Crystallographic characterization of catastrophic shear in submicron nickel at low temperatures

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Abstract. Nickel produced by additive-free pulsed electrodeposition with grain size of about 150 nm was deformed in tension at 320 K and 4 K. At both temperatures the specimens exhibited strong parabolic hardening and good ductility. However, at 4 K a sudden transition from homogeneous deformation to “repeated catastrophic shear” took place. Microstructural characterization of the sheared region showed deformed grains elongated in between tensile axis and growth direction. The texture information from the sheared region was obtained using conical dark field measurements in the transmission electron microscope. The pole figures of the deformed 4 K specimen show a typical shear texture which clearly differs from the starting texture.

1. Introduction
Pure metals with grain sizes between the nanocrystalline (<100 nm) and microcrystalline (>1 μm) state are very promising objects for investigations of the transition from “conventional” deformation behavior to “nano”-behavior. To study this temperature dependent transition, additive-free pulsed electrodeposited (PED) nickel was chosen as model material for face-centred cubic metals [1]. Additive-free PED nickel was pure, fully dense and contained a large fraction of high angle grain boundaries with a narrow grain size distribution.

It was the aim of the present work to extend investigations of grain size induced changes of deformation mechanism already performed on pure polycrystalline nickel with large grain size [2].

2. Experimental
A rectangular plate of PED nickel (70 × 40 × 2 mm³) was produced using a sulfamate bath without additives (e.g. saccharin). Two cylindrical tensile specimens (“dog bone” shaped, gauge length 10 mm and diameter 0.8 mm) were cut from the PED plate by spark-erosion with the tensile axis (TA) aligned perpendicular to the growth direction (GD). Subsequently, the specimens were electropolished to remove the disturbed surface layer. Tensile deformation was carried out in an open continuous flow cryostat (He coolant) permitting to keep the temperature constant within less than 0.05 K [3].
cryostat was mounted in an Instron 4502 load frame. The experiments were performed at constant crosshead displacement rate providing an initial strain rate of $4 \times 10^{-4}$ s$^{-1}$. Electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM) were performed in a scanning electron microscope Zeiss Ultra 55 and a Technai, respectively. TEM foils were cut by focused ion beam (FIB). Conical dark field measurements in the TEM after processing through ACT software yield orientation imaging maps and related pole figures. The pole figures were obtained using LABOTEX software.

3. Results and Discussion

The initial microstructure and texture of the additive-free PED nickel plate is given in figure 1. Few grains are elongated along GD. The average grain size obtained from the EBSD scan is 150 nm. There is a weak $<110>$ fiber texture along GD.

![EBSD map of the initial microstructure, acquisition step size 40 nm, (b) $\{110\}$ and $\{111\}$ pole figures from (a)](image)

Figure 1. (a) EBSD map of the initial microstructure, acquisition step size 40 nm, (b) $\{110\}$ and $\{111\}$ pole figures from (a)

Figure 2 shows the engineering stress (force with respect to the initial cross section) vs. crosshead displacement for the specimens deformed at 320 K and 4 K. The two stress drops at 320 K are the results of stress relaxation tests performed in order to study the deformation mechanisms [2]. Apart from that, the 320 K curve shows homogeneous continuous deformation, but at a rather high stress level of more than 1500 MPa at the end of deformation. Reports in the literature (e.g. [4, 5]) claim (X-ray) grain sizes between 20 nm and 50 nm for such stress levels in PED nickel. Initially, the curve at 4 K behaved similar to that at 320 K, but at 2400 MPa the deformation mode suddenly changed towards repeated shear events. After a first stress drop of 16.9% the deformation continued elastically before a second stress drop of the same order appeared. Also the second event was not fatal for the specimen. Again the deformation continued elastically, but then the specimen failed. The shear events were accompanied by loud acoustic emission.

Similar shear events are reported by Niewczas et al. [6, 7] on copper single crystals deformed in tension at 4.2 K, and are attributed to “adiabatic deformation” (see e.g. [8]). In this case, a dramatic load drop of 16.5 % marks the sudden transition from homogeneous slip on the primary and conjugate slip systems at the beginning of deformation towards twinning alone. Niewczas et al. [6, 7] argue that the onset of twinning acts as trigger for the adiabatic deformation in the way that after a substantial overshoot of the crystal axis the resolved stress in the twinning system reaches the stress necessary to
nucleate twins. It is interesting to note that the relative depth of the load drops in copper single crystals is almost the same as in PED nickel polycrystals.

![Figure 2](image)

**Figure 2.** Engineering stress vs. crosshead displacement at 320 K and 4 K (stress relaxation tests are marked by SR)

After dismounting the 4 K tensile tested specimen no distinct shear band, but a necked region of about 200 µm extension in tensile direction around the crack was recognized which probably represents the sheared region. For microstructural observations of the sheared region, a thin foil cut through FIB parallel to GD was studied by TEM (figure 3). Elongated grains are directed in between TA and GD. While initially the grains were almost equiaxed (figure 1a) this indicates deformation-induced grain shape changes. Obviously, during the shear event localized deformation took place also clearly changing the texture. As figure 4 shows, the texture evolved towards a typical shear texture produced by shear on \{111\}<110> slip systems [9].

![Figure 3](image)

**Figure 3.** (a) Bright field TEM micrograph of the sheared region of the 4 K specimen after fracture, (b) orientation map of part of the region shown in (a), color code with respect to vertical upward direction (red colored), black dots not indexed
Figure 4. (a) Schematic representation of an ideal {111} pole figure for simple shear along Xs on the plane normal to Ys, (b) experimental {110} and {111} pole figures related to figure 3(b)

Interestingly, the pole figures of catastrophic shear at cryogenic temperature are comparable with those obtained for cyclic deformation at room temperature [10]. This indicates that the localized shear observed most probably is carried by dislocations. So far, no sign is found about the nature of the triggering mechanism for catastrophic shear.

4. Conclusions
During uniaxial tension at 4 K, additive-free pulsed electrodeposited nickel containing nearly equiaxed grains of 150 nm in size at an engineering stress of 2.4 GPa changed from homogeneous deformation to localized catastrophic shear. TEM of the sheared region reveals substantial elongation of the grains in between TA and GD. Pole figures show a typical shear texture produced by slip on {111}<110> systems. Thus, localized shear deformation of sub-microcrystalline nickel at low temperatures is carried by dislocation slip. The trigger mechanism for the onset of strain localization is probably related to twinning.

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