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

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The grain-size effect on deformation twinning in nanocrystalline copper is studied. It has been reported that deformation twinning in coarse-grained copper occurs only under high strain rate and/or low-temperature conditions. Furthermore, reducing grain sizes has been shown to suppress deformation twinning. Here, we show that twinning becomes a major deformation mechanism in nanocrystalline copper during high-pressure torsion under a very slow strain rate and at room temperature. High-resolution transmission electron microscopy investigation of the twinning morphology suggests that many twins and stacking faults in nanocrystalline copper were formed through partial dislocation emissions from grain boundaries. This mechanism differs from the pole mechanism operating in coarse-grained copper.
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Nanocrystalline (nc) materials have been reported to have superior mechanical properties such as high strength, which can coexist with very good ductility.[1–3] These superior mechanical properties are attributed to their unique deformation mechanisms, which are different from those in their coarse-grained (CG) counterparts.[4–8] For example, molecular dynamics simulations, which used extremely high strain rates in the order of 10^6 to 10^8 s^{-1}, predict that NC Al deforms via partial dislocation emission from grain boundaries, which consequently produces deformation twins.[7] These predictions have recently been verified experimentally in nc Al powder processed by ball milling at liquid nitrogen temperature[9] and in nc Al film produced by physical vapor deposition.[8] These observations are very surprising because deformation twinning has never been observed in CG Al.

High strain rate, low temperature, and nanometer grain size are major contributing factors for deformation twinning in the ball-milled Al powder.[9,10] In fact, both high strain rate and low temperature are known to promote deformation twinning.[11,12] For example, CG copper does not deform by twinning[13,14] except at very high strain rate[15,16] and/or low temperature.[17] However, the grain-size effect is not so clear. It has been suggested that both the critical slip stress and twinning stress follow the Hall–Petch (HP) relationship, with the HP slope for twinning (k_T) significantly larger than that for slip (k_S) for many CG metals and alloys.[18] For copper, the k_T is about 0.7 MN/m$^{3/2}$, while k_S is about 0.35 MN/m$^{3/2}$.[19] Consequently, dislocation slip rather than deformation twinning is expected to become the preferred deformation mode when the grain is smaller than a certain size. Indeed, Meyers *et al.*[20] reported that shock compression at 35 GPa produced abundant deformation twins in copper samples with grain sizes of 117 and 315 μm, but virtually no twinning in a copper sample with a grain size of 9 μm. On the other hand, it has been well known that the HP relationship fails in nc materials.[4,5] These literature observations raise some fundamental questions on the grain-size effect on deformation twinning. Does the trend that smaller grains are harder to twin extend down to the NC regime? If not, can the nanograin sizes alone produce deformation twinning without the assistance of high strain rate and/or low temperature? In this letter, we will answer these questions by investigating the deformation mechanisms of nc copper.

A 99.99 wt % pure CG copper disk with a thickness of 0.5 mm and a diameter of 10 mm was processed into nc copper via high-pressure torsion (HPT)[21] for five revolutions under 7 GPa at RT and a very low strain rate of about 10^{-2} s^{-1}. HPT produces 100% dense nc materials without introducing any impurity into deformed samples. The sample for high-resolution transmission electron microscopy (HRTEM) investigation was prepared by mechanical grinding of the as-processed nc Cu disk to a thickness of about 10 μm and subsequent ion milling. Special attention was paid to prevent any heating during the HRTEM sample preparation. HRTEM was carried out using a JEOL 3000F microscope operated at 300 kV.

Figure 1 shows two types of grain morphologies exist in the nc copper: (a) elongated and (b) equiaxed. The diameters of most equiaxed grains and the widths of most elongated grains are in the range of 10 to 20 nm. It is believed that, in severely deformed metals and alloys, equiaxed grains, as seen in Fig. 1(b), were evolved from elongated grains.[22]

Twins are ubiquitous in both elongated and equiaxed grains, implying that twinning is a major deformation mechanism in the sample when the grain sizes reach nanometer range. Multifold twins are also abundant. A typical example of a fivefold twin is shown in Fig. 2, in which the twin boundaries are indicated by black arrows and each twin do-

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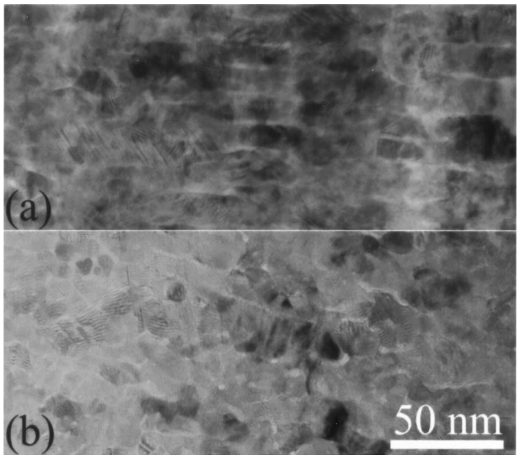

FIG. 1. Two kinds of morphology exist in the HPT copper: (a) elongated and (b) equiaxed.

main in the fivefold twin is marked with a number (1 to 5). The formation mechanism of multifold deformation twins is not clear and needs further study.

Figure 3(a) shows another typical type of twinning morphology in an elongated grain observed from [011]. Most of the twin planes are $(11\bar{1})$ (indicated by white arrows) and one twin plane is $(1\bar{1}1)$ (indicated by a black arrow). A high density of microtwins and stacking faults are seen in areas marked A, B, and C, respectively. Area B, where the narrowest $(11\bar{1})$ planes are less than 10 nm in width, is magnified in Fig. 3(b). There is only one twin boundary at the upper part of Fig. 3(b) that divides twin domains I and II. However, there are high densities of microtwins and stacking faults, which can also be regarded as microtwins with the thickness of only one atomic layer, at the lower part of II. These microtwins and stacking faults do not pass across the whole grain, but stop in the grain interior with Shockley partial dislocations (Burgers vectors $\frac{1}{6}\langle 112\rangle$) located at the front boundaries of the microtwins and stacking faults. It is obvious that these twins were heterogeneously nucleated at a grain boundary and grew into the grain interior via partial dislocation emission from the grain boundary. Such heterogeneous twin nucleation and twin morphology have been predicted in nc Al by molecular dynamics simulations,[7] but have never been experimentally observed in nc materials.

Perfect dislocations (Burgers vectors $\frac{1}{2}\langle 110\rangle$) are found in elongated grains but not in equiaxed grains. For example, there are a few 60° dislocations in the elongated grain shown in Fig. 3(a), which amount to a dislocation density of close to $10^{16}\ \mathrm{m}^{-2}$. Figure 4 shows an equiaxed grain with a diameter of about 10 nm containing some microtwins, but no perfect dislocation. These observations strongly imply that for grains of around 10 nm in diameter, partial dislocation emissions from grain boundaries are still a viable deformation mechanism, but perfect dislocation slip becomes more difficult. These results are reasonably consistent with a recent molecular dynamics simulation on nc copper[23] that predicts a deformation mode transition from dislocation-mediated plasticity to grain-boundary sliding at a grain size of 10 to 15 nm.

These experimental observations demonstrate that copper can deform by twinning at a very low strain rate and RT if its grain size is in the nanometer range. The grain-size effect on deformation twinning in CG copper (that is, smaller grains are less likely to twin), does not apply to NC copper. Our results suggest that the deformation twining in nc copper is due to the grain-size effect, that is, caused by the unique deformation mechanism in nc copper. In CG copper, defor-

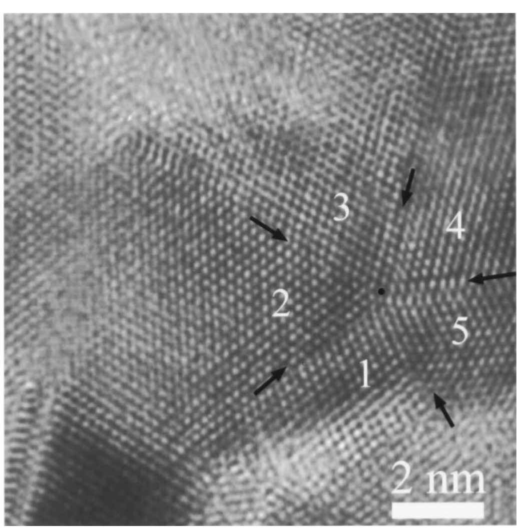

FIG. 2. A typical image of a fivefold twin. The twin boundaries are indicated by black arrows, and each twin domain is marked with 1 to 5, respectively. The twin center is highlighted with a black dot.

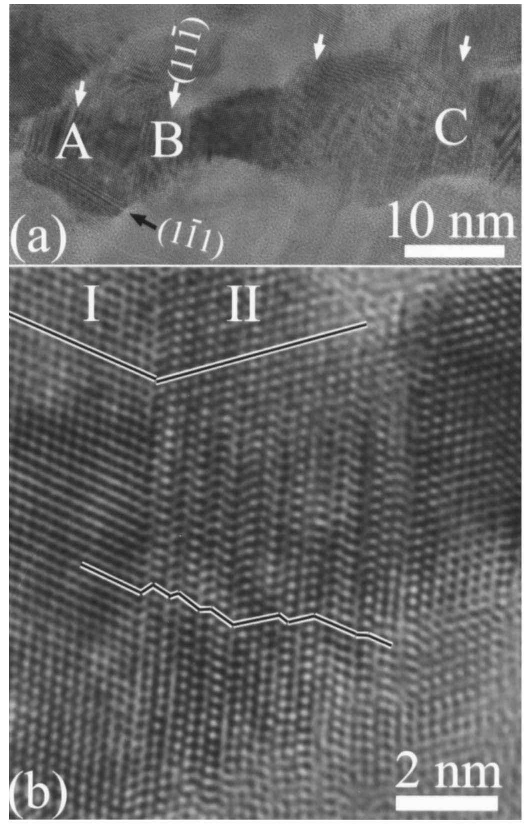

FIG. 3. (a) A typical [011] HRTEM image of an elongated crystallite with width varying from smaller than 10 nm to about 20 nm. Twins are seen in this crystallite, with most of the twin planes being $(11\bar{1})$ (indicated by white arrows), and one being $(1\bar{1}1)$ (indicated by a black arrow). Microtwins and stacking faults are seen in areas marked A, B, and C, respectively; (b) an enlarged image of area B in (a). The upper part of the image shows only two twin domains—I and II—while the lower part of II have a lot of microtwins and stacking faults with one end of the microtwins/stacking faults ends within the crystallite.

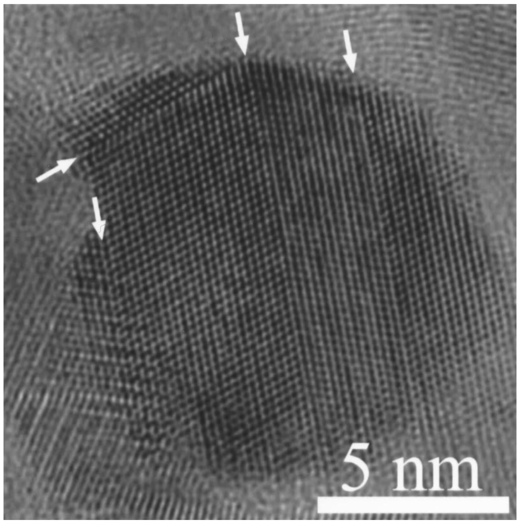

FIG. 4. A typical HRTEM image of an equiaxed grain with microtwins. The twin boundaries are indicated by arrows. No perfect dislocation is seen within the grain.

mation twinning is generally believed to occur via the pole mechanism and requires higher resolved shear stresses that can only be reached under high strain rate and/or low-temperature conditions.[12] In contrast, in nc copper deformation, twins form via partial dislocation emission from grain boundaries instead of the pole mechanism. Therefore, the HP relationship for twinning does not apply to the nc copper. More specifically, in nc copper, the conventional dislocation source (e.g., the Frank–Reed source) in the grain interior may no longer operate and may even no longer exist (see, e.g., Fig. 4). As a consequence, dislocations need to be emitted from the grain boundaries. Smaller grains make it easier to emit partial dislocations than to emit perfect dislocations from grain boundaries.[6] This makes partial dislocation emission from grain boundaries a major deformation mechanism when the grain size is below a certain critical value, and explains the pervasive deformation twins observed in this study.

Our observation clearly shows that the deformation twinning was formed by partial dislocation emissions from grain boundaries [Fig. 3(b)]. However, the process and mechanisms of partial dislocation emission from grain boundaries are not well understood. Molecular dynamics simulations indicate that it involves the removal of a grain-boundary dislocation from the grain boundary and the reorganization of the remaining grain-boundary dislocations.[24] Niewczas and Saada[25] recently proposed a partial dislocation source mechanism based on faulted dipoles. Although such a mechanism is not expected to operate in the interior of nanograins because of the small grain sizes, it is not clear if faulted dipoles exist on the grain boundaries and act as a source for partial dislocation emissions. It is well known that nc materials produced by severe plastic deformation techniques such as HPT have nonequilibrium grain boundaries, which contain extrinsic dislocations.[26] These dislocations might form faulted dipoles via interactions either among themselves or with grain-boundary dislocations. More theoretical and experimental studies are needed to solve this issue.

In summary, twinning via partial dislocation emissions from grain boundaries becomes a major deformation mechanism in NC copper deformed at a low strain rate and room temperature. The Hall–Petch relationship breaks down in NC copper because of the change in deformation mechanism.

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