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

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:** P-V equation of state of deep Earth's mantle-like glasses	**Experiment number**: ES-163
Beamline: ID13	**Date of experiment**: from: 07.05.2014 to: 12.05.2014	**Date of report**: 28.07.2014
Shifts: 15	**Local contact(s)**: Thomas Dane and Micheal Sztucki	*Received at ESRF:*

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Report:

The aim of this experiment is to measure in situ the density of amorphous systems that are composed of light element using a novel technique. The materials investigated are glasses with relevant silicate composition for the earth's interior with: SiO2, MgSiO3 and MgFeSiO3. Such data are crucial to understand the behaviour of matter under high pressure but moreover, phenomenon that occur in glasses can be also extrapolated to melt with the same composition and give more information about the past and actual picture of the Earth interior. Indeed, melting processes have a crucial importance in the evolution and differentiation of the Earth throughout its history. Deep magma ocean of 1000km or more have lead to the segregation of the core during the accretion stage of the Earth formation [1]. A basal magma ocean may have been sustained at the interface between the mantle and core due to an accumulation of high density melts or a hot core [2,3,4]. Still today, the fate, amount and behaviour of melts at this interface have not been clarified.

To achieve our goal, we adapt and develop a new technique for the diamond anvil cell (DAC) inspirited from large volume experiments [5]. The principle of the technique is quite simple and uses the absorption contrast of a focus X-ray beam. It requires the ability to measure precisely the dimension of the sample and get its absorbance at the different pressure conditions. To be able to record the absorbance of the sample, we confined the glasses in Beryllium gasket and squeezed them between the two diamond anvils. The sample was rastered in the beam to obtain maps through the anvils from which we could measure the path length of the sample, as defined by the rim of the sample in the gasket hole. The DAC was then rotated at 90 degrees to collect the absorbance of the sample through the Be gasket. At ID13, the beam was focus to 2x2 micrometers FWHM (bigger than requested in the proposal, see limitation section) and we could measure the sample absorbance up to 130 GPa with this technique with a final sample thickness of 5 microns. For higher pressure or thinner samples, a smaller beam is required.

The data are treated as followed:

- the distance of the sample in the gasket, which correspond to the path length of glass that is exposed to X-rays is measured through the maps collected through the anvils, figure 1.

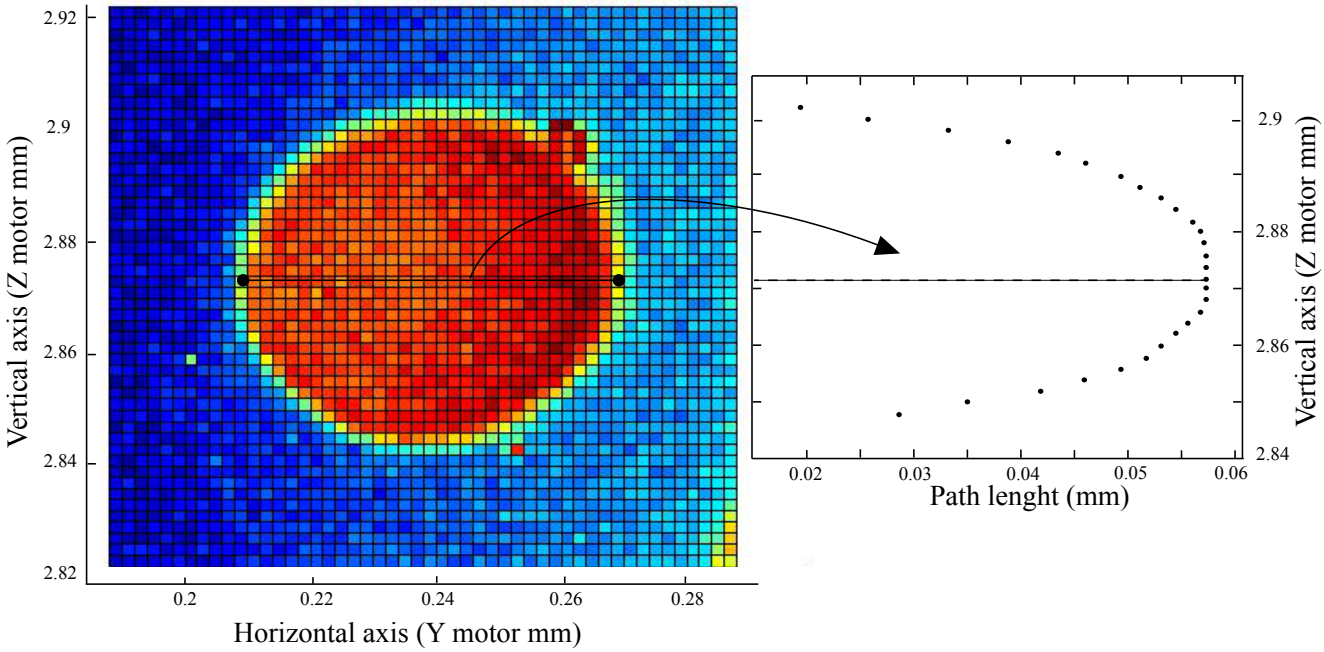

Figure 1. Absorption contrast map of the sample through the diamond anvils. From the map we can set the rim of the sample, which defines the dimension of the sample. The path length is then used in correlation to the absorption scanned at 90 degree to get the absorbance at the relevant PT conditions.

- The maps collected at 90 degrees are used to calculate the corresponding absorbance spectra for the different path lengths obtained in the previous step, figure 2.

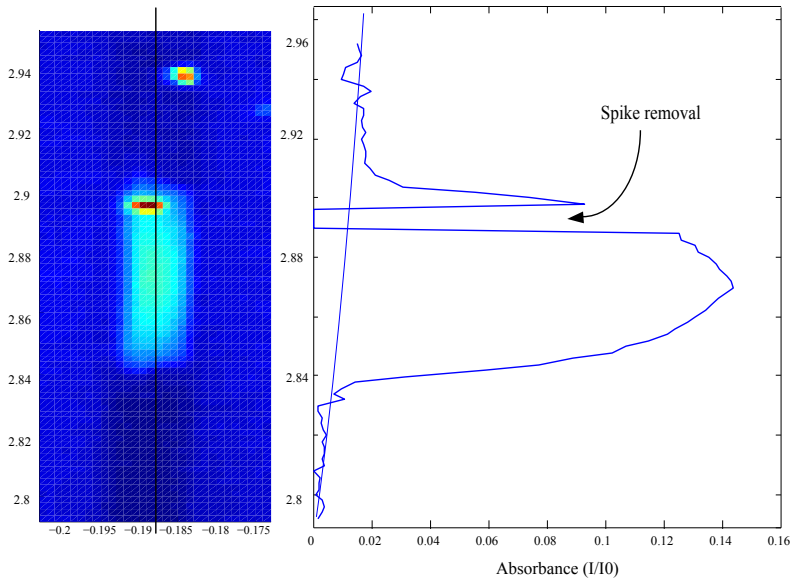

Figure 2. Absorption map and scan through the Be gasket. The spikes are removed from the absortion scan before data treatment. The absorption scan is then correlated with the path length obtained in the previous step to get the absorbance and finally the density

The combination of the path lengths (through the diamonds) with the variation of intensities (through the Be gasket) is used to calculate the absorbance of the sample with a beer-lambert expression:

$$- log_{10} (I / I_0) = (\mu_{HP} x) \quad (1)$$

I and I_0 being the the instensities of the beam after and before the DAC respectivly, x is the path length and μ_{HP} the absorbance of the sample at the P-T conditions of the experiment. Finally, the density is directly calculated through the following expression:

$$\rho_{HP} / \mu_{HP} = \rho_0 / \mu_0 \quad (2)$$

μ_0 is the absorption value for the glass of interest at room conditions measured on double polished thin sections of the staring material. In our case thin sections of 0.7 mm were scanned during the beam time to get this value. ρ_0 was measured after the beam time using a sink/float method on the exact same piece of glasses that were measured with the X-rays.

Results:

Here we show some preliminary results on MgSiO3 glass up to 15 GPa figure 3. A disc of 50 micrometres diameter and 13 micron thickness was cut with an FIB and loaded in the DAC with methanol/ethanol as pressure medium and compressed up to 15 GPa. The data are compared with measurement using Brilliouin spectroscopy in DAC [6] and with recent Ab initio calculations [7].

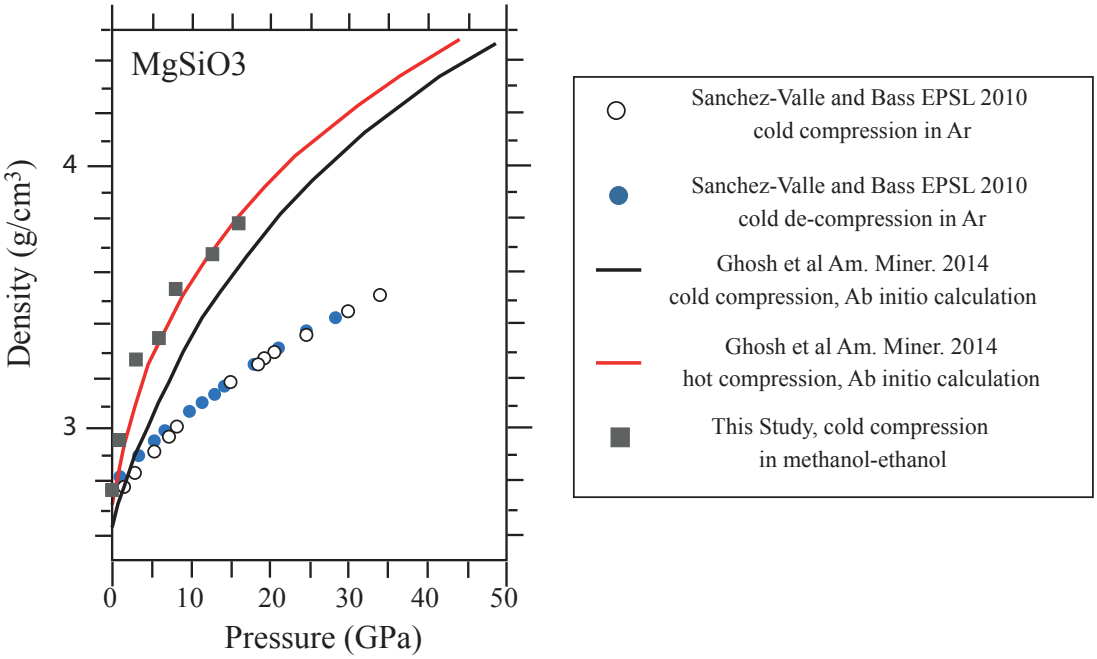

Figure 3. Results for sample 6 in methano-ethanol up to 15 GPa and compared with litterature data.

Main problems and limitations:

- We identify the main limitation of the technique to be the beam size. For this experiment the beam size at 13.2 keV was 2x2 FWHM which means a full beam of about ~ 3x3 micrometres. The ideal situation would be a full beam with a maximum size of 1x1 micrometres, meaning 0.5x0.5 FWHM at an energy of maximum 13 keV, lower would also improve the data quality.

- The possibility to place the sample on the rotation axis at the focal point of the X-ray, while requested several months prior to the beam time, would have saved us precious measurement time as well.

References:

[1] Rubie et al, EPSL, 2012
[2] Labrosse et al, Nature 2007
[3] Andrault et al, Nature 2012
[4] Nomura et al, Nature 2011
[5] Malfait et al, Nature Geoscience 2014
[6] Sanchez-valle et al, EPSL 2010
[7] Ghosh et al, Am. Mineral. 2014