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 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: Application of high resolution x-ray diffraction imaging to the study of the genesis of crystal perfection of bio-molecular crystals**	**Experiment number**: LS1860
Beamline: ID19	**Date of experiment**: from: 20 June 2001 to: 25June 2001 06 February 2002 06 February 2002 06 June 2002 11 June 2002	**Date of report**: 01/09/2002
Shifts:	**Local contact(s)**: Jürgen Härtwig	*Received at ESRF:*

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Vivian Stojanoff

Report:

Biomolecular crystal growth remains an issue with several implications. Be it to enhance crystal diffraction data for structural analysis or the characterization of physical properties for the development of new biomolecular materials. It is the goal of this proposal to use high-resolution X-ray diffraction and imaging methods to: 1.characterize bio-crystals to better understand the nature of growth mechanisms and therefore predict better growth methods; 2.understand defect mechanisms in biomolecular crystals.

To achieve our goals we proposed to develop further the four-circle diffractometer on ID19 to allow for the combination of high-resolution X-ray imaging, high-resolution X-ray diffraction (reciprocal space mapping), and large-field Laue imaging. For our first beam time (June 20 and 25, 2001) we proposed to attach a Mar research image plate detector (MAR345) to the four circle vertical diffractometer to allow to index any given reflection, work the orientation routines and install a Si (111) analyzer crystal.

In June 2001, the first cycle of shifts, the vertical diffractometer had just been moved to its final position in the new ID19 experimental hutch. Most of the beam time was spent to calibrate some of the functions such as the synchronization of the shutter and the spindle axis

(sample rotation) without much success. Several topographs were collected but it was not possible to index them at the time. An example extracted of the 43 films recorded is shown in Figure 1.

The second shift assignment from February 06 to 12, was actually used to work towards our primary goals. The Mar research image plate detector (MAR345) from the ESRF detector pool was successfully attached to the vertical diffractometer and the synchronization of the shutter and spindle axis calibrated. As test crystals we used Hen Egg White Lysozyme crystals grown by the batch method in gel media under different super-saturation conditions. Several rocking curves and X-ray diffraction images from known (hkl) reflections were recorded from 4 different crystals. See Figure 2.

The main goal of the shifts from June 06 to 11 was to integrate the previous setup with the high resolution reciprocal space mapping. A flat Si (111) analyzer crystal was installed on the diffractometer to allow for the reciprocal space mapping. Several maps were registered from three crystals. In order to properly analyze the data we still need to record the experimental function but as can be seen in Figure 3 it is possible to record the reciprocal space map of a given reflection. One problem found was that not always the samples were stationary in the sample holder. Some more though needs to be given on the mounting of the crystals. For the remaining shifts it is our purpose to further develop a user friendly data collection setting and study the strain/stress field around a pre-selected reflection.

The preliminary results obtained in this experiment were presented at the 19th Congress and General of the International Union of Crystallography and will be discussed at the 6th Biennial Conference on High-resolution X-ray Diffraction and Imaging. A paper on the origin of defect contrast is also being prepared for publication.

The shifts to date have been dedicated to tests of the instrument and implementation of a measurement routine. Even though we have not achieved all of the initial goals mentioned above we are at a point where other members of this group could bring their samples and use the experimental setup.

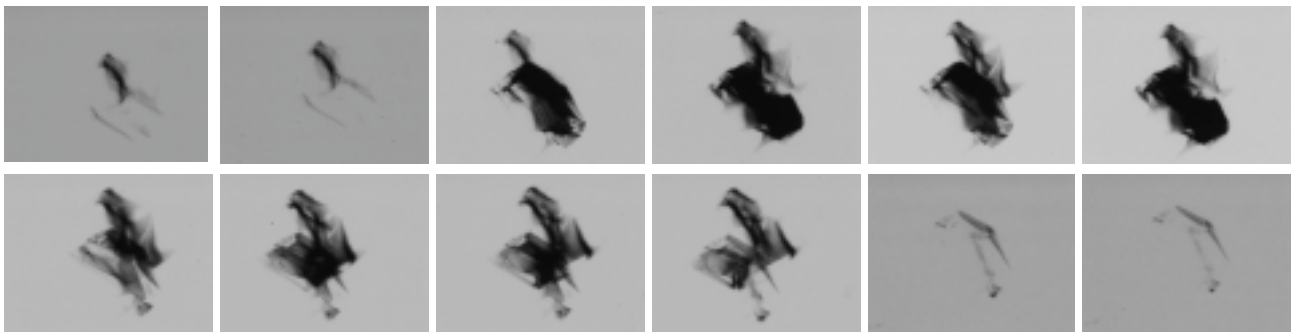
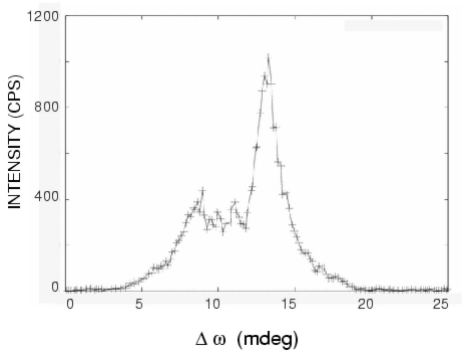
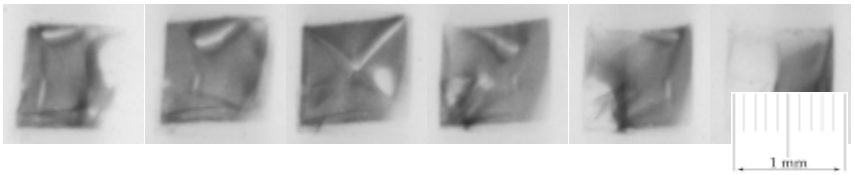


Figure 1.: Series of topographs recorded at 4.1 Å resolution. Angular step between images: 2 mdeg; crystal to film distance 280 mm.



(a)



(b)

Figure 2. Hen Egg White Lysozyme grown through the batch method in 10% agarose media and 3% NaCl. Shown for the (–17 16 0) reflection (a) Rocking curve; (b) series of topographs collected in 0.02 mdeg steps along the rocking curve. Quite visible in the topographs are the growth sector and fine lines originating at the border of the crystal in the topographs taken at the slope above the peak.

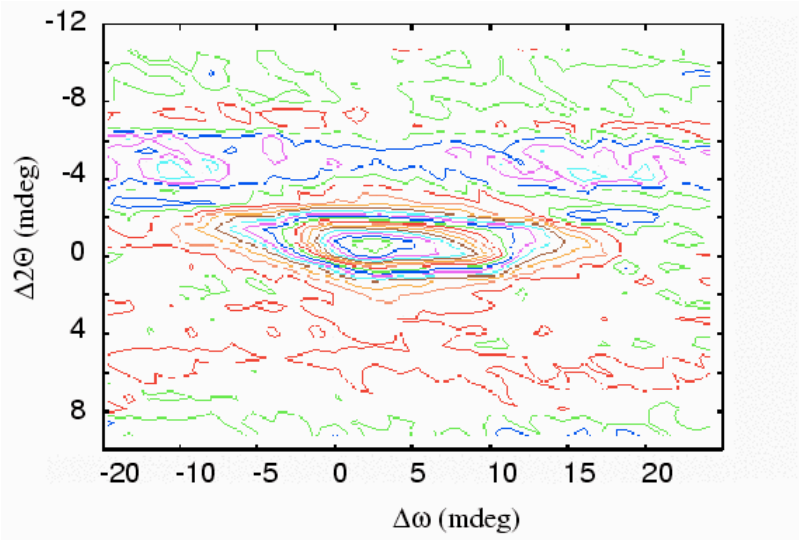


Figure 3. Hen Egg White Lysozyme grown through the batch method in 10% agarose media and 3% NaCl. Shown for the (15 15 0) reflection is the 2D reciprocal space map. The large variation observed in the omega direction corresponds to a relative large mosaic spread. This can mean that lattice fragmentation is favoured over elastic distortions.