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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

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Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

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Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
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Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** ***Deformation-induced change of grain structure and internal strains in micro-, submicro- and nanocrystalline nickel***	**Experiment number**: HS- 1774
Beamline: BM 20	**Date of experiment**: from: 04. 04. 2002 to: 08. 04. 2002	**Date of report**: 11. 11. 2002
Shifts: 15	**Local contact(s)**: Dr. Norbert Schell	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Ellen Thiele*, Robert Klemm*, Lutz Hollang*

Institute of Physical Metallurgy, Technical University Dresden

D – 01062 Dresden
Germany

Report:

Motivation. The aim of the experiment was to examine the influence of cyclic plastic deformation on the change of grain structure and internal strains and stresses in Ni specimens, which exhibit very different mean grain sizes D and defect structures in their initial stages. Six different types of samples were investigated: on the one side fine- (fc) and micro-crystalline (mc) Ni with $D \approx 35\mu m$ and $3\mu m$, respectively and a nearly stress-free initial stage and, on the other side samples produced by equichannel angular pressing at room-temperature (RT-ECAP, $D \approx 0.5\mu m$) and at 250°C (ET-ECAP, $D \approx 0.8\mu m$), by electrodeposition (PED), and by hot-compaction of ball-milled Ni powder (CBM), all of them showing a certain amount of internal stresses already in the as-produced stage. All these samples had been cyclically deformed at constant plastic strain amplitude ε_{pa} at room temperature.

Experimental. Using a wavelength $\lambda = 0.1534nm$ BRAGG-diffraction profiles of seven different {*hkl*}-reflections were measured with a position sensitive detector (3380 channels for 8.28° in 2ϑ) from the {111}- to {331}-type. In order to ensure that a sufficient number of crystallites is in Bragg-position also in the case of fc and mc Ni, the samples were rotated round the surface normal which was parallel to the diffraction vectors g. The evaluation procedures given by Williamson-Hall (WH) [1], Warren-Averbach (WA) [2] and Krivoglaz-Wilkens (KW) (cf. [3]) were used to analyse the BRAGG-profiles with a negligible instrumental broadening.

Results and discussion. It was found that for the fc, mc and CBM samples the size d of coherently scattering regions decreases due to cyclic deformation whereas for all other samples a maximum value d occurs at $\varepsilon_{pa} \approx 2x10^{-3}$ (cf. Fig. 1a, where d_{WA} was determined by the WA-analysis). In all cases, the size D of grains with high angle grain boundaries is at least ten times higher than d. Results of transmission electron microscopy suggest that d could be correlated with the thickness of regions with high dislocation density forming fatigue-induced dislocation patterns.

Taking the root mean square stress $<\sigma^2>^{1/2}$ calculated by a WH-analysis (cf. Fig. 1b) as a global measure for internal stresses of different range, than for the fc, mc and CBM Ni samples an increase of internal stresses was observed at small deformation amplitudes. In contrast, for the RT-ECAP, ET-ECAP and PED Ni samples the initial internal stresses are decreased at first. It seems, that there should be a common value of $<\sigma^2>^{1/2} \approx 50MPa$ for all types of samples at deformation amplitudes $\varepsilon_{pa} \geq 2x10^{-3}$ independently from the grain size of the samples. This means that at sufficient large ε_{pa} the cyclic plastic deformation is able to compensate

the influence of the different prehistory of these samples on $<\sigma^2>^{1/2}$. A similar behaviour was observed also for the root mean square strains $<\varepsilon_L^2>^{1/2}$ determined by the WA-analysis where L is the Fourier-length .
In order to proof the existence and to estimate the density ρ of dislocations a KW-analysis was performed. For that purpose a function ψ was calculated from the Fourier-coefficients A_n of the diffraction profiles with $\Psi = -\frac{\ln A_n}{L^2} = \frac{1}{Ld} + B\left(\ln\frac{R_{eff}}{L}\right)$ with $B = \frac{\pi}{2}(bg)^2 C\rho$, where R_{eff} is an outer cut-off radius for the stress field of the dislocation arrangement, b is the burgers vector and C is a mean contrast factor for the dislocations. From a fit of the ψ-function it follows that dislocations exist after cyclic plastic deformation in all kinds of samples, so that dislocations should assumed to be responsible for the elementary deformation processes even in grain structures with $D \approx 0.5\mu m$. For the RT-ECAP, ET-ECAP and PED Ni samples the high initial dislocation density is reduced by the fatigue, whereas in fc and mc Ni samples the dislocation density increases with increasing ε_{pa} (cf. Fig. 1c).
Considering the shape parameter *IB*/*FWHM* of the diffraction profiles (*IB*… integral breadth, *FHWM*… half with) in dependence on ε_{pa} there is a conspicuous agreement of all kinds of samples. Starting from small values in the initial stage a maximum shape parameter can be observed at $\varepsilon_{\text{pa}} \approx 2.5\text{x}10^{-4}$. For higher deformation amplitudes *IB*/*FWHM* seems to saturate at a value of about 1.35. For comparison, the corresponding values for fatigued single crystals are added [4]. From the agreement of the data it can be supposed that the arrangement of dislocations due to the cyclic plastic deformation is very similar independently from the grain size: at small and medium ε_{pa} there is a large volume fraction of thick regions with a high density of dislocation dipoles. The reduction of the shape parameter could be explained by a decrease of thickness and volume fraction of these regions with increasing ε_{pa}.

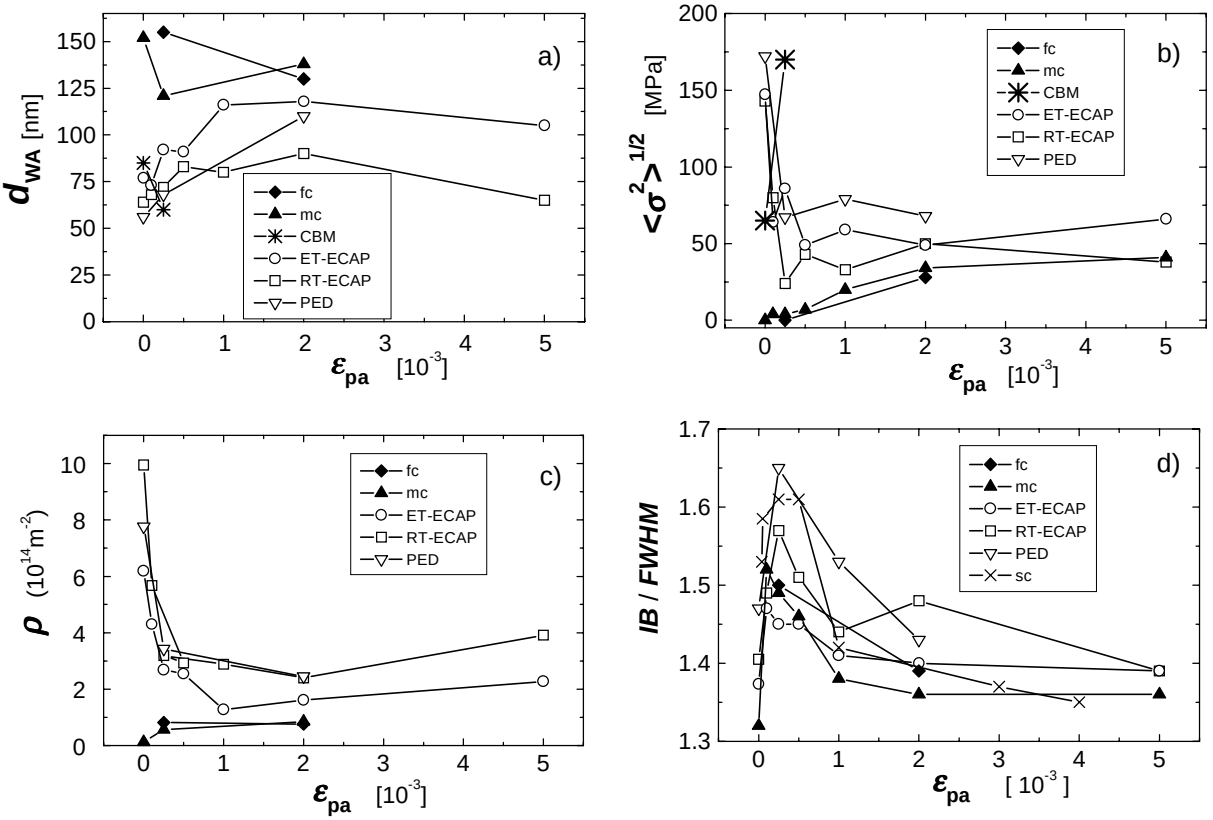

Fig. 1 Dependence of the size d_{WA} of coherently scattering regions (a), of the rms stress $<\sigma^2>^{1/2}$ (b), of the mean dislocation density ρ (c) and of the profile shape parameter *IB*/*FWHM* (d) on the plastic strain amplitude ε_{pa} for the different kinds of samples.

[1] R. Klemm, E. Thiele, C. Holste, J. Eckert, N. Schell: Scripta Mater. **46** (2002) 685
[2] B.E. Warren (1990) *X-Ray Diffraction*, Dover Publications Inc., New York
[3] D. Breuer, P. Klimanek, U. Mühle, U. Martin: Z. Metallkd. **88** (1997) 680
[4] M. Hecker, E. Thiele, C. Holste: Z. Metallkd. **88** (1997) 321