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A STUDY ON THE MICROSTRUCTURE CHARACTERISTIC OF THE COLD-ROLLED DEFORMED NANOCRYSTALLINE NICKEL

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Abstract. In this paper the microstructure characteristic of the cold-rolled deformed nanocrystalline Nickel metal has been studied by transmission electron microscopy (TEM). The results show that there were step structures near by grain boundary (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 became about 100 nm, the deformation occurs only depend upon the moving of the boundary of the stack faults (SFs) which result from the imperfection dislocations emitted from GBs. In the other word, the movement of the boundary dislocations of SFs results to growing-up of the size of the SFs, therefore realizes deformation. 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 stopping the gliding of the partial dislocations, the SFs will stop growing up and leave a step structure behind.

1. INTRODUCTION

When the mean grain sizes in metals are below 100 nm, whether plasticity is still carried by dislocations or not is a question 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. So 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 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]. Kumar *et al.* [4] 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 facilitate the nucleation of voids at boundaries and triple junctions. However, Hugo *et al.* [9] 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, Liao *et al.* [10] found that, in the deformation of nanocrystalline Al, a full dislocation often dissociates into two partial dislocations enclosing a stacking fault (SF) ribbon. They also studied the grain-size effect on deformation twinning

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in nanocrystalline copper [11], and 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. There, they showed that twinning becomes a major deformation mechanism in nanocrystalline copper during high-pressure torsion under a very slow strain rate and at room temperature. Budrovic *et al.* [12] 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, indicating the deformation process does not build up a residual dislocation network and the absence of substantial work hardening in nanocrystalline metals. By using computer simulating, Gutkin *et al.* [13,14] have suggested that the deformation of nanocrystalline metal is by the cooperative action of grain boundary sliding and rotational deformation. Yamakov *et al.* [15] demonstrated that when the grain size is less than the splitting distance, only partial dislocations can nucleate on the grain boundaries, it means that when the single partial dislocations glide through the grain, stacking faults (SFs) are produced, until these single partial dislocations become incorporated into the grain boundaries on the opposite side. These SFs remain in the grains as planar defects. The grain boundaries in the nanocrystalline regime can promote sliding and act as both the source and sink for lattice dislocations that extend throughout the entire grain, leaving behind a stacking fault defect [16-19]. The molecule dynamics simulation about nanocrystalline Al has shown that dislocation–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 [13,20]. Zhu *et al.* [21] presented an analytical model, based on classical dislocation theory, to explain the nucleation and growth of the deformation twins in nanocrystalline Al. Their model suggests that the stress for twin growth is much smaller than that for its nucleation.

However, in some specific experiments, such as ball-milled Cu-Zr with a grain size under 100 nm, dislocations were not found [22]. On the other hand, considering the geometry difference between foil and bulk samples, the authors of [23,24] thought that the results of [4,9] did not indicate that deformation was governed by extended partial dislocations emitted from GBs. Enough and obvious experiment observations, which supports that partial or full dislocation emitted and absorbed by grain boundaries, are still lacking. In this paper the microstructure characteristic of the cold-rolled deformed nanocrystalline Nickel metal has been studied by transmission electron microscopy (TEM) and the results have been discussed.

2. MATERIALS AND EXPERIMENTAL METHODS

The nanocrystalline sheet Nickel investigated were 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 samples were characterized by Riguta D/max–RC X-ray analysis (Cu target: 40 kV/200 mA, Scan: 20.0/70.0/0.2/0.2 sec.). The microstructure of the Nanocrystalline metals has been investigated in the transmission electron microscopy (TEM). The TEM is Philips Tecnai-2000.

3. RESULTS AND DISCUSSION

The observation results of TME show that the shape of nanocrystalline metal has not changed after rolled deformation, however, the size of grains bas grown up. The average size of nanocrystalline deformed were between 50-70 nm. In addition, some contrasts of strips can be seen in some grains or area, and these contrasts have been proved being Morie stripe. Because the size of the grain is too small, the electron beam penetrated area in TEM may be not a single grain but an area in which factually are many grains overlapped together. Therefore the Morie stripe bended results from the interference of diffraction [25]. On the other hand, though dislocations piling-up structure were not found, a few of dislocation have been found in some particular areas. Further more, step structure has been found at and near the grain boundaries as shown in Fig 1. From Fig. 1 it can be also seen that there was contrast of stress field in front of the step corresponding to the step in the shape. Fig. 2 shows the steps at grain boundaries in another area. These results in Fig. 1 and Fig. 2 indicate that, with the growing up of grain size in the later stage of the deformation, the dislocations can nucleate at the GB [13-15], and considering the character of nanocrystaline grain boundary [16-19], it seems that GB emits partial dislocations promoting stack fault, realizing the process of deformation. Muller and Solenthaler [26] discussed how dislocations interact with twins. They figured out that a series of Shockley partial

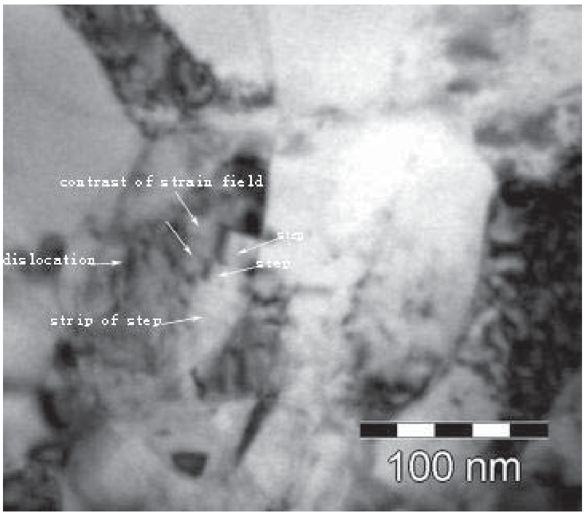

Fig. 1. The step in the microstructure of cold-rolled nanocrystalline Nickel.

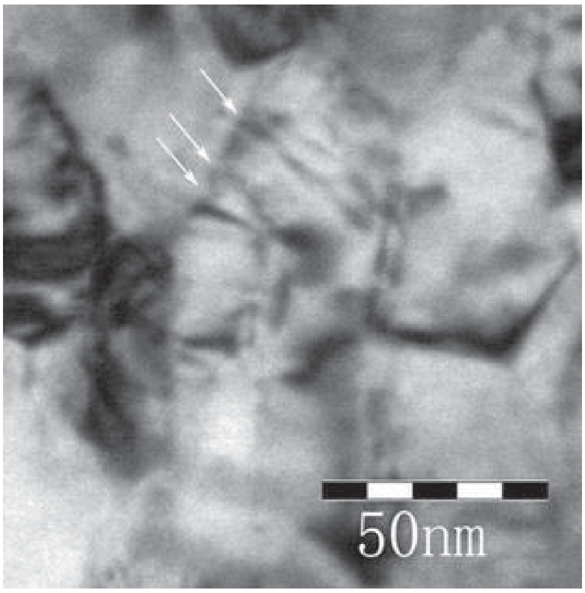

Fig. 2. The step in the microstructure in another area.

dislocations incident on a twin boundary can lead to complete untwining and cutting off of the twin, by sequential untwinning of one atomic layer at a time. This process of untwining results from the dislocation bombardments from a nearby GB. A series of partial dislocations nucleated from the GB leave several traces of extrinsic SFs on their way down to the twin, continuously narrowing the twin by untwinning one atomic layer at a time. The result of the computer simulation for nanocrystaline fcc metal [15] indicated that, when GBs become very active as dislocation sources, the size of the twins would become narrower after the interaction between the imperfection dislocations and the twins. For the bigger size [70-100 nm] of nanocrystaline metal, a more complicated reticulate structure of the twin shall occur.

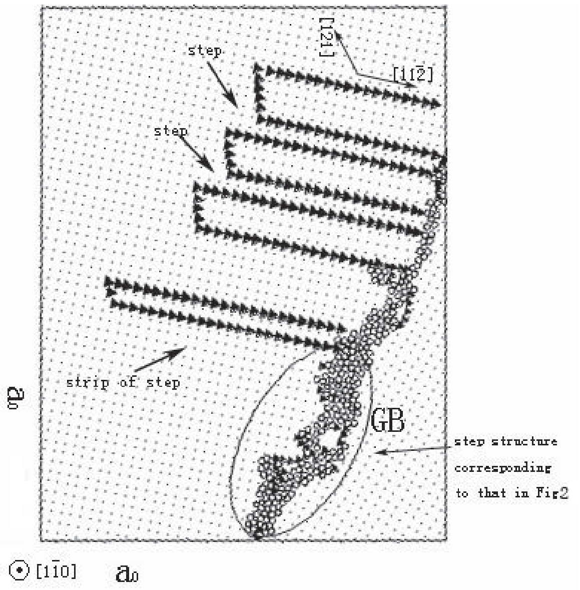

Fig. 3. Partial dislocations nucleate on the grain boundaries producing stacking faults, and stacking faults moved by the partial dislocations gliding (corresponding to Fig. 1 and Fig. 2, the (111) plane and atoms are marked according to [16]).

Fig. 1 indicates that GB emits dislocations, therefore promoting the nucleation of SF, and a few stack faults constitute the steps structure. However, there is no twin in the grains, whereas the contrast of stress field is found ahead of the steps field. It shows that in this experiment the interaction between twins and dislocations [13-15,26] is not a necessary condition to realizing the deformation, and it is very possible that, in the later stage of the deformation when the grain size became to about 100 nm, the deformation occurs only depend upon the moving of the boundary of the SFs which result from the imperfection dislocations emitted from GBs. In the other word, the movement of the boundary dislocations of SFs results to the size growing-up of the SFs, therefore realize deformation. 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 reaches a critical value which stopping the gliding of the partial dislocations, the SFs will stop growing up and leave a step structure behind, as it be seen in Fig. 1 and Fig. 3.

4. CONCLUSIONS

In this paper the microstructure characteristic of the cold-rolled deformed nanocrystalline Nickel metal has been studied by transmission electron microscopy (TEM). The results show that there were step structures near by grain boundary (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. When the grain size became to about 100 nm, the deformation occurs only depend upon the moving of the boundary of the stack faults (SFs) which result from the imperfection dislocations emitted from GBs, in the later stage of the deformation. In the other word, the movement of the boundary dislocations of SFs results to the size growing-up of the SFs, therefore realizes deformation. 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.

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