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 via the User Portal: <u>https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do</u>

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a "preliminary report"),

- even for experiments whose scientific area is different form the scientific area of the new proposal,

- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- > 1st March Proposal Round 5th March
- > 10th September Proposal Round 13th September

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.

Instructions for preparing your Report

- fill in a separate form for <u>each project</u> or series of measurements.
- type your report in English.
- include the experiment number 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: Biomimetic approach to develop a novel environmental- friendly cement inspired by mason bee nests	Experiment number : MA-4865
Beamline:	Date of experiment:	Date of report:
ID16A	from: 29 April 2021 to: 02 May 2021	
Shifts:	Local contact(s):	Received at ESRF:
9	Federico Monaco	
Names and affiliations of applicants (* indicates experimentalists):		
*DA SILVA, Julio Cesar, Laboratory CNRS - Institut Neel		
HAZEMANN, Jean-Louis, Laboratory CNRS - Institut Neel		

Report:

The objective of this beamtime was to characterize the microstructure of the nest of endangered bee species (*Megachile pyrenaica*). Different from the standard hives, those bees build nests which are hard as a rock and remain in place for a year until it is naturally destroyed. Therefore, our idea is to use the biomimetic approach to research new bioinspired building materials from those mason bee nests. We also wish to understand the impact of the nest architecture design on CO_2 exchange, ventilation, and thermoregulation. Additionally, this study may contribute to saving this endangered bee species.

The studies included experiments of near-field ptychographic X-ray Computed Tomography (nf-PXCT) of capillaries of about 200 microns-diameter filled with pieces of the nest. Initially, we wanted to prepare a smaller pillar of the samples with FIB/SEM, but since the sample is not conducting and we did not have access to an ion-beam FIB, we had to adapt the sample preparation strategy. In the end, it was positive to do so since we could investigate a larger volume of the sample, which is very heterogeneous and complex and we could have missed details if done otherwise. The pixel size we used was 150 nm and the estimated resolution is about ~400 nm. Since their structures contain materials of different sizes and even big ones, that resolution was sufficient (figure 1). On top of this, our goal was also to determine the composition of the nest by measuring the electron and mass densities of the different components directly from the reconstructed 3D images, since nf-PXCT is highly quantitative for this, which justifies its need in this study.

We could scan 5 samples during the 3 days of beamtime, which was performed during the lockdown. At least I could partially be at the beamline, but the local contact made most of the work and even came on the weekend when I could not come. So, I would like to thank him a lot. Some problems with the piezo controlling the settings of the first multilayer mirror of the beamline was not working so well, so we lost a bit of time with repeats of parts of the experiments, but nothing crucial. Each 2D scan took about 30 seconds and each tomographic scan took about 9 hours with some overhead. Additionally, at each injection, we had problems with the coherence of the beam, forcing us to put a routine to wait a bit after the injections before starting acquiring again. Despite this, we could still get good results.

About 1000 projections were acquired and the phase retrieval was performed using the software Ptypy (https://ptycho.github.io/ptypy/) using the GPUs of the ESRF cluster. The tomography data processing and reconstruction were performed using our python suite Toupy (https://toupy.readthedocs.io/en/latest/), which is open-source and available to the community in my GitHub (https://github.com/jcesardasilva/toupy). Additonally, Toupy was used to remove unphysical phase vortices appearing during the phase retrieval, which allows us to get the proper quantification we need of the gray level of the images. Figure 1 displays two phase-contrast projections obtained with Ptypy of 2 nests coming from different regions and possibility with different regions build nests with slightly different compositions, possibly due to the availability of the resources, but how they know what to do remains to be understood yet. Complementary studies with micro tomography will be performed at BM05 beamline.

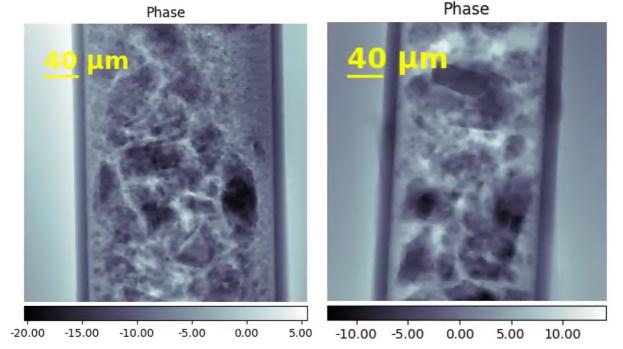


Figure 1 – nf-PXCT 2D projections, after phase retrieval with Ptypy, of two samples from 2 nests coming from two different regions. (Left) The exterior part of the nest which shows rocks of different sizes and composition. (Right) The exterior part of another next shows rocks with more rectangular shapes and less variety of sizes than the first one. One can also notice the range of phase shifts in both cases is about 20 radians, which is consistent.