EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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 via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can 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

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.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

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;
- 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 metals and alloys	**Experiment number**: MA 1353
Beamline: ID15A	**Date of experiment**: from: 26.10.2011 to: 01.11.2011	**Date of report**: 31.08.2014
Shifts: 18	**Local contact(s)**: Veijo Honkimäki	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. Patric Gruber (Main Proposer and *), Dipl.-Ing. Jochen Lohmiller*,

Karlsruhe Institute of Technology, Institute for Applied Materials, Herrmann-von-Helmholtz Platz 1, 76344 Eggenstein-Leopoldshafen, Germany

Prof. Dr. Rainer Birringer (Co-Proposer), Dipl.-Phys. Manuel Grewer*, Dipl.-Phys. Christian Braun*,

Universität des Saarlandes, Lehrstuhl für Technische Physik, Campus D2 2, 66041 Saarbrücken, Germany

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. This proposal was a continuation of the proposal MA 1112 where a novel experimental methodology for *in situ* mechanical testing on miniaturized shear-compression samples (SCS) and compression samples of inert-gas condensated PdAu (igc-PdAu, grain size 10-15 nm) and electro-deposited Ni (ed-Ni, grain size 30 nm) has been established successfully at the High Energy Microdiffraction endstation (HEMD) of beamline ID15A. Special features of the experimental setup are the use of a fast large area detector and the transmission geometry with microbeam. Using this technique, different stages of the unique deformation behavior of nanocrystalline materials could be identified and characterized by analysing the evolution of peak shape parameters along complete Debye-Scherrer rings *in situ* during deformation. Microplastic and macroplastic contributions to the overall deformation behavior could be clearly separated and it could be shown that the microplastic deformation is characterized by an increase in microstrain which is fully reversible while macroplastic deformation yields to irreversible texture formation and grain growth. The characterization of reversible and irreversible deformation processes and their allocation to different deformation stages could be performed for the first time for the grain size regime below 30 nm. In the following exemplary results of *in situ* compression and shear compression tests on ed-Ni as well as shear compression tests on igc-Pd90Au10 are reported.

Compression tests on ed-Ni (Lohmiller et al., Acta Materialia 65 (2014) 295–307):
The deformation behavior of bulk nc Ni (D = 30 nm), investigated by synchrotron-based in situ compression testing and sophisticated peak-shape analysis in combination with complementary ACOM-TEM analysis, can be summarized as follows:

- Not only could the coexistence of dislocation plasticity, grain growth and interfacial deformation modes be demonstrated, but it was also possible to assign them to distinctive strain regimes and estimate their relative contributions to overall deformation.
- The mechanical behavior of electro-deposited Ni is essentially unaffected by the orientation of initially elongated grains with respect to the loading direction, but the subgrain structure seems to be the determining microstructural parameter.
- We have shown that the deformation behavior of nc Ni entails a sequence of different deformation mechanisms (Fig. 1): a crossover from elastic and GB-mediated accommodation processes to the coexistence of GB shear and slip as well as dislocation glide and finally stress-driven grain growth at large strains. Based on the excellent resolution and statistics of the experimental approach, the sequence as well as the individual shares of deformation modes could be discriminated.
- For electro-deposited nc Ni we estimated the relative contributions to the overall deformation as 40% intragranular dislocation plasticity, 15% grain boundary migration and 45% GB-mediated deformation.
- However, it is expected that the onset, sequence and proportion of individual deformation mechanisms may vary with grain size and impurity content. The combination of in situ XRD and ACOM-TEM seems to be the method of choice to gain deeper insight into the deformation behavior of nc metals by systematically varying these parameters.

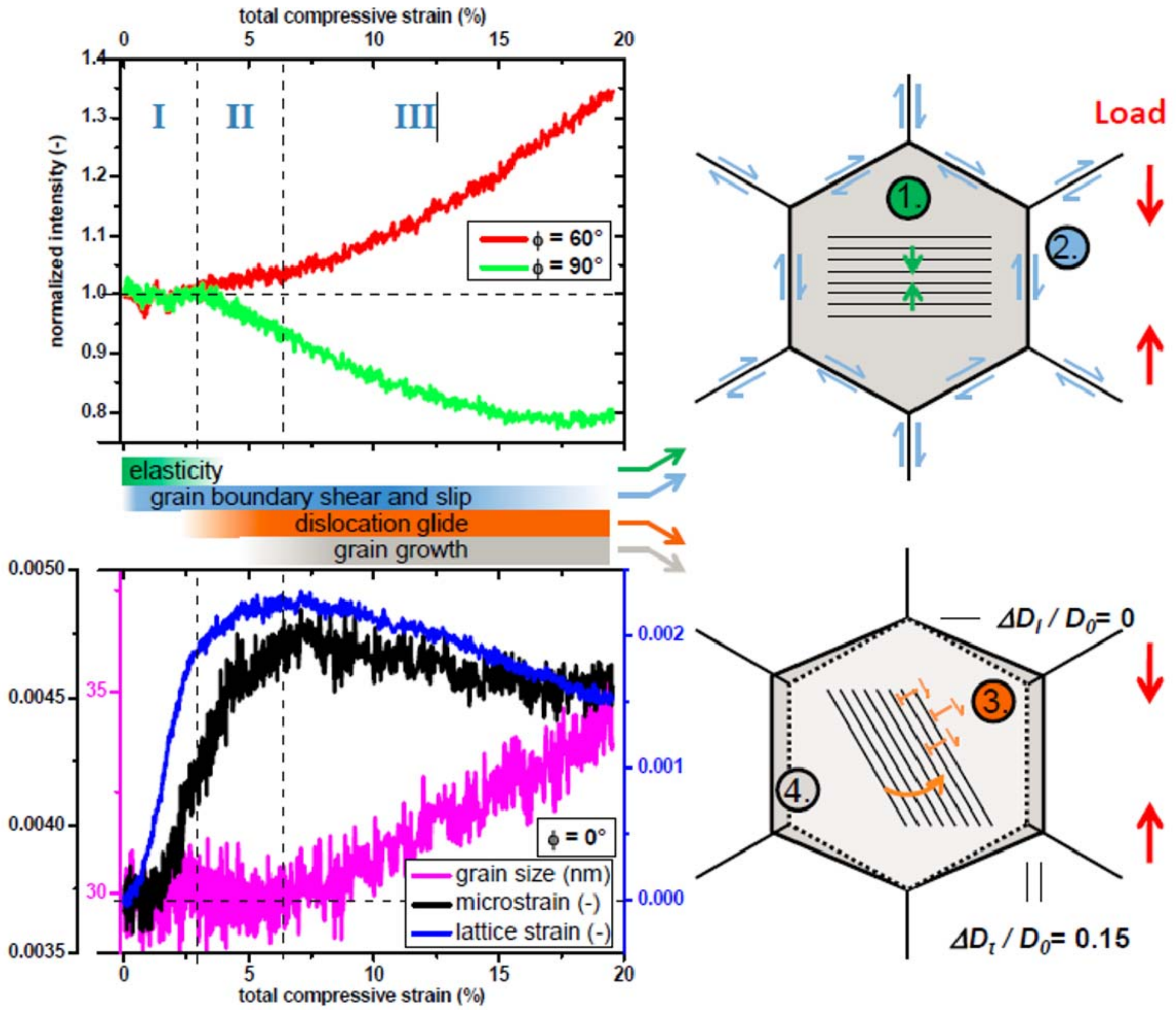

Figure 1: Compilation of results for in situ compression tests on ed-Ni. The most relevant XRD peak parameters are shown as a function of total compressive strain together with a schematic of the identified microscopic deformation mechanisms. An explicit succession of different deformation modes can be derived: (I) inhomogeneous elastic lattice straining and GB accommodation; (II) upcoming dislocation plasticity, inferred from texture evolution; and (III) onset of stress-driven GB migration.

Shear compression tests on ed-Ni (Lohmiller et al., under Review by International Journal of Plasticity):

The effect of shear-dominated deformation on the mechanical behavior of nanocrystalline Ni has been investigated using miniaturized shear-compression testing in combination with synchrotron-based *in situ* XRD. A succession of different deformation mechanisms during loading could be derived by the analysis of the evolution of XRD peak parameters: Lattice elasticity accompanied by accommodation processes in/near GBs and triple lines is dominant during the first regime ($\varepsilon < 2\%$). During the microplastic regime ($2\% < \varepsilon < 6\%$), localized inelastic accommodation processes proceed and dislocation activity starts to evolve leading to the onset of texture formation. At high plastic strains ($\varepsilon > 6\%$) we observe pronounced intragranular dislocation activity, entailing microstrain relief and distinct deformation texture formation. Concomitantly, stress-driven grain boundary migration is observed. The comparison between dominant shear and compression testing reveals that shear-dominated deformation facilitates intragranular dislocation plasticity and texture formation even for grain sizes as small as 20 nm, indicated by the stronger six-fold symmetry of normalized integral intensity (Figure 2a).

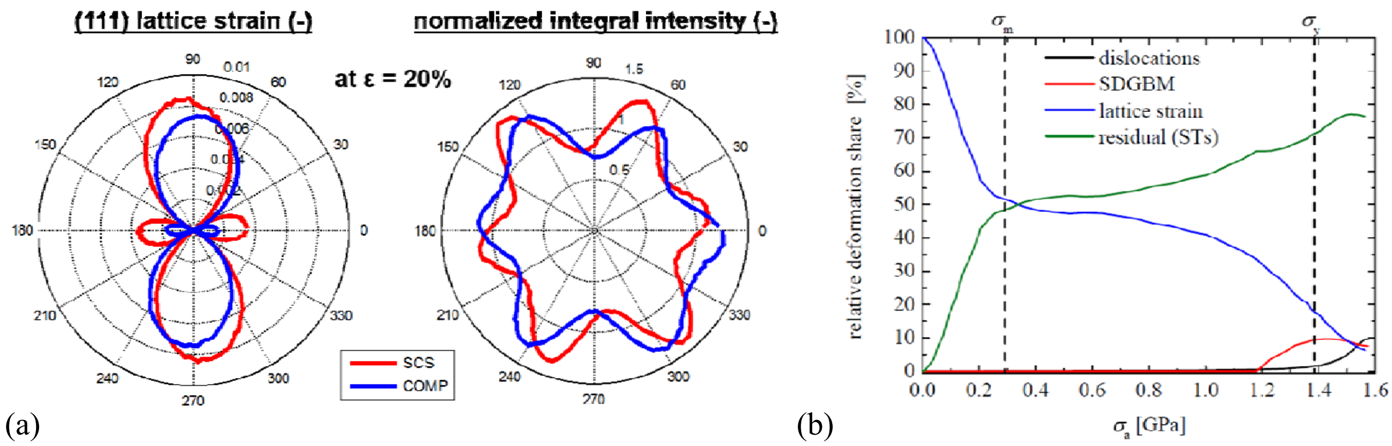

Figure 2: Compilation of results for in situ shear compression tests on (a) ed-Ni and (b) igc-Pd90Au10.

Shear compression tests on igc-Pd90Au10 (Grewer et al., submitted to Phys. Rev. Applied, see arXiv:1408.5049):

In this work, we have utilized in-situ diffraction and post mortem microscopy in conjunction with dominant shear deformation to identify, dissect, and quantify the relevant deformation mechanisms in nc Pd90Au10 in the limiting case of D <10 nm. We could identify lattice elasticity, shear shuffling operating in the core region of GBs, stress driven grain boundary migration (SDGBM), and dislocation shear along lattice planes to all contribute, however, with significantly different and nontrivial stress-dependent shares to overall deformation. We avoid the term GB sliding to specify GB-mediated deformation since shear shuffling or shear transformations (STs), which operate as generic flow defect in metallic glasses, could be unambiguously identified in conjunction with an analysis of activation volume and energy in a previous study (M. Grewer and R. Birringer, Phys. Rev. B 89, 184108 (2014)). In the so-called microplastic regime, STs coexist with lattice elasticity in a way that the share of the latter decreased inversely proportional to the progressive increase of STs (Figure 2b). Dislocation activity is basically missing here. The fact that STs and linear lattice elasticity more or less exclusively operate in this regime, suggests to discard the conventional concept of work- or strain hardening since it is based on intraplanar dislocation interactions. Moreover, STs which propagate strain at/along GBs have been found to carry three quarters of the overall strain in the regime of macroplasticity and so are by far the dominant carrier of strain. The abrupt onset of SDGBM appeared at stress values in the vicinity of the yield stress. Dislocation shear contributed in a very sluggish manner even above yielding. Appreciable and progressively increasing dislocation activity required stress values near the failure stress. Overall, the diverse evolution of the respective deformation shares appears as being just a consequence of a complex hierarchical order of onset/nucleation and upholding stresses of different deformation mechanisms. We expect that the configurational state of the material's microstructure, whether tested in as-prepared or relaxed state, should have a decisive inuence on this hierarchy.