Step Structure in Cold-Rolled Deformed Nanocrystalline Nickel *

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The microstructure characteristic of the cold-rolled deformed nanocrystalline nickel metal is studied by transmission electron microscopy. The results show that there are step structures nearby the grain boundary (GB), and the contrast of stress field in front of the step corresponds to the step in the shape. It is indicated that the interaction between twins and dislocations is not a necessary condition to realizing the deformation. In the later stage of the deformation when the grain size becomes about 100 nm, the deformation can depend upon the moving of the boundary of the stack faults (SFs) which result from the partial dislocations emitted from GBs. However, when the size of SFs grows up, the local internal stress which is in front of the step gradually becomes higher. When this stress reaches a critical value which stops the gliding of the partial dislocations, the SFs will stop to grow up and leave a step structure behind.

When the mean grain sizes in metals are below 100 nm, whether plasticity is still carried by dislocations or not is a problem because the stress to bow out a dislocation approaches the theoretical shear stress.[1] For example, the critical grain size of face-centered cubic (*fcc*) metals lie between 20 nm and 40 nm. Thus it is uncertain that whether dislocation nucleates and piles up in the finest fine grain size.[2] In previous experiment studies on nanocrystalline metals, twin and stack faults (SFs) were found in the face-centered-cubic (*fcc*) structure, such as Al,[3] Ni,[4] Cu,[5] and hexagonal structure Co,[6] and grain boundary (GB) sliding was found in Cu metal.[7,8] By in-site tensile deformation observation in transmission electron microscopy (TEM), Kumar *et al.*[9] have found that dislocation-mediated plasticity plays a dominant role in the deformation of nanocrystalline nickel. Dislocation emission at grain boundaries (GBs) together with intragranular slip and unaccommodated grain boundary sliding facilitates the nucleation of voids at boundaries and triple junctions. However, Hugo *et al.*[10] suggested that the samples investigated are thinned for electron transparency, and the observed dislocation activity might therefore be a result of other dislocation sources such as surface defects. In recent study, Budrovic *et al.*[11] have studied the Bragg peaks in x-ray diffraction of electrodeposited nanocrystalline nickel and found that during plastic deformation, the peak broadening is reversible upon unloading. This result indicates that the deformation process does not build up a residual dislocation network and substantial work hardening does not occur in nanocrystalline metal. By using computer simulation, Gutkin *et al.*[12,13] have suggested that nanocrystalline material deformed by the cooperative action of grain boundary sliding and rotational deformation. Yamakov *et al.*[14] demonstrated that when the grain size is less than the splitting distance, only partial dislocations can nucleate on GBs. This means that the single partial dislocations which produce stacking faults (SFs) can glide through the grain until they become incorporated into the GBs on the opposite side. These SFs remain in the grain as planar defects. Thus the GBs in the nanocrystalline regime can promote sliding and act as both the source and the sink for lattice dislocations that extend throughout the entire grain, leaving behind a stacking fault defect.[15−18] The molecule dynamics simulation about nanocrystalline Al has shown that dislocation and dislocation twin boundary reactions can lead to the formation of complex twin networks, i.e. structures of coherent twin boundaries connected by stair-rod dislocations.[14,19]

However, in some specific experiments, such as in ball-milled Cu-Zr with a grain size under 100 nm, dislocations were not found.[20] On the other hand, considering that the geometry difference between foil and bulk samples,[21,22] the results of Ref. [9,10] did not indicate that deformation is governed by extended partial dislocations emitted from GBs. Sufficient and obvious experiment observations that support GBs emitting and absorbing dislocation are still lacking. In this Letter, the microstructure characteristic of the cold-rolled deformed nanocrystalline nickel metal is studied by TEM and the results have been discussed.

The nanocrystalline sheet nickel investigated was electrodeposited with the purity of 99.8% and the thickness of 0.25 mm. The average grain size of the nanocrystalline metals is 20 nm. The dimension of cold-rolled deformation samples was 12.5×12.5 mm. The samples were rolled by 35% reduction in thickness. All the samples were characterized by a Riguta D/max RC X-ray analyser (Cu target: 40 kV/200 mA, Scan: 20.0/70.0/0.2/0.2 s). The microstructure of the Nanocrystalline metals has been investigated in the TEM of Philips Tecnai-2000.

Figure 1 shows the x-ray diffraction pattern of the nanocrystalline nickel 1 and cold-rolled nanocrystalline nickel 2. For nanocrystalline metals, the peak of x-ray diffraction has an obvious broadening because of the fine grain size. The observation results of TEM show that after rolled deformation, the shape of nanocrystalline metal has not changed. However, the size of grains has grown up. This growth up of the grain size results in the narrowing of the peak of x-ray diffraction (see Fig. 1). The average size of deformed nanocrystalline was between 50–70 nm. In addition, some contrasts of strips can be seen in some grains or areas, and these contrasts have proven that Morie stripe exist. Because the size of the grain is too small, the electron beam penetrated area may not be a single grain but an area in which factually there are many grains overlapped together. Therefore the Morie stripes result from the interference of diffraction.[23] On the other hand, though dislocations piling-up structure was not found, a few of dislocations have been found in some particular areas. Furthermore, step structure has been found at and near the GBs as shown in Fig. 2. From Fig. 2 it can also be seen that there is the contrast of stress field in front of the step. These contrasts correspond to the step in the shape. Figure 3 has shown the steps at GBs in another area. The results of Figs. 2 and 3 indicate that, with grain size growing up in the later stage of the deformation, the dislocations can nucleate at the GB.[12−14] Considering the character of nanocrystalline GB,[15−18] it seems that GB emits partial dislocations promoting SF, therefore realizing the process of deformation. Muller and Solenthaler[24] discussed the dislocations-twins interaction. They figured out that, by sequential untwining of one atomic layer at a time, a series of Shockley partial dislocation incidents on a twin boundary can lead to complete untwining and cutting off of the twin. This process of the untwining results from the dislocation bombardments from a nearby GB. Thus a series of partial dislocations nucleated from the GB can continuously narrow the twin by untwining one atomic layer at a time and leave several traces of SFs on their way down to the twin. The result of the computer simulation for nanocrystalline *fcc* metal[14] indicated that, under deformation

conditions when GBs become very active dislocation sources, the size of the twins would become narrower after the interaction between the imperfection dislocations and the twins. For the bigger grain size (70–100 nm) of nanocrystalline metal, a more complicated reticulate structure of the twin would come out.
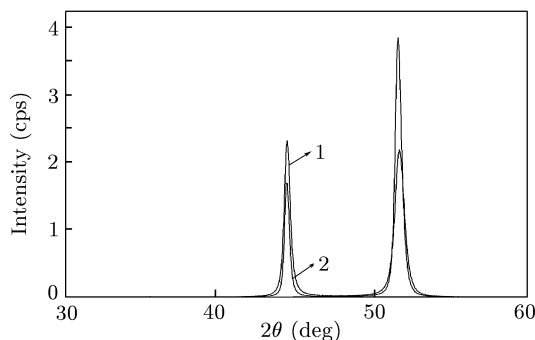

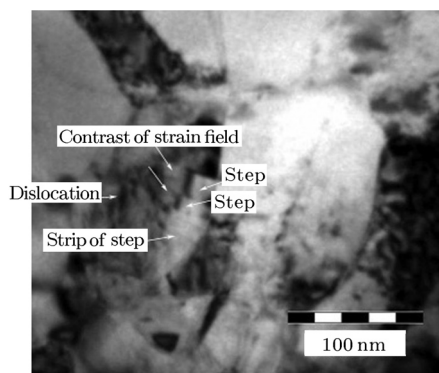**Fig. 1.** X-ray diffraction pattern of the nanocrystalline nickel 1 and cold-rolled nanocrystalline nickel 2.

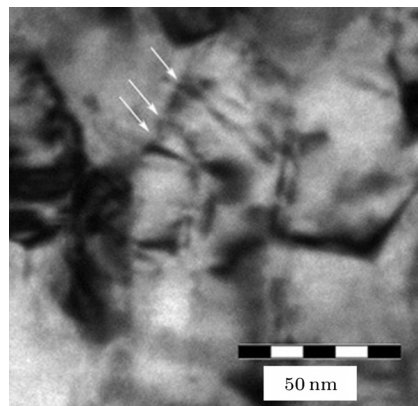**Fig. 2.** The step in the microstructure of cold-rolled nanocrystalline nickel.

Fig. 3. The step in the microstructure in another area.

From Fig. 2, it can be seen that GB emits dislocations and promote the creation of SF, and a few of stack faults constitute the steps structure. However, there is no twin in the grains, and the contrast of

stress field is found in front of the step field. It shows that in this experiment, the interaction between twins and dislocations (or SFs)[12−14,24] is not a necessary condition to realize the deformation. It is very possible that, in the later stage of the deformation when the grain size became to about 100 nm, the deformation only depends upon the moving of the boundary of the SFs which result from the imperfection dislocations emitted from GBs. In other words, the movement of the boundary dislocations of SFs results in the growing-up of the SFs, therefore realizes deformation. This process is illustrated in Fig. 4. The orientation and plane of the atom lattice of Fig. 4 are according to the computer simulation results of Ref. [14] on the deformation mechanism of the *fcc* structure nanocrystalline metal. However, when the size of stack faults grows up, the local internal stress which is in front of the step gradually becomes higher. When this stress reach a critical value which stopping the gliding of the partial dislocations, the SFs will stop growing up and leave a step structure behind, as is seen from Figs. 2 and 4.
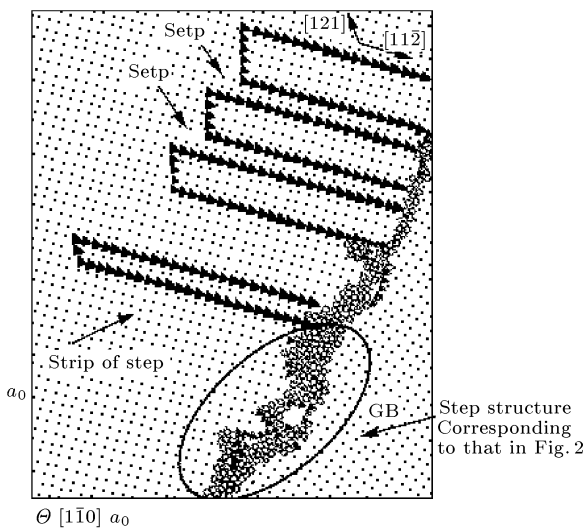

Fig. 4. Partial dislocations nucleate on the grain boundaries producing stacking faults, and stacking faults moved by the partial dislocations gliding (corresponding to Figs. 5 and 6, the (111) plane and atoms are marked according to Ref. [14]).

In summary, the microstructure characteristic of the cold-rolled deformed nanocrystalline nickel metal has been studied by TEM. The results show that there are step structures nearby GB, and the contrast of stress field in front of the step corresponds to the step

in the shape. It indicates that the interaction between twins and dislocations is not a necessary condition to realizing the deformation. In the later stage of the deformation when the grain size become about 100 nm, the deformation can depend upon the moving of the boundary of the SFs which result from the partial dislocations emitted from GBs. However, when the size of SFs grows up, the local internal stress which is in front of the step gradually becomes higher. When this stress reaches a critical value which stop the gliding of the partial dislocations, the SFs will stop growing up and leave a step structure behind.

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