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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

Reports supporting requests for additional beam time

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

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:** 3D mapping of the local elastic strain field in single grains of a Cu blanket film with coherent x-ray diffraction.	**Experiment number**: Si-1463
Beamline:	**Date of experiment**: from: 04/11/07 to: 04/17/07	**Date of report**: 07/20/07
Shifts: 18	**Local contact(s)**: Cristian MOCUTA	*Received at ESRF:*

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Report:

In this experiment, the 3D coherent x-ray diffraction pattern is successfully measured on a single Cu crystal embedded in a polycrystalline Cu thin film. As Cu exhibits a very strong elastic anisotropy (a ratio of 3.2 exists between the Young moduli along the [111] and [001] orientations), very large grain-to-grain interactions are expected between the (001) and (111) grains. It results in large strain in-homogeneities depending on the grain aspect ratios. The aim is to investigate the local elastic strain field in a single grain in a matrix.

Coherent x-ray diffraction is a powerful technique for imaging the 3D strain field in a single grain [1]. The method is based on the oversampling of the coherent pattern, allowing in principle to the phase restoration [2] using phase retrieval algorithm without the need for a priori hypothesis [3,4]. In the presence of strains, the method is more challenging as an effective complex-valued electron density is introduced, whose amplitude is the true electron density and whose phase is related to the atomic displacement field projected onto the chosen Bragg vector. In the weak strain regime, the method is limited by the divergence of the beam while the inversion of the diffraction patterns from highly inhomogeneous strained systems relies on the adaptation of the iterative algorithms.

The Cu films (0.5 μm thick) are deposited onto a silicon substrate. The mixing with the substrate and the oxidation of the film are avoided by the depositions of two thin silicon nitride layers at the substrate/Cu film interface and onto the Cu film surface. The grains are mostly <111> oriented (*i. e.* (111) planes parallel to the surface). However, about 10% are <100> oriented (estimated from Electron Back-Scattered Diffraction analysis). After several thermal cycles up to 400°C, the structure is stabilized with in-plane grain size typically in the 0.1−1 μm range, for both orientations.

An un-focused 8 keV beam is used. The longitudinal coherence length is ensured by the double crystal Si(111) monochromator while the transverse coherence length is given by the apertures of the SS2/SS4 slits, located at about 10 m and 1 m from the sample position, respectively. A special attention is paid to the fine characterization of the beam divergence, which is about 3 mdeg in both horizontal and vertical directions. The sample is mounted horizontally on the diffractometer under a neutral atmosphere. A 0D detector and two 2D-CCD's camera are used for the setting and the measurements. Two kinds of investigations are performed: measurements of the Debye-Scherrer rings on a grain assembly and 3D coherent x-ray diffraction on a single grain. For the first ones, a wide view acquisition (6° × 6° with the 2D CDD) is performed at the 111, 200, 222 and 400 Debye-Scherrer rings, obtained with a large spot size at the sample position. By varying the incident angle and the spot size and by probing different positions on the sample, it is possible to discriminate

between two strain in-homogeneities: with a large spot size (200×200 μm²) the measurement reveals the homogeneity of the average strain distribution along the sample, while a smaller spot size (10×10 μm²) allows to separate the diffraction patterns from individual grains and to estimate therefore the distribution of the average strain from one grain to another.

In the second part of the experiment, which is also the more challenging one, the detailed shape of the coherent diffraction pattern from individual Cu grains ((100) oriented) is obtained. When the diffracted intensity is collected in a solid angle of about 1°, the grain density decreases down to about 1 for 10 μm^2. One single grain is selected by scanning the sample through the beam spot and the coherently diffracted intensity is measured with a direct illumination CCD detector (384×576 pixels of 22×22 μm^2 area), used in a single photon counting mode [5]. This provides noise free results with a precisely known number of detected photons, making possible statistic studies. The final 2D pattern results from the accumulation of thousands of frames, each obtained with an acquisition time of about 2 seconds. The 3D Bragg spot is fully described by scanning the incident angle on a few tenths of a degree. Typical patterns can be seen on Fig. 1. A visibility as large as 80% is reached, showing the good coherence properties of the experimental set-up. The full 3D measurements of the 200 reflection are obtained for two crystals (with 21 and 41 frames measured along the rocking curve, respectively). An online estimation of the crystal sizes leads to about 300 nm. In addition, we monitor the decrease of the 2D intensity distribution, while scanning the grain through the x-ray beam. The simultaneous vanishing of the different parts of the diffraction pattern allows to conclude that the whole diffraction pattern is arising from one individual grain. Finally, for one of the two crystals, the second order (*i. e.* the 400 reflection) is also investigated. Further analysis are now in progress in order to attempt 3D shape and strain inversion.

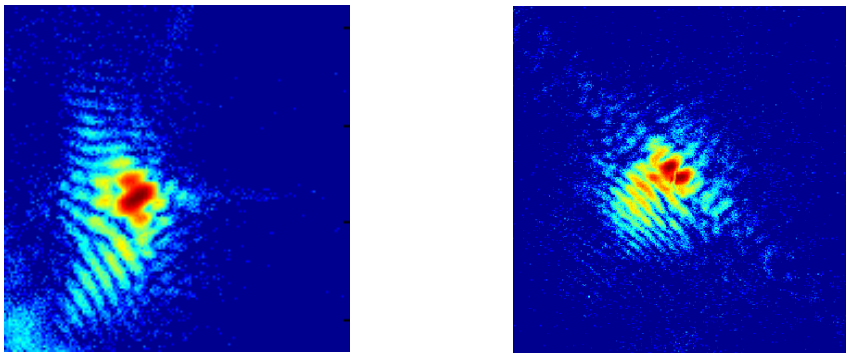

Figure 1: Coherent x-ray diffraction patterns from two individual grains in a Cu polycrystalline thin film, measured at the Cu 200 Bragg reflection.

[1] M. A. Pfeifer, G. J. Williams I. A. Vartanyants, R. Harder and I. K. Robinson, *Nature* **442**, 63 (2006).
[2] D. Sayre, *Acta Cryst.* **5,** 843 (1952).
[3] J. R. Fienup J. Appl. Opt. **21,** 2758 (1982).
[4] R. W. Gerchberg and W. O. Saxton, *Optik* **35**, 235 (1972).
[5] F. Livet, F. Bley, J. Mainville, R. Caudron, S. G. J. Mochrie, E. Geissler, G. Dolino, D. Abernathy, G. Grübel and M. Sutton, *Nucl. Instr. Meth A* **451** 596 (2000).