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Microstructural evolution in Mg-3Gd during accumulative roll-bonding

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Abstract: An Mg-3Gd (wt. %) alloy has been deformed by accumulative roll bonding through four cycles to an equivalent strain of 3.2. The deformed microstructure has been fully characterized by advanced electron microscopy techniques and the mechanical properties are determined by tensile testing. Three characteristic microstructures have been identified and characterized as: cell blocks, twin blocks and nanograins, all with boundary spacings of 100-200 nm. These structures illustrate a structural subdivision by low angle dislocation boundaries and high angle boundaries, which shows a clear resemblance with deformation microstructures in fcc and bcc metals deformed to high

strain. The structural evolution has its origin in dislocation and twinning based plasticity which is quantified. The structure-strength relationship of this low-alloyed Mg-3Gd is analysed.

Keywords: Mg alloy, Accumulative roll bonding; Grain subdivision; Microstructure; Mechanical properties.

1. Introduction

Magnesium and its alloys are superior materials for lightweight automotive applications due to their high strength-density ratio [1,2]. However, a low strength and formability at ambient temperature limits their application. A characteristic of Mg alloys is their weaker response to precipitation and solid solution strengthening compared to fcc and bcc metals [3,4], but a large grain boundary strengthening points to grain size as an important strengthening parameter [5-7]. It follows that microstructural refinement by severe plastic deformation (SPD) [8,9], such as accumulative roll-bonding (ARB) [10,11], equal channel angular extrusion [12], high-pressure torsion [13], cyclic extrusion and compression [14], and repetitive extrusion-upsetting [15], may be an effective way to strengthen magnesium alloys.

In plastic deformation, dislocation glide is the dominant deformation mechanism supplemented by a limited amount of twinning in medium to high stacking fault energy fcc and bcc metals, such as Al [16], Ni [17] and Fe [18]. For magnesium, due to the low crystal symmetry and high mechanical anisotropy, both basal slip and twinning are important deformation modes at room temperature [19,20]. However, in Mg-RE (rare earth) alloys, many slip systems can be activated during plastic deformation [21-24],

which may be affected by both processing and materials parameters [25], are expected to influence the microstructural refinement and mechanical properties.

Although SPD processed Mg alloys were extensively reported in the literature [11-13], limited transmission electron microscopy (TEM) studies have been done to quantify their microstructural evolution during SPD. It is therefore the objective of the present study to understand the microstructural refinement during SPD for a selected Mg alloy based on morphological observations and quantification of structural parameters by means of TEM. The analysis and the discussion of the experimental observations are guided by the hypothesis that the many slip systems activated in an Mg-RE alloy may produce deformation microstructures which have comparable features to those formed by plastic deformation of fcc and bcc metals [26-28], where multiple glide systems are activated during loading. Tensile testing is also conducted to investigate the effect of structural refinement on the mechanical properties.

2. Experimental procedure

An Mg-3Gd (wt. %) was selected for the present study. The addition of 3 wt.% Gd is mainly based on a previous study [29] which showed that Mg-Gd alloys with Gd concentrations in the range of 2.75% - 4.65% exhibit good combinations of strength and tensile ductility, and similar and weak textures. An Mg-3Gd (wt. %) rod ingot was produced with a diameter of 100 mm by casting pure Mg and Mg-Gd master alloy in a columnar steel mould cavity using an electrical resistance furnace under Ar atmosphere. The cast ingot was homogenized at 450 °C for 24 h, followed by preheating at 450 °C for 30 min and extrusion to a plate with a thickness of 3 mm. Following steps were hot rolling to 1 mm and annealing at 500 °C for 3 h, producing initial sheet samples with an equiaxed recrystallized structure. The microstructure of the initial sheet sample with an average

grain size of 45 μm is shown in Fig. 1(a). A pole figure shows a weak $\{0002\}$ basal texture, see Fig. 1(b).

The 1 mm thick recrystallized sheet samples were used for ARB processing to apply high strains. Specimens with a length of 70 mm, a width of 25 mm and a thickness of 1 mm were prepared from the sheet. Two such specimens were stacked and roll-bonded without lubrication. The sheet samples produced from the first ARB cycle were preheated at 400 °C for 8 min and roll-bonded again, keeping the rolling direction (RD) the same as the previous cycle. During preheating, the specimens were recrystallized to a grain size of about 10 μm . This procedure was repeated up to four ARB cycles and the samples produced were dubbed after the number of cycles 1c, 2c, 3c and 4c.

Uniaxial tensile tests were performed in an AGX-50 tester system with a strain rate of 10^{-4} s^{-1} at room temperature. A laser extensometer was used to measure the sample strain during loading. Dog-bone shaped tensile specimens with a gauge length of 13 mm and cross-section of $5 \times 1 \text{ mm}^2$ were cut with the tensile direction parallel to the RD. For each sample condition, three tensile specimens were tested.

Microstructures of ARB processed samples were characterized by scanning electron microscopy (SEM) and transmission electron microscopy (JEM-2100, FEI Tecnai F20). Both the SEM and TEM samples were cut from the longitudinal section containing the normal direction (ND) and RD. The SEM samples were prepared by mechanical polishing followed by electrochemical polishing in the AC2 solution. Thin foils for TEM were grinded and then ion-milled using Gatan PIPS 691.

Fig. 1 (a) EBSD inverse pole figure showing the microstructure in the RD-ND section of the initial sheet sample (500 °C, 3h). The colours represent the crystallographic direction of the RD. (b) {0002} pole figure showing the initial texture.

3. Results

3.1 Microstructure

Typical TEM images of the Mg-3Gd samples processed from 1c to 4c are shown in Fig. 2. As seen in Fig. 2(a), the microstructure of the 1c sample has been refined to the sub-micrometer scale. The microstructure is inhomogeneous with a relatively high concentration of deformation twins. However, the microstructures after 2c to 4c (Fig. 2(b-d)) all show typical lamellar structures with lamellar boundaries (LBs) aligned along the RD. The 3c and 4c samples are therefore not further investigated leaving deformed 1c and 2c samples, respectively, where the latter shows a homogeneous microstructure subdivided by dislocation boundaries and twin boundaries. Therefore the 2c sample is chosen for a full characterization.

Fig. 2 TEM images of ARB samples, (a) 1c, (b) 2c, (c) 3c and (d) 4c. The RD and ND are marked on the images.

Fig. 3(a) shows an ECC image of the 2c sample. The structure is fully refined and the majority of the dislocation boundaries are aligned along the RD. Fig. 3(b, c) show TEM images of the lamellar structure and equiaxed grains in the 2c sample. Regular lamellar boundaries parallel to the RD are seen in Fig. 3(b). Fig. 3(c) shows a complex contrast including lamellae and equiaxed grains. More precise TEM characterization along $\langle 20 \rangle$ in Fig. 3(d) shows that the matrix contains a small amount of deformation twins (marked by white lines), stacking faults (SFs) (marked by red arrows) and low angle

boundaries (LABs, marked by yellow arrows). Inset in Fig. 3(d) shows the selected area electron diffraction (SAED) pattern of the circled area including the matrix and T1, and a LAB segment in the matrix is seen to approximately parallel to the basal plane (marked in blue).

Fig. 3 (a) SEM ECC image showing the microstructure of the 2c sample, (b, c and d) TEM images of the 2c sample with characteristic dislocation structures (b and c) and twin structure (d). Note the different magnifications in the figures.

The deformation microstructure of the 2c sample as analyzed by TEM shows three characteristic features classified as: cell blocks, twin blocks and nanograins. The

structural subdivision to the 100-200 nm scale is characterized by the boundary spacing distribution shown in Fig. 4. The three average spacings are comparable 190, 138 and 93 nm.

Fig. 4 The lamella (a) and twin (b) thicknesses measured perpendicular to boundaries. For equiaxed nanograins (c), the grain size is the equivalent circle diameter.

3.1.1 Cell blocks

A typical feature in fcc and bcc metals deforming by multiple glide is the cell block structure defined as a group of dislocation cells which is enclosed by extended boundaries. It follows that the lamellar structures shown in Fig. 2(b-d) can be classified as a cell block structures. An inspection of the volume between the lamellar boundaries shows a high density of low angle boundaries present in imperfect cell blocks [27]. Fig. 5 shows two examples of the lamellar structure. Dislocation boundaries (LABs and LBs) are found in deformed samples in a high area fraction. In Fig. 5, the structure is subdivided by parallel LABs on a very fine scale (~ 200 nm). The basal plane is marked by white lines, and the insets show SAED patterns. Due to the activation of more slip systems in Mg-3Gd than in magnesium, non-basal slip is activated at room temperature, and two sets of slip-plane-aligned cell block structures (CB1 and CB2) were found in the deformed sample in Fig. 5(b). The finding that cell blocks form on different slip planes is in accord with previously characterized fcc and bcc metals [28].

Fig. 5 TEM micrographs showing cell block structures including (a) basal-plane-aligned CB1 indicated by white dashed line, (b) two sets of dislocation structures (basal CB1 and non-basal CB2 indicated by white and red dashed lines, respectively).

3.1.2 Twin blocks

In magnesium alloys, twinning is typically observed together with dislocation structures after plastic deformation at room temperature [19,20]. Fig. 6 shows TEM images of twin structures in the 2c sample. Abundant twins and also twin-twin interactions can be seen. Such interactions are on a fine structural scale and may be part of the structural subdivision by twinning. The TEM structure in Fig. 6(c) is sketched in Fig. 6(d), which illustrates a structure of similar appearance as a cell block structure. A typical feature of a twin block is a volume enclosed by twin boundaries. An inspection of the volume between the twin boundaries shows a high density of low angle boundaries supplemental by twin boundaries and stacking faults (see Fig. 6(d)).

Fig. 6 TEM micrographs showing twin blocks (a, b and c) in the 2c sample. (d) A sketch showing adjacent twins (marked with white lines), SFs (red lines) and LABs (yellow lines) in a twin-block corresponding to Fig. 6c.

To further investigate the twin structure, high resolution TEM (HRTEM) has been applied. Within the twin blocks, nanograins can be identified, as shown in Fig. 7. Pronounced twin-twin interactions apparently take place during grain subdivision and may be origin of the nanograins. However, such a structural transition is for future research.

Fig. 7 TEM micrographs showing nanograins observed in the twin-blocks. Twin boundaries are indicated by red dashed lines, and profiles of the nanograins are highlighted with white dashed lines.

3.1.3 Equiaxed nanograins

Fig. 8 illustrates equiaxed nanograins area in a deformed sample. Inset shows the SAED pattern corresponding to this region, indicating the presence of equiaxed nanograins with a mean grain size of ~ 93 nm, see Fig. 4(c). These equiaxed grains are different from the nanograins in twin blocks in Fig. 7, which points to a different origin to be investigated. Note that these equiaxed nanograins only occupy a small volume fraction of the deformed structures (see Table.1).

Fig. 8 TEM image showing the equiaxed nanograins area in a 2c sample.

3.1.4 The volume fraction of structural features

The volume fraction of the three structural features, cell blocks, twin blocks and equiaxed nanograins are given in Table.1. In this table, the volume fraction of cell blocks and twin blocks are calculated based on a quantification of the distance between lamellar boundaries and twin boundaries, respectively. This table illustrates the significant difference between 1c and 2c samples and comparable parameters of 2c, 3c and 4c samples. Note the significant decrease in the density of twins after first and second cycle.

Table 1. Microstructural parameters

ARB samples	Equivalent von Mises strain	Microstructure					
		Cell blocks		Twin blocks		Equiaxed-nanograins	
		Lamella thickness (nm)	Vol. (%)	Twin thickness (nm)	Vol. (%)	Grain size (nm)	Vol. (%)
1c	0.8	$\lambda=237$	59	$\lambda=152$	41	/	/
2c	1.6	$\lambda=191$	65	$\lambda=138$	27	d=93	~8
3c	2.4	$\lambda=182$	67	$\lambda=132$	25	d=106	~8
4c	3.2	$\lambda=182$	66	$\lambda=126$	26	d=109	~8

3.2 Mechanical properties

Engineering stress-strain curves of the initial sample and ARB samples are shown in Fig. 9 where two stress-strain curves for each condition are include for the sake of clarity. The initial sample shows a high elongation of 21%, but a low yield stress of 93 MPa. Compared with the initial sample, the elongations of the ARB samples show a large reduction; the yield stress has however increased to about 3 times as a result of the structural refinement. ARB samples show similar high yield stress. However, early fracture was clearly found in 1c sample with a total elongation about 1.2%, which is lowest in all curves. 2c samples show best combination of strength and elongation, with a yield stress about 244 MPa and total elongation about 5.3%. With the increasing ARB cycles, 3-4c samples show a slow increase in yield stress and decrease in elongation. All mechanical property data and spacing of LBs or grain sizes are listed in Table 2.

Fig. 9 Tensile stress-strain curves of the initial sample and the samples after ARB processing to 1c, 2c, 3c and 4c. Tensile curves of two samples for each condition are included in the plot.

Table 2. Mechanical properties

	$\sigma_{0.2}$ (MPa)	σ_{UTS} (MPa)	$\epsilon_{uniform}$ (%)	ϵ_{total} (%)	Vol. (%) of twins	Grain size or lamella thickness
Initial	93	168	16.6	21.1	-	d=45 μ m
1c	259 $\pm$ 5	266 $\pm$ 2	1.2 $\pm$ 0.1	1.2 $\pm$ 0.1	41	λ =237 nm
2c	244 $\pm$ 3	257 $\pm$ 2	2.2 $\pm$ 0.1	5.3 $\pm$ 0.4	27	λ =191 nm
3c	248 $\pm$ 4	260 $\pm$ 3	2.3 $\pm$ 0.5	3.9 $\pm$ 0.1	25	λ =182 nm
4c	253 $\pm$ 2	265 $\pm$ 2	2.5 $\pm$ 0.5	4.0 $\pm$ 0.1	26	λ =182 nm

4. Discussion

4.1 Microstructural evolution

The microstructure of Mg-3Gd has been significantly refined by ARB processing to a total equivalent strain of 3.2. Three characteristic microstructural morphologies have been identified and characterized as: cell blocks, twin blocks and nanograins, all with boundary spacings of 100-200 nm, which illustrate a structural subdivision by low angle dislocation boundaries and high angle boundaries. The cell block structure reflects their evolution by dislocation based plasticity with activation of multiple glide systems. This structure has a clear resemblance to the cell block structure observed in bcc and fcc metals rolled to high strain. The twin block structure shows a morphology comparable to the cell block structure, a volume delineated by medium and high angle twin boundaries subdivided by low angle dislocation boundaries. Nanograins as a new structural feature are observed as part of the twin block structure and as equiaxed nanograins. Moreover, the structural evolution has its origin in dislocation and twinning based plasticity is quantified. The quantified microstructural results show a significant difference between

1c and 2c samples and comparable parameters of 2c, 3c and 4c. In addition, the significant decrease in the density of twins after first and second cycle can be seen in Table.2. This differences may have its cause in a reduction in twin frequency when the grain size is reduced [30-33], which is the case as the average grain size of preheated specimens for 1cycle ARB rolling is about 40 μm and about 10 μm for 2cycles ARB rolling.

4.2 Structure-strength relationship

A comparison of 1c data with 2c-4c data shows that the strength is the highest and the elongation is the lowest for the 1c sample, which may reflect an inhomogeneous microstructure and a high twin density in the 1c sample. This may suggest that twin boundaries show a stronger resistance to dislocation glide than lamellar boundaries. In addition, it is known that a high fraction of twins usually lead to premature fracture in magnesium alloys, which has its cause in the high stress concentration by twin-twin/twin-boundary interaction [34]. With increasing ARB cycles, the volume fraction of twin blocks decreases from 41% to 26%, while the volume fraction of cell block structure increases from 59% to 66%, which is conducive to ductility improvement. In parallel, the structural refinement with increasing ARB cycles results in improvement in the strength and decrease in elongation for the 3c and 4c samples (Table 2). However, as both the volume fraction and the boundary spacing in the twin blocks differ from those in the cell blocks, a firm conclusion cannot be drawn based on the present data sets, which will be explored in future work.

A comparison of the present tensile results with the previous study [29] on the binary Mg-(2.75-4.65) Gd alloys (grain size: 10 μm) shows that the yield strength is increased from about 150MPa to 250 MPa, as a result of structural refinement obtained in the present study. However, the tensile elongation is significantly reduced from 20%

to less than 5%. Clearly this is not the optimal combination of strength and ductility achievable from the present alloy. Very recently, we investigated the grain size effects on the deformation mechanisms and tensile behaviour of the Mg-3Gd alloy and found that fine grained samples (grain size $<10\text{ }\mu\text{m}$) show significantly improved combination of strength and ductility as compared with the samples of coarser grain sizes [35].

5. Conclusions

The microstructure of Mg-3Gd has been refined by ARB processing through four cycles to a total equivalent strain of 3.2. The deformed microstructure has been fully characterized by electron microscopy techniques and the conclusions are the following:

1. By plastic deformation the microstructure is refined to the nanometer level through formations of low angle dislocation boundaries and high angle boundaries. Three different morphologies have been identified and characterized as: cell blocks, twin blocks and nanograins, all having an average boundary spacing of 100-200 nm.

2. Nanograins as a new structural feature are observed as part of the twin block structure and as equiaxed nanograins.

3. A characterization of the cell block structure reflects their evolution by dislocation based plasticity with activation of multiple glide systems. The structure has a clear resemblance to the cell block structure observed in rolled bcc and fcc metals.

4. A characterization of the twin block structure shows a morphology comparable to the cell block structure, a volume delineated by medium and high angle twin boundaries subdivided by low angle dislocation boundaries. A similarity is also found in their contribution of boundary strengthening to the flow stress.

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Data availability

The processed data required to reproduce these findings are available by email to the corresponding author: wugl@cqu.edu.cn.

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