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Deformation mechanism in nanocrystalline Al: Partial dislocation slip

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We report experimental observation of a deformation mechanism in nanocrystalline face-centered-cubic Al, partial dislocation emission from grain boundaries, which consequently resulted in deformation stacking faults (SFs) and twinning. These results are surprising because (1) partial dislocation emission from grain boundaries has not been experimentally observed although it has been predicted by simulations and (2) deformation stacking faults and twinning have not been reported in Al due to its high SF energy. © 2003 American Institute of Physics.

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Nanocrystalline (nc) materials are believed to deform via mechanisms that are fundamentally different from those present in their coarse-grained counterparts.¹⁻⁷ A deformation mechanism, via partial dislocation emission from grain boundaries or free surfaces, has been predicted to operate in nc fcc metals by simulations,⁵⁻¹⁰ but has never been experimentally verified. Such a mechanism, if proven true, could help us understand the effect of length scale (from micrometer to nanometer) on the deformation mechanism in fcc metals. It could also shed light on the unique, superior mechanical properties of nc materials,¹¹⁻¹⁴ some of which exhibit both high strength and excellent ductility.¹¹⁻¹³ It is the objective of this investigation to experimentally verify this deformation mechanism.

Al powder with a purity of ~99.9 wt% (Valimet) was ball milled in liquid nitrogen. The starting powder consists of equiaxed particles with diameters ranging from 0.5 to 1 μm . The ball milling was performed in a modified Union Process 1-S attritor (Szegvari) with a stainless steel vial and stainless steel balls (6.4 mm in diameter) as milling media. The vial is a vertical cylindrical tank in which the powder and balls are charged. The ball-to-powder weight ratio is 32:1. Prior to milling, 0.2 wt% stearic acid [$\text{CH}_3(\text{CH}_2)_{16}\text{CO}_2\text{H}$] was added to the powder as a control agent. No adhesion of the powder to the milling tool occurred during the process. During the milling, liquid nitrogen was added into the vial to maintain a complete immersion of the milling media at the temperature of -196°C . The balls and powder were stirred by horizontal impellers attached to a vertical shaft rotating at 180 rpm. The milling duration was 8 h. The composition of the milled powder was analyzed at a commercial laboratory (Luvak Inc., Boulston, MA) and is listed below in weight percent: 0.05 Fe, 0.013 Cr, 0.003 Mn, 0.002 Mo, 0.05 Si, 0.2 C, 0.4 N, 1.45 O. The iron content did not show measurable increase after the ball milling, indicating little contamination from the milling media. However, there could be contamina-

tion from C, N, H, and O. The ball-milled powder, with typical agglomerate diameters from 20 to 50 μm , was pressed to form a small pellet, which was then mechanically ground to a thickness of $<30 \mu\text{m}$. Further thinning to a thickness of electron transparency was carried out using Gatan precision ion polishing system with Ar^+ accelerating voltage of 4 kV. Transmission electron microscopy (TEM) investigation was carried out using a JEOL 3000F transmission electron microscope operating at 300 kV.

Figure 1 shows that in the milled powder some grains are elongated [Fig. 1(a)] while others are equiaxed [Fig. 1(b)]. The diameters of most equiaxed grains and the widths of most elongated grains are smaller than 100 nm.

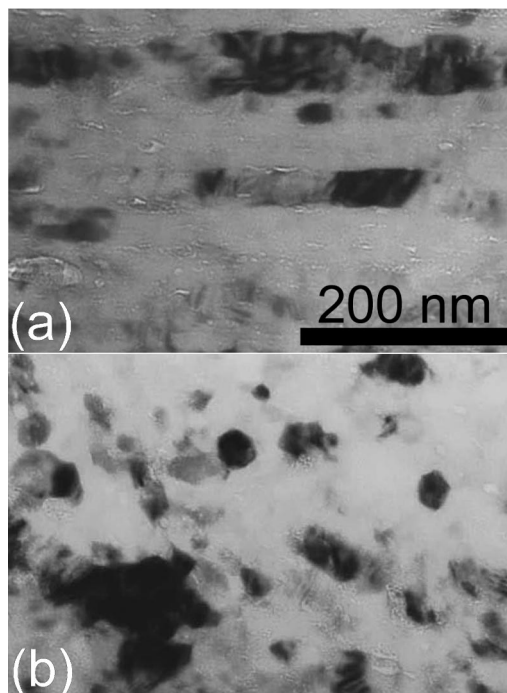


FIG. 1. Nanograins in Al powder ball-milled in liquid nitrogen. (a) Elongated grains and (b) equiaxed grains.

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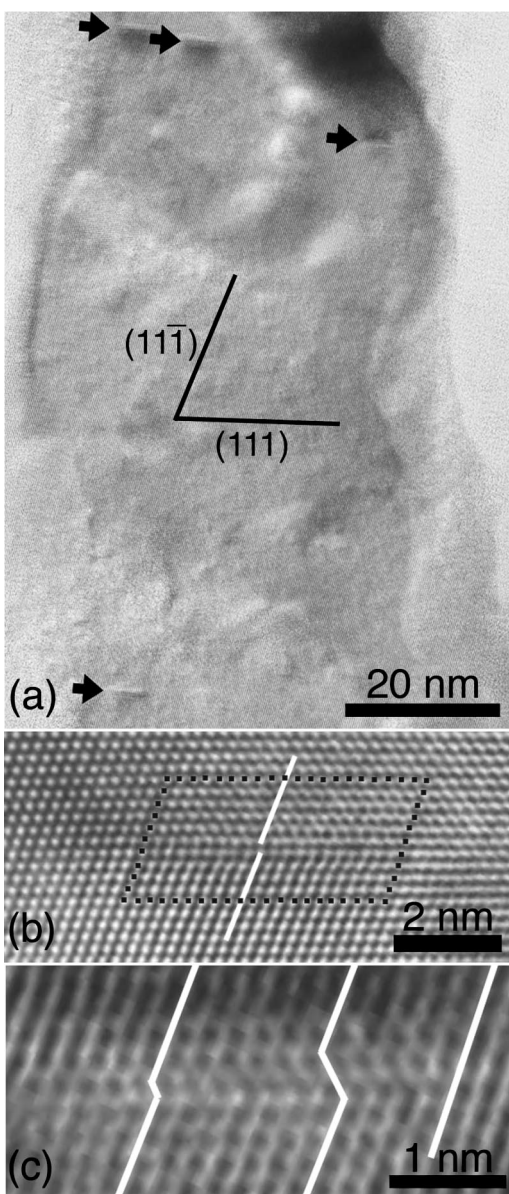


FIG. 2. (a) SFs on (111) planes, which are marked by black arrows. Only full dislocations were observed on (11 $\bar{1}$) planes, which have a much longer path length than the (111) planes. (b) Atomic level image of a typical SF. A Burger's loop around the partial dislocation pair revealed no edge dislocation component, indicating that the two partial dislocations have a combined screw dislocation characteristics. (c) A deformation twin formed by the overlapping of two extended dislocations on adjacent slip planes.

Figure 2 shows high-resolution TEM micrographs of (a) stacking faults (SFs) (marked by arrows), (b) the atomic level image of a typical SF, and (c) a deformation twin in nc Al. On (111) slip planes that have a path length for dislocation motion of ~ 50 nm SFs are observed [Fig. 2(a)]. The SF density is $6 \times 10^{14} \text{ m}^{-2}$. In contrast, on (11 $\bar{1}$) planes with a path length of ~ 150 nm, only full dislocations with a high-density of $3 \times 10^{16} \text{ m}^{-2}$ are observed. The SFs are formed by the grain boundary emission of leading and trailing Shockley partial dislocations, which is consistent with the molecular dynamics simulation.⁸ In addition, the partial dislocation emission depends on the dislocation path length on the slip planes on which they glide. This result suggests a transition from full dislocation slip to partial dislocation slip with decreasing grain size.

The SF width shown in Fig. 2(b) was likely affected by both resolved nucleation stress¹⁵ and the diffusion of impurities to the SF after ball milling. The former can only account for a very small fraction of the SF width shown in Fig. 2(b). We believe that the latter is mostly responsible for the wide SF. As the milled powder sample was kept at room temperature, impurities may have diffused into the SF, lowering the SF energy, which led to a wider stacking fault.

Most of the partial dislocation pairs that are connected by a SF would become a compact screw dislocation if forced to collapse together. As shown in Fig. 2(b), a Burgers loop around the SF reveals no edge component. The partials were not necessarily formed by the dissociation of a compact screw dislocation. Molecular dynamics simulations indicated that grain boundaries successively emit the leading $1/6[112]$ and trailing $1/6[\bar{1}21]$ Shockley partial dislocations connected by a stacking fault.^{6–8} More investigation is needed to understand why these partial dislocation pairs mostly have a combined screw characteristics instead of edge characteristics.

Figure 2(c) shows a deformation twin with a thickness of two atomic planes. Such a twin was formed by the dynamic overlapping of two extended partial dislocations with SFs on adjacent slip planes.⁸ As shown, the two SFs are only partially overlapped. This twinning mechanism is different from the well-known pole mechanism,^{16,17} in which one partial dislocation forms a whole twin via climbing a screw dislocation pole to adjacent slip planes. In contrast, the twin shown in Fig. 2(c) was formed by the coincidental overlapping of two partial dislocation pairs (SFs) on adjacent planes. The twin can grow thicker by adding more SFs on either side of the twin.⁸

We did not observe partial dislocation in grains smaller than 45 nm. This could be caused by one of the following two factors. First, partial dislocation pairs traveling inside a very small grain could have been attracted to the grain boundary and disappeared on it after the ball milling stopped. Such dislocation-grain boundary interaction becomes stronger with decreasing grain size. In fact, it has been reported that nanograins generated by severe plastic deformation are dislocation free in their interior when their sizes are smaller than a critical value.^{18–20} Second, the resolved shear stress to emit partial dislocations increases with decreasing grain size.⁸ The processing condition in the current experiment might not have generated shear stress high enough to activate the emission of partial dislocations from boundaries of grains smaller than a certain critical size.

The resolved shear stress to move a *full* dislocation from a grain boundary into a grain has been suggested by an analytical model²¹ to be greater than the stress required to extend a *partial* dislocation into the same grain for fcc metals with a small grain size. The model predicts a stress obtainable under ball-milling conditions (e.g., 410 MPa for 50 nm Al grains) for partial dislocation emission, and below 15 nm it indicates that partial dislocations are emitted at a lower stress than full dislocations. Although the model provides only preliminary insight, it does qualitatively explain the deformation mechanism transition observed in this study.

In summary, we have observed a deformation mechanism, partial dislocation emission from grain boundaries in

nc Al powder produced via ball milling in liquid nitrogen. Consequently, SFs and twins are also observed. Almost all observed partial dislocation pairs would form compact screw dislocations if combined. The observed deformation mechanism demonstrates that when the size scale is reduced down to the nano range, nature will facilitate deformation via mechanisms that are not accessible at conventional length scales.

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