

# Partial dislocation emission in a superfine grained Al–Mg alloy subject to multi-axial compression



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## ABSTRACT

We report partial dislocation emission from subgrain boundaries with subgrain size being significantly larger than 100 nm in an Al–Mg alloy. The dislocation density in subgrain boundary has significant effect on partial dislocation emission. The high stacking fault density inside a subgrain is related to the large dislocation density in the subgrain boundary.

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## 1. Introduction

Grain size plays a significant role in determining the deformation mechanisms of metallic materials and dramatic change in the deformation mechanisms occurs when the grain size is reduced to the nanometer regime [1,2]. For example, while deformation in coarse-grained (grain size larger than 1  $\mu\text{m}$ ) face-centered cubic (f.c.c.) metals occurs mainly via intragranular dislocation activities including dislocation multiplication and slip, partial dislocation emission from grain boundaries (GBs) [3,4], deformation twinning [5,6], GB sliding and grain rotation [5] dominate the deformation process in nanocrystalline materials (grain size smaller than 100 nm). Grain boundaries become the primary dislocation source and sink when the grain is smaller than a critical size [7,8]. The critical grain size for partial dislocation emission from grain boundaries is in fact a function of stacking fault energy (SFE). Reducing SFE via alloying could increase the critical grain size to the sub-micrometer regime, leading to simultaneous operation of intragranular full dislocation slip and partial dislocation emission from grain boundaries in ultrafine-grained (grain size in the range of 100 nm and 1  $\mu\text{m}$ ) materials [9].

It is well known that the addition of Mg to Al (with SFE of about 166  $\text{mJ}/\text{m}^2$  [10]) can greatly reduce the SFE [11] and hinder the

dislocation slip during plastic deformation due to the interaction between Mg atoms and dislocations. In fact, shock loading experiments at low temperature have shown that the addition of Mg to Al can facilitate twinning as a consequence of solute effects on dislocation behaviors [12]. In addition, direct experimental evidences have shown that stacking faults and microtwins were frequently observed in severe plastic deformed Al–Mg alloys with a grain size larger than 100 nm [13]. Therefore, it is possible to investigate the deformation mechanism transition during grain refinement process by severe plastic deformation at room temperature and low strain rate. Molecular dynamics (MD) simulations of nanoscale f.c.c. metals have illustrated that partial dislocation emission from grain boundary and formation of deformation twins occur in a grain with only several tens of nanometers at high strain rate and extremely high stress [1]. Experiments conducted by Chen et al. [14] and Liao et al. [15] also indicated that twinning occurs only in nano-grained Al and Al–Mg alloys during plastic deformation. However, the formation and evolution of stacking faults or microtwins during grain refinement process by severe plastic deformation have not been well understood till now. In this paper, we report that partial dislocations are emitted from locally distorted subgrain boundaries that contain local, deformation-distorted fragments being rich in dislocations induced by plastic deformation. Experiments also show that the dislocation density in the subgrain boundary has a positive influence on the stacking fault density. The stacking faults with high density inside the subgrain can easily form microtwins by

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overlapping on adjacent parallel slip planes and be blocked on intersecting slip planes.

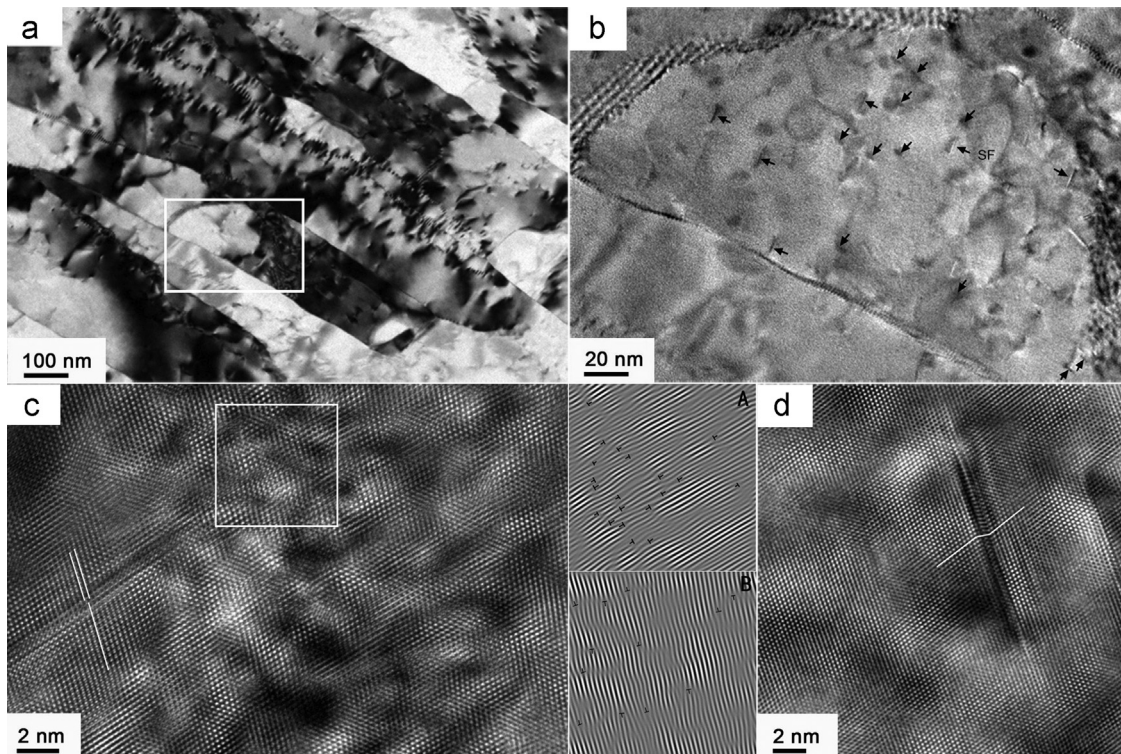
## 2. Experimental

The material used in present work is an Al-4 wt% Mg alloy (with SFE of about  $120 \text{ mJ/m}^2$  [16]) fabricated by ingot metallurgy, in which commercially pure Al and pure Mg (both are 99.9% in purity) were used as the raw materials. The alloy was then homogenization annealed at 733 K for 10 h. Equiaxed grains with an average grain size of  $\sim 500 \mu\text{m}$  were observed in the annealed alloy by optical microscope. Specimens with a dimension of  $12 \times 12 \times 6 \text{ mm}^3$  were cut from the annealed alloy and were then subjected to multi-axial compression (MAC) at room temperature using Instron 8802 testing machine with a compressive speed of  $0.05 \text{ mm s}^{-1}$ .  $\text{MoS}_2$  was used as the lubricant to reduce the friction between the samples and the steel die. The direction of the sample was changed after each MAC pass with a  $90^\circ$  angle and the detailed information can be found elsewhere [17]. The strain per pass during MAC can be calculated by equation  $\varepsilon = 2 \ln(H/W)/\sqrt{3}$  [18]. In the present study, the accumulated strains are 0.8 and 4.0 after 1 and 5 MAC passes, respectively. The deformed samples were cut parallel to the last loading direction from the center position of the specimens. The microstructures of the samples were studied using a JEOL-2100F transmission electron microscope (TEM) operated at 200 kV. The TEM samples were prepared by twin-jet polishing technique in a mixture of 30% nitric acid and 70% carbinol at  $-30^\circ\text{C}$ .

## 3. Results and discussion

Fig. 1a presents a TEM image of the alloy after 1 MAC pass. The

microstructure mainly consists of parallel lamellar boundaries (LBs) with strongly elongated subgrains (mean spacing of  $\sim 200 \text{ nm}$ ), inside which equiaxed subgrains with a size of several hundred nanometers were observed. By statistical measurements of about 50 equiaxed subgrains inside the lamellar boundaries using TEM, the average size of the equiaxed subgrain after 1 MAC pass is about 280 nm in length and 170 nm in width. The obvious characteristics of the equiaxed subgrains are the high SF density and dislocation density inside the subgrain interior. A typical equiaxed subgrain structure marked by a rectangle in Fig. 1a was enlarged in Fig. 1b. A lot of the stacking faults (marked by black arrows in Fig. 1b) were observed inside the subgrain and the density reaches about  $8.0 \times 10^{14} \text{ m}^{-2}$ . Obviously, the width of stacking fault is larger than the theoretically calculated spacing of about 1.1 nm without external stress ( $r_0 = Kb^2/\Gamma$ , where  $K$  is a factor depended on elastic constants and  $\Gamma$  is the stacking fault energy) [19]. Fig. 1c shows a high-resolution TEM (HRTEM) image of the subgrain boundary along [110] direction. A stacking fault was observed close to the equiaxed subgrain boundary. The inverse Fourier transformation images A and B of the region marked by a white box in Fig. 1c shows that a lot of dislocations, marked by black "T", are stored in the subgrain boundary with many dislocation dipoles on different  $\{111\}$  planes. The stacking faults here were proposed to be formed via partial dislocation emission from the subgrain boundary. The mechanism for partial dislocation emission from the subgrain boundary is clarified as follows. LBs with elongated subgrains are formed at the early stage of deformation, and a high density of the dislocations distribute unevenly within each elongated subgrain and present in the form of tangles or dislocation cells that maybe evolve into equiaxed subgrains with wide dislocation boundaries [20,21]. With increasing strain, a greater stress is required for the equiaxed subgrain inside the LBs to participate in further plastic deformation. Because the size is several hundred nanometers for the equiaxed subgrain in



**Fig. 1.** (a) TEM image of the Al-4%Mg alloy after 1 MAC pass, (b) the enlarged view of the subgrain marked by white square box in (a), (c) HRTEM image of the subgrain boundary along [110] direction showing partial dislocations bounding stacking fault emitted from subgrain boundary, where a high density of dislocation was shown by inverse Fourier transformation images (A and B) from the area marked by white box in (c) on  $(111)$  and  $(\bar{1}\bar{1}\bar{1})$  planes, respectively, and (d) a microtwin in the cell formed by stacking faults overlapping.

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