EUROPEAN SYNCHROTRON RADIATION FACILITY

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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:** Coherent x-ray diffraction for investigating the mechanocs of sub-micrometer size metallic crystals	**Experiment number**: si1740
Beamline: ID01	**Date of experiment**: from: 19/11/08 to: 25/11/08	**Date of report**: 25/02/09
Shifts: 18	**Local contact(s)**: Ana Diaz	*Received at ESRF:*

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Report:

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[2] of the coherent pattern, allowing phase retrieval by using appropriate algorithms [3,4].

In this experiment, three 3D coherent X-ray diffraction patterns were successfully measured on a Au single crystal embedded in a polycrystalline film for three different temperatures. Contrary to previous experiments Si1660 and Si1470, the position of the diffracting grain, can be find thanks to a special design of the sample (Fig.1). The sample was a polycrystalline film, 200 nm thick, mainly <111> oriented. A 150 x 150 µm² area was etched by FIB (Focused Ion Beam). In the center of this area, a polycrystalline film of 10x10 µm² was preserved. This large area without Au grains was quite easy to find thanks to the microscope and the focused X-ray beam provided by the Beryllium lenses (beam size on the sample: 3x3 µm2).

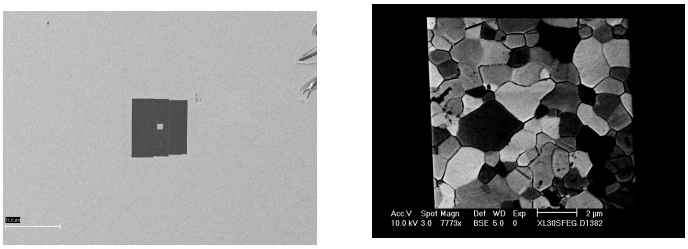

Fig.1: Special design of the sample allow us to know which grain is mapped

To select a grain, the Debye ring of the 10x10µm sample was recorded with a wide angle camera while scanning the sample in omega (0,01° step), x and y (1µm step) . This first measurement will give us the

Pole figure of the sample (about 100 grains) and could be compared with Laue Microdiffraction and EBSD data.

To study mechanical properties of the sub-micrometer grain, a mechanical loading is very interesting. In the Si1660 experiment, thermal loading appears to be the more convenient mechanical in-situ test. However during this experiment Si1660, we used Antor Paar furnace on the Huber Tower. It was not convenient to move the incident beam angle. That is why in this new experiment, a transmission furnace (coming from sample environment group) used in tomography was adapted to in-plane diffraction geometry, and was mount on the hphi arm. This furnace was successfully link to spec with Eurotherm controller. It was easy to launch thermal cycle. The sample was heated up to 150°C.

The loading of the film was performed in compression via the thermal expansion mismatch between Au and SiO_2. As we can see on the three rocking curves taken at three different temperatures (Fig.2), the Bragg is moving towards smaller scattering vector. That is expected for a thermo-elastic behaviour.

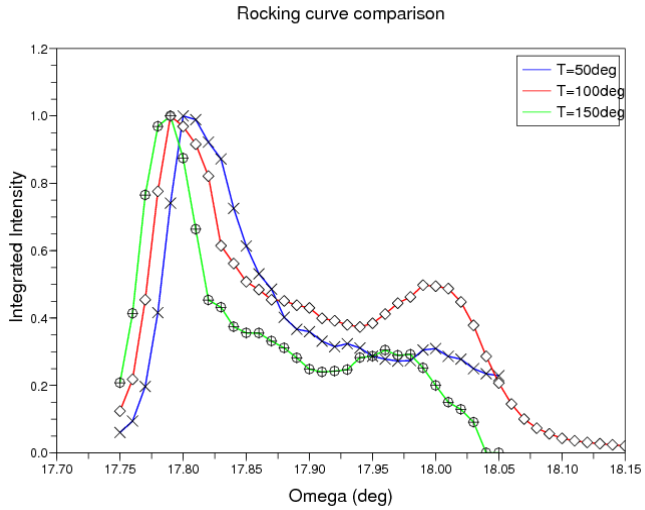

Fig.2: Rocking Curve Evolution in Tempearture

The coherent x-ray diffracted intensity was measured with a direct illuminated CCD camera (384x576 pixels 22x22 µm² area), used in a single photon mode [5]. Some tests with the new MaxiPix camera were performed but were not successful. We find a isolated grain (isolated in orientation- "alone" in the Debye ring), with a lot of fringes(Fig.3 and Fig.4). The diffraction pattern was measured from 50°C to 150°C with 25°C step. We performed three large 3D with 0,01° omega step (8 hours each).

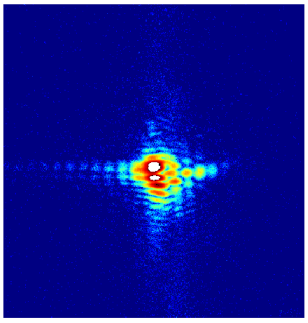

Fig.3: 2D slice of (111) Bragg Peak of an <111> oriented grain at T=50°C

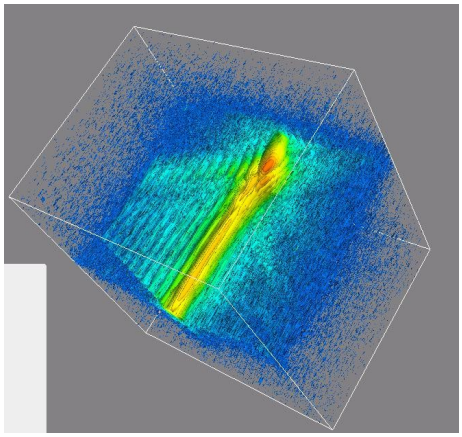

Fig.4: 3D Bragg peak of the same grain (31 2D slices in omega)

In order to investigate the effect of the wavefront of the incident beam on the diffraction pattern , the same diffraction pattern were recorded with different optics.
A summary of the different acquisition is drawn in Tab. 1 and Tab. 2 .

With lenses	ss4	T	Slices
g3	40	50°	31
g3	20	75°	3
g3	20	100°	41
g3	10	100°	5
g3	10	125°	3
g3	10	150°	31
g3	5	150°	1

Tab. 1: Summary of Temperature and Optic Configuration Acquisitions

Without lenses	ss4	T	Slices
g4	10	100°	8
g4	20	100°	5
g4	10	125°	3

Tab .2: Optic Test without Lenses

[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 et al., Nucl. Instr. Meth A **451** 596 (2000).*