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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.

ESRF	**Experiment title:** **Density of volatile-bearing, alkaline magmas determined by X-ray radiography**	**Experiment number:** HS 4483
Beamline: ID27	**Date of experiment:** from: 26/08/2011 to: 30/08/2011	**Date of report:** 7th Feb 2012
Shifts: 12	**Local contact(s):** Sylvain Petitgirard, M. Mezouar	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Summary:

The goal of the experiments was to provide in-situ density data and to determine the equation of state of dry and volatile-bearing (water and CO_2) phonolitic liquids for the pressure-temperature (P-T) range related to midocean ridges, subduction zones and continental rifting using X-ray absorption. During the allocated beamtime at ESRF ID27, experiments were performed using a panoramic Paris-Edinburgh Press (PE Press). We were able to systematically measure the absorption contrast of dry, hydrous and carbon-bearing phonolitic melts as a function of pressure (1 to 3.1 GPa) , temperature (1600 to 2000K) and water content of 0 and 4.7 wt% as well as a CO_2 content of 1.7 wt%. All samples had been synthesized in a piston-cylinder apparatus and characterized (compositional analyses, density measurements and FTIR analyses) prior the beamtime at ETH Zurich. The run products of the experiments have been analysed for water and carbon using FTIR spectroscopy and electron microprobe. Unfortunately, these analyses indicate that CO_2 and water were (partially) lost from the capsules during the experiments. As a consequence, we have successfully derived the equation of state from the measured X-ray absorption contrast for volatile-free phonolite liquids, but need additional experiments to do so for the volatile-bearing liquids. A proposal with this aim will be submitted for the next call for proposals.

Experimental Setup:

High pressure and temperature experiments were generated in a panoramic PE press, using 7 mm tungsten carbide anvils. The sample containers consisted of natural single crystal diamond cylinders (Almax Industries, Belgium) with $Ø_{in}$ = 0.5 mm, $Ø_{out}$ = 1.5 mm and a height of 1.0 mm. The capsule was sealed by Pt disks (200 μm) on both sides and enclosed in an hBN cylinder and two hBN caps were placed on both ends acting as pressure-transmitting medium. The furnace assembly is placed inside a standard 7 mm boron gasket. The absorption scans were collected with two ionization chambers and at a relatively low X-ray energy (Mo edge, 20.0 keV) for an optimal absorption contrast. Two different pressure marker (hBN and Pt) were used to determine the pressure and temperature by X-ray diffraction from the respective equations of state. After verifying the liquid state of the sample by X-ray diffraction, absorption scans of the assembly (Fig. 1a and b) were collected.

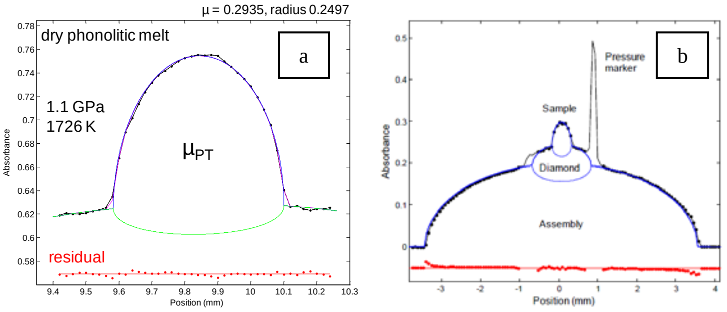

Fig. 1 Fit of absorption profiles a) Dry phonolite at 1.1 GPa and 1726 K as a function of the position of the sample. b) The density was determined from the X-ray absorption contrast between the sample and the diamond capsule.

Results and discussion:

We performed a total of 7 experiments on volatile free, hydrous and carbon-bearing phonolitic melt, 5 runs in June and 2 runs in August. We successfully and systematically collected absorption scans of dry, hydrous and CO_2-bearing melts in the PT range from 0.6 to 3.1 GPa and 1600 to 2000 K (Fig. 2). Contrary to previous experiments (report HS-4216), we had no problems with failed experiments and this allowed us to collect more data more efficiently. We fitted the dry density data using the 3rd order BM equation to get the EoS for the dry phonolitic melt and this equation allows us, for the first time, to predict the density of phonolitic liquids within 2%. The collected absorption scans for the hydrous and carbon-bearing phonolitic melts were of excellent quality, but we had a significant loss of volatiles, in contrast with the hydrous rhyolite samples (HS-4216). The FTIR spectra in Fig. 3a demonstrate the loss of water from 4.7 to 2.7 wt% after the experiment. We suspect the loss of water happens when a fluid phase is exsolved during heating/crystallization and expect to overcome this problem by applying a faster heating rate. The FTIR spectra in Fig. 3b indicate the complete loss of CO_{2mol} and CO_3^{2-} and addition of a significant amount of water. We suspect that the experiments with the carbon-bearing phonolitic melts were performed in a too reduced environment. A possible explanation could be the conversion of CO_2 into C by the following equation:

$4 H^+ + 2 CO_2 = 2 H_2O + C$. Thus, to avoid this problem in future experiments, we need to keep the system more oxidized, for example by replacing hBN by MgO and/or adding Fe_2O_3 to the assembly.

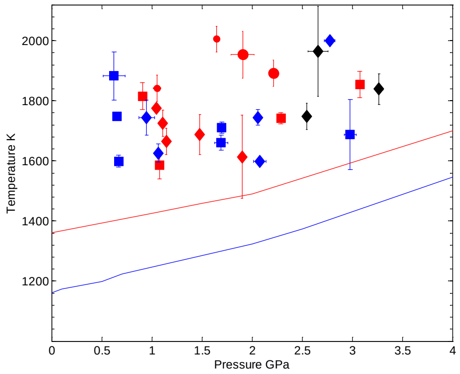

Fig. 2: PT diagram of experimental data for dry (red), hydrous (blue) and carbon-bearing (black) phonolitic melts in the range of 1-3.1 GPa and 1600-2000K. Red solid line indicates the liquidus T for the dry composition, blue for hydrous.

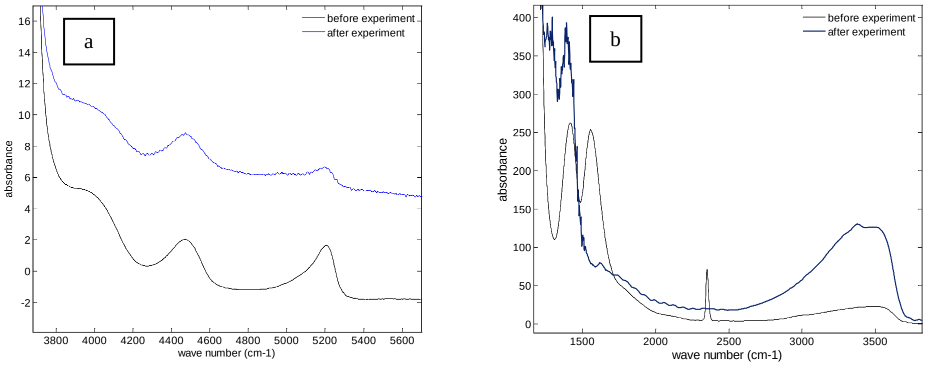

Fig. 3 FTIR spectra before and after the experiment a) of water bearing phonolite b) of carbon-bearing phonolite

Implications:

The performed experiments provide a first experimentally derived equation of state of dry phonolitic melts. We can now accurately predict the density of dry phonolitic melts in the range of 1-3 GPa and 1400K-2000K. With these data we can better understand magma migration in the continental rift zone settings. A paper on the dry phonolite density is in preparation for Geophysical Research Letters. In order to determine the partial molar volume of water and CO_2 more data are needed for volatile bearing melts.

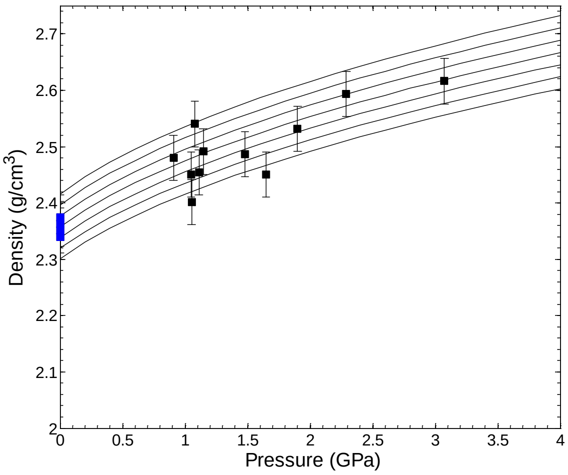

Fig. 3 Phonolite melt density as a function of pressure. Isothermal compression curves of dry phonolitic melt at temperatures of 1400K- 2000K are displayed. Black squares denote our data, blue squares represent the density at one atmosphere calculated using Lange&Carmichael (1987).