alloys were processed in the as extruded condition. Both the extruded bar and the CEC die were held for 10 minutes at 100˚C and coated with a graphite powder lubricant before heating to 225˚C for about 2 hours. The sample was then put into the die to start the CEC processing carried out at 225˚C and an extrusion rate of 7mm/s. After CEC 7 passes, i.e. an accumulated strain of 10.5 [2], longitudinal sections at the representative center position were prepared for electron backscattered diffraction (EBSD) analyses in the scanning electron microscope. The successful sample preparation for EBSD investigations involved mechanical grinding with successively SiC papers to 2400 ASTM MESH followed by mechanical polishing to 1μm before final polishing with a diluted OPS solution (10%). Thereafter, electropolishing was performed with an AC2 solution using a voltage of 15V for 10-20s at temperature -30˚C before cleaning with methanol and acetone.

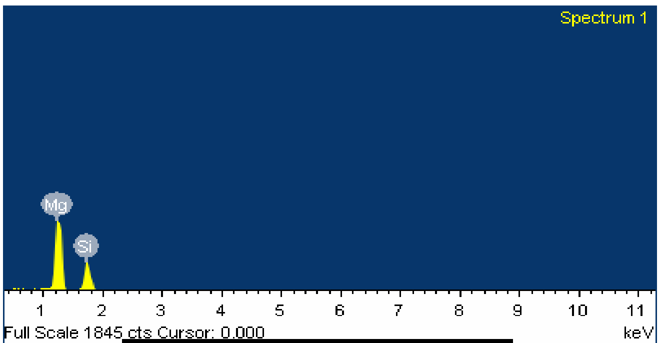
Table 1 Composition of the studied alloys (wt, %)

Alloy	Al	Zn	Mn	Si	Mg
AZ31	3.091	1.023	0.421	-	Bal.
AZ31-1Si	2.971	0.687	0.377	1.32	Bal.
AZ91	8.282	0.754	0.223	-	Bal.

Results and discussion

The microstructure of the AZ31-1Si contained numerous relatively large particles of the type Mg_2Si. Fig.1 shows a Mg_2Si particle in the extruded alloy and the corresponding EDAX analysis results, hence confirming this phase composition. The Mg_2Si particles in the as-extruded condition were located in clusters and had a lot of cracks. Even after CEC processing, the distribution of Mg_2Si particles remained very similar.





Element	Weight%	Atomic%
Mg K	7.16	66.34
Si K	4.20	33.66
Totals	11.35	100

Fig.1 Mg_2Si phase in extruded AZ31-1Si and the corresponding EDAX analysis.

Fig.2 shows the overall grain structures of the three alloys after CEC 7 passes at 225˚C. Here, the boundaries with relative misorientations less than 2^o were removed due to the resolution limitations of the EBSD technique in this case. In alloy AZ31 a typical bimodal grain size distribution was quite evident (Fig. 2a) and the fraction of small grains constituted a dominating part. Further, the fine grains tended to form network structures [2] and this alloy seemed to be well suited for formation of grains at the smallest scale. Also, in alloy AZ31-1Si the grain size distribution exhibited a bimodal nature although the coarse grains dominated over the finer grains (Fig. 2b). On the other hand, the grain structure obtained in alloy AZ91 tended to be reasonable equiaxed as evident from the micrograph in Fig.2c. These observations indicate that additional alloying in the AZ31 alloy system do not increase the number fraction of fine grains, hence larger amounts of second phase particles seemed to promote less bimodality. However, comparing alloys AZ31 and AZ31-1Si versus AZ91 it was evident that the latter alloy developed a more homogeneous and fine grain size distribution. Moreover, the fractions of low angle grain boundaries (LAGBs) and high angle grain boundaries (HAGBs) measured from Fig.2 are listed in Table 2. These results show that low fractions of LAGBs, i.e. below 10% can be obtained after CEC processing at 225˚C in the present three alloy systems. Coarse Mg_2Si particles (AZ31-1Si) seemed not to promote formation of more fine scaled grains [5] and the population of coarser grains dominated over the fine grains. However, the fine particles of the type $Mg_{17}Al_{12}$ present in alloy AZ91 promoted grain refinement and increased the fraction of HAGBs.

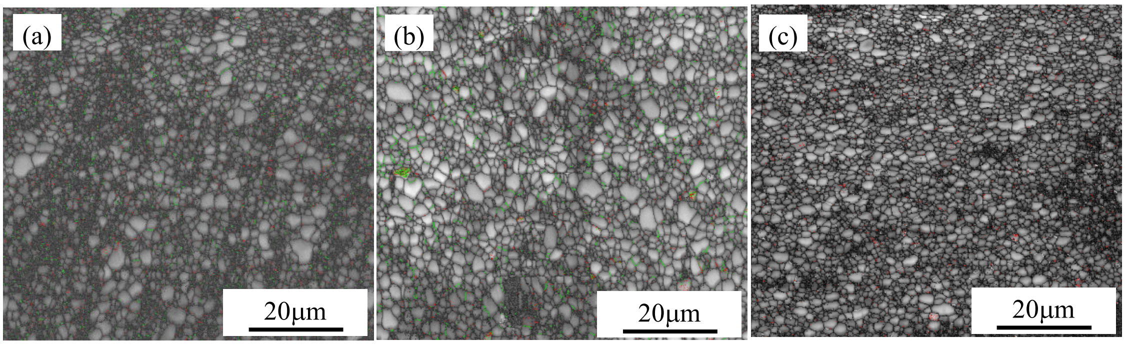


Fig.2 Grain structure maps viewed in the longitudinal section (SEM-EBSD) after CEC 7 passes at 225˚C, (a) AZ31, (b) AZ31-1Si and (c) AZ91.

Table 2: Fraction (%) of LAGBs and HAGBs in the present alloys after CEC 7 passes at 225˚C.

Misorientation angles	AZ31	AZ31-1Si	AZ91
LAGBs (2^o-5^o)	1.7	3.2	1.7
LAGBs (5^o-15^o)	5.6	5.8	2.3
HAGBs (>15^o)	92.7	91.0	96.0

The grain size area fraction distribution of the present alloys can be seen in Fig. 3. Fig.3a presents that the maximum grain size for AZ31 was ~5.2μm and the average grain size was about 1.3μm. Similarly, Fig.3b shows that the maximum grain size for AZ31-1Si was ~5.6μm and the average grain size was close to 1.5μm. Compared with AZ31, the AZ91 alloy contained more particles of the type $Mg_{17}Al_{12}$. The coarse grains were obviously refined by these particles and they also trigged a relative fine average grain size, i.e. ~1.4μm (Fig. 3c). It should be remarked, however, that the population of fine grains having a size below 1μm in the AZ91 alloy were obviously less than present in the AZ31 alloy. In other words, the present results showed that the $Mg_{17}Al_{12}$ particles seemed to promote a refinement of coarse grains but did not stimulate a more extended grain refinement.

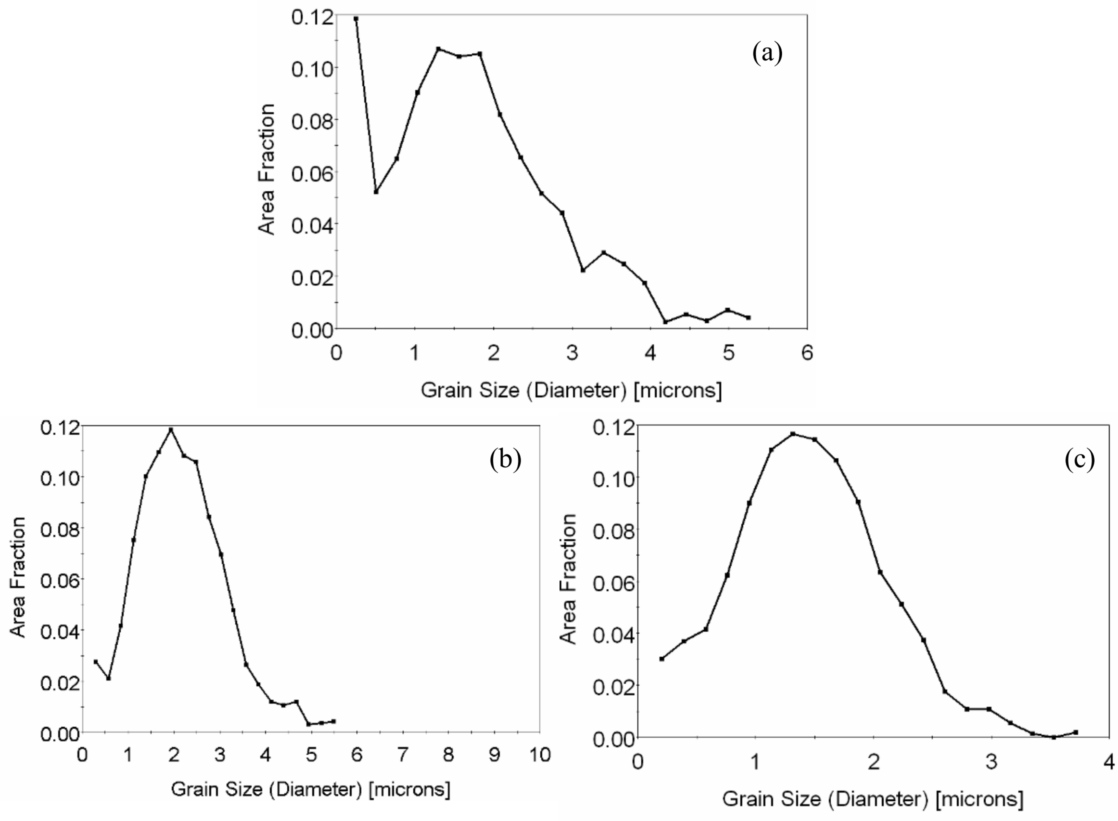


Fig.3. Grain size area fractions in the present alloys after CEC 7 passes at 225˚C, (a) AZ31 (b) AZ31-1Si and (c) AZ91.

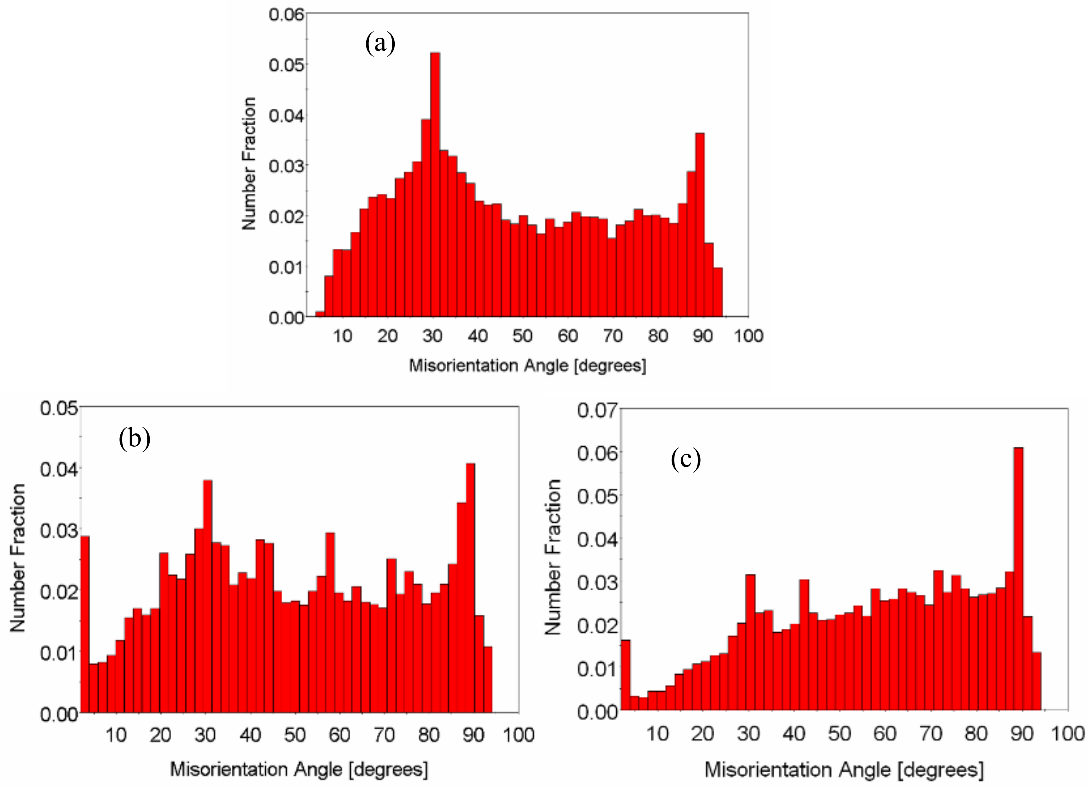


Fig.4 Grain boundary misorientations in the present alloys after CEC 7 pass at 225 ˚C, (a) AZ31 (b) AZ31-1Si and (c) AZ91.

The grain boundary misorientation data of the present alloys after CEC are shown in Fig.4. In alloy AZ31 two different frequency peaks could be distinguished around angles 30° and 90° respectively (Fig. 4a). Similar results have earlier been reported for this alloy after ECAP [6]. However, the reasons behind this observation remain unclear. It should also be noted that the observed average grain misorientation was ~50˚ after CEC 7 passes of alloy AZ31. Further, there were more LAGBs in AZ31-1Si than in AZ31, as shown in Fig.4b and Table 2. Concerning the average grain boundary misorientation, alloy AZ31-1Si had a similar value as in AZ31. The present results also displayed that the average misorientation in AZ91 was significantly larger than in the other two alloys, i.e. ~57˚ (seen in Fig.4c). This indicated that the fine $Mg_{17}Al_{12}$ particles contributed to an increase in the average grain boundary misorientation.

Summary

(1) The microstructures of magnesium alloys AZ31, AZ31-1Si and AZ91 could be refined when subjected to CEC 7 passes at 225˚C and the average grain size was 1.3μm, 1.5μm and 1.4μm, respectively.

(2) The finest grains tended to form network structures and the potential to produce fine grains decreases with the increase of second phase particles of the type Mg_2Si in the AZ31 alloy system.

(3) The fine phase $Mg_{17}Al_{12}$ contributed to an increase of relative grain misorientations and to the formation of HAGBs and finer grains.

(4) The grain size, grain shape and grain boundary structures in the present alloys seemed to be little affected by the coarse phase Mg_2Si.

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