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

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Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

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Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

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:** In situ characterization of pure shear deformation of nanocrystalline Pd and Pd alloys	**Experiment number**: MA 1112
Beamline: ID15A	**Date of experiment**: from: 21 Oct 2010 to: 26 Oct 2010	**Date of report**: 24 Feb 2011
Shifts: 15	**Local contact(s)**: Veijo Honkimäki	*Received at ESRF:*

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Report:

Nanocrystalline (nc) materials with grain sizes well below 100 nm show different deformation behavior as compared to their coarsegrained counterparts. Understanding the underlying plastic deformation mechanisms is essential to make use of the unique properties of these materials. Therefore *in situ* mechanical tests were conducted on so-called miniaturized shear-compression samples (m-SCS) of different nanocrystalline materials, while diffraction patterns were recorded continuously during deformation.

The setup is based on a method established by Bohm *et al.* (*Rev. Sci. Instrum. 75 (4), 1110*) for *in situ* lattice strain and stress measurements during tensile tests of thin metallic films on compliant substrates. The setup and methodology was modified and implemented to the HEMD beamline of the ESRF, see Fig. 1(a)-(b). A mechanical testing device (Kammrath & Weiss) was mounted on the diffractometer, and the incident X-ray beam was oriented perpendicular to the sample surface. Complete Debye Scherrer rings were recorded on a area detector placed in transmission geometry with fast data aquistion (2 patterns per second). The m-SCS samples were loaded in compression with a strain rate of about 10^{-3} s^{-1}. Numerical analysis shows that the dominant deformation mode in the gauge section is shear (Ames *et al.* (*Mater Sci Eng, A 528, 526*). Due to the small dimensions of the spark-eroded slit of the m-SCS samples, a beam size of less than 25x25 μm^2 was nessecary to monitor the deformation behavior in the gauge section. On the other hand, a high energy X-ray beam was required to penetrate through the slit (thickness ≈ 100 μm) and still receive a good signal-to-noise ratio. The characteristics of HEMD together with the availability of an area detector with fast data acquisition makes the beamline perfectly suited for our *in situ* experiments. The macroscopic displacement of the samples was determined by digital image analysis, where the displacement of the two parallel edges of the slit was monitored, see Fig. 1(c). By use of the finite element method equivalent stress – equivalent strain curves were calculated, see Fig. 1(d).

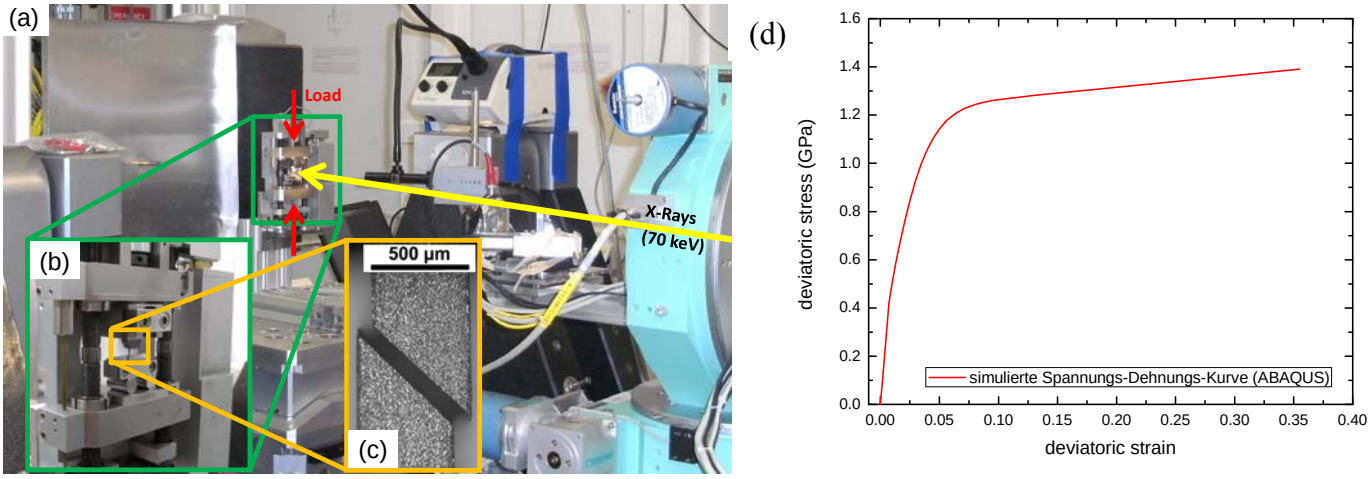

Figure 1: (a) Setup overview at HEMD. (b) Mechanical testing device with skid allowing for lateral sample movement for shear deformation. (c) Micro shear-compression sample with spark-eroded slit. (d) Representative stress-strain curve obtained by FEM analysis.

Different alloys of the binary, continuous miscible Pd-Au system with gold contents ranging from 10 to 75 at% were tested. The samples were fabricated by inert-gas condensation (Birringer *et al.* (*Phys Lett A 102 (8), 365*) and had an initial grain size of 10 nm. In addition, electrodeposited Nickel (Natter *et al.* (*J Mater Res 13 (5), 1186*) with an initial grain size of 20 nm was tested. Samples of both systems were initially texture-free. Peak shape analysis was performed by fitting splitted Pearson VII distributions to the peaks. This function includes a peak shape factor to differentiate between Gaussian and Lorentzian peak shape as well as an asymmetry parameter. In the following the main findings are explained exemplarily for a Pd-70at%Au alloy. In Fig. 2(a) a representative diffraction pattern is shown. The inner 5 rings - from (111) to (222) - are evaluated by radial line scans with an azimuthal angle increment of 2°. This enables to follow the evolution of peak parameters for selective azimuthal directions, as shown in Fig. 2(b) for the (111) peak. The directions parallel ($\varphi = 90°$) and perpendicular ($\varphi = 0°$) to the external loading direction show the maximum elastic compressive and tensile strain, respectively. The directions parallel ($\varphi = 46°$) and perpendicular ($\varphi = 136°$) to the sample slit show the same behavior until 3% strain where the perpendicular direction remains almost strain free, while the parallel direction shows compressive behavior. During initial loading the integral width of the (111) peak is increasing in all φ-directions, where the strongest increase is observed in loading direction, see Fig. 2(c). During macroplastic deformation (characterized by constant elastic strain) the peak width decreases in all directions, while in all directions except of the loading direction the peak width returns almost to its initial value. Plotting the evolution of (111) integral peak intensity normalized by its initial value for all azimuthal directions, a strong intensity redistribution is oberved, indicating the formation of an in-plane texture with 6-fold symmetry during deformation, see Fig. 2(d). Noteworthy is that the texture development can be clearly assigned to the macroplastic regime ($\varepsilon > 9\%$). The formation of texture is observed for all monitored *hkl*-families. Line broadening analysis by use of a simple Single Line Method (de Keijser *et al.* (*J Appl Crystallogr. 15, 308)* was performed on the integral breadth. By separating the integral peak breadth in Lorentzian and Gaussian contributions, grain size and microstrain can be determined as shown in Fig. 2(e)-(f). Again it can be clearly differentiated between 2 different stages of deformation. During the first, so-called microplastic regime, solely the microstrain evolves while grain size remains constant. In contrast, during the macroplastic regime, the grain size starts to increase, preferably in the directions of large elastic strain, while the microstrain remains constant. Upon unloading the microstrain is almost fully reversible besides the direction parallel to loading.

In summary, different stages of deformation were observed during *in situ* testing of m-SCS samples. The stages can be separated by analysing the peak shape evolution (breadth and intensity) and derived parameters, like grain size and microstrain. In the first regime, microplastic deformation is dominant, where no texture evolves and the peak broadening is caused by an increase in microstrain. The second regime, is represented by an elastic strain plateau indicating macroplastic deformation. During macroplastic deformation an in-plane texture evolves and the grain size increases slightly, while microstrain remains constant. These results indicate that the dominant macroplastic deformation mechanism is grain boundary mediated. The preservation of the equiaxed grain shape and the texture formation suggest grain boundary sliding accompanied by grain rotation as the prevailing macroplastic deformation mechanism. This is supported by the fact the the grain interior (microstrain, constant elastic strain) is not affected during

macroplastic deformation. Experiments on PdAu alloys with lower Au content show that the deformation mechanisms depend on alloy composition, as e.g. Pd-10at%Au samples show almost no texture formation. However, the results are conformable with existing work on nc materials (e.g. Budrovic *et al. (Science 304, 273),* Ivanisenko *et al.(J. Mater Sci 45, 4571))*, but moreover reveal novel and unique aspects of plastic deformation in the nc regime, which in our opinion justify publication in *Physical Review Letters*. The manuscript is under preparation and will be submitted shortly.

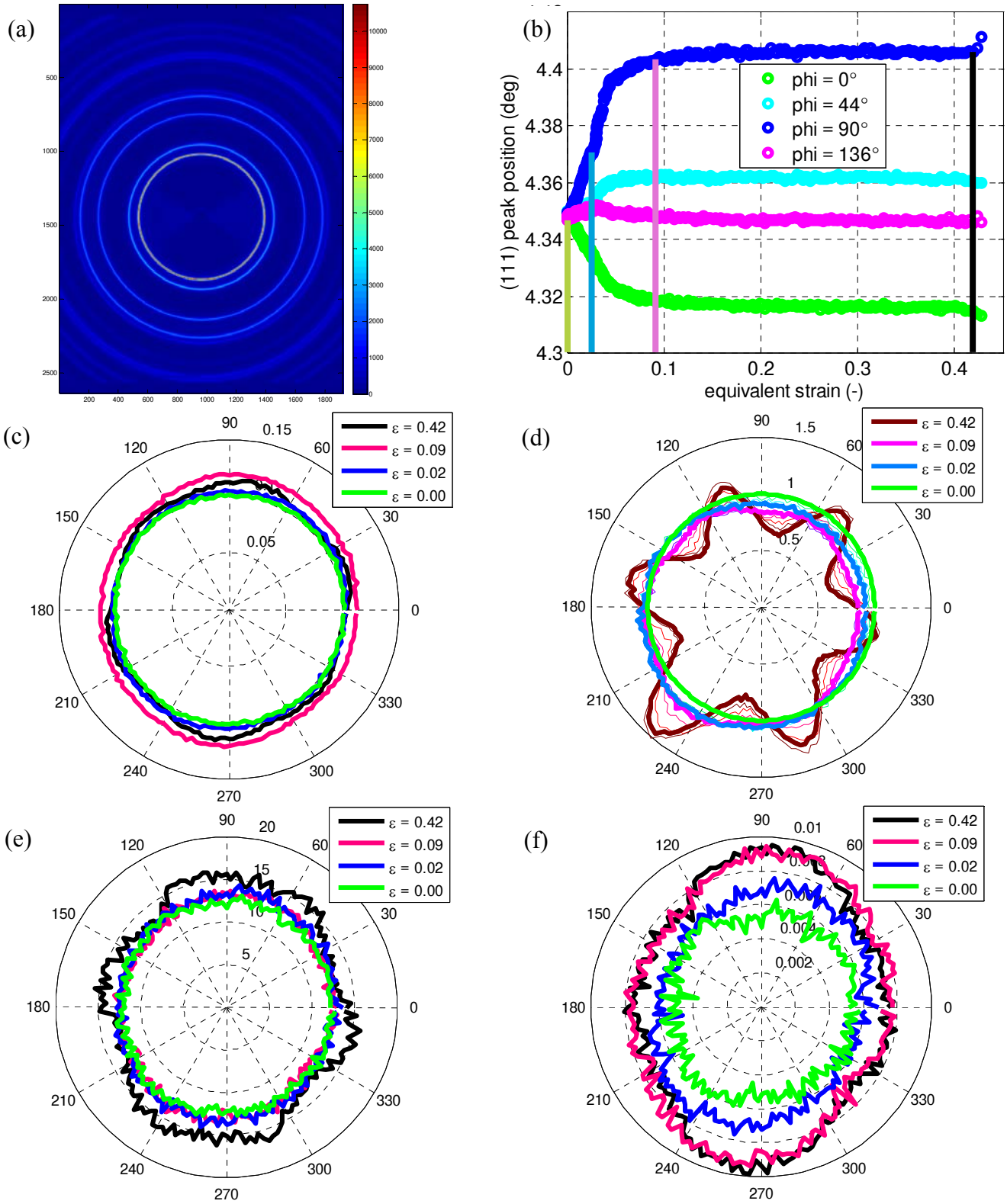

Figure 2: (a) Representative diffraction pattern for a Pd-70at%Au alloy sample. (b) (111) peak position as function of total strain. Different parameters over azimuthal angle φ for different total strains, indicated by vertical lines in (b): (c) (111) integral peak breadth [°] and (d) (111) normalized integrated peak intensity as well as (e) grain size [nm] and (f) microstrain [-] derived by the Single Line Method performed on the integral peak breadth of the (111) peak.