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The microstructure and tensile properties of pure Ni single crystal irradiated with high energy protons

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Abstract

The microstructure and tensile behavior of pure single crystalline Ni irradiated with 590 MeV protons are investigated. First results have been obtained for a damage level of 0.13 dpa at room temperature and 0.002 dpa at 523 K. The irradiation induced defect microstructure observed under transmission electron microscopy consists of loops and stacking fault tetrahedra in a lower density. Tensile tests were performed at room temperature and show observable radiation induced hardening already at 0.002 dpa. Dislocation channels and cells were observed and the transition from the initial channel formation to the development of the deformation cells is investigated. First results are presented here.

1. Introduction

Ni, Cu, Au and Pd are recognized as typical face-centered cubic metals for radiation effect studies, with convenient variations of relevant parameters such as the stacking fault energy. Both the defect and deformation structures of Cu and Pd are now well documented [1,2]. In particular dislocation channeling has been shown to be one of the main initial deformation modes in fcc materials, with characteristic channel width and separation. The transition from the initial channel dominated deformation to the formation of dislocation cells which are generally observed at the end of the plastic regime is not yet fully understood.

Previous observations on Ni after irradiation with neutrons or energetic ions [3–5] at temperatures up to 503 K have shown that the defect density accumulates at a lower rate in this metal as compared to either Cu or Pd at a similar homologous temperature. Zinkle and Snead [5] have reported a transition from a stacking fault tetrahedra (SFT)-dominated microstructure at low doses to an interstitial dislocation loop one at higher doses at 503 K, with a threshold dose between 0.01 and 0.1 dpa.

In the present investigation, a part of an ongoing program investigating the relationship between the tensile properties and the microstructure developed in irradiated metals, Ni single crystals were irradiated with high energy protons and the details of the resulting microstructure is quantified. Observations were also performed after tensile testing at room temperature and information regarding both the dislocation channels and the dislocation cell structure is sought after. First results are presented here, for an irradiation conducted to 0.002 dpa at 523 K and to 0.13 dpa at room temperature.

2. Experimental procedure

Single crystal rods of pure 99.999% nickel of 12 mm in diameter were provided by Goodfellow Cambridge Ltd. Plates 350 μm thick with the desired orientation were cut by spark erosion. From there tensile specimens of 2.5 mm gauge width and 5.5 mm gauge length were cut from the plates by spark wire saw. The tensile orientation was close to (0 1 1), 5° towards (0 0 1) and 3° towards (−1 1 1). After mechanical polishing of the surfaces, the resulting thickness of specimen was about 300 μm, which was further reduced to about 250 μm to

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remove the work-hardened layer by electro-chemical polishing. All specimens were annealed at 1273 K for 2 h followed by a slow cooling to room temperature in high vacuum.

The tensile specimens were irradiated in the PIREX facility [6] installed in the 590 MeV proton beam of the accelerator located at the Paul Scherrer Institut in Switzerland. The typical dose rate is 5×10^{-7} dpa/s. The doses obtained were 0.13 dpa at room temperature (320 K) and 0.002 dpa at 523 K.

The tensile tests were executed at room temperature in air on a computer controlled micro tensile machine allowing a maximum load of 500 N. All tests were performed at a speed of 0.3 μm/s, corresponding to a strain rate of about 5×10^{-5} s^{-1}.

Transmission electron microscopy (TEM) specimens of the deformed unirradiated specimen and of the deformed irradiated ones were prepared. In order to observe undeformed microstructure and due to a lack of specimens at this stage of the study, some deformed irradiated specimens were cut in regions outside of gage length. This should ensure that the observations are conducted in an undeformed region of the irradiated specimens. In the high dose specimen it is assumed that in regions where no channels are visible the sample is not strained. In the low dose specimen this cannot be anymore considered, as channels were not evidenced, and information on the irradiation induced microstructure is taken from regions were the dislocation density is the lowest.

As Ni is magnetic, the volume of the TEM specimen was reduced by the following technique. First, discs were punched with a diameter of 1 mm and then concentrically glued into a stainless steel ring with an outer diameter of 3 mm. The composite specimens were first mechanically polished and then jet-electro-polished at 0 °C in a solution of 12% H_2SO_4 in methanol with 15 V. TEM examinations were performed in a JEOL 2010 operated at 200 kV.

3. Results

3.1. Irradiation induced defect microstructure

Fig. 1 shows weak beam micrographs of the general microstructure of nickel following irradiation to 0.002 dpa at 523 K and 0.13 dpa at 320 K after tensile test in a region outside of gage length. At 0.13 dpa (Fig. 1(b)) a high density of dislocation loops and SFTs can be observed. Radiation induced defects are difficult to detail because of their small size and especially because of the oxide present at the surfaces. The measured cluster density was 2.1×10^{23} m^{-3} after 0.13 dpa at 320 K and 7.8×10^{21} m^{-3} after 0.002 dpa at 523 K. About 39% of the defect clusters are resolved as SFT in the 0.13 dpa specimen irradiated at room temperature, while a lower fraction of SFT, about 13%, was found in the 0.002 dpa specimen irradiated at 523 K. The majority of the defects observed in both cases were dislocation loops. The mean size of loops was 2.5 nm at 0.13 dpa and 320 K and 5.4 nm at 0.002 dpa at 523 K while the mean sizes of SFTs were, respectively, 1.6 and 3.8 nm.

3.2. Tensile testing and microstructure after deformation

It has already been shown that the type of tensile small specimen used here reproduces well the bulk tensile properties [7]. Fig. 2 shows the resulting curves after

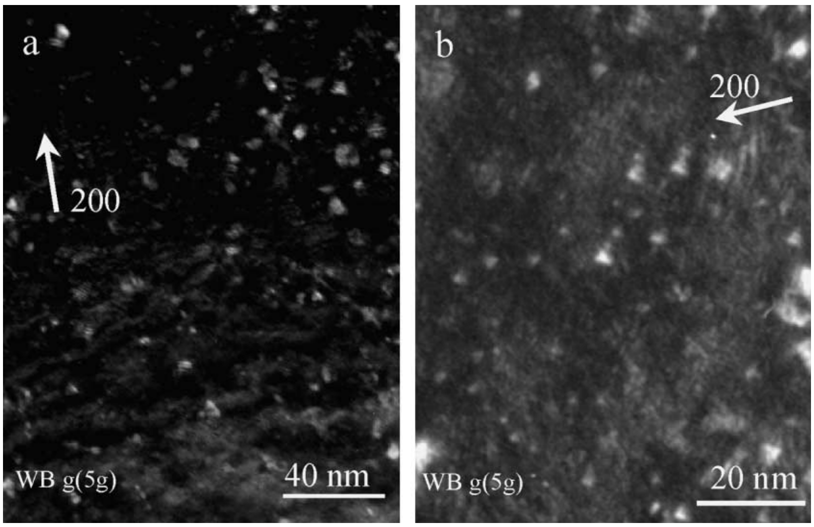

Fig. 1. TEM micrographs showing the morphology of irradiation induced defects at (a) 0.002 dpa at 530 K and (b) 0.13 dpa at RT.

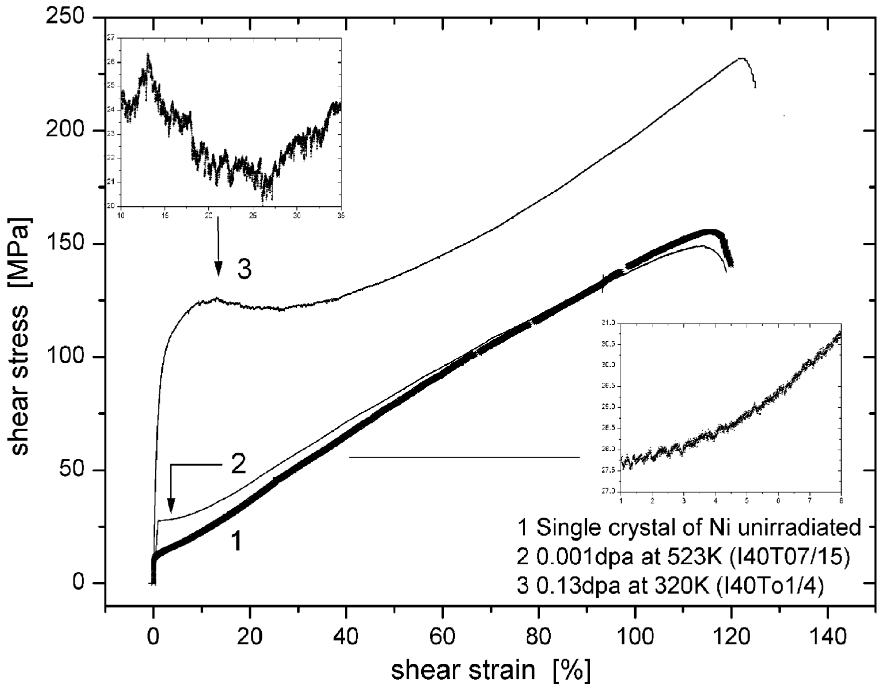

Fig. 2. Room temperature shear stress–shear strain curves obtained at room temperature for single crystal of unirradiated Ni and specimens irradiated at 320 K to 0.13 dpa and at 523 K to 0.002 dpa.

Table 1
Results of tensile test of Ni at room temperature as a function of dose and size of deformation cells as a function of dose

	Unirradiated	0.002 dpa	0.13 dpa
Critical resolved shear stress (MPa)	8	28	100
Work hardening rate measured at 50% strain (MPa)	135	110	90
Length of deformation cells (μm)	1–4	0.6–2	0.6–2
Mean width deformation cells (nm)	700	500	400

tensile test at room temperature for the irradiated and unirradiated specimens. The results are summarized in Table 1. It appears that the critical resolved shear stress (CRSS) increases with increasing dose. The CRSS at 0.13 dpa is almost 12 times higher than the CRSS of unirradiated Ni and, at 0.002 dpa, it is 3 times higher than the unirradiated Ni. The elongation at failure of irradiated specimens is similar to that of the unirradiated Ni. At the highest dose of 0.13 dpa in Ni, there is a yield region showing serrations and no hardening after a marked yield point. The serrations of stress in the yield region increase from a maximum of 0.7 to 3.5 MPa following an increase in dose from 0.002 to 0.13 dpa. The higher dose presents a wide serrated yield region. Moreover, the work hardening rate tends to decrease with dose (Table 1).

The deformation microstructure of the irradiated and unirradiated samples was examined by TEM. For irradiated TEM samples cut in a region outside of gage length, the 0.002 dpa specimen shows a dense dislocations structure forming deformation cells, as presented in Fig. 3(a), while the 0.13 dpa specimen shows defect free channels as presented in Fig. 3(b). Further deformation leads to the development of a deformation cell structure with rectangular shapes, as seen in TEM specimens cut close to the neck region. The morphology of these cells is shown in Fig. 4(a)–(c), discussed more in detail later. The average cell diameter was measured and is given in Table 1.

4. Discussion

4.1. Irradiation induced defects

The dose dependence of defect clusters density in Ni following irradiation at temperatures between room temperature and 523 K including results from [3,5] shows that the defect density increases with dose. The total defect cluster density formed in the 0.13 dpa at 320

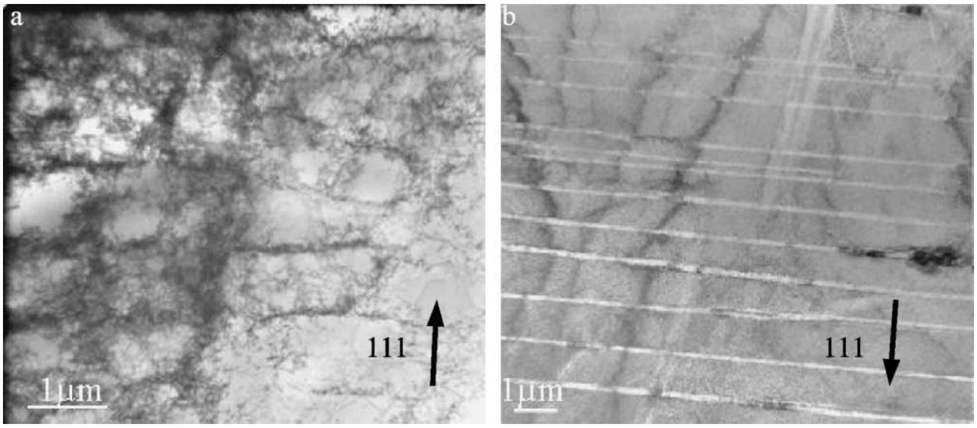

Fig. 3. Deformation cells were in (a) 0.002 dpa at 530 K specimens and deformation channels in (b) 0.13 dpa at RT specimens. Both TEM samples were obtained outside of gage length of deformed specimens.

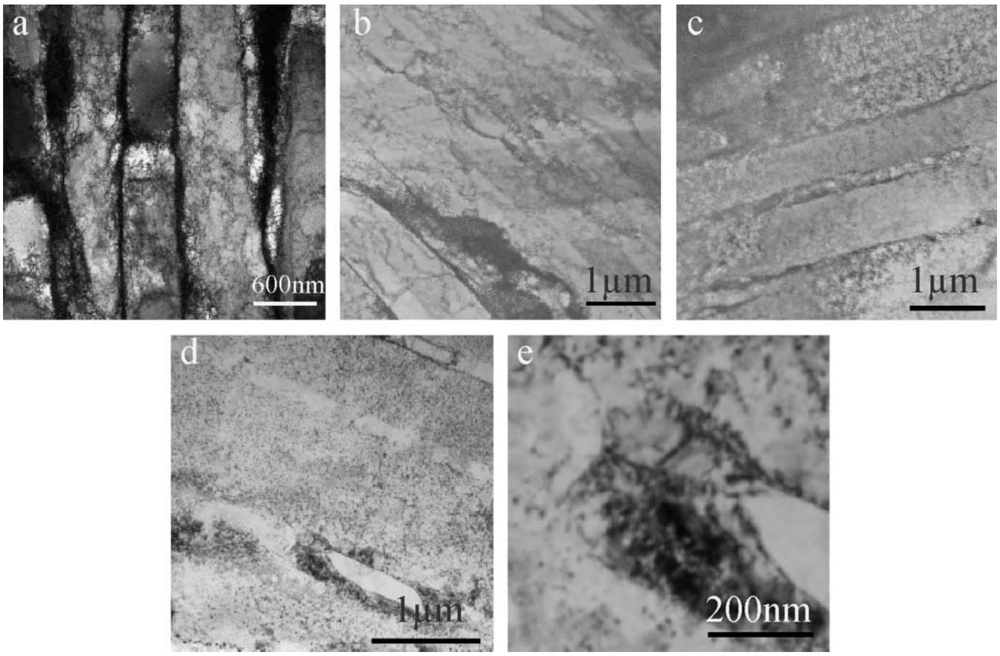

Fig. 4. TEM micrographs showing deformation cells in (a) unirradiated, (b) 0.002 dpa at 530 K and (c) 0.13 dpa at RT samples which were obtained close to fracture region, and in 0.13 dpa specimen showing (d) channels containing blocked dislocations and (e) a detail of it showing a channel seemingly evolving to deformation cell.

K specimen is higher than the results reported earlier in [5] for 0.1 dpa at 503 K, probably because the temperature is lower. It is also a little higher than the value reported for 0.25 dpa at 503 K, which was reported as the saturation value for a density of about 2×10^{23} m^{-3} [5]. The higher defect density at 0.13 dpa relative to that at 0.002 dpa can be attributed partially to the higher dose but also to the lower irradiation temperature. It is interesting to note that the proportion of SFTs in the 0.002 dpa specimen is much lower than that in the 0.13 dpa specimen. Earlier results [5] on Ni irradiated by neutrons showed, that the SFTs should be more than 90% of the total defect clusters at low doses but again comparison should be taken with care as in that case irradiation temperature was 503 K. The temperature difference is not large, but it is very close to the temperature of stage III (490 K [8]) when vacancies start to move. The fraction of SFTs formed in the present work at 0.13 dpa is 39%, comparable to the 33% reported [5] for 0.1 dpa.

The density of visible defects in single crystal Ni below 523 K is almost five times to one order of magnitude lower than that in copper, as well as Pd and Au, at these two damage levels. It means that when the defects density of copper reaches a saturation value of about 1×10^{24} m^{-3} the defects density of Ni is only about 2.1×10^{23} m^{-3} for a similar damage level. Interestingly, it appears that even if the defect density is much lower in Ni Cu shows a similar hardening for a similar dose of 0.13 dpa. The lower proportion of SFTs formed in Ni relatively to the one formed in Cu can be explained by the higher stacking fault energy, 130 mJ m^{-2} [9], as compared to 73 [10], which could imply that their formation is more difficult. The lower defect density however cannot be related to the stacking fault energy, as Pd, which has similarly a high stacking fault energy (180 mJ m^{-2} [11]), shows a similar defect density as Cu. At this stage of study however it is only a suggestion. Other parameters, such as irradiation temperature, dose, flux, have also to be considered to account for the differences [12].

4.2. Mechanical properties and deformation microstructure

Note that the amplitude of serrations just after the yield point increases from 0.7 to 3.5 MPa when the dose increases from 0.002 to 0.13 dpa (Fig. 2). This can be reasonably explained by the fact that the higher the defect density, the greater the magnitude of the stress drops, as the dislocations are pinned by a higher density of defects. Indeed the dislocations would be released from a higher stress level to initiate channels, events that trigger stress drops and hence lead to the serrations.

As shown in Figs. 4(d) and 3(b), the defects free channels are about 100 nm in mean width and with a mean separation distance of about 1 μm. Channels have the orientation of slip planes that in the FCC structure are the planes $\{1\,1\,1\}$. As shown in Fig. 3(b), Fig. 4(d) and (e) the dislocation channels are straight, and in some cases, present localized dark contrast, which arises from a tangle of dislocations. Dislocations in the channels can be divided into two groups [2]: first, mobile dislocations in the channels, and second, dislocations pinned by defects at the edges of channel. The crowded nodes observed in the present study can be considered as dislocations strongly pinned at the channels' edges. This may be important in explaining as to how the deformation structure evolves.

Table 1 shows relative slopes and stress levels of these strain curves. In unirradiated Ni, the curve shows three stages of single crystal deformation. At a shear strain of about 70%, for the unirradiated and 0.002 dpa cases, the deformation changes from stage II to stage III. At this transition the slope of the tensile curve decreases. It means that at this stress, of about 100 MPa, the dynamical recovery will reduce the hardening rate. But for 0.13 dpa, the upper-yield point is 123 MPa, which is above this recovery stress level. Plastic deformation begins at a level of stress higher than that of stage III of the unirradiated Ni. Deformation cell sizes were measured and are reported in Table 1. Because cells have rectangular shape, measurements of mean width and length are made. Results show that the mean width and length in the 0.13 dpa specimen of Ni are smaller than that in the unirradiated specimens. For a similar length, the mean width in 0.13 dpa Ni is smaller than that in the 0.002 dpa Ni specimen. Note that the above mentioned higher upper-yield point relatively to the irradiated Ni for the 0.13 dpa sample correlates well with the fact that deformation cells are smaller. Our observations suggest that channels could be the origin of the deformation cells' morphology as explained in the following. Early deformation leads to the formation of channels, which are the only vector of plasticity at this stage. When deformation is increased, dislocation in the channels experiences more and more pinning events by dislocation–dislocation interaction (Fig. 4(d)). Our observations suggest that the channel widths remain constant, at about 100 nm. When channels are blocked (Fig. 4(e)) they would trigger the formation of new channels instead of increasing in width. As deformation proceeds, deformation cells develop. They would develop in the channels as they are the only locations where dislocation can move. Deformation cells size could increase with further deformation. When the volume is filled with them, their size would remain smaller as measured experimentally (Table 1) than in the unirradiated case because their density, inherently related to channel width, is higher from the beginning of their formation. Work is continued to repeat observations of radiation induced defects in undeformed irradiated specimens. Also, observations will be made on specimens irradiated at the same temperature in order to investigate the dose dependence, as well as the temperature dependence for a given dose.

5. Conclusions

This preliminary work shows that irradiated pure single crystal of Ni exhibits a lower defect clusters density than other FCC crystals, such as Cu, Pd and Au, at comparable doses and comparable temperatures. Compared with previous data, SFTs proportion in 0.002 dpa specimen at 523 K is much lower than what is found in the literature. It may be attributed to the slightly higher irradiation temperature.

While strain hardening rate is similar to other FCC crystals, the CRSS change of Ni at 0.13 dpa is the same as that for Cu.

It appears that in the 0.13 dpa specimens dislocation channeling is the initial deformation mode.

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