Special nanostructures in Al-Mg alloys subjected to high pressure torsion

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Abstract: Deformation twins and stacking faults have been observed in nanostructured Al-Mg alloys subjected to severe plastic deformation. These observations are surprising because deformation twinning has never been observed in their coarse-grained counterparts under normal conditions. This paper reviews experimental evidences on non-equilibrium grain boundaries, deformation twinning and partial dislocation emissions from grain boundaries. Some of these features can be explained by the results reported from molecular-dynamics simulations of pure fcc metals. Special emphasis is laid on the recent observations of high density hexagonal and rhombic shaped nanostructures with an average size of 3 nm in the Al-Mg alloys processed by high pressure torsion. A possible formation process of these nanostructures is proposed based on molecular-dynamics simulations.

Key words: Aluminum alloys; Severe plastic deformation; High pressure torsion; Grain boundary structure; Deformation twinning; Nanostructures

1 Introduction

In the last decade, there has been a considerable interest in the development of bulk nanostructured materials processed by severe plastic deformation (SPD) [1–4]. Although outstanding progress has been made in this area in recent years, genesis of the structural features in SPD-processed metals is not yet fully understood [1, 3–5]. These features are quite complex and the presence of non-equilibrium grain boundaries (GBs) [2–4], deformation twins, stacking faults (SFs) [6–10], severe lattice distortions and other nanostructures [11–13] in these materials may have profound effects on the deformation mechanisms and the mechanical behavior. High pressure torsion (HPT) is one of the most promising SPD techniques because it has the potential to produce nanostructures with grain sizes of less than 100 nm [12]. The purpose of this paper is to further explore the formation mechanisms of the typical nanostructures in Al-Mg alloys subjected to HPT based on our high-resolution transmission electron microscopy (HRTEM) observations.

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2 Experimental

Two Al-Mg alloys including an Al-0.5Mg alloy (by wt.%) and a commercial AA5182 alloy (Al-4.1Mg-0.35Mn-0.13Si-0.32Fe, by wt.%) received in the as-cast and homogenized condition were subjected to HPT to five turns under pressure of 6 GPa at room temperature. The deformed HPT samples had dimensions of 20 mm in diameter and 0.2 mm in thickness. Small disks with diameters of 3 mm were punched from the outer edge of these HPT samples. The structural characterization was performed by both conventional TEM and HRTEM in a JEM-2010 high-resolution TEM operated at 200 kV. Thin TEM foils were prepared from the small disks by means of disc grinding, dimpling and finally ion polishing with Ar^+ at an accelerating voltage of 3 kV.

3 Results and discussion

3.1 Grain boundary structure

Our previous works confirm that the low angle grain boundaries (LAGBs) in the HPT alloys can be in either non-equilibrium or equilibrium state [4]. Fig.1a is an HRTEM image of an equilibrium sub-boundary within a large grain of the HPT AA5182 alloy [4]. The misorientation between these two subgrains is about 3.5º. As shown, five 60° perfect dislocations on the $(11\bar{1})$ plane are found to be almost periodically distributed at the sub-boundary. These dislocations are geometrically necessary and the subgrain boundary can be described by the simplified Frank formula [4]. In other words, this sub-boundary does not contain extrinsic dislocations and is in an equilibrium state.

Fig.1b shows an example of a non-equilibrium sub-boundary generated within a larger grain in the HPT Al-0.5Mg alloy [4]. The misorientation angle is here about 1º. It is clearly seen that two types of 60° dislocations are present in the sub-boundary region on the (111) and $(11\bar{1})$ planes, respectively. Based on the simplified Frank formula, several extrinsic dislocations which are not geometrically necessary exist in the sub-boundary region. Therefore, this subgrain boundary is in a high energy state (i.e., non-equilibrium).

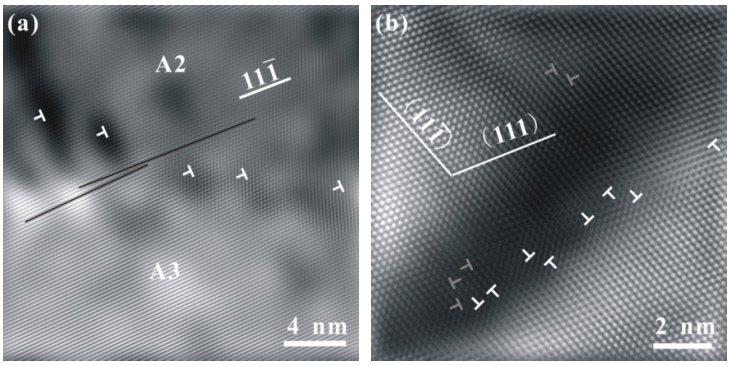

Fig.1 (a) HRTEM $[1\bar{1}0]$ image of an equilibrium sub-boundary [4]; (b) HRTEM $[1\bar{1}0]$ image of a non-equilibrium sub-boundary (inverse Fourier image) [4]

The mechanism for development of high angle grain boundaries (HAGBs) in SPD materials is still unclear [4]. Some special HAGBs have been frequently observed after deformation in our previous works

[4, 13]. Fig.2a shows an example of an equilibrium Σ9 GB in the HPT AA5182 alloy [13]. As shown, the Σ9 GB between grain A2 and C is clearly confirmed by the HRTEM image and the selected area diffraction (SAD) pattern (the inset in Fig.2a). The neighboring grains share a common $[1\bar{1}0]$ axis and have a misorientation of 38.9° (the ideal angle of Σ9 is 38.94º). As described by the coincidence site lattice (CSL) model [13], one ninth of the lattice points at this grain boundary are shared by the two neighboring crystal lattices and the distortion of the atom array at the boundary is smaller than that at any other randomly oriented HAGBs. Therefore, the Σ9 boundary has lower boundary energy and can be referred to as an equilibrium grain boundary [13].

In particular, non-equilibrium HAGBs have also been observed in the HPT alloys. As shown in Fig.2b, a very high density of dislocations is found near a non-equilibrium HAGB in the HPT AA5182 alloy [4]. The neighboring grains have a misorientation of about 18.5º, which transcends the limiting angle 15º for that of a LAGB. The local dislocation density measured from Fig.2b is as high as ~ 3.8×10^{17} m^{-2}, which is considerable higher than the average dislocation density 1.3×10^{15} m^{-2} as measured from the X-ray line profile analysis in the same sample [4]. In addition, most dislocations in Fig.2b appear as dipoles. Interstitial loops (marked by black circles) and vacancy loops (marked by white circles) also exist near the GB (Fig.2b). The introduction of dipoles near GBs is likely to increase the stored elastic energy [4] and make the GB energy higher. Therefore, it is reasonable to believe that such grains with so severe lattice distortion and extreme density of dislocations are still in a high energy state albeit the GB plane is almost straight.

Non-equilibrium GBs might play a role in the formation of deformation twins and SFs shown in this investigation [4, 7]. The "non-equilibrium" dislocations are probably candidates ready for emission into partials [7] and the stress concentrations caused by the severe lattice distortions are so high that they could overcome any energy barriers for nucleating partial dislocations and twins [4, 8, 13].

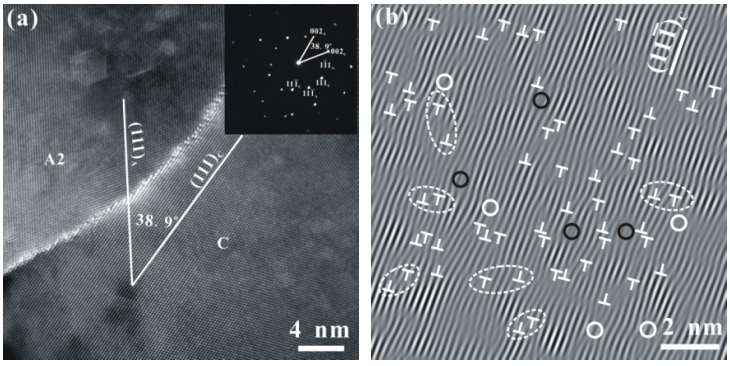

Fig.2 (a) HRTEM image of an equilibrium Σ9 GB [13]; (b) A high density of 60° perfect dislocations on the $(11\bar{1})$ plane near a non-equilibrium HAGB (inverse Fourier image) [4]

3.2 Deformation twins and stacking faults

A high density of micro-twins and SFs was often detected within smaller grains and subgrains with sizes of 20−50 nm. The density varies from 10^{16} to 10^{18} m^{-2}. Fig.3a shows a typical HRTEM image of such micro-twins and SFs observed in the subgrain A1 in the HPT AA5182 alloy [13]. The width of the subgrain is about 20 nm. The planar defects are indicated by white arrows. It is clearly evident that the planar defects

have a habit plane of (111), as the white solid line indicated. The fast Fourier transform (FFT) pattern (the inset in Fig.3a) demonstrates the twin relationship between the twin and matrix. The thickness of the twins spans only 1–4 atomic layers (0.2–1 nm). These microtwins and SFs are believed to be formed behind the moving partial dislocations which are emitted from the sub-boundary [13]. Therefore, it is reasonable to conclude that such microtwins and SFs observed in Fig.3a are formed through the heterogeneous mechanism as predicted by the molecular dynamics (MD) simulation [14].

In fcc metals, stacking faults and twins can be formed from the dissociation of either a screw dislocation or a 60° dislocation [5, 8, 11]. SFs formed by two 30° Shockley partials dissociated from end-on 0° screw dislocations were frequently observed in the HPT alloys. Fig.3b shows a stacking fault formed from the dissociation of a screw dislocation in the HPT Al-0.5Mg alloy [5]. If Burgers circuits of the two partials were drawn as indicated in Fig.3b, one can easily find that the two partials are 30° Shockley partials [5, 8, 11]. It is suggested that the stacking fault between the two partials is intrinsic and the width of the SF is 5.8 nm.

As predicted by the MD simulation [7], a twin can be also formed by the homogeneous mechanism involving the dynamic overlapping of the stacking faults of dissociated dislocations in the grain interiors. Deformation twins formed by such homogeneous mechanism were in fact observed by HRTEM in the HPT alloys [4–5, 13]. Fig.3c shows a deformation twin with a thickness of four atomic planes (about 1 nm) [5]. Such a twin was formed by the dynamic overlapping of four SFs of dissociated dislocations on adjacent slip planes. The twin can grow thicker by adding more SFs on either side of the twin. Note that such a four-layer twin formed by four SF ribbons has never been experimentally observed by HRTEM. The 2-layer version of such twins was first observed by LIAO et al. in 2003 [7]. Interestingly, as indicated by the white Ts in Fig.3c, a high density of 60° perfect dislocations exists around the twin boundary. These dislocations are believed to be related to the twin formation process [7].

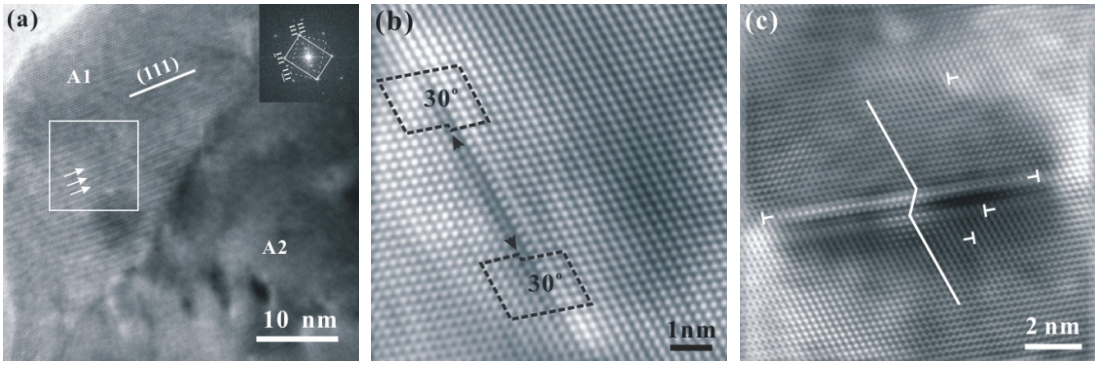

Fig.3 (a) A high density of micro-twins and SFs formed by partial dislocations emitted from a sub-boundary [13]; (b) A stacking fault formed by two 30º Shockley partials dissociated from an end-on 0º screw dislocation [5] ; (c) A four-layer twin formed by the dynamic overlapping of four SF ribbons [5]

Recently, MD simulations by SWYGENHOVEN et al. reported that generalized planar fault energy (GPFE) curves significantly affect the partial-dislocation-mediated deformation processes [15]. Our experimental findings suggest that the MD simulations based on GPFE curves could explain the formation of perfect dislocations, SFs and twins in the HPT Al-Mg alloys. However, further investigations are necessary to reveal the dominant dislocation activity in the nanostructured Al-Mg alloy.

3.3 Special nanostructures

Surprisingly, high density hexagonal and rhombic shaped nanostructures (referred as special nanostructures) were frequently observed in the HPT Al-Mg alloys. These special nanostructures are analogous to our previous observations in a commercial Al-Mg-Si alloy processed by equal channel angular pressing [11]. Figs.4a and 4b are HRTEM images of the special nanostructures observed inside a grain with a size of about 50 nm in the HPT AA5182 alloy. As shown, an extremely high density of planar defects exists within the grain. The average size is about 3 nm and the local density is about 10^{18} m^{-2}. As revealed by the FFT of the images (the inset in Fig.4b), these planar defects are present along two {111} planes and one (001) plane and are therefore here referred to as {111} and (001) interfaces, respectively. An interesting feature in these images is that these planar defects appear with two kinds of regular shape, one is rhombic and another is hexagonal [11].

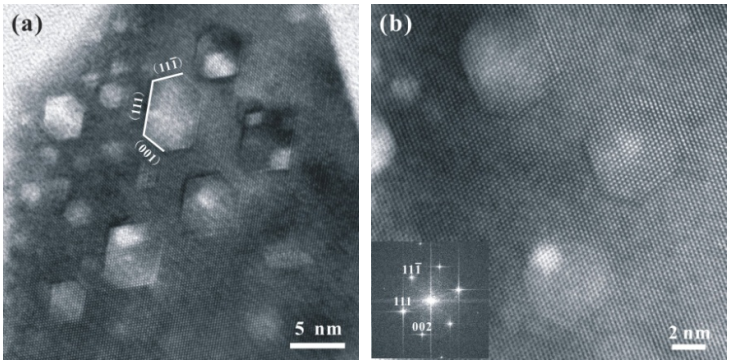

Fig.4 (a) HRTEM [1$\bar{1}$0] image showing planar hexagonal and rhombic shaped nanostructures; (b) HRTEM [1$\bar{1}$0] image of the special nanostructures at higher magnification (The inset shows a FFT of the images)

These surprising nanostructures could be caused by reactions between dislocations on different slip systems [6, 11, 16]. A high density of perfect dislocations (Fig.2b), partials and SFs (Fig.3) observed in the HPT alloys are probably candidates ready for the reactions. As predicted by MD simulations [6], when two partials of two SFs on different slip planes meet, they can react to form a triangular structure with an angle of 70.53° or 109.47º. Therefore, the present rhombic structures could be formed by the reaction of such four SFs [11]. Furthermore two trailing partials can react with a stair-rod dislocation to form a perfect dislocation capable of gliding on {001} planes [16]. Thus, the hexagonal structures could be formed by the slip of perfect dislocations on {001} planes after the triangular structures formed [11].

The dislocation reactions can be understood by the unfolded Thompson's tetrahedron (Fig.5) [6]. Based on MD simulations by YAMAKOV et al. [6], the possible formation process of these nanostructures is illustrated in Figs.6a–e. As shown, the formation process probably includes the following sequent stages.

(i) One pair of two intrinsic stacking faults (ISFs) are generated from the dissociation of two perfect dislocations 1/2[101] (**DA**) and 1/2[01$\bar{1}$] (**AC**) on two (11$\bar{1}$) and (111) slip planes (Fig.5 and Fig.6a), i.e.,

$$\mathbf{DA} \rightarrow \mathbf{D\gamma} + \mathbf{\gamma A} \qquad (1)$$

$$\mathbf{AC} \rightarrow \mathbf{A\delta} + \mathbf{\delta C} \qquad (2)$$

These two reactions produce four partials, i.e., 1/6[112] (**Dγ**), 1/6[2$\bar{1}$1] (**γA**), 1/6[$\bar{1}$2$\bar{1}$] (**Aδ**) and 1/6[11$\bar{2}$] (**δC**).

(ii) A triangular structure with two ISFs is formed from the reaction of the partials **δC** and **Dγ** (Fig.5 and Fig.6b), i.e.,

$$\boldsymbol{\delta C + D\gamma \rightarrow \delta D/C\gamma} \qquad (3)$$

A stair-rod dislocation 1/3[110] (**δD/Cγ**) is produced from this reaction. The two ISFs meet at an angle of 109.47º, i.e., the angle between $(11\bar{1})$ and (111) planes.

(iii) Another triangular structure is formed (Fig.6c) from repeating the above reactions in equations (1), (2) and (3).

(iv) A rhombic structure is obtained from the reactions of the trailing partials 1/6[$2\bar{1}1$] (**γA**) and 1/6[$\bar{1}2\bar{1}$] (**Aδ**) (Fig.5, Figs.6c and 6d), i.e.,

$$\boldsymbol{\gamma A + A\delta \rightarrow \gamma\delta} \qquad (4)$$

A stair-rod dislocation 1/6[110] (**γδ**) is produced from this reaction.

(v) A hexagonal structure will be acquired if the reactions between the trailing partials 1/6[$2\bar{1}1$] (**γA**), 1/6[$\bar{1}2\bar{1}$] (**Aδ**) and a stair-rod dislocation 1/3[110] (**δD/Cγ**) take place (Fig.5, Figs.6c and 6e), i.e.,

$$\boldsymbol{\gamma A + A\delta + \delta D/C\gamma \rightarrow DC} \qquad (5)$$

Thus, the slip of perfect dislocations **DC** on (001) plane may occur due to this reaction.

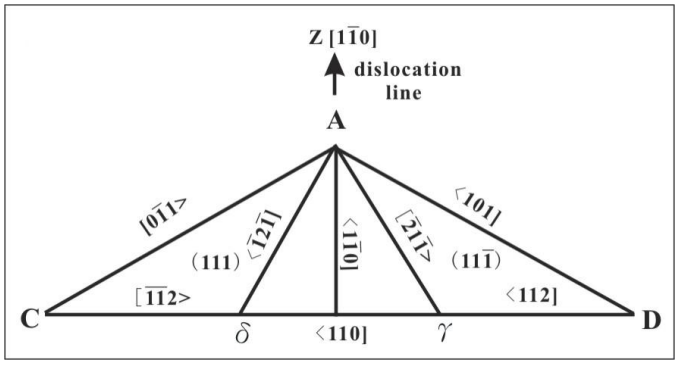

Fig.5 The unfolded Thompson's tetrahedron (after YAMAKOV et al. [6])

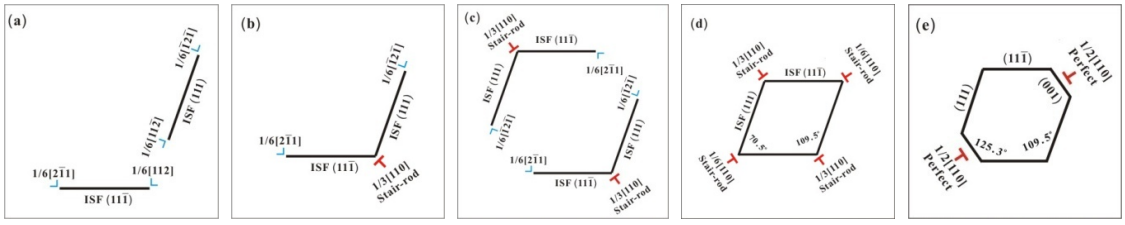

Fig.6 (a) Generation of two ISFs resulting from the dissociation of two perfect dislocations; (b) Formation of a triangular structure due to the reaction of the partials; (c) Formation of another triangular structure from repeating the reactions in (a) and (b); (d) Acquisition of a rhombic structure from the reactions of the trailing partials; (e) Acquisition of a hexagonal structure due to the reactions between the trailing partials and the stair-rod dislocations

4 Summary

Non-equilibrium GBs, deformation twinning, SFs and partial dislocation emissions from grain boundaries are observed in the nanostructured Al-Mg alloys. Two twinning mechanisms predicted by MD simulations are verified. A four-layer twin formed by the dynamic overlapping of four stacking faults is observed. The MD simulations based on GPFE curves could explain the formation of perfect dislocations, SFs and twins in the HPT Al-Mg alloys. Specially, high density hexagonal and rhombic shaped nanostructures are observed in the HPT Al-Mg alloys. A possible formation process of these nanostructures is proposed based on MD simulations. However, further investigations are necessary to reveal the exact nature of non-equilibrium GBs, deformation twinning, SFs and the special nanostructures, as well as the interactions between these features in the nanostructured Al-Mg alloys.

Acknowledgments

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