Available online at www.sciencedirect.com

SCIENCE DIRECT®

Acta Materialia 54 (2006) 655–665

Acta MATERIALIA

www.actamat-journals.com

Deformation twinning in polycrystalline copper at room temperature and low strain rate

C.X. Huang, K. Wang, S.D. Wu *, Z.F. Zhang, G.Y. Li, S.X. Li

Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China

Received 29 July 2005; received in revised form 30 September 2005; accepted 5 October 2005
Available online 22 November 2005

Abstract

Deformation twins were widely observed in polycrystalline Cu with grain sizes varying from micrometers to nanometers during the process of equal channel angular pressing at room temperature and low strain rate ($\sim 10^{-2}\ s^{-1}$). The microstructures of deformation twins were characterized by a transmission electron microscope (TEM) and a high-resolution TEM. It was found that deformation twinning in coarse-grained Cu occurred mainly in shear bands and their intersections as a result of the very high local stress resulted from the severe plastic deformation, and followed the well known pole mechanism. With a decrease in the grain size down to submicrometer (<1 μm) and nanometer (<100 nm) dimensions, twinning was observed to take place via partial dislocation emission from grain boundaries and grain boundary junctions, which is different from the pole mechanism operating in coarse-grained Cu. These observations are consistent with the predictions of recent molecular dynamic simulations for nanocrystalline face-centered cubic materials. The deformation conditions required for twinning and the formation mechanism of deformation twins varying with grain size in Cu are discussed.
© 2005 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Keywords: Deformation twinning; Cu; Nanostructured material; Equal channel angular pressing; Shear band

1. Introduction

Deformation twinning is a common and important mechanism for the plastic deformation in hexagonal close-packed metals, such as Ti and Mg. In body-centered cubic metals and alloys, such as pure Fe, twinning takes place at low temperature because the stress for dislocation slip increases sharply with decreasing temperature, which makes twinning become a favorable deformation mode [1–3]. In face-centered cubic (fcc) metals, deformation twinning is rarely observed, especially in those with medium or high stacking fault energy (SFE), such as Cu, Ni and Al [3,4].

Since Blewitt et al. [5] reported that deformation twinning occurred in Cu single crystals put under tension at 4.2 and 77.3 K, numerous experimental investigations on coarse grained (CG) fcc metals with medium–high SFEs have indicated that deformation twinning did not take place under normal deformation conditions, such as tension, compression and torsion, at room temperature (RT) and low strain rate ($<10^{0}\ s^{-1}$) [4,6]. For example, there are no deformation twins in CG-Cu and Cu-0.2 or 2 wt.%Al alloys quasi-statically compressed at room temperature (RT) to a strain of 0.8 [7]. The absence of deformation twinning in CG fcc metals at RT and low strain rate is mainly due to two reasons: (a) the existence of many independent slip systems making slip a very sufficient deformation mode and (b) much higher critical twinning stress [4] (more than several times higher than slip stress).

At the same time, the experimental observations have also demonstrated that deformation twinning in CG-Cu or Ni took place only after sufficient strain hardening has occurred [4,6,8]. Recently, El-Danaf et al. [9] indicated that a certain critical amount of dislocation density is required for the nucleation of deformation twins. For those materials with relatively high SFE, these deformation conditions

* Corresponding author. Tel.: +86 24 8397 8271; fax: +86 24 2389 1320.
E-mail address: shdwu@imr.ac.cn (S.D. Wu).

1359-6454/$30.00 © 2005 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.
doi:10.1016/j.actamat.2005.10.002

can be obtained under a very low temperature and/or a high strain rate ($\sim 10^3\ s^{-1}$), where dislocation-slip processes are suppressed. For example, deformation twins were usually observed in Cu that was tensioned or rolled at liquid nitrogen temperature [5,10]. Also, a very high strain rate promotes the formation of deformation twinning. There are many experiments which show that CG-Cu does not twin at moderate strain level under RT quasi-static deformation, but twins readily when subjected to shock deformation at sufficiently high pressures [11–14]. By way of exception, Dymek et al. [15,16] recently observed deformation twins in Cu single crystal, which was first uniformly rolled to 50% reduction and additionally compressed from different directions in a channel die to a further 30% reduction at RT. They attributed the formation of deformation twins to a martensitic transformation [16].

As far as the mechanism is concerned, deformation twinning in CG fcc metals is generally believed to occur via the well-known pole mechanism proposed by Venables in 1961 [17]. In this mechanism, deformation twin is nucleated from the dissociation of a prismatic dislocation into partial dislocations ($(a/2)[1\,1\,0] = (a/3)[1\,1\,\bar{1}] + (a/6)[1\,1\,2]$) and grows via climbing a Shockley partial dislocation (Burgers vector $(1/6)[1\,1\,2]$) helically to the adjacent slip plane [17]. It requires a high critical resolved shear stress that can only be reached at very low temperature and/or high strain rate for those materials with medium to high SFEs [4,6,17,18]. Moreover, the critical twinning stress follows the Hall–Petch relationship, with a slope for twinning (K_T) significantly larger than that for slipping (K_S) [3,18]. For example, the K_T of Cu is about 0.7 M $N/m^{3/2}$, which greatly exceeds K_S ($\sim$0.35 M $N/m^{3/2}$) [19]. While in recent investigations on CG 70/30 brass and MP35N alloy (a multiphase Co–Ni–Cr–Mo alloy with low SF), the authors found a power-law increase of the twinning stress, σ_t, with the decrease in homogeneous slip length, L, ($\sigma_t \propto L^{-k}$, with $k = 0.89$) [9]. The L is directly related to the grain size and dislocation density. They found no twinning for MP35N alloy with the grain size smaller than 1 μm [9]. For Cu, Meyers at al. [20] performed shock compression experiments at 35 GPa and obtained profuse twinning for grain sizes of 117 and 315 μm, but virtually no twinning for a grain size of 9 μm. These findings imply that deformation twinning in fcc materials becomes very difficult as the grain size decreases down to submicrometer scale.

Recently, molecular dynamic (MD) simulations predicted that Al and Cu with grain size of several tens of nanometers should twin via the process of successive partial dislocation emission from grain boundaries (GBs) and GB junctions [21–23]. These predictions were, subsequently, experimentally verified in nanocrystalline (NC) Al film deformed by slow microindentation [24] and NC-Cu processed by high-pressure torsion at RT and low strain rate ($\sim 10^{-2}\ s^{-1}$) [25]. In addition, abundant deformation twins were also observed in NC Pd [26] and NC fcc Co [27] with a very low SFE. Two other twinning mechanisms predicted by the MD simulations, i.e., homogeneous nucleation of twins inside grains by the dynamic overlapping of SFs and the twin lamella formation via the dissociation and migration of GB segments [28], were also confirmed in NC Al produced by cryogenic ball milling [29,30]. All of the above three twinning mechanisms in NC fcc metals are different from the well known pole mechanism operating in their CG counterparts. These reports clarify a fact that the twinning mechanism is dependent on the grain size. However, it is not known at what grain size the twinning mechanism transforms from the pole mechanism to partial dislocation emission from GBs and GB junctions.

A review of the above literature shows that the deformation conditions and mechanisms of deformation twinning in fcc materials vary with grain size. For those CG materials with medium–high SFE, low temperature and/or high strain rate are required; pole mechanism is the fundamental twinning mechanism. While in the NC case, deformation twins can be formed at RT and low strain rate; partial dislocation emission from GBs and GB junctions controls deformation twinning. Among these deformation conditions, however, large plastic deformation prior to twinning is needed in both cases. Up to date, there has been no published results describing the formation of deformation twins under the same external deformation conditions (temperature, strain and strain rate) in the same material, with grain sizes varying from micrometers to nanometers. In the present work, we used the equal channel angular pressing (ECAP) technique, one of the most promising methods of severe plastic deformation [31], to deform CG-Cu and to produce ultrafine grains (UFG, <1 μm). The objectives of this paper focus on the following issues: the first is to find whether CG-Cu can deform by deformation twinning under ECAP deformation at room temperature and low strain rate and what deformation conditions are required if it can; the second, whether UFG-Cu can deform by twinning and what is the deformation twinning mechanism if it can; the third, what is the grain size effect on the deformation twinning mechanisms.

2. Experimental

Pure (99.97%) polycrystalline Cu rods (∅ 10 mm × 85 mm) annealed at 600 °C for 40 min were used for the ECAP. A scanning electron microscope-electron channeling contrast (SEM-ECC, Cambridge S-360) technique was used to characterize the microstructure of the initial CG-Cu. Before the SEM-ECC observations, the sample was electronically polished. Fig. 1 shows a SEM-ECC micrograph of the initial CG-Cu rod. Annealing twins with micrometer-dimension lamellae were observed frequently. The average grain size was estimated to be about 57 μm by the linear intercept method, excluding annealing twin boundaries.

The ECAP processes were performed at RT, using a die with two columned channels intersecting at an angle of 90°.

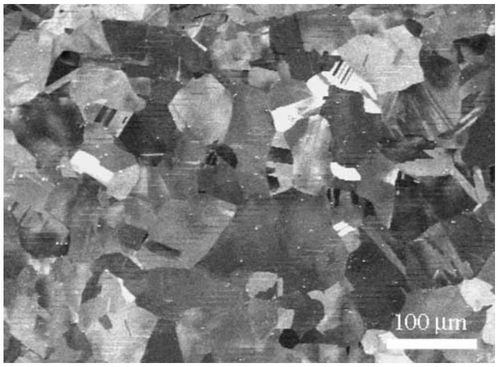

Fig. 1. SEM-electron channeling contrast micrograph of the initial CG-Cu rod.

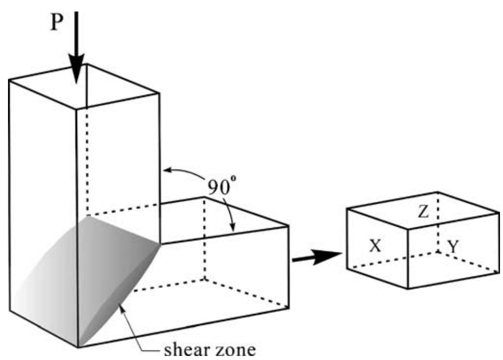

Fig. 2. Schematic illustration of ECAP process.

Fig. 2 is a schematic representation of the ECAP process. For simplicity, only two channels with rectangular shape are presented. Load is applied from the top of vertical channel and the billet is deformed in the narrow shear zone and extruded from the horizontal channel. The shear strain after each pass is about 1 [32]. The pressing speed was about 10 mm/min, which leads to a maximum equivalent strain rate on the order of $10^{-2}\ s^{-1}$ based on the calculation of finite element method [33,34]. The rods were pressed for 1, 2, 4, 8, 16, 20 and 24 passes with route Bc, i.e., the rods were rotated round the longitudinal axis by 90° anti-clockwise before each pass [35].

After ECAP, the microstructure characterization was performed on a transmission electron microscope (TEM; JEM-2000FXII) and a high-resolution TEM (HRTEM; JEM-2010) operated at 200 kV. The finest aperture size from which the selected-area diffraction (SAD) pattern can be taken is 100 nm. Thin foils for TEM and HRTEM observations were cut from both the *X* and *Y* planes in the centers of the pressed rods as shown in Fig. 2. The *X* plane is perpendicular to the longitudinal axis of the rods and the *Y* plane is parallel to the side faces. The prepared thin foils were then first mechanically ground to about 50 μm thick and finally thinned by a twin-jet polishing method in a solution of 25% phosphoric acid, 25% ethanol and 50% water at a voltage of 8–10 V and at RT.

3. Experimental results

3.1. Deformation microstructures

Fig. 3(a)–(c) present bright-field TEM micrographs of the microstructures and the corresponding SAD patterns of Cu ECAPed for: (a) 1, (b) 8 and (c) 24 passes, respectively. After the first pressing, the microstructures in the *Y* plane, as shown in Fig. 2(a), mainly consist of parallel subgrain bands, which lie essentially parallel to the shear direction of 45° from the top to the bottom edges of the sample in the *Y* plane, as also observed in Al previously [35,36]. With an increase in the pressing passes, ultrafine grains were formed via the processes of grain subdivision evolving dislocation accumulation, tangling and rearrangement [35,36]. Careful examination showed that there were no apparent differences in the microstructures in the *X*

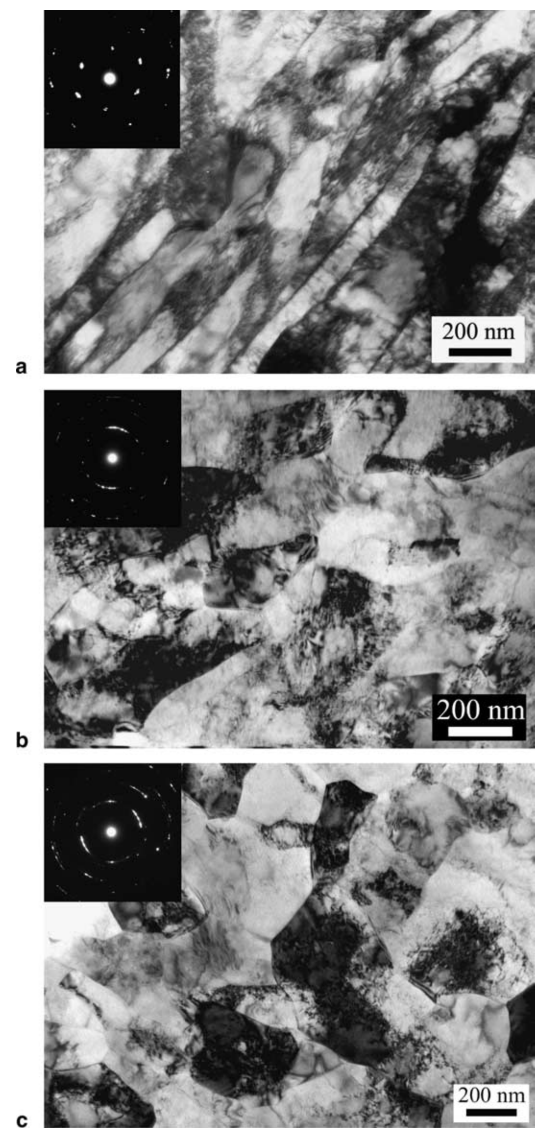

Fig. 3. Bright-field TEM micrographs of deformation microstructures of CG-Cu after ECA pressing for: (a) one pass in the *Y* plane; (b) eight and (c) 24 passes in the *X* plane.

and Y planes after eight passes. As shown in Fig. 3(b) of the microstructures in the X plane, ultrafine grains with mean grain size of ~300 nm are formed after being ECAPed for eight passes. The SAD pattern with discontinuous rings obtained from the region with a diameter of 1 μm indicates that a number of different orientations exist within the selected area. Further pressing does not refine the grains obviously but increases the misorientation between adjacent grains. Fig. 3(c) shows the ultrafine grains with mean size of ~270 nm formed in the X plane in the sample after being ECAPed for 24 passes. The SAD pattern consists of more continuous rings, indicating a higher population of boundaries separated by large angle of misorientation compared with that in Fig. 3(b). It can be seen that there are more sharp and narrow grain boundaries, suggesting a more equilibrium state of the boundaries. Also, the grains are more equiaxed and uniform in size. Such homogeneous UFG microstructures are believed to evolve a kind of continuous reaction that is continuous dynamic recovery under very large accumulative strain [37,38].

3.2. Microstructures of deformation twins in CG-Cu

Deformation twins were observed in CG-Cu after ECAP for one pass. Fig. 4(a) shows a bright-field TEM micrograph of the microstructures in the Y plane near GB. Several parallel lenticular-shaped bands (within black frame) with thickness of only several tens of nanometers are found in the vicinity of a GB. Fig. 4(b) is the SAD pattern taken using a [0 1 1] zone axis from the area containing parallel narrow bands. The mirror spots appear with respect to the $(1 1 \bar{1})$ plane, indicating that the bands constitute a $(1 1 \bar{1})$ twin relationship.

Fig. 5(a) is a bright-field TEM micrograph of deformation microstructures in the grain interior. A severely deformed area (in the center of the micrograph) is visible. Deformation twins (marked with black and white arrows) are detected in this area as demonstrated by the corresponding SAD pattern. Hence, it is reasonable to believe that deformation twinning is triggered because of a very high local stress resulted from the significant plastic deformation. Fig. 5(b) is the corresponding dark-field TEM micrograph of Fig. 5(a), taken using a $(\bar{1} 1 \bar{1})$ diffraction spot of matrix. The twin lamellae are indicated by arrows.

Large numbers of TEM observations show that most of the deformation twins are localized in shear bands and their intersections, both of which are the places undergoing severe plastic deformation. A typical example of a shear band viewed from the [0 1 1] direction of the sample ECAPed for four passes in the Y plane is shown in Fig. 6. The SAD patterns, A and C, with zone axis [0 1 1] taken from the regions marked with white letters A and C in the micrograph, indicate that dislocation slip dominates the plastic deformation in these areas. In contrast, deformation twinning is activated in region B, as indicated by the SAD pattern of B. The twin lamellae are limited to a

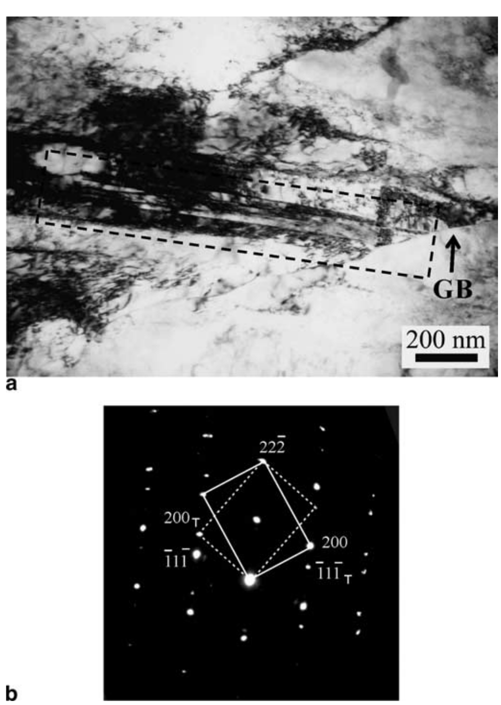

Fig. 4. TEM micrographs of deformation twins near grain boundary in CG-Cu ECAPed for one pass in the Y plane: (a) bright-field image; (b) corresponding SAD pattern with a zone axis [0 1 1].

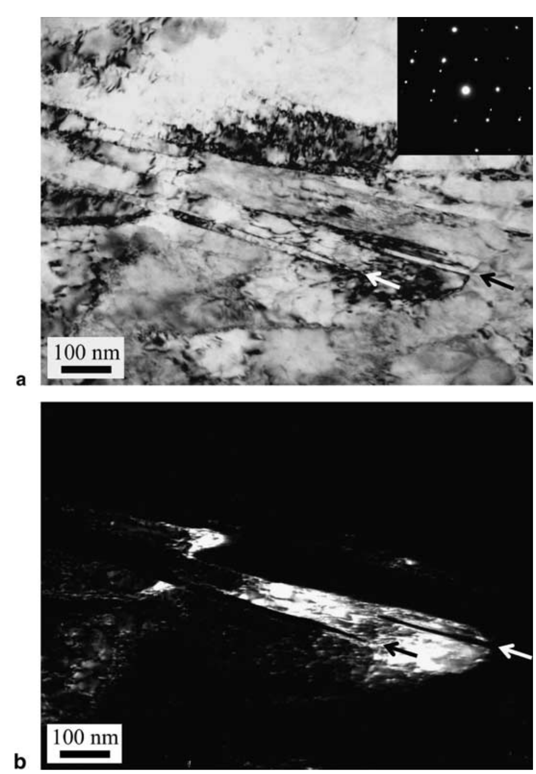

Fig. 5. TEM micrographs of deformation twins in grain interior of the sample ECAPed for one pass: (a) bright-field image; (b) dark-field image. The inset in (a) shows SAD pattern with a zone axis [0 1 1] taken from the area containing narrow bands.

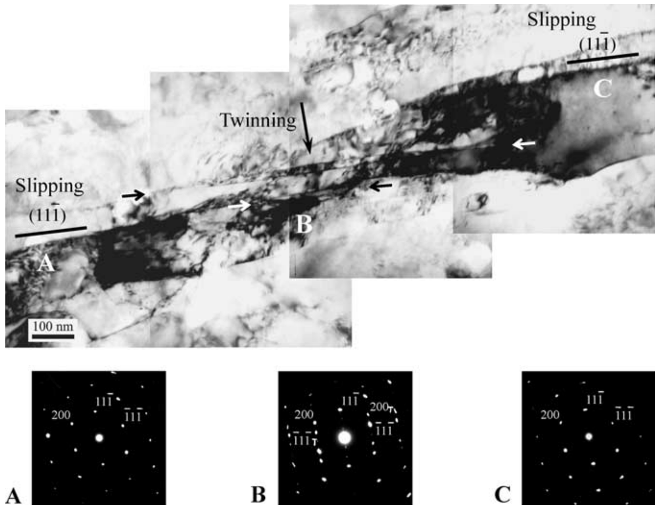

Fig. 6. TEM micrographs of deformation twins formed in a shear band. The SAD patterns with zone axis [0 1 1] taken from the regions marked with white letters A, B and C in micrograph, respectively.

narrow region of the shear band and grow to be lenticular as marked with two pairs of white–black arrows.

Fig. 7 is an example showing the interaction of two shear bands in the *Y* plane in CG-Cu sample ECAPed for two passes. Twins are found in the vicinity of the site where shear bands interact each other, as indicated by the black arrows in Fig. 7. The inset is the SAD pattern with a [0 1 1] zone axis taken from the region containing two acicular bands as marked with arrow A. It is obvious that such deformation twins are formed through the strong interactions of shear plastic deformation from different directions.

In order to view the twin morphology more clearly, HRTEM observations were carried out. Fig. 8 is a HRTEM micrograph of a twin front viewed from the [0 1 1] direction. It can be seen that the twin is advanced by twin dislocations at the tip (marked with white arrows) and thickened by passage of some steps as shown by black arrows. The steps are associated with a few Shockley partial dislocations. The tendency to be lenticular has become obvious. Such deformation twin is believed to grow via the well-known pole mechanism in CG fcc materials [6,17]. As suggested by Venables, once a twin is nucleated, the twin dislocation (Shockley partial dislocation) moves from one slip plane spirally to the next, making the twin thicker and finally lenticular-shaped [17].

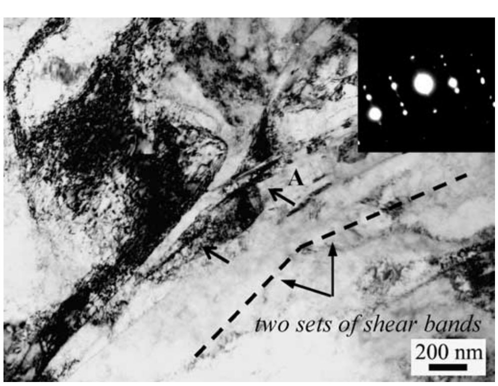

Fig. 7. TEM micrograph of deformation twins formed in the intersection of two shear bands. The inset shows SAD pattern with a zone axis [0 1 1] taken from the area as denoted by arrow A.

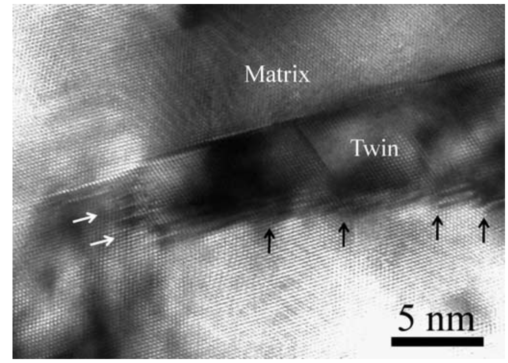

Fig. 8. HRTEM micrograph of a lenticular twin front viewed from the [0 1 1] direction.

3.3. Microstructures of deformation twins in UFG- and NC-Cu

3.3.1. Microstructures of deformation twins in UFG-Cu

With increasing pressing passes, ultrafine grains of several hundred nanometers in size were formed. At the same time, deformation twins were also observed in some of these grains. Fig. 9 shows the formation of deformation twins in ultrafine grains of the samples ECAPed for (a) 16, (b) 20 and (c) 24 passes. In Fig. 9(a) and (b), twins are nucleated at GBs or GB junctions and extend into grain interior as indicated by the white arrows. While in Fig. 9(c), deformation twins have extended to the entire grain. It is probably that such deformation twins in UFG-Cu are formed via partial dislocation emission from GBs and GB junctions as predicted by MD simulations [21,28].

Fig. 10(a) is an HRTEM micrograph of a curved GB (marked by black asterisks) with several steps taken from the white frame in Fig. 9(c). The steps are probably formed by the migration of GB under stress. Further deformation makes dislocations nucleate easily at the corners of the steps because of the high stress concentration around there. As indicated by the black arrows in Fig. 10(a), twins and SFs can usually be detected in the vicinity of the steps. The twin boundaries are determined to be parallel to one set of (1 1 1) planes. Fig. 10(b) is an atomic scale image of the twin boundary enlarged from the white frame in Fig. 10(a). It clearly shows that a partial dislocation (marked by a white arrow on the right) is nucleated at a step and propagates into the grain interior, leaving behind a SF (labeled as SF1) connected with GB. While in its upper part, a twin boundary together with an adjacent SF (labeled as SF2) is formed. It is reasonable to suppose

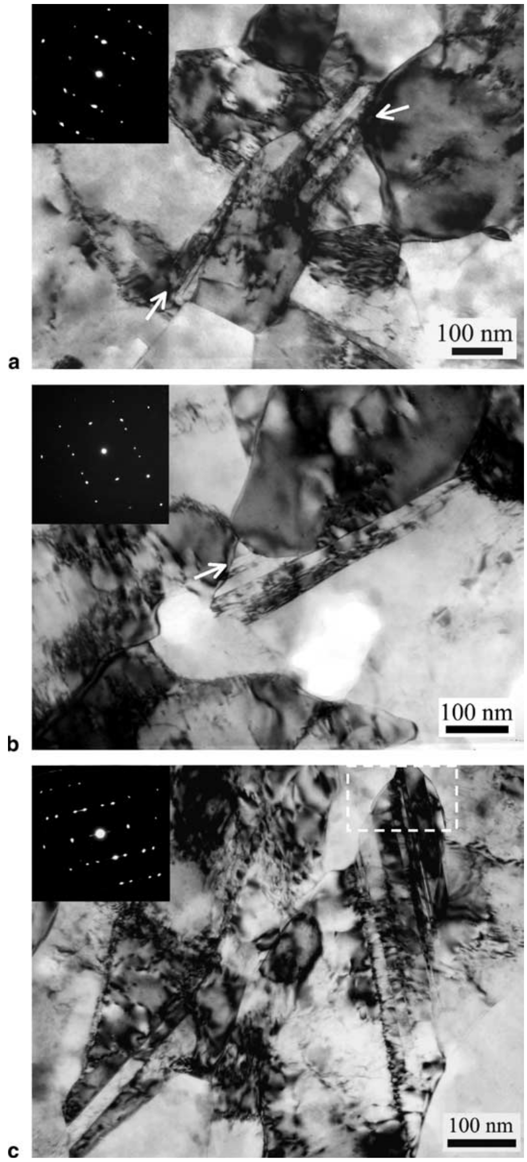

Fig. 9. Bright-field TEM micrographs of deformation twins in ultrafine grains in Cu ECAPed for: (a) 16; (b) 20 passes in the *X* planes and (c) 24 passes in the *Y* plane.

that the twin should be heterogeneously nucleated at GB and grow via successive emission of partial dislocations from GBs on adjacent slip planes [28].

Fig. 11(a) and (b) exhibit several other kinds of deformation twins in ultrafine grains. As shown in Fig. 11(a), deformation twins transect the region between A and B as indicated by arrows in a grain with size of ~200 nm in the cross direction. While in the region between B and C, twins propagate only several tens of nanometers and are terminated by a subgrain boundary. Also, a nanocrystallite of ~90 nm as marked with arrow D is completely twinned. In Fig. 11(b), an ultrafine grain of ~250 nm is divided into a few elongated subgrains by dislocation walls. The narrowest width of the subgrains is less than 100 nm. Deformation twinning tends to take place at these places as indicated by arrows in Fig. 11(b).

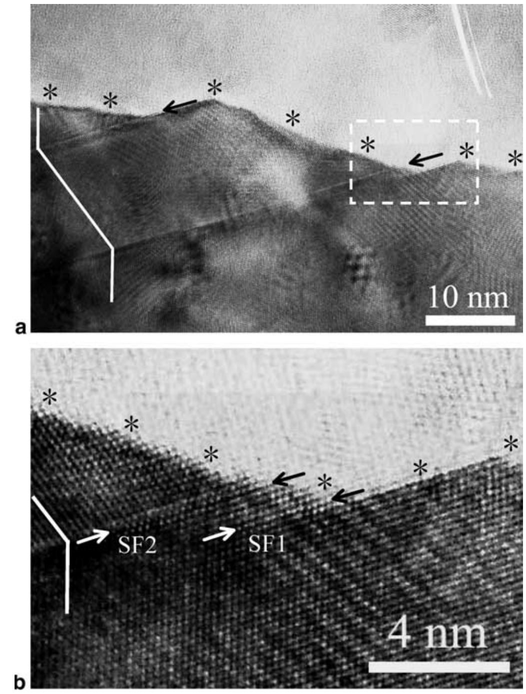

Fig. 10. [1 1 0]-direction HRTEM micrographs taken from the white frame in Fig. 9(c). (a) A curved GB segment (marked by black asterisks) with twin boundaries. The {1 1 1} planes that form the twin relationship among the bands are indicated by white lines. (b) An enlarged image of the white frame in (a).

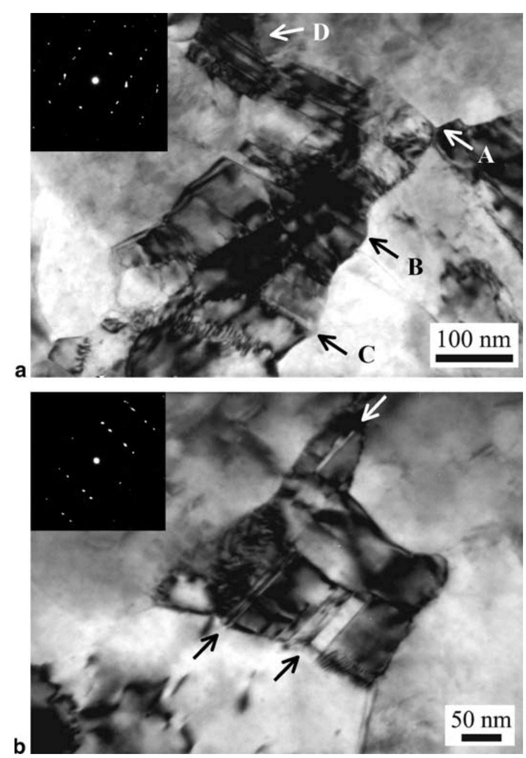

Fig. 11. Bright-field TEM micrographs of deformation twins: (a) in an ultrafine grain and a nanocrystallite; (b) in subgrains of an ultrafine grain.

3.3.2. Microstructures of deformation twins in NC-Cu

There is a small amount of nanocrystallites in Cu produced by ECAP. Fig. 12(a) and (b) show two elongated

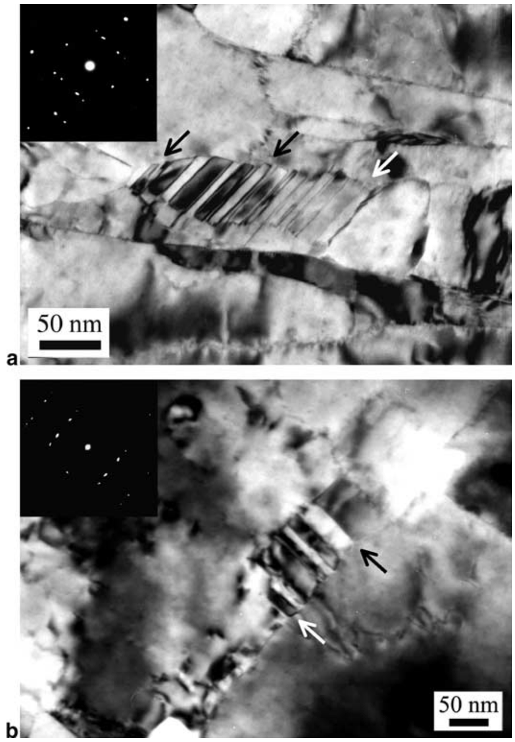

Fig. 12. Bright-field TEM micrographs of deformation twins in nanocrystallites.

nanocrystallites, as indicated by arrows. The width of the grains is 50–70 nm. Along the cross direction, several planar defects characterized with two flat interfaces parallel to each other are visible. The SAD patterns show that they are deformation twins. It seems that deformation twinning is more likely to occur within a short spacing along the cross direction, but not the longitudinal direction. Both MD simulations and experiments have confirmed that the twinning mechanism in NC fcc metals is partial dislocation emission from GBs and GB junctions [21,24,25,28–30].

4. Discussion

The above TEM and HRTEM observations provide experimental evidence that deformation twinning in polycrystalline Cu can take place at RT and low strain rate during the process of ECAP. Twinning in Cu often has the following characteristics. (i) Deformation twinning in CG-Cu mainly occurs in shear bands and their intersections, which are places experiencing severe plastic deformation. Pole mechanism is the fundamental twinning mechanism. (ii) Deformation twinning occurs in UFG- and NC-Cu via partial dislocation emission from GBs and GB junctions, which is different from that in their CG counterpart.

4.1. Deformation twinning in CG-Cu

It has been known for a long time that the local stress required to nucleate deformation twinning in fcc materials is high, e.g., 150 MPa for Cu single crystal [5] and 300 MPa for Ni [39]. In order to reach such a high local stress, significant plastic deformation is generally needed. For instance, the increment of residual strain of shock-loaded CG-Cu was observed to correlate with a change in the deformation substructure from dislocation cells to microbands and deformation twins [13]. The present TEM investigations (Figs. 4–7) also confirm that appreciably plastic deformation by slip takes place prior to activating deformation twinning. Thus, it is necessary to consider a twinning criterion which might account for the change of deformation mode in shear band. According to the pole mechanism [17,18], the critical shear stress, τ_T, required to activate a twinning source of length l is

$$n \cdot \tau_T = \frac{\gamma}{b_1} + \frac{Gb_1}{l}, \tag{1}$$

where n is a stress concentration factor. In the initial stages of nucleation, n of the order 2–4 is needed [17,40]. γ is the SF energy of metal, while b_1 is the Burgers vector modulus of the Shockley partial dislocation, and G is the shear modulus.

With increasing plastic deformation, the dislocation density increases, relating to l', the length of dislocation, in terms of $l' = 1/\sqrt{\rho}$. Assuming that $l = l'$, then the critical twinning stress can be written as

$$n \cdot \tau_T = \frac{\gamma}{b_1} + Gb_1\sqrt{\rho}. \tag{2}$$

On the other hand, for fcc metals which have a high strain hardening ability, the local shear stress, τ, increases with increasing dislocation density according to Taylor dislocation hardening model [41,42],

$$\tau = \alpha' Gb\sqrt{\rho}, \tag{3}$$

where b is the Burgers vector modulus of the full dislocation and α' is an empirical material constant. For CG-Cu, α' measured by Bailey is 0.5 through tensile experiments [43].

During the process of severe plastic deformation, when the stress concentration resulting from dislocation pile up increases up to the level of the critical twinning stress, deformation twinning might take place. Therefore, the necessary local dislocation density, ρ_n, required for activating deformation twinning can be obtained by combining Eqs. (2) and (3),

$$\rho_n = \frac{\gamma^2}{G^2 b_1^2} \cdot \frac{1}{(n\alpha' b - b_1)^2}. \tag{4}$$

In the case of Cu, the parameters in Eq. (4) are given as follows [44]: $\gamma = 45 \times 10^{-3}$ J/m^2; $G = 48.3$ GPa; $b = (\sqrt{2}/2)a$, where $a(= 3.6 \times 10^{-10}$ m) is the lattice parameter; $b_1 = (\sqrt{6}/6)a$. For $n = 2$ and $\alpha' = 0.5$: $\rho_n = 3.4 \times 10^{15}$ m^{-2} and $\tau_T = 360$ MPa. In shear bands and their intersections, the stress level is raised after severe plastic deformation. It should be noted that present observations of deformation twins in CG-Cu is in a much narrow scale, typically within a length of ∼100 nm, which is different from prior observations in the literature of twinning in Cu single crystal and CG-Cu at a large length scale of 10–100 μm [3–14]. Accordingly, the stress estimated above to trigger such nanoscale twins might also apply to the level at a scale of nanometers, but not to activating twinning at a mesoscale according to previous findings, such as 150 MPa for Cu single crystal [5].

As to the dislocation density, Dalla Torro et al. [45] measured by TEM analysis that it was $1.6 \times 10^{14}\ \mathrm{m}^{-2}$ within cells and $1.5 \times 10^{15}\ \mathrm{m}^{-2}$ in the cell walls of CG-Cu ECAPed for only one pass. It should be noted, however, that these values are the lower limit of the density, since they only measured the visible dislocations and besides, there might be some release of dislocations during the preparation of foils. Consequently, it is reasonable that the dislocation density in shear band should be on the order of $10^{15}\ \mathrm{m}^{-2}$ and easily reach the estimated value ($3.4 \times 10^{15}\ \mathrm{m}^{-2}$) of local dislocation density. However, it does not mean that deformation twinning will be activated once the necessary local dislocation density is achieved. Our experimental observations show that twins are not ubiquitous but are dispersed in the matrix with high dislocation density (Figs. 4–7). The selective twin formation in certain places might be attributed to limited twin sources and grain orientation. It has been suggested by Venables [17] that only long jogs (>5 nm) in a secondary slip dislocation are a source of twins on the primary slip plane in work-hardened fcc metals. The jogging of dislocations in the two conjugate slip systems for cold-worked materials produces these long jogs. Such long jogs are not prevalent, but localize at places where severe plastic deformation occurred.

Fig. 13(a) schematically shows the nucleation of a long jog as a twin source in the pole mechanism as suggested by Venables [17]. Using the notation of the Thompson tetrahedron, there is a dislocation with a Burgers vector AC lying in plane b except for a long jog N_1N_2 (along CB) lying in plane a. The dislocation in plane a (jog N_1N_2) dissociates into a Shockley and a Frank partial according to

$$AC = A\alpha + \alpha C. \tag{5}$$

Under the action of a shear stress, the glissile Shockley partial αC moves away from the sessile Frank partial $A\alpha$ on the plane a, leaving an intrinsic fault. With the continuous operation of the twin source, the twin grows to be a biconvex lenticular shape [17]. To activate such twin source, the shear stress must be high enough along the [1 1 2] direction and there must be some stress concentration on the primary slip plane a. A high stress concentration can only be maintained if cross-slip is unfavourable and this is the case when the shear direction is around [1 1 2]. In the case of ECAP operation, the schematic illustration of twin source is shown in Fig. 13(b). In the Thompson tetrahedron ABCD, the plane AFD, i.e. {1 1 0} plane, is parallel to the shear plane of ECAP and the long jog along CB is perpendicular to the shear direction. Under this grain orientation, double-slip is active. Our experiments clearly demonstrate that CG-Cu deform readily by deformation twinning if the shear stress is high enough.

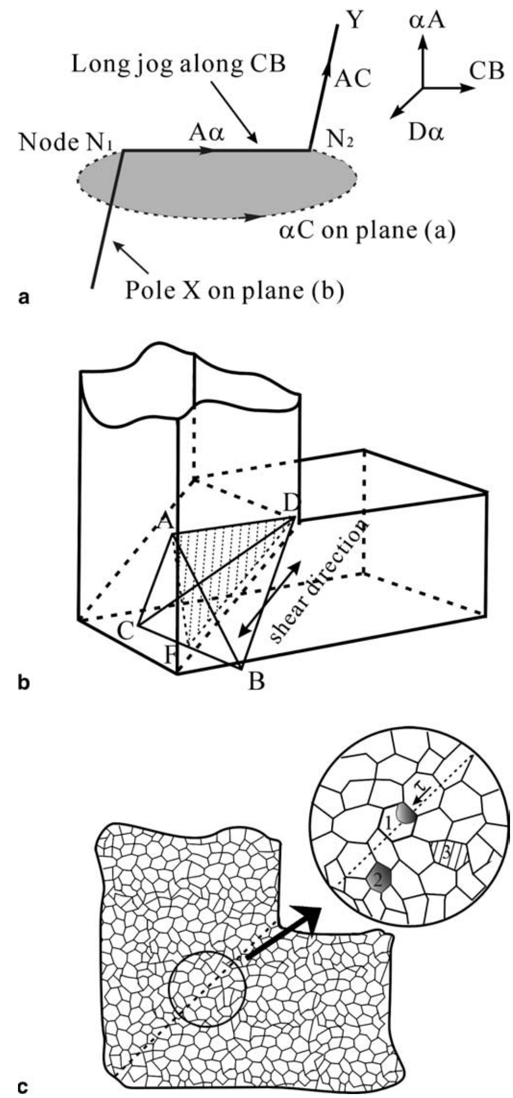

Fig. 13. (a) Pole mechanism of deformation twinning for fcc metals; (b) grain orientation for twinning in ECAP pattern; (c) twinning of ultrafine and nanometer-sized grains under ECAP shear stress.

4.2. Deformation twinning in UFG- and NC-Cu

The twinning stress becomes extremely high when the grain size is below micrometer dimensions as deduced from the Hall–Petch relationship ($\tau_T \sim d^{-1/2}$). This means that deformation twinning should be very difficult or even absent in ultrafine and NC grains. However, both previous works [24–27,29,30] and present observations show that deformation twinning does take place in ultrafine and NC grains via partial dislocation emission from GBs and GB junctions (Figs. 9–12). These investigations reflect the inability of the pole mechanism to provide sources for deformation twinning in ultrafine and NC grains.

It is well known that the dislocation nucleation mechanism, the Frank–Read mechanism, can only operate down to a minimum grain size of typically about 1 μm [44]. Below this size, GBs and GB junctions should therefore provide the necessary dislocation sources. Computer simulations have predicted that, with decreasing grain size, the dislocation nucleation at GBs transforms from full dislocation emission to partial dislocation emission [46]. In order to elucidate this transition (slip to twinning) in NC Al, Chen et al. [24] proposed a simple model based on the shear stresses required to initiate a full 1/2[1 1 0] dislocation and a Shockley 1/6[1 1 2] partial dislocation from GBs. In the model, the shear stresses are expressed as

$$\tau_S = \frac{2\alpha\mu b}{d}, \tag{6}$$

for a full dislocation, and

$$\tau_P = \frac{2\alpha\mu b_1}{d} + \frac{\gamma}{b_1}, \tag{7}$$

for a Shockley partial dislocation, where μ is the shear modulus, d is the grain size, γ is the SF energy, b and b_1 are the Burgers vector modulus of full dislocation and Shockley partial dislocation, respectively. The parameter α reflects the character of the dislocation ($\alpha = 0.5$ and 1.5 for edge and screw dislocations, respectively [44]) and contains the scaling factor between the length of the dislocation source and the grain size.

In this dislocation model, the influence of elastic anisotropy, the small Peierls–Nabarro stress, localized stress concentration and the interactions of dislocations with grain boundaries are not included. In our case, deformation twinning takes place during the processes of severe plastic deformation, in which stress concentration should play an important role. Here, we consider that localized stress concentration promotes deformation twinning. Then, Eq. (7) can be written as

$$n \cdot \tau_P = \frac{2\alpha\mu b_1}{d} + \frac{\gamma}{b_1}, \tag{8}$$

where n is a stress concentration factor.

Fig. 14 shows the grain size dependence of the stresses, τ_S and τ_P, according to Eqs. (6) and (8). It is clear that, with decreasing grain size, τ_P will be smaller than τ_S and deformation twinning should occur via partial dislocation emission from GBs. The critical grain size, d_c, at which deformation modes transform from slip to twinning, can be obtained by comparing Eqs. (6) and (8)

$$d_c = \frac{2\alpha\mu(nb - b_1)b_1}{\gamma}. \tag{9}$$

Using the parameters of Cu and $\alpha = 1$, for $n = 2$ and 4, d_c is estimated to be 115 and 277 nm, respectively. These predictions are consistent with our observations that Cu with grain size of several hundred nanometers twins via partial dislocation emission from GBs and GB junctions, but not the pole mechanism.

Partial dislocation emission from GBs or GB junctions, as indicated by MD simulations, involves the removal of a GB dislocation and the reorganization of the remaining GB dislocations [47]. Yamakov et al. [48,49] used the splitting distance, r, between two Shockley 1/6[1 1 0] partial dislocation (assuming a 1/2[1 1 0] edge dislocation is dissociated into two partials) to compare with grain size, d. They found that if d is larger than r, both a leading and a trailing Shockley partial dislocation connected with a SF are emitted from GB, traveling across the grain and finally being absorbed in the opposite GB. There is nothing reserved in the grain. If d is smaller than r, however, only the leading partial dislocation is emitted from GB. There is an SF, which can also be considered as a micro-twin with thickness of only one atomic layer, transecting the grain. A schematic illustration of the process is shown in Fig. 13(c). As shown, a Shockley partial dislocation is emitted from the GB in nano-grain 1 when it passes through the ECAP shear plane, leaving behind a SF connected with the GB (the shadowed area). While in nano-grain 2, a SF has completely transected the entire grain. For twinning, an approximate grain orientation similar to that in the CG case as shown in Fig. 13(b) might also be needed. The creation of such a SF corresponds to the heterogeneous nucleation of a deformation twin at GB as predicted by MD simulations [47,48]. In this case, if later partial dislocations are successively emitted from the adjacent (1 1 1) slip plane, the deformation twin grows thicker [28], as illustrated in nano-grain 3 in Fig. 13(c). However, all the deformation twins that can be experimentally found in various NC metals are formed after the metals have experienced severe plastic deformation by one of following methods: microindentation [24], high-pressure torsion [25], cryogenic ball milling [29,30], cold rolling [26], surface mechanical attrition treatment [27] and present ECAP. It is well known that UFG and NC materials processed by severe plastic deformation methods have nonequilibrium GBs, which contain lots of extrinsic GB dislocations [50]. The interactions of the extrinsic and intrinsic GB dislocations might form various defaults, such as jogs and faulted dipoles. These defaults together with GB dislocations might act as the twin source. However, the detailed process and mechanisms of partial dislocation emission from GB and GB junctions are not well understood yet. More theoretical

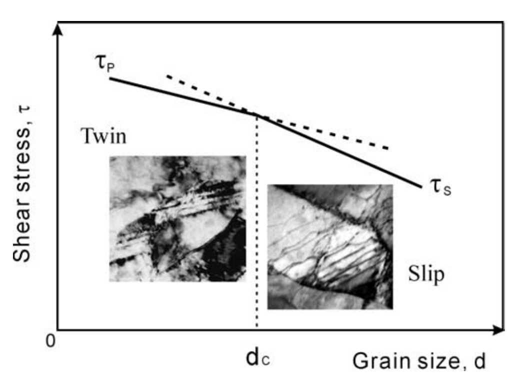

Fig. 14. Grain size dependence of the shear stress required for dislocation emission from GB of UFG- and NC-Cu.

and experimental investigations are needed to solve this issue.

5. Conclusions

Deformation twins have been observed in polycrystalline Cu with grain sizes varying from micrometers to nanometers during the process of ECAP at room temperature and low strain rate ($\sim 10^{-2}\ s^{-1}$). Based on the experimental investigations, the following conclusions are drawn.

(1) Deformation twinning takes place in CG-Cu mainly in shear bands and their intersections, which are places undergoing severe plastic deformation. It is a localization effect of plastic deformation. Twinning triggered in these sites is due to the very high local stress up to the level of the critical twinning stress. The fundamental twinning mechanism is the pole mechanism.

(2) Deformation twins in UFG- and NC-Cu are nucleated from GBs and GB junctions and grow via successively partial dislocation emission from the adjacent (1 1 1) slip plane. At grain sizes of several hundred nanometers, the twinning mechanism transforms from the pole mechanism in the CG case to partial dislocation emission from GBs and GB junctions.

Acknowledgements

The authors thank the financial support from NSFC under Grant Nos. 50171072, 50371090 and 50471082. The authors express their appreciation to Professor K. Lu for his valuable suggestions. Z.F. Zhang thanks the "Hundred of Talents Projection" of the Chinese Academy of Sciences for financial support.

References

[1] Harding J. The yield and fracture behaviour of high-purity iron single crystals at high rates of strain. Proc Roy Soc 1967;299A(1459):464–90.
[2] Bolling GF, Richman RH. Continual mechanical twinning. Acta Metall 1965;13(7):709–57.
[3] Meyers MA, Vöhringer O, Lubarda VA. The onset of twinning in metals: a constitutive description. Acta Mater 2001;49(19):4025–39.
[4] Venables JA. In: Reed-Hill RE, Hirth JP, Rogers HC, editors. Deformation twinning. New York (NY): Gordon and Breach; 1964. p. 7.
[5] Blewitt TH, Coltman RR, Redman JK. Low-temperature deformation of copper single crystals. J Appl Phys 1957;28(6):651–60.
[6] Christian JW, Mahajan S. Deformation twinning. Prog Mater Sci 1995;39(1–2):1–157.
[7] Rohatgi A, Vecchio SK, Gray III TG. The influence of stacking fault energy on the mechanical behavior of Cu and Cu–Al alloys: deformation twinning, work hardening, and dynamic recovery. Metall Mater Trans A 2001;32(1):135–45.
[8] Nolder RL, Thomas G. The substructure of plastically deformed nickel. Acta Metall 1964;12(2):227–40.
[9] El-Danaf E, Kalidindi SR, Doherty RD. Influence of grain size and stacking-fault energy on deformation twinning in fcc metals. Metall Mater Trans A 1999;30(5):1223–33.
[10] Wang YM, Jiao T, Ma E. Dynamic processes for nanostructure development in Cu after severe cryogenic rolling deformation. Mater Trans 2003;44(10):1926–34.
[11] Johari O, Thomas G. Substructures in explosively deformed Cu and Cu–Al alloys. Acta Metall 1964;12(10):1153–9.
[12] Crimp MA, Smith BC, Mikkola DE. Substructure development in shock-loaded Cu–8.7Ge and copper: the role of temperature, grain size and stacking fault energy. Mater Sci Eng 1987;96:27–40.
[13] Gray III GT, Follansbee PS, Frantz CE. Effect of residual strain on the substructure development and mechanical response of shock-loaded copper. Mater Sci Eng A 1989;111:9–16.
[14] Murr LE, Esquivel EV. Observations of common miscrostructural issues associated with dynamic deformation phenomena: twins, microbands, grain size effects, shear bands, and dynamic recrystallization. J Mater Sci 2004;39(4):1153–68.
[15] Wróbel M, Dymek S, Blicharski M. The effect of strain path on microstructure and texture development in copper single crystals with $(1\,1\,0)[0\,0\,1]$ and $(1\,1\,0)[1\,\bar{1}\,0]$ initial orientations. Scripta Mater 1996;35(3):417–22.
[16] Dymek S, Wróbel M. Polytypism in copper single crystals deformed at room temperature. Mater Chem Phys 2003;81(2–3):552–4.
[17] Venables JA. Deformation twinning in face-centred cubic metals. Philos Mag 1961;6(63):379–96.
[18] Venables JA. The nucleation and propagation of deformation twins. J Phys Chem Solids 1964;25(7):693–700.
[19] Vöhringer O. Flow stress for twinning of α-copper-alloys. Z Metallkd 1976;67(8):518–24.
[20] Meyers MA, Andrade RU, Chokshi HA. The effect of grain size on the high-strain, high-strain-rate behavior of copper. Metall Mater Trans A 1995;26(11):2881–93.
[21] Yamakov V, Wolf D, Phillpot SR, Mukherjee AK, Gleiter H. Dislocation processes in the deformation of nanocrystalline aluminium by molecular-dynamics simulation. Nature Mater 2002;1(1):45–8.
[22] Schiøtz J, Jacobsen KW. A maximum in the strength of nanocrystalline copper. Science 2003;301(5):1357–9.
[23] Van Swygenhoven H. Grain boundaries and dislocations. Science 2002;296(5):66–7.
[24] Chen MW, Ma E, Hemker KJ, Wang YM, Cheng X. Deformation twinning in nanocrystalline aluminum. Science 2003;300(5623):1275–7.
[25] Liao XZ, Zhao YH, Srinivasan SG, Zhu YT, Valiev RZ, Gunderov DV. Deformation twinning in nanocrystalline copper at room temperature and low strain rate. Appl Phys Lett 2004;84(4):592–4.
[26] Rösner H, Markmann J, WeissmÜller J. Deformation twinning in nanocrystalline Pd. Philos Mag Lett 2004;84(5):321–34.
[27] Wu X, Tao N, Hong Y, Lu J, Lu K. $\gamma \rightarrow \varepsilon$ martensite transformation and twinning deformation in fcc cobalt during surface mechanical attrition treatment. Scripta Mater 2005;52(7):547–51.
[28] Yamakov V, Wolf D, Phillpot SR, Gleiter H. Deformation twinning in nanocrystalline Al by molecular-dynamics simulation. Acta Mater 2002;50(20):5005–20.
[29] Liao XZ, Zhou F, Lavernia EJ, Srinivasan SG, Baskes MI, He DW, et al. Deformation mechanism in nanocrystalline Al: partial dislocation slip. Appl Phys Lett 2003;83(4):632–4.
[30] Liao XZ, Zhou F, Lavernia EJ, He DW, Zhu YT. Deformation twins in nanocrystalline Al. Appl Phys Lett 2003;83(24):5062–4.
[31] Valiev RZ, Islamgaliev RK, Alexandrov IV. Bulk nanostructured materials from severe plastic deformation. Prog Mater Sci 2000;45(2):103–89.
[32] Segal VM. Materials processing by simple shear. Mater Sci Eng A 1995;197(2):157–64.
[33] Baik SC, Estrin Y, Kim HS, Hellmig RJ. Dislocation density-based modeling of deformation behavior of aluminium under equal channel angular pressing. Mater Sci Eng A 2003;351(1–2):86–97.
[34] Kim HS, Hong SI, Seo MH. Effects of strain hardenability and strain-rate sensitivity on the plastic flow and deformation homogeneity

during equal channel angular pressing. J Mater Res 2001;16(3):856–64.
[35] Iwahashi Y, Horita Z, Nemoto M, Langdon TG. The process of grain refinement in equal-channel angular pressing. Acta Mater 1998;46(9): 3317–31.
[36] Iwahashi Y, Horita Z, Nemoto M, Langdon TG. An investigation of microstructural evolution during equal channel angular pressing. Acta Mater 1997;45(11):4733–41.
[37] Belyakov A, Gao W, Miura H, Sakai T. Strain-induced grain evolution in polycrystalline copper during warm deformation. Metall Mater Trans A 1998;29(12):2957–65.
[38] Belyakov A, Sakai T, Miura H, Tsuzaki K. Grain refinement in copper under large strain deformation. Philos Mag A 2001;81(11):2629–43.
[39] Haasen P. Plastic deformation of nickel single crystals at low temperature. Philos Mag 1958;3(28):384–418.
[40] Thornton PR, Mitchell TE. Deformation twinning in alloys at low temperature. Philos Mag 1962;7(75):361–75.
[41] Taylor GI. The mechanism of plastic deformation of crystals. Proc Roy Soc 1934;145(A855):362–87.
[42] Mott NF. The work hardening of metals. Trans Met Soc AIME 1960;218:962–8.
[43] Bailey JE. The dislocation density, flow stress and stored energy in deformed polycrystalline copper. Philos Mag 1963;8(86):223–36.
[44] Hirth JP, Lothe J. Theory of dislocations. 2nd ed. New York (NY): Wiley; 1982.
[45] Dalla Torre F, Lapovok R, Sandlin J, Thomson PF, Davies CHJ, Pereloma EV. Microstructures and properties of copper processed by equal channel angular extrusion for 1–16 passes. Acta Mater 2004;52(16):4819–32.
[46] Cheng S, Spencer JA, Milligan WW. Strength and tension/compression asymmetry in nanostructured and ultrafine-grain metals. Acta Mater 2003;51(15):4505–18.
[47] Van Swygenhoven H, Derlet PM, Frøseth AG. Stacking fault energies and slip in nanocrystallie metals. Nat Mater 2004;3(6):399–403.
[48] Yamakov V, Wolf D, Phillpot SR, Mukherjee AK, Gleiter H. Length-scale effects in the nucleation of extended dislocations in nanocrystalline Al by molecular-dynamics simulation. Acta Mater 2002;49(14):2713–22.
[49] Yamakov V, Wolf D, Phillpot SR, Mukherjee AK, Gleiter H. Deformation-mechanism map for nanocrystalline metals by molecular-dynamics simulation. Nat Mater 2004;3(1):43–7.
[50] Horita Z, Smith DJ, Nemoto M, Valiev RZ, Langdon TG. Observations of grain boundary structure in submicrometer-grained Cu and Ni using high-resolution electron microscopy. J Mater Res 1998;13(13):446–9.