ESRF	Experiment title: Topographic studies on thin protein crystals	Experiment number: LS-716
Beamline: ID-19	**Date of experiment:** from: Sept-12 1997 to: Sept-13 1997	**Date of report:** Feb-10 1998
Shifts: 3	**Local contact(s):** Angel Mazuelas	*Received at ESRF:* 31 AOUT 1998

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Report:

The topographic images previously obtained from protein crystals show low contrast and an overall blurring of the image due to

a) the mechanical properties of the protein crystal (a very soft material) which produce low contrast, continuous intensity gradients instead of sharp contrast features imaging crystal defects and
b) the thickness of crystals that gives rise to an averaging of three dimensional, low contrast features through the crystal volume when using transmission topography.

In order to overcome these problems, very high quality crystals of tetragonal lysozyme were grown inside flat (50x4x0.1 mm) capillaries by using the Gel Acupuncture Method. Such thin crystals produced very low diffracted intensity and imposes the use of the ESRF's ID-19 beamline because their high brillance and excellent equipment for imaging techniques.

The experimental set-up (figure 1) was simple: the capillaries were fixed on the axis of the horizontal goniometer at ID-19 and, then, a fast theta scan was performed to look for intense reflections at a given and fixed angle (d-spacing) using the intensified Sofretec camera. When an intense reflection was found, their Bragg angle and width were estimated, an high resolution X-ray film was mounted in front of the camera on a two axes positioning stage and a series of topographies was taken by exposing the sample during a given time and, then, rotating the crystal (typically by **1/1000º)** and displacing the film to take another image displaced some millimetres. This procedure was repeated until the whole rocking curve of the reflection was scanned. The final dataset obtained consist in several films recording such scans for different reflections from several different crystals.

The "rocking curve" concept has been generalised to that of "rocking map" by computing rocking curves for different windows into the stack of consecutive images from each scan. These maps (figure 2) show the non-uniform distribution of the mosaic spread along the crystal volume. This non-uniformity seems to be due to the changing crystal growth rate when using this growth method and to the mechanical stress introduced by the walls of the capillary and small cracks. The excellent quality of the images obtained allows us to perform detailed studies on the origin of the contrast in topographies. No single defects have been observed, the mayor source of contrast being produced the surfaces separating different growth sectors of very different mosaic spread (figure 3). These surfaces can be followed and localised into the crystal volume and produce a continuous change in the misalignment of the domain blocks that separate. These surfaces, when appear parallel to the largest crystal dimensions, produce moiré fringes.

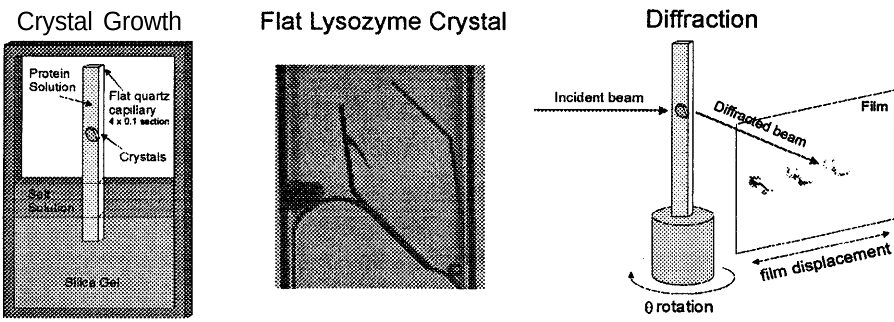

Figure 1.

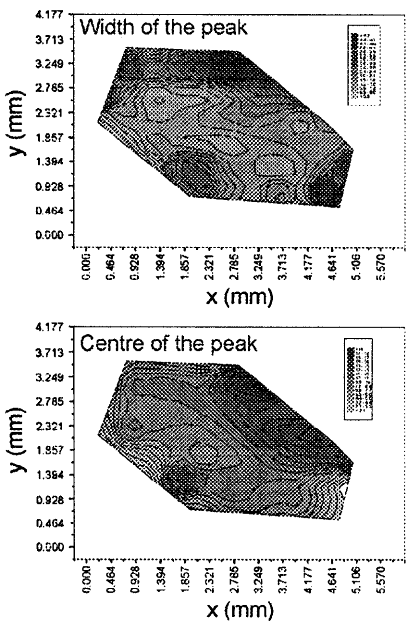

Figure 2.

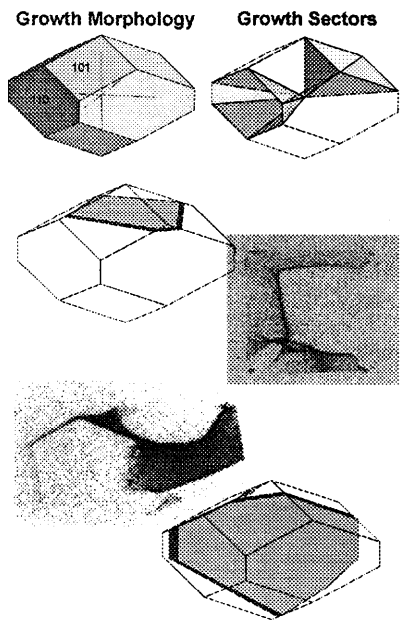

Figure 3.