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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 via the User Portal:

https://wwws.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.

ESRF	**Experiment title:** Structure investigation of sub-micron sized synthetic and biogenic hemozoin crystals with a focused nano beam	**Experiment number**: SC-3329
Beamline: ID13	**Date of experiment**: from: 30. Nov. 2011 to: 3. Dec. 2011	**Date of report**: 1. Mar. 2012
Shifts: 9	**Local contact(s)**: Manfred Burghammer	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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all affiliated with

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Report:

The purpose of the experiment was to get structural information on crystalline biogenic hemozoin and its synthetic counterpart β-hematin. Our recent X-ray Powder Diffraction study indicated that β-hematin crystallized in two phases, whereas hemozoin consists of only a single phase. We concluded that the two compounds were not identical and proposed that they consisted of different polymorphs. Due to the rather large, triclinic unit cell the peaks in the powder patterns highly overlap revealing only three reflections from the second phase of β-hematin. By probing micrometer sized single crystals with a nano beam we would gain much more information on the different crystal structures.

At ID13 we obtained data of very high quality from both hemozoin and β-hematin. With a beam size of only 130 x 180 nm we could produce a map of the sub-micron sized crystals by Fe fluorescence and subsequent obtain diffraction data from different positions inside a single grain.

We found that the mosaicity of the crystals was surprisingly high as seen in Figure 1 (left panel).

An important question was if it the identification of a possible second phase of β-hematin, which indeed was present. Figure 1 shows a maxiprojection of a series of diffraction images, where a cluster of crystals is being probed. The {100} reflections of the two phases are of particular interest as they are by far the most intense reflections and their d-spacings should give separated peaks on the detector. It is clear from Figure 1 that pure major-phase crystals exist, as the {100} reflection of the minor phase only is present at a small fraction of the time the {100} reflection of the major phase is present. Further analysis is required to confirm the unit cell of the minor phase and if pure minor-phase crystals exist as well.

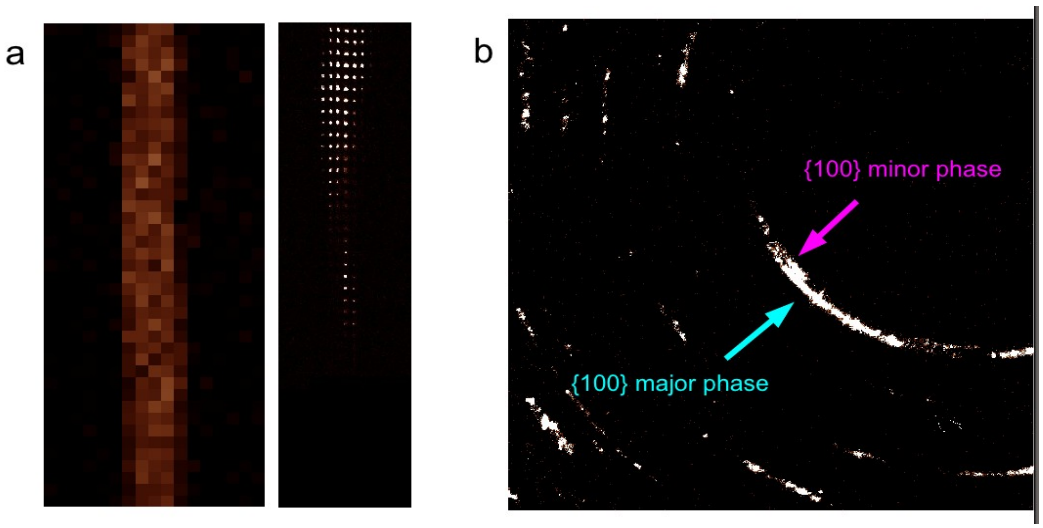

Figure 1: ***a)*** *left: The fluorescence signal as a function of (vertically) theta ranging 20 degrees and (horizontally) y-range (crystal position) ranging 4 microns and, right: the corresponding {100} reflection.* ***b)*** *The {100} reflection of the minor phase of β-hematin is only present at a fraction of the time the {100} reflection of the major phase is present, which indicates the two phases crystallize in separate crystals/domains.*

The purification process of biogenic hemozoin may have caused some cluster formation. Furthermore the amount of crystals was very sparse and the crystals died in the beam. But we managed to obtain several data sets of hemozoin. In figure 2 is seen a maxiprojection of hemozoin. We only see the most intense {100} reflection in this case. More crystals are a requisite in a future experiment. It seems as the crystals are aligned with a certain angle between them, as they are expected to do in the malaria parasite. It could however also be a result of the purification process and should be looked further into.

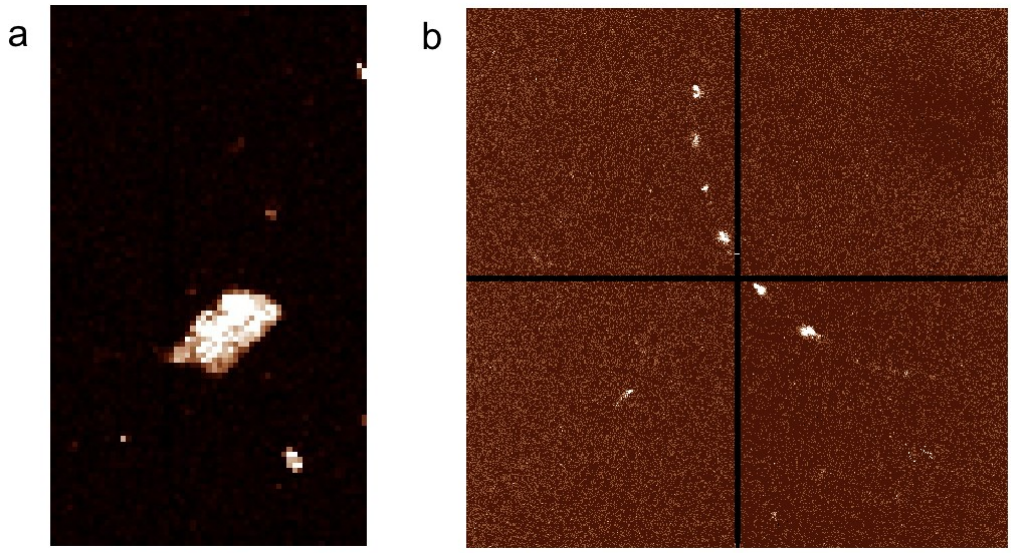

Figure 2: ***a)*** *A real space Fe fluorescence map of a cluseter of hemozoin crystals.* ***b)*** *A maxiprojection of a data set of hemozoin shows that the crystals seems to be aligned. If this is due to the purification process or a consequence of aligned growth within the malaria parasite is uncertain at this stage.*

The analysis is not yet finished, but we can conclude that we get good data with nano beam set-up at ID13, despite the fact that the crystals are considered to be weakly scattering. This experimental set-up is splendid for structure determination of small crystals and opens up for new possibilities. Next step will be to treat the malaria parasites with commonly used anti-malarial drugs. By combining the changes in crystal structure with fluorescence spectroscopy measurements of labeled drugs, it may be possible to reveal the mode of action of the drugs.