

Dislocations in nanocrystalline grains

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Dislocation behavior inside the very small grains of nanocrystalline metals has so far eluded high-resolution transmission electron microscopy (TEM) examinations. We have identified full dislocations using high-resolution TEM, and the different ways they reside in the 20–30 nm Ni grains. The thermally activated propagation of dislocations, their interactions with twin/grain boundaries and dislocation loops, as well as their storage in nanocrystalline grains are demonstrated and discussed. © 2006 American Institute of Physics. [DOI: 10.1063/1.2210295]

Dislocations are well known to be the carriers of plastic deformation in conventional metals. Various types of dislocations have been directly observed in transmission electron microscope (TEM) examinations.^{1,2} For nanocrystalline (NC) metals, the role of dislocations is a fundamental question for understanding the deformation behavior of NC grains, but remains a matter of debate in recent years. The suggestion for dislocations as a major contributor to plastic deformation in NC grains came from computer molecular dynamics (MD) simulations,^{3–5} for grain sizes in the range of approximately 8–50 nm. But in these MD cases the dislocations are activated at strain rates and stress levels much higher than those in laboratory experiments. In postmortem TEM examinations of plastically deformed NC grains, few dislocations were detected.^{6–10} However, *in situ* TEM observations revealed rapid changes in diffraction contrast in NC grains,^{8–13} which have been interpreted as due to dislocation movements upon straining.^{8–13} But the contrast can come from other sources, and it is not possible to obtain high-resolution images and identify Burgers vectors for dislocations during *in situ* TEM experiments. One should also bear in mind that in such ultrathin TEM foils the actions are in regions right at the tip of an advancing crack. Meanwhile, when only a few grains are sitting on top of each other near the free surfaces, dislocation activities, diffusional processes, and changes in the grain boundary structures may have been enhanced. As such, it is questionable if the *in situ* observations can be taken as proof of dislocation activities in bulk deformation.

Therefore, it is important to unequivocally identify dislocations and their Burgers vectors under high-resolution TEM, carefully specify their types (e.g., full versus partial dislocations), and systematically explore their configurations inside the tiny NC grains. In this letter, we address these important issues via extensive postmortem TEM observations in bulk-deformed NC Ni. An electrodeposited NC-Ni foil was acquired from Goodfellow Inc. The as-received 150 μm thick foil was observed to have an average grain size of 24 ± 6 nm, similar to those reported before.^{8,9,14,15} The samples were deformed either in uniaxial tension to failure

(to a strain of $\sim 4\%$, at which point the maximum stress was about 1500 MPa) at a strain rate of $3 \times 10^{-3} \text{ s}^{-1}$, or by cold rolling to a thickness reduction of 30%, both at the liquid-nitrogen temperature (LNT). The tensile behavior was similar to those published before for electrodeposited NC Ni.^{8,9,14,15} The low deformation temperature was employed not only to suppress possible grain growth assisted by stress during deformation¹⁶ but also to retain some of the dislocations for postmortem TEM examination (as mentioned above, dislocation storage is negligible in NC Ni during room temperature deformation but significant in LNT deformation^{17–19}). High-resolution TEM (HRTEM) observations were made in a JEM2010F microscope operating at 200 kV. The TEM sample preparation procedures have been discussed elsewhere.¹⁹

We first examined several as-received NC-Ni samples. Preexisting dislocations and growth twins were few and far between, consistent with previous TEM observations.¹⁹ A comparison of grain size distribution before and after LNT deformation indicated little grain size change during plastic deformation.¹⁹

Figure 1(a) is a HRTEM image showing a NC-Ni grain after tensile deformation. A deformation twin is visible as indicated by an arrow. An enlarged view of the white framed region A, Fig. 1(b), clearly reveals the twin boundary (TB). Details about deformation twinning via partial dislocation mediated processes have been reported elsewhere.¹⁹ These twins are believed to form during deformation and acted as either sources or barriers to dislocations. We observed that full dislocations were activated in addition to partial dislocations, during both tensile and rolling deformations at LNT. In the following, we focus on the normal full dislocations to illustrate in which ways they reside and store in NC grains.

The first example is shown in Fig. 1(c) for region B in Fig. 1(a), where several dislocations are found near the TB. Burgers circuits are drawn to enclose the cores for two dislocations marked by “T.” The magnitude and direction of the vector needed to connect the end to the beginning of the circuit give the Burgers vectors as $\mathbf{b}_1 = 1/2[011]$ and $\mathbf{b}_2 = 1/2[10-1]$, respectively. This indicates that these are full dislocations. We also observed dislocations blocked by the TB, such that a full dislocation is stuck right next to the TB. An example of this, from a sample after cold rolling, is given

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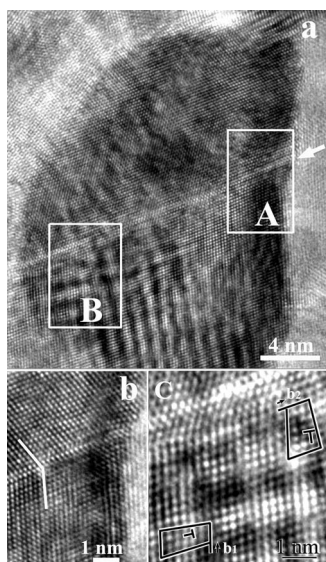


FIG. 1. (a) HRTEM micrograph of a NC-Ni grain after LNT tensile test. A deformation twin boundary is indicated by an arrow. (b) High magnification view of the white framed region A in (a) showing the details of the twin. (c) High-magnification view of the white framed region B in (a) showing the presence of several full dislocations near the twin boundary and grain boundary. The Burgers circuits were drawn to identify Burgers vectors: $b_1 = 1/2[011]$ and $b_2 = 1/2[10-1]$

in Fig. 2. Note that we often observe closely spaced dislocations, causing distortions and difficulties in the identification of their characters and Burgers vectors.

There are other dislocation configurations observed in the NC grains. Figure 3 shows dislocations near a grain boundary (GB) after tensile deformation. These are identified also to be perfect dislocations with Burgers vector $1/2[011]$, in this case in pairs near the GB. We also observed full dislocation loops. Figure 4(a) is a TEM micrograph showing several NC grains (labeled A–C) after LNT rolling. Figure 4(b) is an enlarged lattice image of grain B. The white-boxed area in (b) is further enlarged in Figure 4(c). Clearly visible in the interior of the NC grain is a dislocation loop, consisting of two dislocations with opposite signs on the same slip plane (marked by the double-ended arrow). A Burgers circuit starting at S and ending at F was drawn to enclose one dislocation core marked by a “T.” Assuming the electron beam and the dislocation line are parallel to $[1-10]$, the Burgers vector of the dislocation is determined to be $1/2[101]$ or $1/2[011]$, which has an angle of 60° (or 120°) with respect

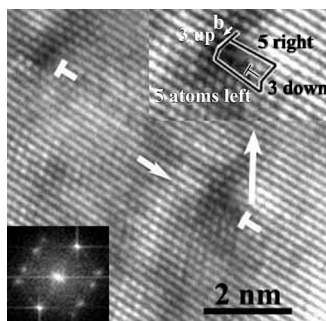


FIG. 2. HRTEM micrograph of a NC grain after LNT rolling showing full dislocations right at the twin boundary. The top-right Burgers circuit was drawn to enclose one dislocation core marked by a “T.” The Burgers vector is $b = 1/2[011]$ or $1/2[101]$. The bottom-left inset gives the diffraction pattern showing the twin relationship.

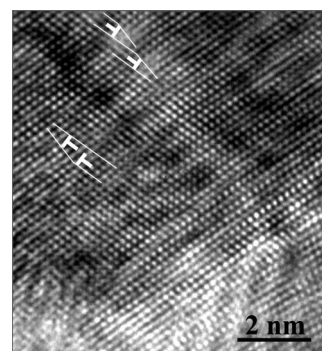


FIG. 3. HRTEM micrograph of a NC grain after LNT tensile test. Note the presence of full dislocations near the grain boundary.

to the dislocation line. Such a dislocation is referred to as a 60° dislocation. Interestingly, several dislocations are found to pile up against the loop, as seen in Fig. 4(d).

These different dislocation configurations share one common feature: the dislocations trapped inside the NC grains appear to be stabilized by forces from other defects, in particular, TB/GB and dislocation loops. At LNT, the interaction (pinning) forces are apparently sufficient to keep some of the dislocations from propagating and disappearing into the opposing GBs; the depinning of propagating dislocations is a thermally activated process according to recent MD

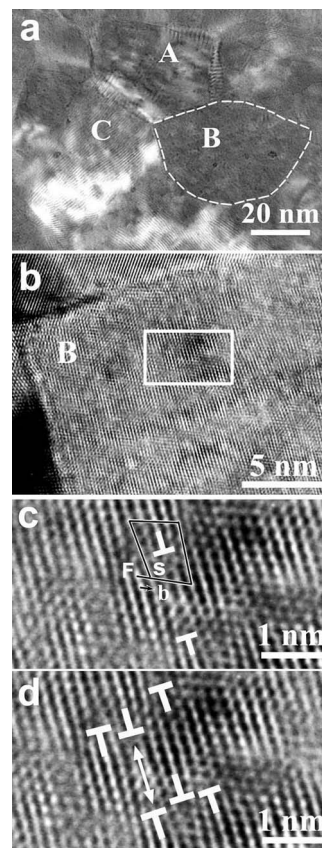


FIG. 4. (a) A TEM micrograph of several NC grains labeled A–C after LNT rolling. (b) HRTEM lattice image of grain B. (c) High-magnification image for the white-boxed area in (b). Note one full dislocation loop consisting of two dislocation cores indicated by the double-end arrow. A Burgers circuit starting at S and ending at F was drawn to enclose one dislocation core marked by a “T.” The electron beam and the dislocation line are parallel to, $[110]$ and the Burgers vector $b = 1/2[011]$ or $1/2[101]$. (d) HRTEM image showing several nearby full dislocations piling against the dislocation loop.

simulations^{14,20} and experiments.¹⁸ We surveyed a number of typical NC grains having full dislocations, five grains in the tensile sample and four grains for the rolled sample. The number of full dislocations was counted, including using Fourier filtered images (not shown due to space limitations). Discounting those right inside the GBs, the (local) dislocation density inside the NC grains is estimated to be in the range of $4.7 \times 10^{15} - 1.3 \times 10^{16} \text{ m}^{-2}$.

Recent MD simulations have revealed that looplike dislocations nucleate from GBs (or the leading partial comes from the GB, but the trailing partial on the same plane arises from the stacking fault left behind by the leading partial),²¹ or from TBs.²² This scenario for the formation of dislocation loops is somewhat different from that in conventional coarse-grained metals. For the latter materials, dislocation loops often form from Frank-Read sources inside the grains. In the tiny NC grains, the population of Frank-Read sources is expected to be low and their operation would require high stresses due to the large curvatures.²³ As such, defect (GB/GB)-assisted nucleation of loops may be more favorable.²⁴⁻²⁶ Another conventional way to form the dislocation loops is the condensation of vacancies and interstitials created during plastic deformation or irradiation. This mode of loop nucleation is not expected to be easy inside NC grains, as the point defects have easy access to sinks provided by the abundant GBs nearby.

This work answers several questions regarding dislocation behavior in NC grains. First of all, we have directly imaged and identified 60° full dislocations, providing unequivocal evidence that they are contributing to plastic deformation, not only in submicron (ultrafine) grains²⁷ but also in truly NC grains. The full dislocations are highly active, perhaps more so than the previously reported partial dislocation mediated processes leading to stack fault formation and deformation twinning.¹⁹ Note that our observation is for relatively simple uniaxial tensile and rolling deformation (stress) conditions, for samples that experienced relatively small plastic strains, and in a material with starting NC grains (rather than in a severely deformed material in which the NC structure comes from accumulation of excessive strains and extraordinarily high densities of dislocations²⁸). Note also that for Ni, MD simulations were not able to reveal dislocation processes after the emission of a leading partial dislocation from GBs, due to the very limited simulation time.²⁹ Second, the full dislocations do not all traverse the grains during deformation leaving no debris behind, but also are capable of accumulating inside NC grains.^{17,20} Our observations, showing dislocation storage only at LNT but not at RT, indicate that the propagation (or depinning) of dislocations is a thermally activated process.^{14,18,20,30} Third, our postmortem examinations revealed the formation of full dislocation loops, possibly emanating from GBs as suggested by MD simulations. Fourth, we have specified several ways in which the dislocations reside inside the tiny grains, postmortem. Note that dislocations have often been perceived not to be sufficiently stable inside NC grains to allow detection after the loading stresses are removed.^{11,31,32} The dislocation configurations observed, including the dislocation piles near TB/GB and dislocation loops, as well as dislocation pairs and dipoles³³ suggest that the interactions with other defects

(perhaps decorated by trace amounts of impurities) may be the origin of stabilizing forces that trap dislocations inside NC grains.

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