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

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Abstract: Deformation twins and stacking faults were observed in nanostructure Al-Mg alloys subjected to high pressure torsion. These observations are surprising because deformation twinnings have never been observed in their coarse-grained counterparts under normal conditions. Experimental evidences are introduced on non-equilibrium grain boundaries, deformation twinnings 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 nanostructure materials processed by severe plastic deformation (SPD)[1−4]. This interest arises because the use of different SPD technologies provide new opportunities for developing nanostructures in metals and alloys with unusual properties that are very attractive for various structural and functional applications[1−4]. Although outstanding progress has been made in this area in recent years, the 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 mechanical behavior. High pressure torsion (HPT) is one of the most promising SPD techniques because it has the potential to produce nanostructures with grain size less than 100 nm[12]. The purpose of this work 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.

2 Experimental

Two Al-Mg alloys including an Al-0.5Mg alloy (mass fraction, %) and a commercial AA5182 alloy Al-4.1Mg-0.35Mn-0.13Si-0.32Fe (mass fraction, %) received in the as-cast and homogenized condition were subjected to HPT and twisted five turns with a rotation speed of 1 r/min under imposed pressure of 6 GPa at room temperature. The deformed HPT samples had dimensions of d20 mm×0.2 mm. Small disks with diameters of 3 mm were punched from the outer edge of

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these HPT samples. The equivalent strain at the outer edge of the HPT samples was about 906[2]. 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.1(a) shows an HRTEM image of an equilibrium sub-boundary within a large grain of the HPT AA5182 alloy. The misorientation between these two subgrains is about 3.5°. As shown, 5 perfect dislocations of 60° on the $(11\bar{1})$ plane are found to be almost periodically distributed on 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 it is in an equilibrium state.

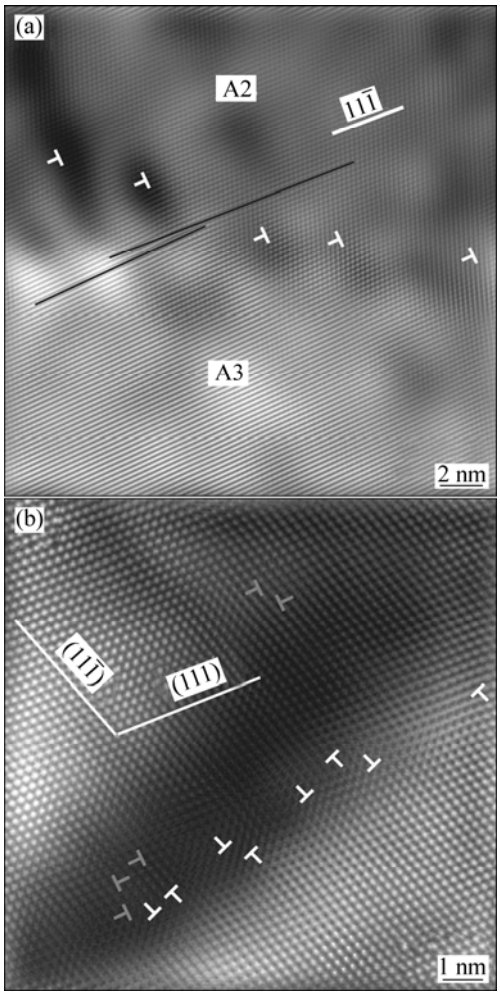

Fig.1 HRTEM $[1\bar{1}0]$ images of equilibrium sub-boundary (a) and non-equilibrium sub-boundary (inverse Fourier image) (b)

Fig.1(b) shows an example of a non-equilibrium sub-boundary generated within a larger grain in the HPT Al-0.5Mg alloy. The misorientation angle here is about 1°. 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).

The mechanism for development of high angle grain boundaries (HAGBs) in SPD materials is still unclear[4]. Some special HAGBs were frequently observed after deformation in our previous work[4, 13]. Fig.2(a) 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.2(a)). The neighboring grains share a common $[1\bar{1}0]$ axis and have a misorientation of 38.9° (the ideal angle of Σ9 is 38.94°). As described by the coincidence site lattice (CSL) model[13], 1/9 of the lattice points on this grain boundary are shared by two neighboring crystal lattices and the distortion of the atom array on the boundary is smaller than that on 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 are also observed in the HPT alloys. As shown in Fig.2(b), a very high density of dislocations is found near a non-equilibrium HAGB in the HPT AA5182 alloy. The neighboring grains have a misorientation of about 18.5° which transcends the limiting angle 15° of a LAGB. The local dislocation density measured from Fig.2(b) is as high as 3.8×10^{17} m^{-2}, which is considerably higher than the average dislocation density of 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.2(b) appear as dipoles. Interstitial loops (marked by black circles) and vacancy loops (marked by white circles) also exist near the GB (Fig.2(b)). 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 grains with such severe lattice distortion and extremely dense dislocations are still in a high energy state though the GB plane is almost straight.

Non-equilibrium GBs might play a role in the formation of deformation twins and SFs shown in the

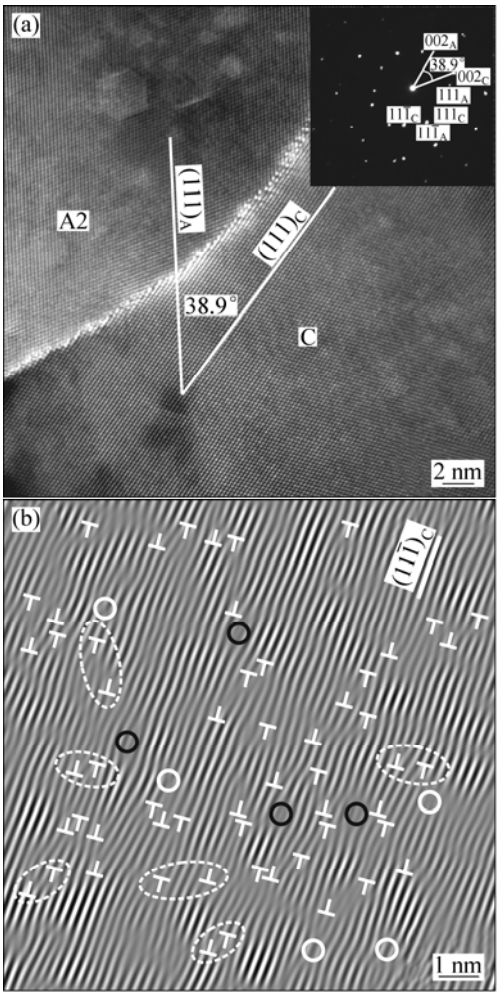

Fig.2 HRTEM images of equilibrium Σ9 GB (a) and high density of 60° perfect dislocations on $(11\bar{1})$ plane near non-equilibrium HAGB (inverse Fourier image) (b)

investigation[4, 7]. The non-equilibrium dislocations are probably candidates 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].

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.3(a) 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 evident that the planar defects have a habit plane of (111), as the white solid line indicated. The fast Fourier transform (FFT) pattern demonstrates the twin relationship between the twin and matrix, as shown in Fig.3(a). The thickness of the twins spans only 1−4

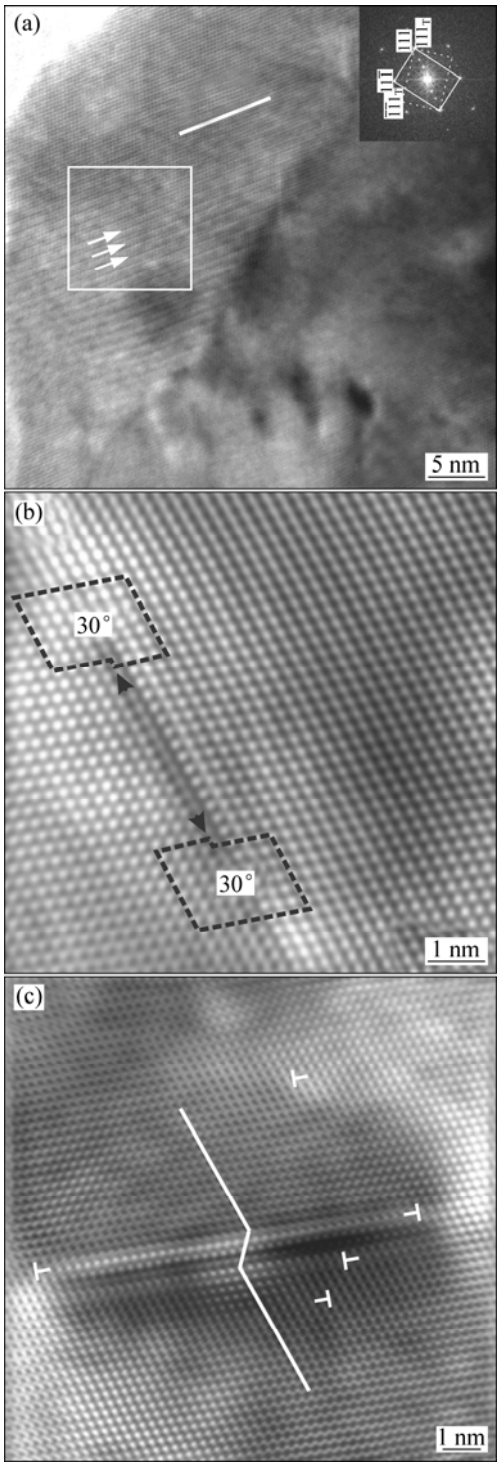

Fig.3 HRTEM images of high density of micro-twins and SFs formed by partial dislocations emitted from sub-boundary [13] (a), stacking fault formed by two 30° Shockley partials dissociated from end-on 0° screw dislocation[5] (b) and four-layer twin formed by dynamic overlapping of four SF ribbons[5] (c)

atomic layers (0.2−1 nm). These microtwins and SFs are believed to form 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.3(a) form through the heterogeneous mechanism as predicted by the molecular-dynamics (MD) simulation[14].

In FCC metals, stacking faults and twins form from the dissociation of either a screw dislocation or a 60° dislocation[5, 8, 11]. SFs formed by two Shockley partials of 30° dissociated from end-on 0° screw dislocations were frequently observed in the HPT alloys. Fig.3(b) 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.3(b), it can be easily found 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 observed by HRTEM in the HPT alloys[4−5, 13]. Fig.3(c) shows a deformation twin with a thickness of 4 atomic planes (about 1 nm)[5]. The twin was formed by the dynamic overlapping of 4 SFs of dissociated dislocations on adjacent slip planes. The twin grows thicker by adding more SFs on either side of it. Such a 4-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 ZHU et al in 2003[7]. Interestingly, as indicated by the white "⊤" in Fig.3(c), 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].

Recently, MD simulations by SWYGENHOVEN et al[15] reported that generalized planar fault energy (GPFE) curves significantly affect the partial-dislocation-mediated deformation processes. 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 nanostructure 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.4(a)−(b) show 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

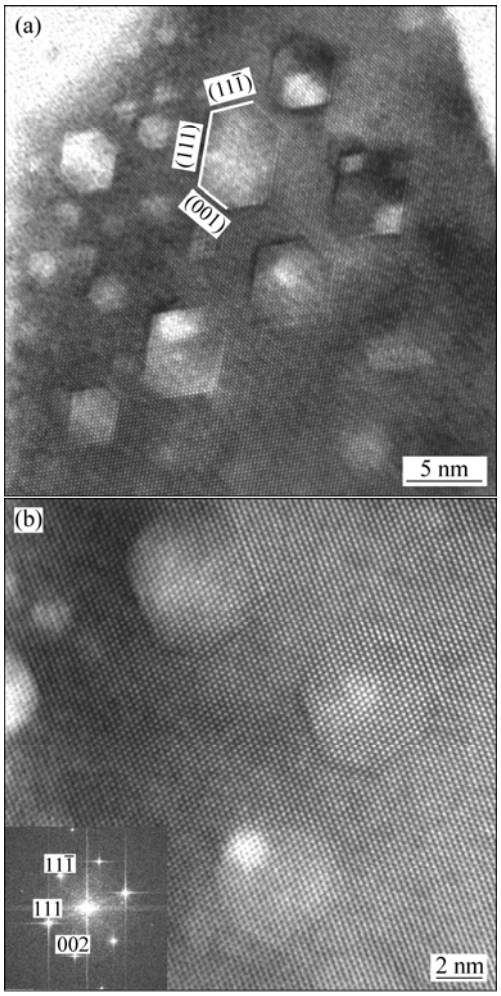

Fig.4 HRTEM $[1\bar{1}0]$ images showing planar hexagonal and rhombic shaped nanostructures (a) and special nanostructures at higher magnification with inset showing FFT (b)

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 in Fig.4(b), these planar defects are present along two {111} planes and one (001) plane and are therefore 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 the other is hexagonal[11].

These surprising nanostructures may be caused by reactions between dislocations on different slip systems[6, 11, 16]. High-density perfect dislocations (Fig.2(b)), partials and SFs (Fig.3) observed in the HPT alloys are probably candidates 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 109.47°. 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 form[11].

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

1) 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, as shown in Fig.5 and Fig.6(a).

$$DA \longrightarrow D\gamma+\gamma A \quad (1)$$

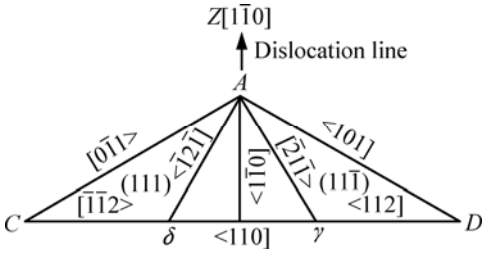

Fig.5 Schematic diagram of unfolded Thompson's tetrahedron[6]

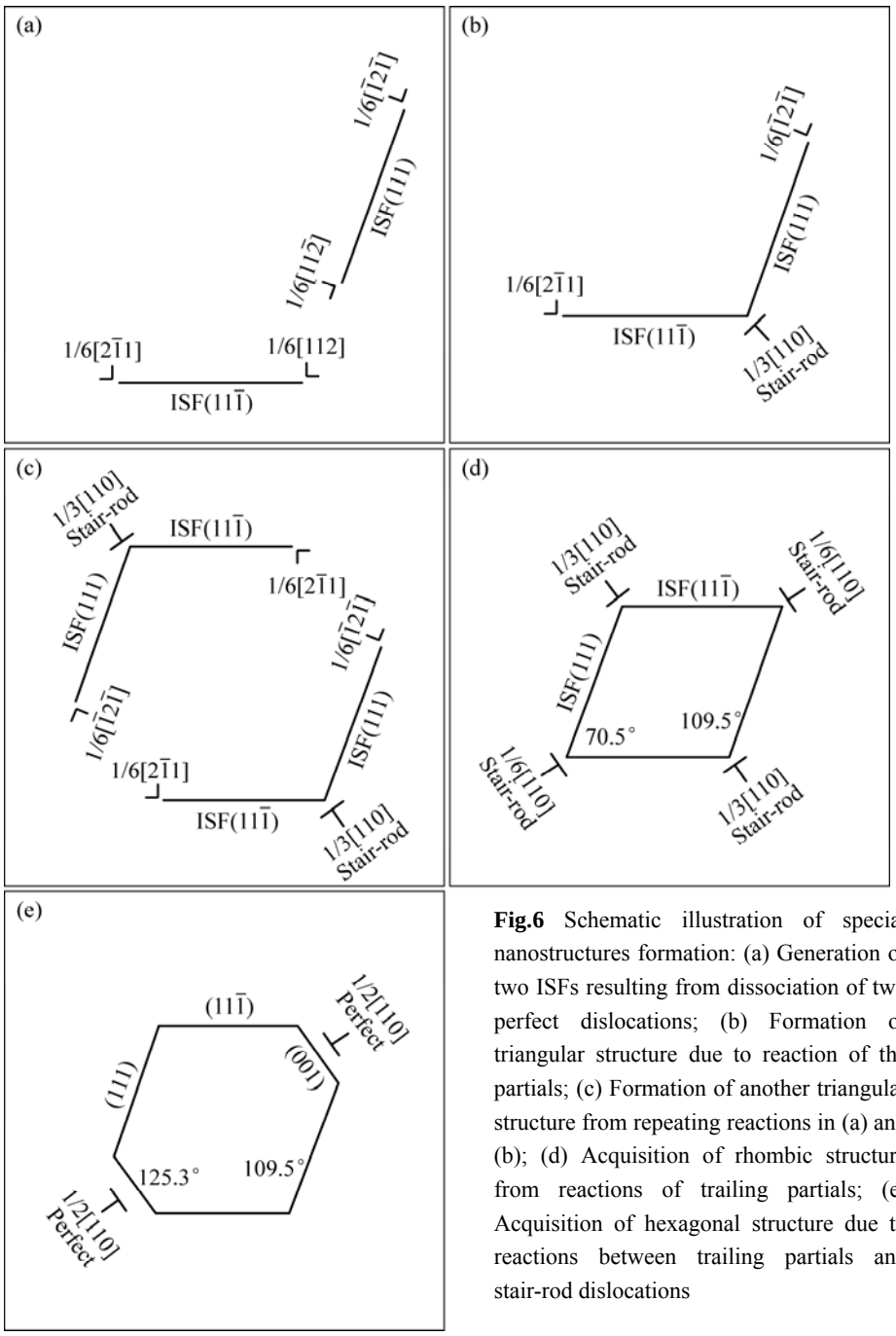

Fig.6 Schematic illustration of special nanostructures formation: (a) Generation of two ISFs resulting from dissociation of two perfect dislocations; (b) Formation of triangular structure due to reaction of the partials; (c) Formation of another triangular structure from repeating reactions in (a) and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

$$A\mathrm{C}\longrightarrow A\delta+\delta C \quad (2)$$

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

2) A triangular structure with two ISFs is formed from the reaction of the partials δC and $D\gamma$ as shown in Fig.5 and Fig.6(b).

$$\delta C+D\gamma\longrightarrow\delta D/C\gamma \quad (3)$$

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

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

4) A rhombic structure is obtained from the reactions of the trailing partials 1/6 $[2\bar{1}1]$ (γA) and 1/6$[\bar{1}2\bar{1}]$ ($A\delta$), as shown in Fig.5 and Figs.6(c)−(d).

$$\gamma A+A\delta\longrightarrow\gamma\delta \quad (4)$$

A stair-rod dislocation 1/6[110] ($\gamma\delta$) is produced from this reaction.

5) A hexagonal structure will be acquired if the reactions between the trailing partials 1/6$[2\bar{1}1]$ (γA), 1/6 $[\bar{1}2\bar{1}]$ ($A\delta$) and a stair-rod dislocation 1/3[110] ($\delta D/C\gamma$) take place, as shown in Fig.5 and Figs.6(c)−(e):

$$\gamma A+A\delta+\delta D/C\gamma\longrightarrow DC \quad (5)$$

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

4 Conclusions

1) The grain boundaries can be either in the non-equilibrium or the equilibrium sate in the nanostructure Al-Mg alloys processed by HPT. A high density of extrinsic 60° dislocations in the form of dipoles are frequently located at non-equilibrium grain/subgrain boundaries.

2) 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.

3) 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.

4) 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 nanostructure Al-Mg alloys.

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