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:** Novel time-resolved imaging of core formation processes in terrestrial planets	**Experiment number**:
Beamline:	**Date of experiment**: from: 22/10/2013 to: 29/10/2013	**Date of report**: 26/02/2014
Shifts:	**Local contact(s)**: Mohamed Mezouar	*Received at ESRF:*

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Report:

Aim: The aim of this series of experiments was to conduct novel *in-situ* tomographic imaging of partially molten geological material at extreme (PT) conditions analagous to the deep Earth. Melt migration and extraction from a solid matrix is a key part of many fundamental geological processes and has played an important role in planetary evolution, from core formation in the early solar system to modern day, large scale volcanism. In order to constrain these processes we need data on the rate at which partially molten systems segregate; in other words on the segregation veolocity of melts from a solid matrix in geologically relevant systems under the extreme conditions of planetary interiors. To date, assessment of melt segregation velocities is based on very poorly constrained and untested numerical models and limited analysis of quenched-experiments. Using the newly developed rotational tomography Paris-Edinburgh cell (rotoPEC), a combination of facilities available on the ID27 beamline (in-situ, large volume diffraction and tomogrpahic imagining under extreme conditions) and a novel experimental design we aimed to provide the first direct measures of melt migration velocities under geologically relevant conditions.

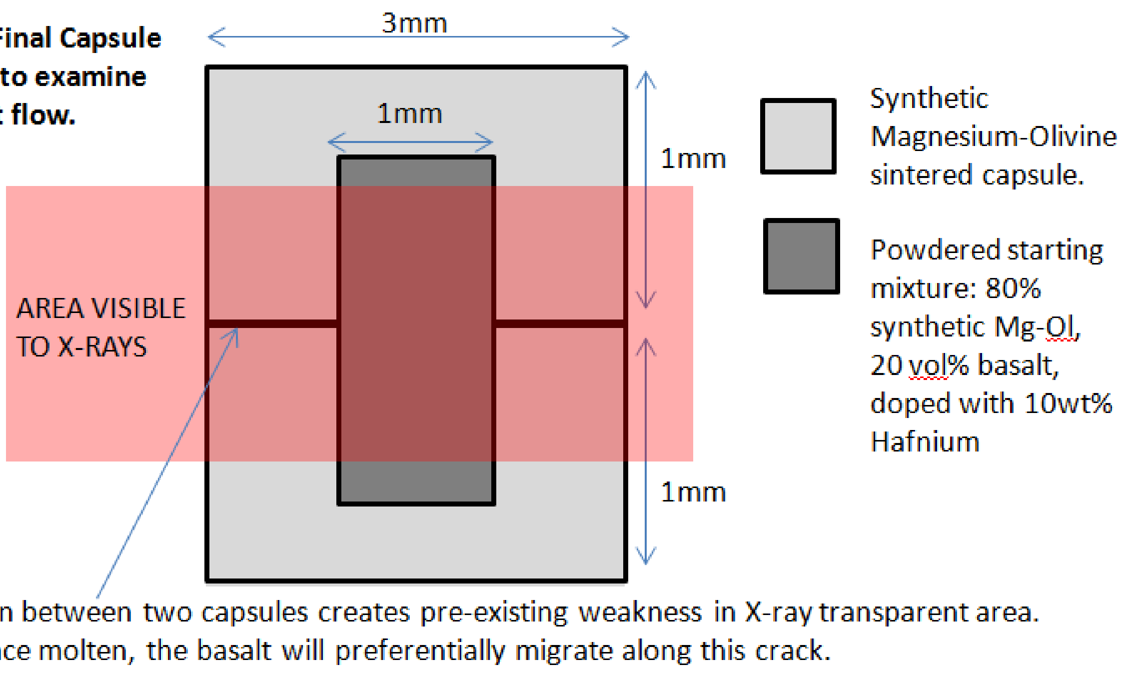
Methods: In-situ tomography at extreme conditions is made possible by the portable rotoPEC equipment which we installed in beamline ID27, allowing access to X-ray imaging and diffraction techniques (see ESRF ES33 and MI1086 reports). The rotoPEC is a uniquely designed Paris-Edinburgh cell with two opposing anvils that have their own independent motors – both rotate fully while samples are held under high temperature and pressure. This allows samples to be deformed (via torsion) and/or rotated in an X-ray beam passing through the transparent B-epoxy gasket and cell assembly to allow full computed X-ray tomography. Diffraction data can also be collected from samples throughout experiments.

The first half of beam time was used in developing an automated tomography system linking the ID27 beamline and software controlling the motors of the rotoPEC equipment. This allowed full automation of data collection for the present study but also for future imagining studies on ID27, thus representing a significant development of the capacity of ID27. Scripting routines now allow shutter control and data collection to be synchronised with step-wise rotation of the sample, increasing the resolution and data quality

in CT images. Initial scans demonstrate that this setup is capable of obtaining voxel resolution of 8 microns for samples at high-PT, which is at present only constrained by the quality of the detector.

In the original experimental design we used polycrystalline olivine+ Fe-S melt to examine melt migration rates related to core formation in the proto-Earth. Preliminary experiments demonstrated that grain-scale mobility of Fe-S melt was far lower than expected and melt velocities could not be assessed using the resolution available and limited run duration. Therefore we adjusted the experimental design as shown in **Figure 1**. We used a more mobile basaltic melt to ensure melt migration took place over the timescale of experiments. This melt was doped with Hf to greatly increase absorption contrast between melt and matrix which would otherwise remain difficult to segment in reconstructed images. A re-designed capsule assembly provided a conduit for melt migration (the join between capsule top and bottom) centred in the volume of the sample visible to X-rays. The width of this conduit during melt flow is significantly greater than melt conduits observed during grain flow, meaning that melt migration could more easily be constrained at the available sample resolution. Samples were pressurised then step-heated, and in-situ diffraction and power-temprature calibrations from previous experiments were used to determine run conditions and assess when melting of the basalt had occurred. Once molten, tomographic images clearly demonstrated extrusion of melt along the capsule join, and we were able to view the first melt movement directly at high P-T (2GPa/1400°C).

Fig.1 : Final Capsule Design to examine conduit flow.



Results:

Following some initial run failures during which experimental design was optimised we successfully imaged melt movement in-situ in 3D. The melt moved along the weak plane of the gap between the capsules, as expected, although only along one particular direction, possibly indicating melt migration following overpressure due to melting. We were able to capture the melt before and after the large-scale movement through high resolution CT imaging. The main melt movement occurred over a period of minutes, significantly shorter than the time required to obtain a full CT scan (~45 mins). However, radiographic images captured melt migration in full and can be used, in combination with full 3D CT models based on data obtained before and after melt movement, to determine melt migration velocity. An example of a 3D reconstruction of the basaltic melt (olivine capsule and matrix rendered transparent in this image) is shown in Figure 2, from a scan taken 2 hours after the majority of melt movement had taken place along the crack. In those areas where movement was taking place, the intensity of the reconstructed image is not as high as those areas in which there was little change in melt fraction. These are represented by the yellow and orange areas respectively. Data processing is on going (coupled with ex-situ characterisation of run products), and it

may additionally be possible to use the imaged blurring caused by melt motion to estimate melt migration velocities.

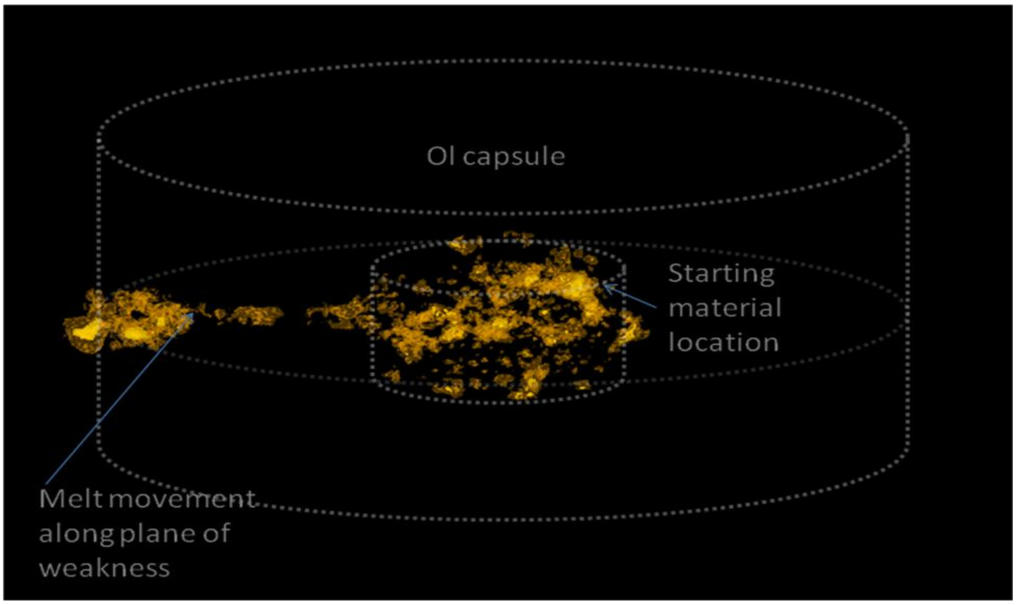


Figure 2 *3D reconstruction of the basaltic melt in the experimental sample from an* in-situ *tomographic scan. The majority of melt motion has taken place, but the material is still in motion shown by the reduced intensity of total attenuation in some areas (darker orange areas) compared to where the melt seems stationary (opaque yellow). The olivine capsule and matrix has been rendered transparent, but its original outline overlain for clarity. Once molten, the basalt moved very quickly (<1minute) along the previously existing plane of weakness (the join between two capsule halves). These images represent the first 3D in-situ images of basaltic melt at extreme temperatures and pressures.*

These are the first *in-situ* 3D images of melt migrating at high P-T and should yield the first preliminary data on melt segregation velocities. Rapidity of melt movement may imply fundamentally different mechanisms controlling melt movement compared to grain flow, although data first need to be scaled to conduit size. However, for basaltic melt, conduit flow is likely to be more applicable to melt movement in planetary interiors following mantle melting. Although we were only able to successfully image one sample during this preliminary beam time, we believe we have developed a method to effectively examine the behaviour of melt at *in-situ* high temperature and pressure conditions.

Future Work:

On-going development at ID27 and installation of the rotoPEC has demonstrated the potential for *in-situ* CT imaging and diffraction for providing new insight into key Earth processes and for characterising complex, multi-phase geomaterials under extreme conditions. Installation of the rotoPEC and development by ESRF staff of a coupled, automated control system for data collection represents a significant enhancement in the capabilities of the ID27 beamline and demonstrates the potential for future, state-of-the-art in-situ studies. Future studies will focus on fully characterising melt migration velocities and their dependence on P-T conditions and melt structure. We are also now testing, off-line, a modified capsule design which will also allow us to compare conduit- and grain-flow melt segregation velocities.