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:
https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

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 each project 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:** In situ study of the growth mechanism of Cu2OSeO3 nanostructures in solution at low temperature	**Experiment number**: 01-02-1205
Beamline: BM01	**Date of experiment**: from: 11 May 2018 to: 15 May 2018	**Date of report**: 07 Oct 2020
Shifts: 15	**Local contact(s)**: CHERNYSHOV Dmitry	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Priya Ranjan Baral *, Crystal Growth Facility, Institut de Physique, EPFL

Wen Hua Bi *, Crystal Growth Facility, Institut de Physique, EPFL

Benoit Truc *, Crystal Growth Facility, Institut de Physique, EPFL

Arnaud Magrez *, Crystal Growth Facility, Institut de Physique, EPFL

Report:

Cu_2OSeO_3 is the first discovered insulator material exhibiting skyrmions under magnetic field at 60K. All of the studies on skyrmions in Cu_2OSeO_3 have been performed on bulk crystals. The effect of crystal size, specifically when it reaches the skyrmion size i.e. 60nm, has never been explored. We have established a low temperature process based on a hydrothermal treatment of $CuSeO_3.2H_2O$ to grow narrow size distribution crystals of Cu_2OSeO_3 with diameter ranging from 30nm to 25microns.

In order to study the process at ESRF in same hydrothermal conditions than the ones running at the Crystal Growth Facility, we designed and developed a novel furnace. The hydrothermal process takes place in a 70micron diameter sealed capillary, which is filled at 80% of its volume. During the XRD experiments, the capillary is sitting on a high thermal conductivity aluminum holder ensuring temperature homogeneity in the entire volume of the capillary. The holder is placed in double silica tube. Between the silica walls, a metal wire is coiled and serves as heating element. During the XRD measurements, three thermocouples probe the temperature at each extremity of the capillary and one in the center underneath the X-Ray beam.

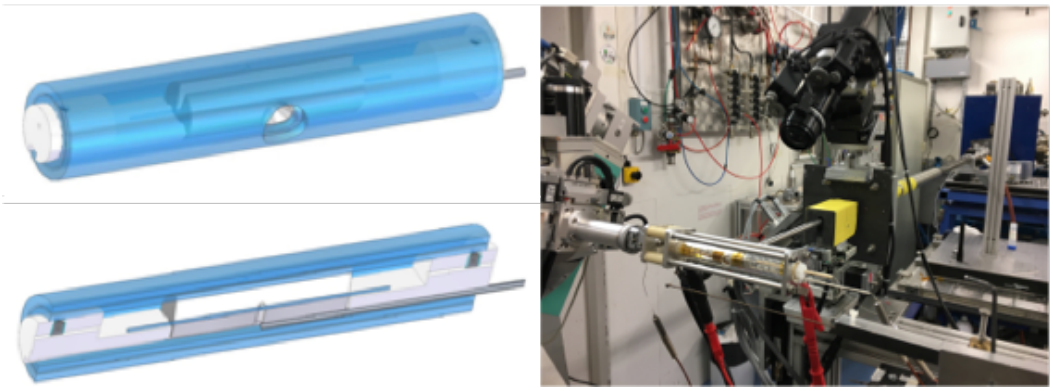

Figure 1: Furnace designed for *in-situ* investigation of hydrothermal process. (left) drawing of the device. The length of the furnace is 15cm and the diameter is 15mm. (right) the furnace installed on the goniometer for *in-situ* XRD measurements at BM01 of ESRF**.**

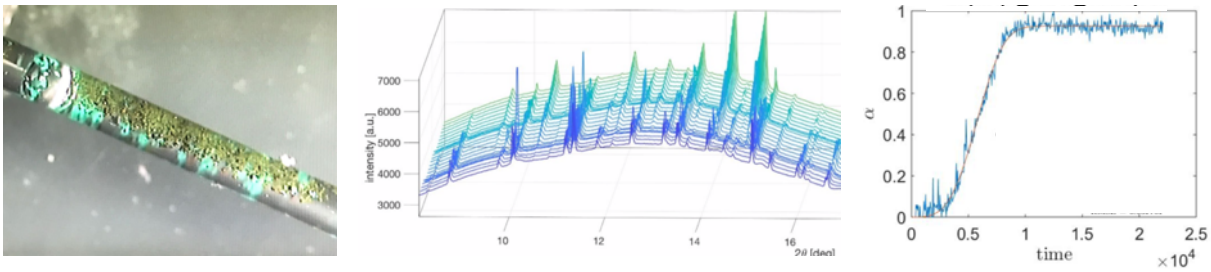

Figure 2: (left) Capillary filled with the hydrothermal medium. Cu_2OSeO_3 crystals appears in green in the capillary. (middle) time dependent XRD pattern recorded during the hydrothermal treatment of $CuSeO_3$. (right) Cu_2OSeO_3 conversion factor versus hydrothermal time

We successfully run measurements up to 350°C and recorded 500ms patterns during hydrothermal treatment (figure 2).
We could follow the chemical conversion of $CuSeO_3.2H_2O$ into Cu_2OSeO_3 by X-Ray diffraction. From these data, we measured temperature dependent kinetic and obtained the activation energy of the chemical processes (figure 3).

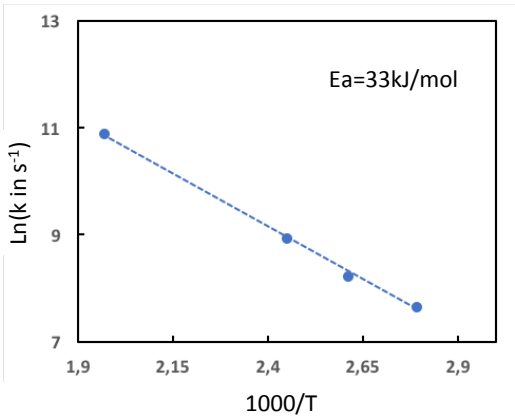

Figure 3: Arrhenius plot of the temperature dependent reaction deduced from the conversion versus time curve (figure 2-left) at different temperature. Inset: calculated activation energy of the reaction obtained from the Arrhenius plot

One paper is in preparation on the furnace design and performances as well as the first in-situ investigation of hydrothermal treatment using the furnace. These first sucessful results lead to the submission of a regular proposal (01-02-1206). We were allocated 15 more shifts to complete the parametric study of the growth process. This chemical study will be reported in a second paper.