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.

	Experiment title: *A RedOx log of the Oceanic crust from Plagioclases Fe^{3+}/Fe^{2+} Micro-XANES in-situ measurements.*	**Experiment number**: *EC-623*
Beamline: *ID-21*	**Date of experiment**: from: *23/06/2010* to: *29/06/2010*	**Date of report**: *29/08/2010*
Shifts: *18*	**Local contact(s):** *Jakub Szlachetko*	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):
Lydéric France (CNRS-CRPG-UHP; Nancy, France)*

Report:

Scientific background:

The oceanic crust covers ~2/3 of the earth surface, and is generated at mid-oceanic ridges. Geophysical studies of fast-spreading ridges like the East Pacific Rise (e.g., Sinton and Detrick, 1992) have shown that the ridge axis is composed of a magma chamber at depth, displaying a thin, narrow, nearly continuous melt lens at its top, and of an upper part formed by the sheeted dykes and the volcanics that seems to be injected from this melt lens. The melt lens plays a key role in the genesis of oceanic crust, as it may be a source of melt for both the upper and the lower crust. It is also a major exchange interface between the ocean and the deep magmatic crust during seawater penetration since it is located at the root zone of the sheeted dike complex, where the hydrothermal convective system and the magmatic one meet, and act together (Nicolas et al., 2008). Assimilation of fragments of hydrothermalized dikes (and therefore of hydrothermal fluids) from above, within the melt lens have been documented (France et al., 2009). Such processes should result in redox variations of the magma as this parameter is related to the presence of water. These variations should consequently be recorded in the rocks crystallized from the melt lens, above in the upper crust and/or below in the lower crust. Constraining the redox conditions present during the crystallization of the lower crust rocks (from top to bottom: upper gabbros, foliated gabbros, and layered gabbros) should consequently help to decipher if the partially hydrous upper melt lens has a role in the lower crust genesis or not.

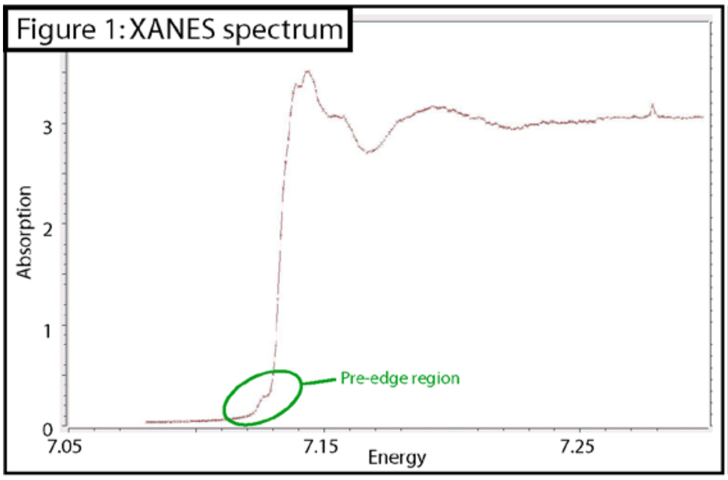
Objectives:

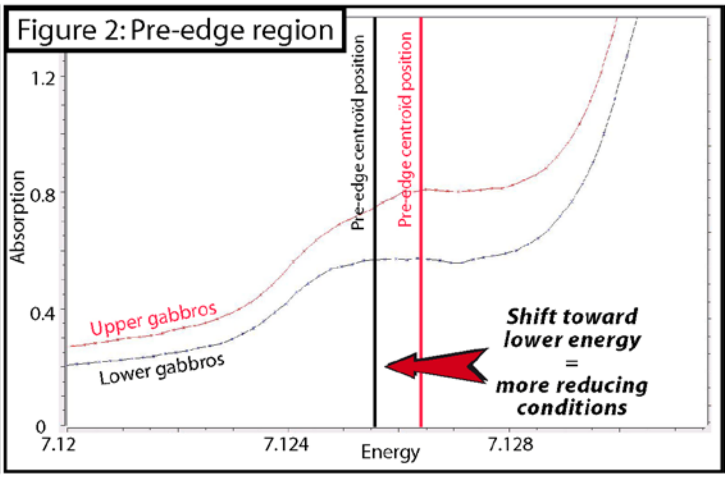
The objective of the experiment was to perform the first in-situ measurements of the Fe^{2+}/Fe^{3+} ratio in minerals (clinopyroxene and plagioclase) from the different lithologies of the oceanic crust. The elaboration of the first redox Log of a whole oceanic crust section should finally bring strong constraints on the oceanic crust accretion models. The comparison between samples from the Oman ophiolite (sultanate of Oman), i.e. a fragment of fossil oceanic crust tectonically brought on the continental margin, with samples from present-day oceanic crust (samples from Hole 1256D of the Integrated Ocean Drilling Program) should help to build a convincing model.

Experimental method:

Many of the rocks that we have analyzed are very small grained, and often largely altered, with even smaller residual fresh mineral parts (down to a few µm). Therefore, it was critical to use a method that allows very precise in-situ measurements on very small areas (~1µm²). Hence we have measured in-situ the Fe^{3+}/Fe^{2+} ratios using µ-XANES on the ID21 beamline of ESRF, the only experimental facility able to provide in-situ measurements with the appropriate spatial resolution. To proceed, µ-XANES data have been collected at the iron K-edge (7112eV) in the fluorescence mode. The pre-edge region of the XANES spectra are used as the centroïd position of this pre-edge is function of the Fe^{3+}/Fe^{2+} ratio (or redox value) in the measured sample. In details, a centroïd shifted toward higher energy corresponds to more oxidizing conditions.

Preliminary results:
As these experiments were only preliminary on ID-21 beamline, several tests had to be performed in order to get the best settings for a given measurement. A large number of standards have also been analysed in order to compare our experimental session with literature data. Finally ~10 shifts have been necessary all along the experiment for measurement improvements and standards analysis. This experimental session will be used as a basis for future experiments using µ-XANES on ID-21 beamline, and a largely shorter time will be therefore necessary to set the beamline. Finally clinopyroxenes, and plagioclases have been analyzed in ~15 samples from the Oman ophiolite.
The first result has been to show that µ-XANES measurements can be performed in-situ in minerals from rock thin-sections, and that reliable measurements can be obtained with a beam-size of 1µm² (Figures 1-2). We have also shown that the thickness of thin sections should be as small as possible as the beam penetration within the sample is important. The second important preliminary result has been to show that the redox signature of oceanic samples is heterogeneous, and depends on the sample position within the oceanic crust Log (Figure 2). The third, and important preliminary result has been to show that the centroïd position is at higher energy for upper gabbros, and at lower energy for lower gabbros, indicating that hydrous fluids (hydrothermal fluids) play an important role in magmatic processes at the top of fast spreading magma chambers. This result will be a key to constrain the accretion models of oceanic crust, and to constrain the magmatic-hydrothermal interactions.





Perspectives:
To finalise this project, samples representing the whole thickness of the magma chamber should be analyzed. The redox variation from the upper samples (crystallized from contaminated-oxidized melts) to the lower samples (crystallized from mantle reduced melts) has to be determined. More precisely, the foliated gabbros should be analyzed to constrain the redox conditions present during their crystallization. This last step of measurements will allow us to bring new strong constraints on how the oceanic crust is accreted. The comparison between fossil oceanic crust samples (Oman ophiolite), and samples from present day oceanic crust (IODP Hole 1256D) has furthermore only been started in this preliminary experiment, and more analyses should be obtained in order to valorised this part of the project.

References :
France et al., 2009 *Geochemistry Geophysics Geosystems* **10-10**: Q10O19, doi:10.1029/2009GC002652
MacLeod and Yaouancq, 2000 *Earth Planet. Sci. Lett.* **176**: 357-373, doi:10.1016/S0012-821X(00)00020-0
Nicolas et al., 2008 *Geochemistry Geophysics Geosystems* **9-5**: Q05001, doi:10.1029/2007GC001918
Nicolas et al., 2009 *Earth Planet. Sci. Lett.* **284**: 76-87, doi:10.1016/j.epsl.2009.04.012
Sinton & Detrick, 1992 *Journal of Geophysical Research* **97(B1)**: 197-216, doi:10.1029/91JB02508