EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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: PDF computed tomography: a tool to understand precipitation in porous media** | **Experiment number**: |
|---|---|---|
| **Beamline**: | **Date of experiment**:<br>from: 10 oct 2015 to: 14 oct 2015 | **Date of report**:<br>15/01/2016<br><br>*Received at ESRF:* |
| **Shifts:**<br>12 | **Local contact(s)**:<br>Nicolas Harker (nicholas.harker@esrf.fr) | |

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## Report:

Diffraction tomography (Bleuet et al., 2008) with a resolution of 10 µm voxel size has been acquired, at ID 11, on three different porous media. The first one was a sea sand with barite and celestite that have precipitated inside its porosity (as described in Chagneau (2015)). Initially, the experiment aimed at determining in situ and time-resolved barite and celestite precipitation. However, it appeared that the precipitation kinetics were to slwo, and this could not be measured. To circument kinetics problems, we measured samples which were previously prepared in the lab. Several aquisions were made to ensure a statistically robust analysis. In addition, a few additionnal samples where measured, in other to ensure that data aquired on the beamline could lead to a publication. These two additonnal samples consisted in  a cement paste and a manganese nodule, sampled in the central pacific Ocean (Wegorzewski et al., 2015), that contains different nickel and copper content. On this latter sample aquision time was optimized to allow the use of Pair Distribution Function computed tomography which enables (1) to account for the 3D spatial distribution of poorly crystalline phases with micrometre resolution and (2) to determine the nature of the poorly crystalline phase (Jacques et al., 2013) has been used (figure 1).

The results obtained on the different porous media are now being analyzed and prepared for publications. A special attention will be paid to tomographic reconstruction in the case of cement. Indeed has already outlined by Voltolini et al. (2013), the problem with cement materials whenever applying routine filtering procedure is linked to the huge difference in crystallite size of the different phases and to the subsequent presence of preferential orientation, which needed to be accounted for in the raw data before tomographic images could be reconstructed. This data processing procedure is currently being performed, following a method previously published by Voltolini et al. (2013). Finally, we expect that on our recent work (Grangeon et al., 2013a; Grangeon et al., 2013b) on deciphering the structure of C-S-H (the main phases forming during cement hydration) will allow us to refine filtering procedure and to be able to spatialize C-S-H structure, in particular refine and spatialize C-S-H Ca/Si ratio distribution.

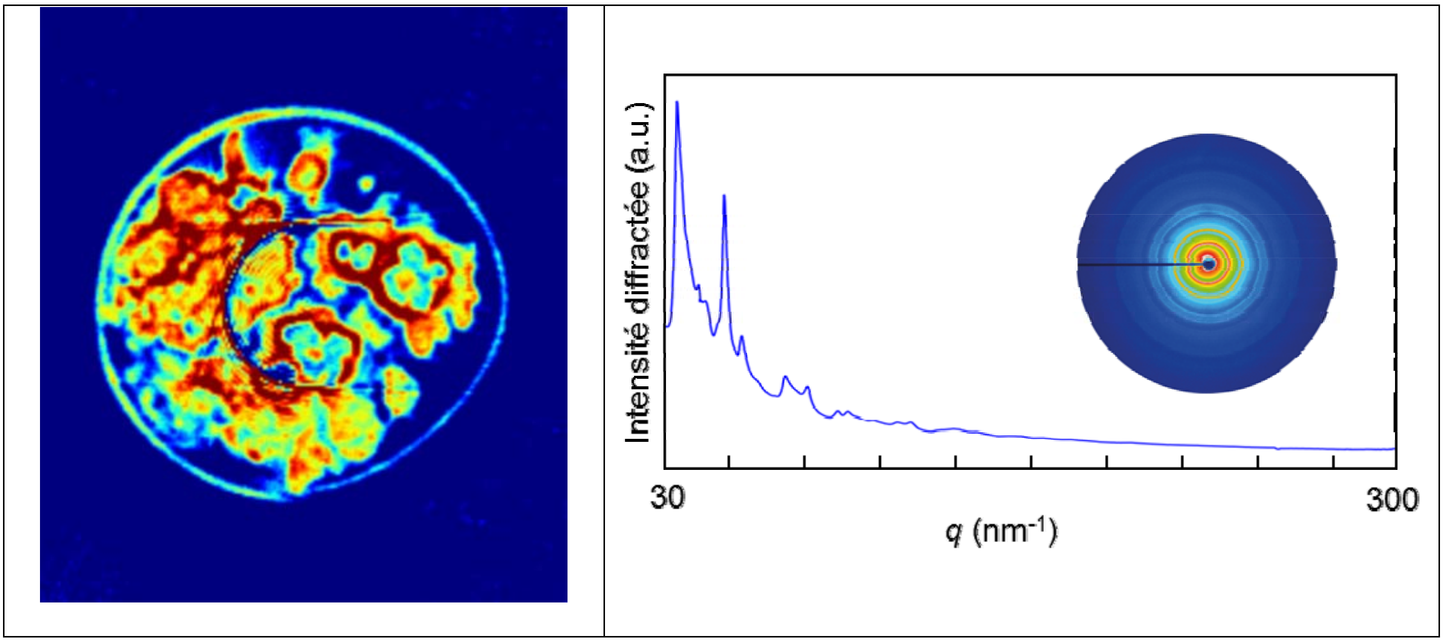


*Figure 1: Analysis of the manganese nodule. Left: reconstruction of the image for a given value of q. Some intensity artefacts persist (we are working on this issue, which should be solved by writing a software that allows for data normalization). Righ:, XRD pattern obtained for a given position inside the nodule.*

Bleuet, P., Welcomme, E., Dooryhee, E., Susini, J., Hodeau, J.-L., Walter, P., 2008. Probing the structure of heterogeneous diluted materials by diffraction tomography. Nat Mater 7, 468-472.

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Grangeon, S., Claret, F., Linard, Y., Chiaberge, C., 2013b. X-ray diffraction: a powerful tool to probe and understand the structure of nanocrystalline calcium silicate hydrates. Acta Crystallogr B B69, 465-473.

Jacques, S.D.M., Di Michiel, M., Kimber, S.A.J., Yang, X., Cernik, R.J., Beale, A.M., Billinge, S.J.L., 2013. Pair distribution function computed tomography. Nat Commun 4.

Voltolini, M., Dalconi, M.C., Artioli, G., Parisatto, M., Valentini, L., Russo, V., Bonnin, A., Tucoulou, R., 2013. Understanding cement hydration at the microscale: new opportunities from `pencil-beam' synchrotron X-ray diffraction tomography. Journal of Applied Crystallography 46, 142-152.

Wegorzewski, A.V., Kuhn, T., Dohrmann, R., Wirth, R., Grangeon, S., 2015. Mineralogical characterization of individual growth structures of Mn-nodules with different Ni+Cu content from the central Pacific Ocean. Am Mineral 100, 2497-2508.