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 using the **Electronic Report Submission Application:**

*http://193.49.43.2:8080/smis/servlet/UserUtils?start*

***Reports supporting requests for additional beam time***

Reports can now 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.

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|---|---|---|
| ESRF | **Experiment title:**<br>A 4D in-situ tomography study of the evolution of porosity in fluid-driven reactive systems | **Experiment number**:<br>ES-1159 |
| **Beamline**:<br>ID-19 | **Date of experiment**:<br>from: 29 September 2022 to: 02 October 2022 | **Date of report**:<br>07 Sep 23 |
| **Shifts:**<br>15 | **Local contact(s)**:<br>Benoit Cordonnier | *Received at ESRF:* |


**Names and affiliations of applicants** (* indicates experimentalists):

Andrew Putnis, University of Münster; Fang Xia*, Khairul Khairudin*,

Murdoch University;

Joel Brugger*, Barbara Etschmann*, Yanlu Xing*, Jonathan Hamisi*,

Monash University


# Report:

## Aim

The aim of this experiment was to perform in situ X-ray μCT experiment (up to 200 °C, 20 MPa) to investigate the effect of pore fluid pressure, confining pressure, and temperature on the dynamics of reaction-induced porosity during the replacement of calcite by magnesite, brucite, gypsum, and anhydrite. This should help more accurate reactive transport modelling for minerals exploration, sustainable metal extraction, and CO2 sequestration.

## Experimental

The X-ray μCT study was carried out at the ID19 microtomography beamline in September 2022 – October 2022 using the Hades deformation rig. The calcite precursors were particle size of 53-150 μm, fitted in a Viton sample jacket with a 5 mm inside diameter and a length of 10 mm. Data were collected for calcite at two temperatures (60 and 200 °C) and two pore pressures (3 and 6 Mpa). We acquired tomograms with all three offered resolutions of 1x, 5x and 10x magnification (6, 1.25 and 0.6 μm/voxel side length, respectively). This is to observe the porosity changes within the particulates at a high resolution alongside the full view of the entire core sample with a low resolution.

During the original experimental time in Sep/Oct 2022, communications broke down between the beam line and the control system, this was not possible to fix during the remainder of our beam time. Ben generously used some of his commissioning time to complete this set of data collection in April 2023.

These in situ experiments were incredibly challenging. A data processing protocol has been developed to get total porosity as a function of reaction time, sample volume as a function of reaction time, reaction extent as a function of reaction time and spatial location of the core sample, and the mechanism of wormhole formation during the reaction. Data processing and interpretation is on-going.

The solid samples from the in-situ experiments were recovered and have been analyzed by X-ray diffraction to confirm mineral phases, scanning electron microscopy to examine texture, and energy dispersive spectroscopy to measure elemental distribution. These data will be used to assist the inteptretation of in situ experiments.

## Impact

In conjunction with ex-situ data, the data from in-situ experiments will be published and will form a major chapter of Khairul Khairudin's PhD thesis, which will be submitted in 2024.