A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 53 2008 Issue 1

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COMPARISON OF MICROSTRUCTURE AND TEXTURE DEVELOPMENT OF ECAPED PURE Mg WITH A Mg-SI ALLOY

PORÓWNANIE ROZWOJU MIKROSTRUKTURY I TEKSTURY CZYSTEGO Mg I STOPU Mg-SI W PROCESIE ECAP

The evolution of microstructures and texture during ECAP processing of pure Mg and Mg-Si alloy with route A were investigated. The results showed, that the degree of grain size refinement for pure Mg was limited after 2 passes, because of a relatively high processing temperature. Two different types of Mg_2 Si were obtained: firstly, relatively coarse polygonal Mg_2 Si grains and secondly, the Chinese type eutectic mixture of Mg with Mg_2Si. These hard particles of Mg_2 Si have an high influence on the development of texture and microstructure during ECAP. A kind of basal-plane-type texture was formed for both materials, where the (0002) pole figure showed a strong orientation maximum, normal to the deformation plane. The misfits from the ideal 45° tilting angle related to the 90° die geometry come from the construction details of the ECAP device. The monoclinic deformation symmetry by shearing was not reached because of friction, which resulted in a simple rotation along the ECAP-direction.
Keywords: ECAP, Mg_2Si alloy, texture, route A

Wyciskanie w kanale kątowym (ECAP), obiecująca technika dużych odkształceń plastycznych jest z sukcesem stosowana do szerokiego zakresu metali i materiałów metalicznych. Teoretycznie prosty model odkształcenie-ścinanie spowodował szerokie badania unikalnej mikrostruktury i tekstury po procesie ECAP i ich odniesienie do polepszenia własności. Jednakże, wpływ parametrów przetwarzania – takich jak: kąt rozwarcia kanału, liczba przejść przez kanał i obrót próbki pomiędzy przejściami – na mechanizm rozdrobnienie ziarna nie są w pełni zrozumiałe. Ponadto, wpływ drugiej fazy na rozwój tekstury podczas procesu ECAP był dotychczas rzadko badany.

W prezentowanych w pracy badaniach odkształcanie ECAP czystego Mg i stopów Mg-Si, o składzie chemicznym 3.26%Si, 0.04%Mn, 0.03%Zn, 0.02%Fe (proc. wag.) z dopełniającym udziałem Mg przeprowadzono dla jednego do czterech przejść, stosując przeciskane przez kanał prostokątny na drodze A (bez obrotu pomiędzy przejściami). Teksturę próbki przy odkształceniu ECAP po każdym przejściu, analizowano techniką dyfrakcji neutronów w TEX-2 Geesthacht w Niemczech. Funkcje rozkładu orientacji wyliczono metodą rozwinięć szeregowych do rzędu rozwinięcia L_{max} = 22.

Obserwacje wykonane przy pomocy mikroskopii orientacji pokazały, ze wielkość ziarna w czystym Mg maleje od około 900 μm dla odlewu, do około 100 μm już po jednym przejściu dla obydwóch materiałów oraz, że "chiński typ eutektyki" (twarda faza metaliczna Mg_2Si w stopie Mg-Si) zaczął pękać i rozprzestrzeniać się wzdłuż kierunku pod kątem około 45° do kierunku wyciskania. Po dalszym odkształcaniu, do 4 przejść, zmniejszenie wielkości ziarna w czystym Mg nie było tak widoczne i nastąpił pewien wzrost ziarna. Dendrytyczna eutektyka Mg_2Si niemal całkowicie się rozpadła, podczas gdy pękanie pierwotnych wydzieleń gruboziarnistego poligonalnego Mg_2Si było bardzo ograniczone. Wskutek negatywnego wpływu Mg_2Si wielkość ziaren macierzystego stopu Mg-Si dalej się zmniejszała.

Systematyczna analiza rozwoju tekstury wykazała, że dla obu materiałów formowała się tekstura bazalna, wówczas gdy kąt nachylenia do kierunku wyciskania był mniejszy niż idealny 45°, co można uważać raczej za złożone odkształcenie niż za proste ścinanie, a niekontrolowalne warunki tarcia mogą być także odpowiedzialne za obserwowany dodatkowy obrót głównego bieguna bazalnego. Płaszczyzna bazalna poruszała się niemal równolegle do kierunku wyciskania wraz ze wzrostem liczby przejść przez kanał. Intensywność (ostrość) tekstury w wyciskanym w kanale kątowym czystym Mg była wyższa niż w stopach Mg-Si, co wskazywało, że istnienie Mg_2Si "randomizuje" odkształcenie podczas przetwarzania materiału.

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1. Introduction

Equal channel angular pressing (ECAP) – a promising severe plastic deformation (SPD) technique – has been successfully applied to a very wide range of metals and metallic materials [1–4]. Due to its theoretically simple shear deformation model, unique microstructures and texture after ECAP processing were widely investigated and related to the improved properties [3–6]. However, the influence of processing parameters – such as channel die angle, the number of pressing passes and billet rotation angle between two passes – on the grain refinement mechanism and their influence on grain refinement and homogeneous grain distribution are still debatable [7, 8]. Moreover, the correlation between texture development and the deformation mechanism during ECAP under different conditions was rarely investigated for hexagonal materials [2].

Magnesium offers a large potential in applications requiring weight reduction, because of its low density of about 1.73 g·cm^{-3}. However, the relatively low strength and plasticity greatly hinder its wide applications [9]. An effective way of improving the mechanical properties of Mg consists in producing ultra fine grained materials [5, 10, 11]. Magnesium easily becomes anisotropic, because of its inner HCP crystallographic structure (HCP), which requires a combination of different glide systems and twinning. A homogenous and fine grained microstructure should reduce this anisotropy. The unique texture developed by ECAP processing are investigated and discussed in other materials, mainly cubic metals [3–7, 12, 13]. It was also known, that the crystallographic texture has a strong influence on the behaviour of materials under tension and compression [14, 15]. The present ECAP studies have shown, that a remarkable grain refinement can be obtained as a result of a shearing [2]. Those researches have shown, that the developed textures were similar to those in torsion, in which deformation also occurred by simple shearing [2, 16]. While recent studies indicated, that the new texture was formed not simply as a result of simple shearing, but also due to an additional strain component [13, 17]. The reason for this texture development is still debatable. Apart from severe plastic deformation, a grain refinement can also be caused by a co-deformation with a second, harder phase. The investigations of composites also showed, that reinforcements will decrease the texture sharpness in case of most reinforcement systems [18–20]. Si is a candidate, which forms hard intermetallic particles as Mg_2Si.

Texture analysis makes it possible to estimate the mechanism responsible for the progress of plastic straining during ECAP processing. The present study is aimed to investigate comparative the effect of the second phase on the evolution of microstructure and texture in pure Mg and Mg-Si alloy. The nature of the texture evolution will be analyzed and co-related to the shearing deformation mode during ECAP, which could contribute to a better understanding of deformation during ECAP.

2. Experimental procedures

The material used for the current investigation is the commercially cast pure Mg and Mg-Si alloy, prepared by a normal casting method. The Mg-Si alloy has the following chemical composition: 3.26%Si, 0.04%Mn, 0.03%Zn, 0.02%Fe, and Mg balance (in wt %). A rectangular billet for ECAP with a dimension of 12×12×60 mm^3 was machined from a cast ingot. ECAP processing was carried out through a half-separated die made of SKD60 with an internal die angle ϕ of 90°. Molybdenum disulphide (MoS_2) was used as lubricant. The billet was first inserted into the die and heated up to 350°C for about 30 min, and pressed thereafter under a speed of 20 mm·min^{-1}. Processing route A – where there is no rotation of the billet around its axis between each pass – was performed from 1 pass to 4 passes for both materials with a final equivalent strain of about 4 [1].

Optical microscopy (OM) was used to observe the microstructures of the ECAPed materials. The OM projection plane was parallel to the ND-ED plane (Y plane as shown in Fig.1). The polished surface was etched by a solvent, consisting of 10 ml of nitric acid, 30 ml of acetic acid, 40 ml of water, and 120 ml of ethanol. In this work, neutron four-circle diffractometer at TEX-2 with the wave length of 1.239 Å(GKSS Research Center, Germany) was used to characterize the crystallographic texture. Due to it is high transmission, neutron diffraction is powerful for texture analysis of different types of bulk materials [21]. A bulk sample with the dimensions 6×12×12 mm^3 was cut from the central part of the ECAPed billet for texture measurement. Measurements covered four complete pole figures (0002), $(10\bar{1}0)$, $(10\bar{1}1)$, $(11\bar{2}0)$ and average texture over the whole sample results, caused by the large beam cross section of about 16 mm. The direction of the neutron beam diffraction is parallel to the extrusion direction X. The definition of the coordinate system for texture characterization is shown in Fig. 1. All four measured pole figures were used to recalculate the complete pole figures and the orientation distribution function (ODF) by the iterative series expansion method, based on Bunge's system definition of Euler angles φ_1, Φ, φ_2 with the expansion degree of l_{max} = 22 [21].

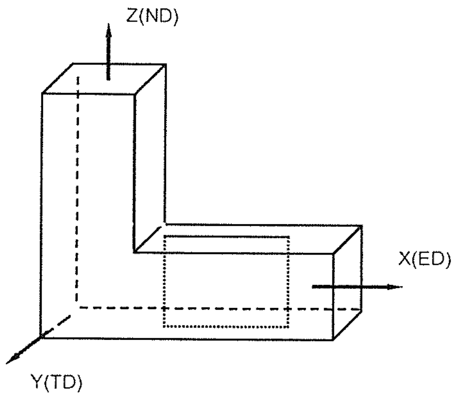

Fig. 1. Definition of the sample coordination system X, Y and Z are extrusion, transverse and normal directions of the ECAPed billet

3. Results and discussion

3.1. Microstructures

Optical microstructures in the ECAPed pure Mg and Mg-Si alloy from 1 pass up to 4 passes are shown in Fig. 2, respectively. For pure Mg, the grain size was decreased from 900 μm in the cast condition to grains between 20 μm and 150 μm after 1 pass, see in Fig. 2 (a) and (b). Large grains were surrounded by the smaller grains, which formed due to the dynamic recrystallization (DRX). The microstructure is still very inhomogeneous. Additional decrease of the grain size after 2 passes of processing is observed. However, a slight increase of the grain size is obtained by recrystallization after 4 passes of processing. As can be seen, the degree of grain size refinement for the pure Mg is very limited in case of 2 passes of ECAP processing. The main reason could consist in the relatively high processing temperature, which makes it easy for the grains to grow. Otherwise, the billet would be broken if the ECAP processing temperature was decreased.

In the case of the cast Mg-Si alloy, two types of intermetallic Mg_2Si are found. Firstly, the primary precipitated polygonal grains, that can be seen in Fig. 2 (e). The grain size is < 20 μm. The grains are inhomogeneously accumulated. The second type is the eutectic Mg_2Si with typical dendritic or fish-bone microstructure of Mg grains of about 150 μm. During ECAP, the hard and more or less round Mg_2Si grains rotate without any deformation. This can bee seen during all passes of the processing. Long dendritic Type II Mg_2Si is broken during ECAP and orients along grain boundaries of Mg-grains, which shows an inhomogeneous distributions of grain sizes, with a large number of grains less than 10 μm.

It can be noted, that the hard Mg_2Si precipitation results in a much finer grain distribution, compared to pure Mg. After 4 passes of processing, all large Mg-grains in the Mg-Si alloy are refined and the slight grain growth observed in pure Mg after 4 passes of processing is not found in Mg-Si alloy (Fig. 2 (d) and (h)). Mg2Si works as a barrier for DRX. It must be indicated, that Type I Mg_2Si is very hard and the hardness difference between Mg_2Si and pure Mg [22] is too large for a refinement. Therefore, we propose to use Si grain refiner to reduce the volume fraction to obtain only Type II Mg_2Si.

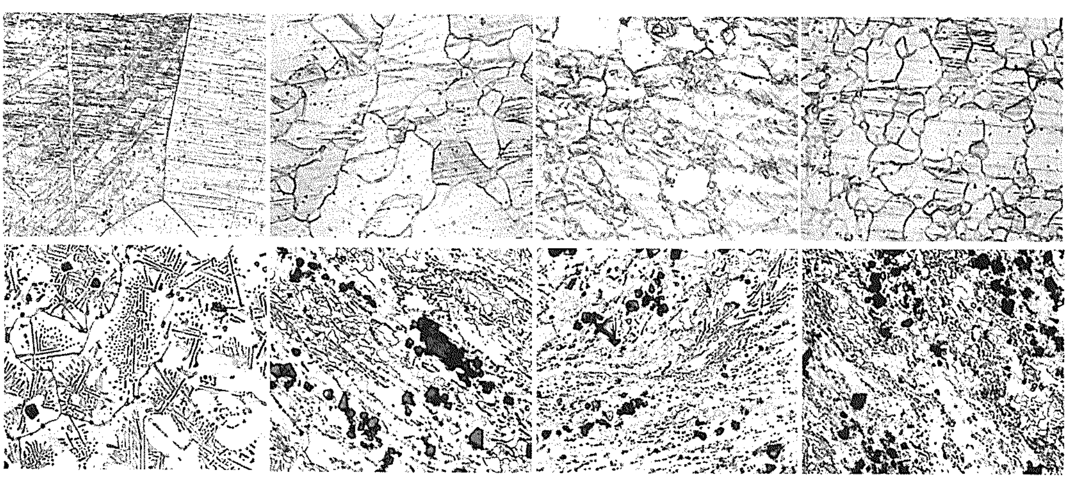

Fig. 2. Optical microstructures of the ECAPed pure Mg (the first row) and Mg-Si alloy (the second row) after different passes: (a) and (e) – 0 passes, (b) and (f) – 1 pass, (c) and (g) – 2 passes, (d) and (h) – 4 passes

3.2. Texture evolution

Fig. 3 (a) and (b) show the complete (0002) and (10$\bar{1}$0) pole figures of the ECAPed pure Mg and Mg-Si alloy, obtained by 1 to 4 passes via route A, respectively. The pole figures are obtained by means of the orientation distribution function ODF, and the corresponding ODF sections at constant φ_2 of 0°, 30° and 60° are shown in Fig. 4. There was no preferred orientation in the initial as-cast pure Mg and Mg-Si alloy; and some high intensity points in pole figure of the cast samples were obtained due to the strong diffraction of coarse grains.

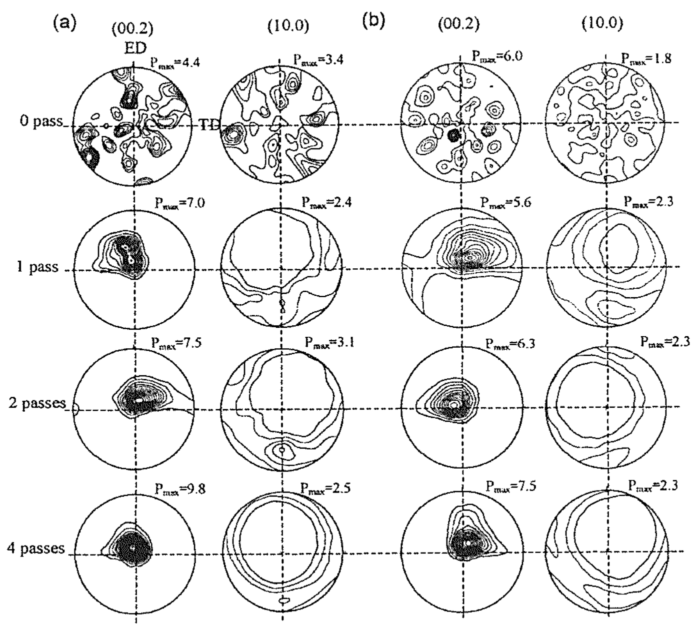

Fig. 3. Recalculated (00.2) and (10.0) completely pole figures (00.2) and (10.0) of the ECAPed (a) pure Mg and (b) Mg-Si alloy from 1 pass to 4 passes, respectively. Contour levels: 1.0×, 1.5×, 2.0×,...

The main texture component in the ECAPed pure Mg splits into a double maximum in pole figure (0002), which is similar to such figure obtained for rolled Mg. This texture can be attributed to the more activations of the $< c + a >$ dislocation sliding [12, 14, 23]. It can also be seen, that this double maximum is rotated due to the shearing caused by ECAP, and there existed a small off titling angle to the transverse direction. This double maximum is reduced to a single maximum after 2 and 4 passes, but the degree of texture intensity increases from F_{max} = 7 mrd (1 pass) to F_{max} = 9.8 mrd (4 passes). Together with the increase of the preferred orientation, the central maximum orients closer to the normal direction (centre of the pole figure).

In the Mg-Si alloy, the texture of the cast sample is dominated by large grains. After 1 pass, a more complex texture was obtained, compared to pure Mg. A strong single maximum is added by a girdle, which is not present in pure Mg. The texture is little weaker, see Fig. 3 (a) and (b). Thereafter, the texture develops in a similar manner in case of Mg-Si alloy and pure Mg. A single maximum with increasing orientation density develops from F_{max} = 5.6 mrd (1 pass) to F_{max} = 7.5 mrd (4 passes), as shown in Fig. 3 and Fig. 4.

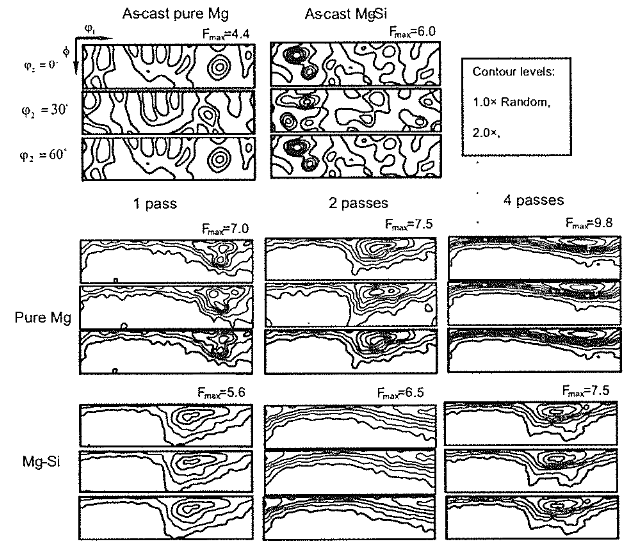

Fig. 4. ODF sections at constant φ_2 of 0°, 30°, 60° of the ECAPed pure Mg and Mg-Si from 1 pass to 4 passes, respectively

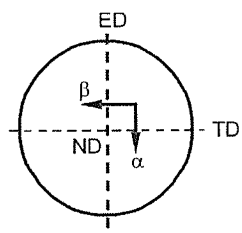

Fig. 5. Definition of the tilting angle α and β in pole figure (0002)

TABLE 1

Tilting angle α and β (defined in Fig. 5) in (0002) pole figure of the ECAPed pure Mg and Mg-Si

pass	Cast pure Mg			Cast Mg-Si		
	P_{max}	α	β	P_{max}	α	β
1	6.0	30	−10	5.0	20	17
2	7.0	17	10	5.5	5	−13
4	9.0	13	5	7.0	17	11

It is of particular interest, that the deviation of the main maximum from the ND-direction in pole figure (0002). Table 1 summarizes this evolution for both ECAPed materials from 1 pass to 4 passes. Firstly, it is found, that the tilting angle α is less than 45°, which is expected theoretically by a 90° die angle of the ECAP-device. Secondly, there is also a misfit to the symmetry axis between ECAP-direction and TD-direction. It is remarkable, that this misfit varies between −13° and +17°. Similar results were already published, but the reasons are still under discussion [1, 17, 24].

A smaller angle α indicates, that the shearing plane is not strictly oriented at 45° to the shearing direction, which could be mainly due to the curvature angle of two channels. This curvature is part of the device geometry, aimed to reduce the pressure force. The tilt angle goes close to 0° with the increasing number of passes. Another explanation can also be related to the high ECAP-temperature and the subsequent recrystallization. α for 1 pass is much higher (30°) than for Mg-Si (20°), that would mean there is also a material-dependent component, not only the curvature angle between the two channels responsible for α. More experiments are necessary to give better conclusions.

The misfit angle β shows a variation, which cannot be explained by the ECAP process itself. Theoretically, ECAP will lead to monoclinic pole figure symmetry with a symmetry axis form ECAP-direction to TD-direction. Texture simulation shows, that a second shear plane is necessary to describe the evolution of these textures. If this second shear plane is based on the ECAP-device by the construction, the misfit angle β should always be positive or negative. A variation of β between −13° and +17° is more related to friction conditions during ECAP. One consequence is that the sample preparation between the passes is very important. Current simulation work should contribute to a better understanding of the complex deformation process and the explanation of asymmetric texture with the evolution of tilt angle β.

4. Conclusions

Grain size in the ECAPed pure Mg was greatly decreased after 1 pass ECAP processing, but tended to be stable after 2 passes because of a relatively high processing temperature, which forces dynamic recrystallization. The Mg-Si alloy forms two types of Mg_2Si precipitations, globular grains about 10 μm in size and eutectic Mg_2Si. Mg_2Si is much harder. Only the dendritic Mg_2Si was broken during ECAP. The other grains showed no deformation, but they rotated. The texture develops in both materials in a similar manner, but with lower texture sharpness in Mg-Si alloy, while the grain refinement is lager in Mg-Si alloy. The variation in the main texture maximum of the (0002) pole figure is remarkable. It leaves some unanswered questions about the influence of the detailed die geometry, friction conditions and a material-dependent component.

Acknowledgements

The author highly appreciates the finical support granted to the research in the form of the DAAD-Helmholtz scholarship. We are grateful for the permission to use the beam time at TEX-2 FRM-I, Geesthacht, Germany. This work is supported by the National Natural Science Foundation of China under the awarded scholarship No.50571031.

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Received: 3 December 2007.