

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://www.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.



Experiment title:

Full Field Diffraction Imaging on InGaN nano-LED devices of varying optoelectronic properties

Experiment number:
MA-3317

Beamline:

ID01

Date of experiment:

from: 06/05/2017 to: 08/05/2017

Date of report:

Shifts:

9

Local contact(s):

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Report:

We used the new Full Field Diffraction X-ray Microscope on the ID01 beamline to study the different lattice strain/tilt both among individual nanopillars and among the differently (opening, pitch) patterned areas. The experiment was a great success. We have now completed the quantification of the tilts, and show some of this results below (the analysis is still ongoing).

The sample was mounted on an aluminium pin (fig 1a) (id:run466) and contains 16 patterned areas of different pitch and opening for the mask (fig 1b). By recording images at the intersections of four zones, labelled 1-4 in the image on the right, we get information on four zones in one shot, fully exploiting the large field of view of the microscope.

Some time was spent at the beginning of the beamtime to optimize the energy and Bragg reflection used for imaging. The best images could then be recorded while doing a rocking scan of the sample thus imaging different angles through the (103) reflection.

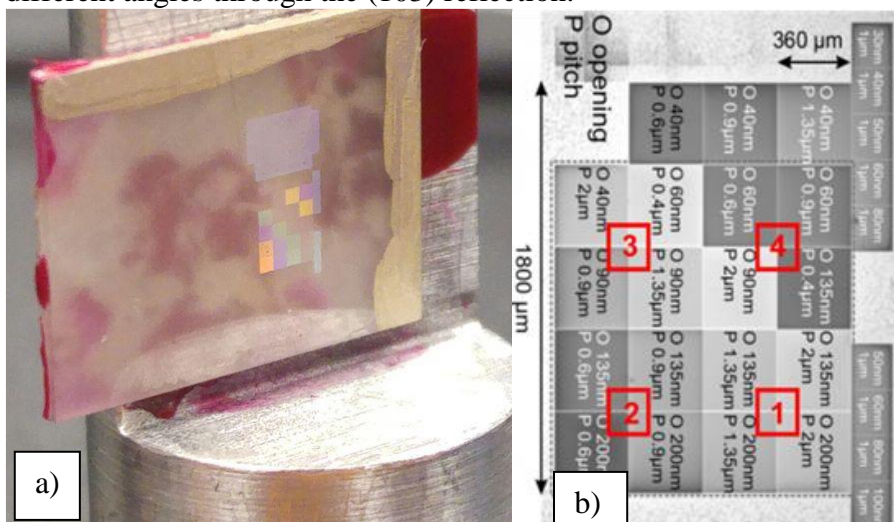


Figure 1: a) the sample mounted on a pin, b) the pyramid patterned zones.

On Fig. 2 is shown one FFDXM image taken at the InGaN(103) Bragg reflection. The Bragg reflection is chosen to increase surface sensitivity (small incident angle 1.8°) and minimize projection issue (large exit angle 67.4°), we have also adjusted the energy (from 8 to 7.7 keV) a little bit to optimize the above condition.

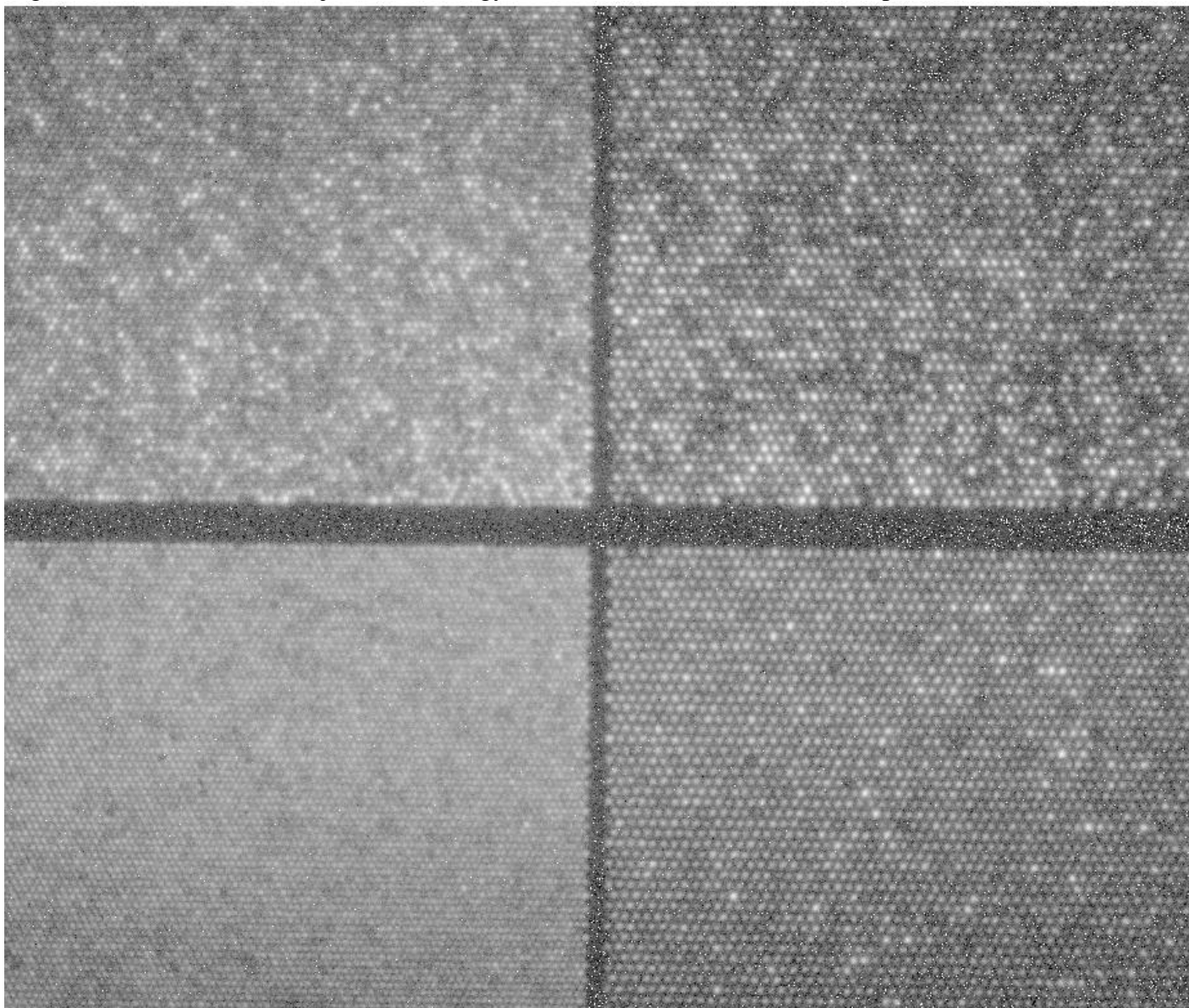
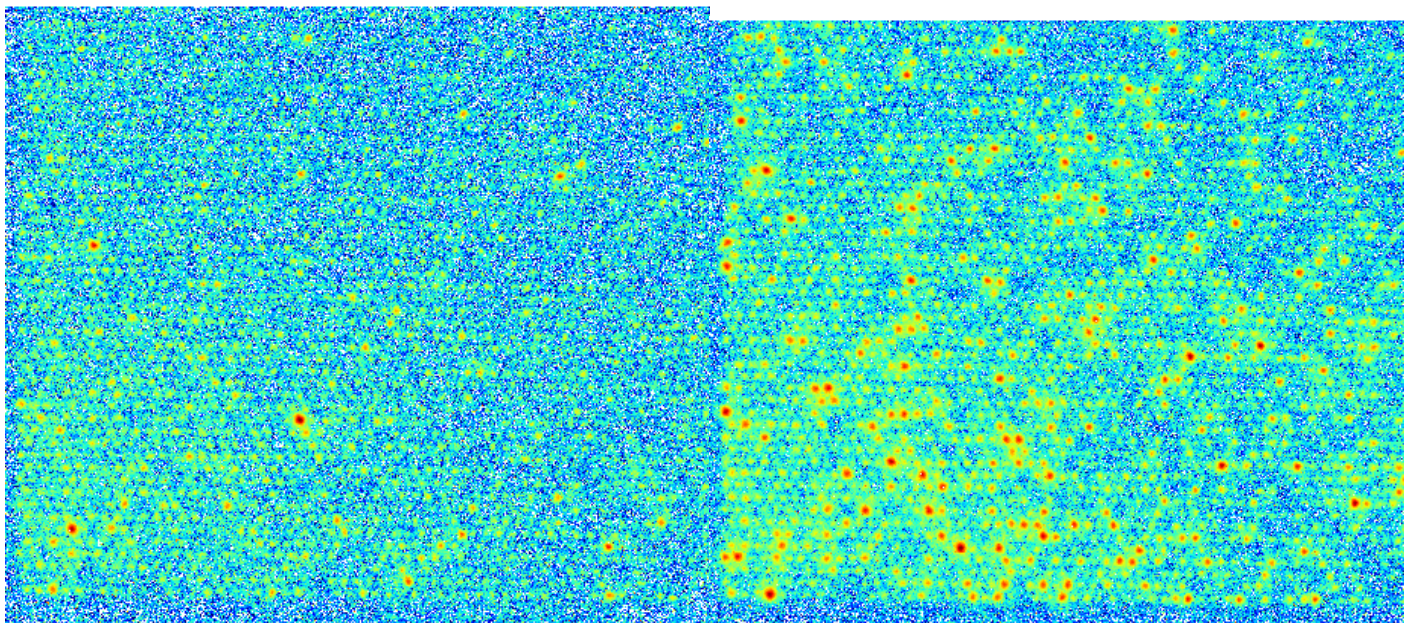


Figure 1: FFDXN image on zone 1, InGaN (103) Bragg reflection



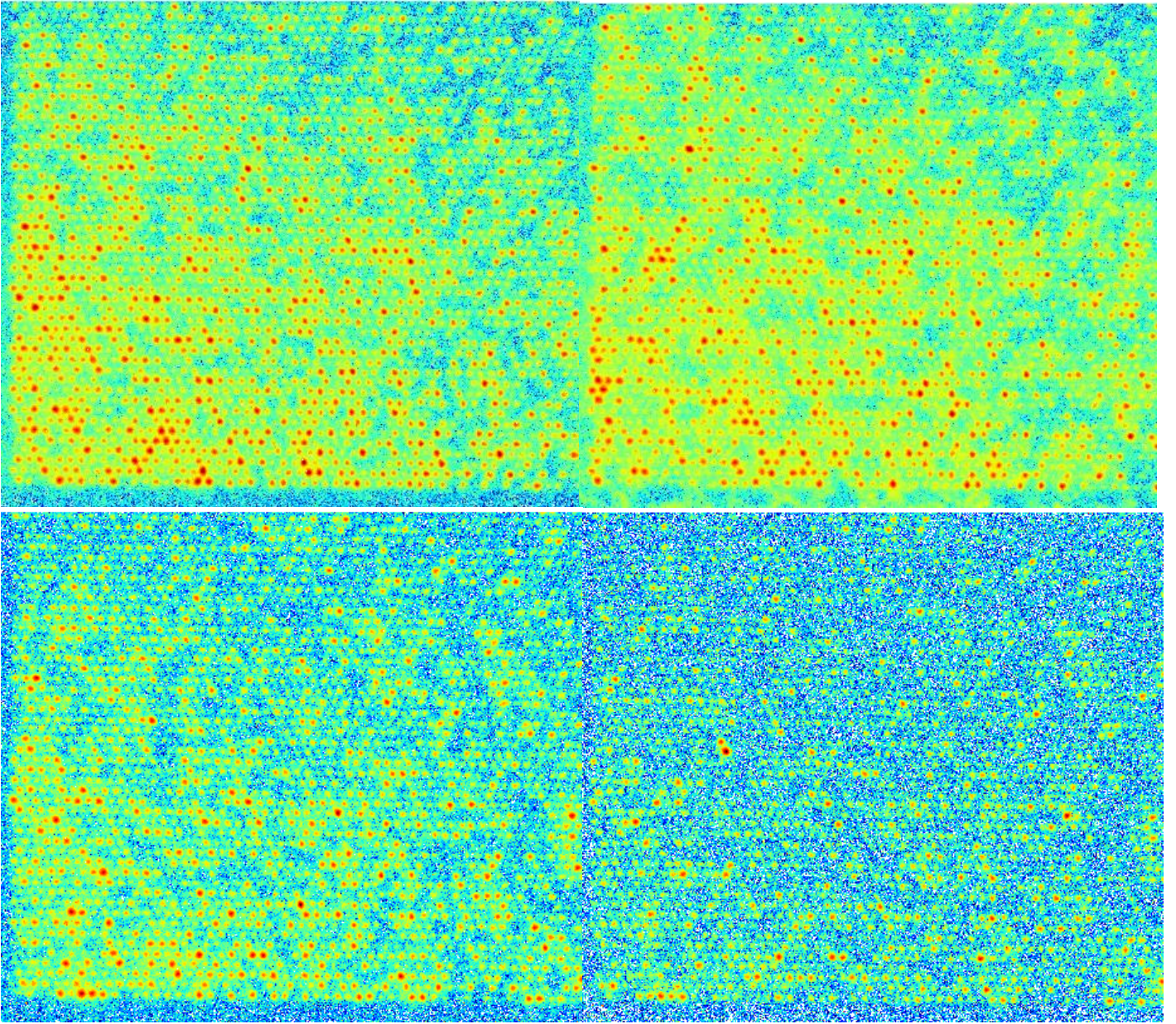


Figure 3: FFDXM image of the top right corner of zone 1 during a rocking curve. The 6 images are taken from theta angle $= -0.3^\circ, -0.2^\circ, -0.1^\circ, 0, +0.1^\circ, +0.2^\circ$, respectively relative to the Bragg angle. The log intensity is expressed in fake color scale.

On the gray scale image, the small and close nanopyramids can be clearly observed. However, not all the pyramids are visible. This is because some of the pyramids have different tilt angles and can only be observed on the shoulder of the rocking curve. On fig. 3 is shown some images taken from the rocking scan, of one patterned area inside zone 1 (the rest are now shown here for clarity). In fact, individual pyramids were observed as far away as $\pm 0.7^\circ$ relative to the Bragg angle. Our analysis strategy is the follows, for each pyramid, the tilt angle is expressed in colors, as the hue(H) in the HSV geometry. The log of the intensity contributes to both the saturation(S) and the value(V) in the HSV. The noise is then removed by setting the value(V) of pixels with too low std. var. to 0. This allows us to produce some nice composite image (tilt map) of the nanopyramids as shown in Fig 4.

Our last effort involves the removal of the influence of the GaN substrate image, visible as a the light orange cloud in Fig. 4. The result is shown in Fig. 6.

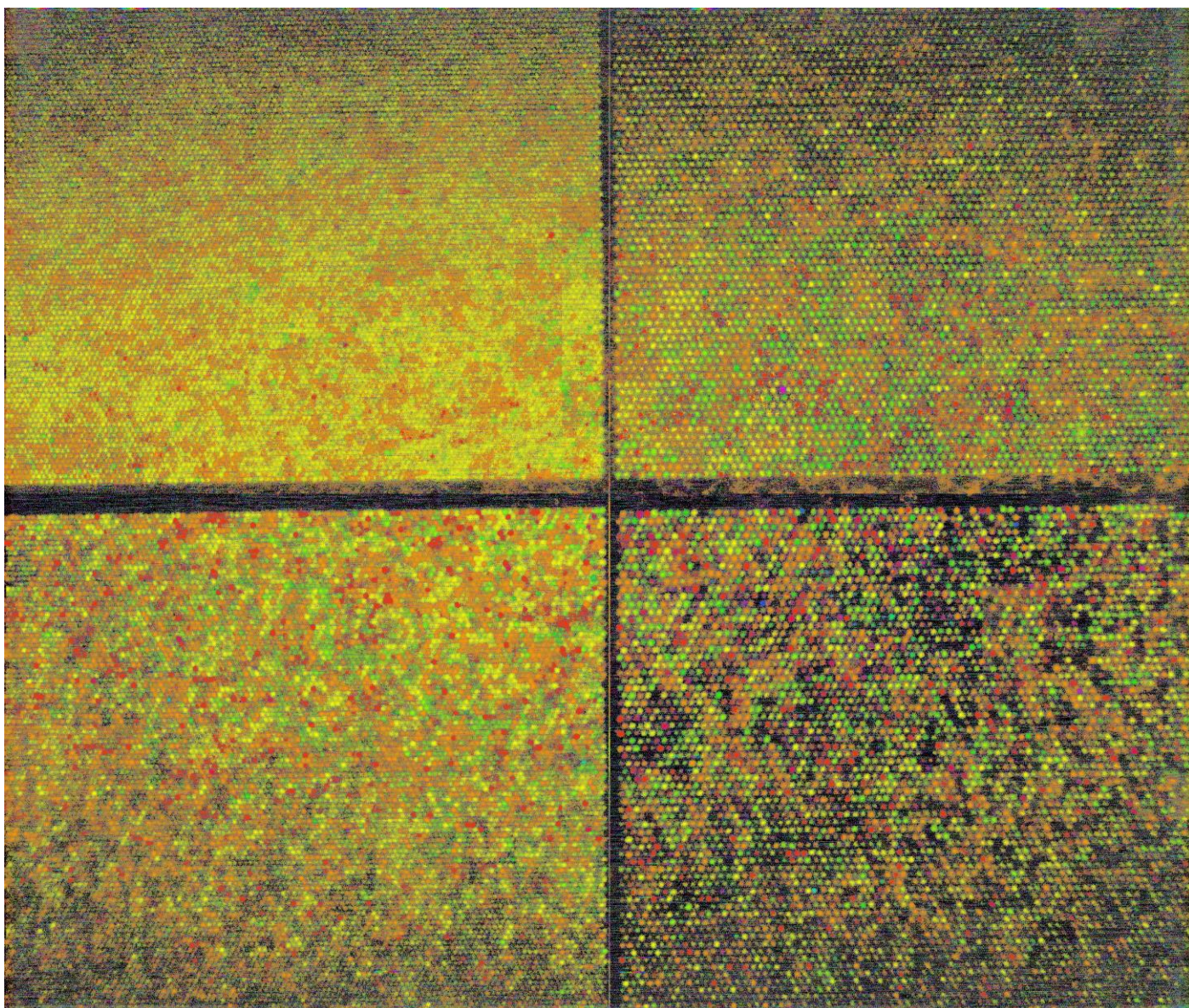


Figure 4: Composite tilt map of zone 1. The corresponding colormap is shown in Fig 4.

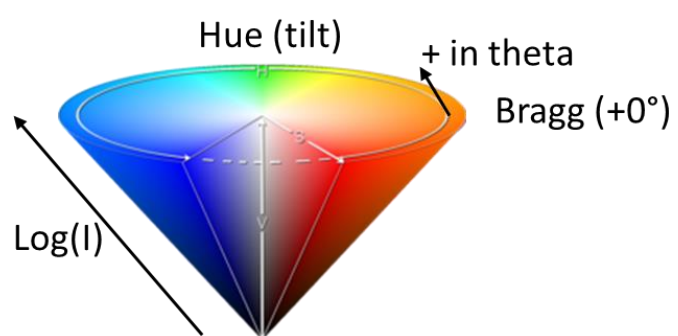


Figure 5 : Tilt map colormap removal

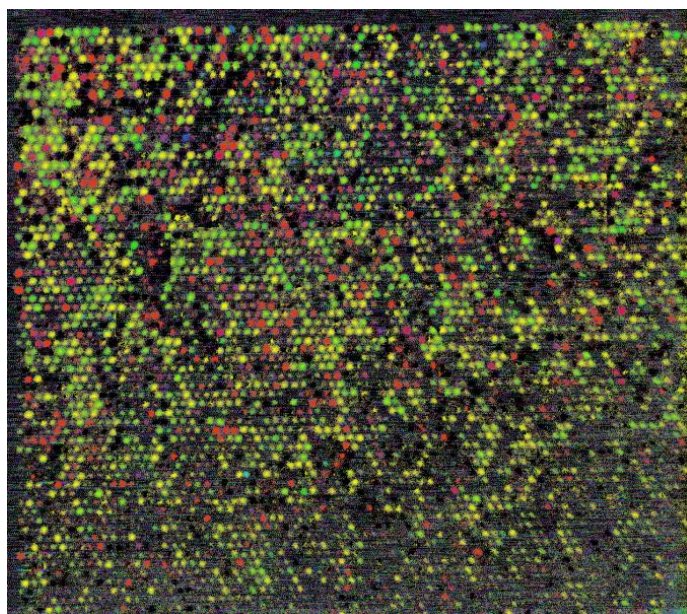


Fig. 6 Bottom left corner of zone 1 after substrate removal

To summarize, we have successfully mapped out the different lattice tilt both among individual nanopyramids and among the differently (opening, pitch) patterned areas. FFDXM is very well adapted to our study thanks to its large FoV, nice resolution, easy and well established analysis routine.

We managed to measure completely one sample in the 9 shifts that were given. While we lost some time trying to find the best measurement strategy (which energy, which optics, which reflection, what kind of scans, etc.), it is understandable considering that this is our first experiment and that we are among the first users for this new instrument. The best conditions to image this sample are now known and hopefully this can help us become more efficient in the future.

We wish to express our gratitude to the beamline staff for their invaluable help throughout the beamtime.