ESRF	**Experiment title:** High pressure density of volatile-bearing silicate melts determined by X-ray radiography	**Experiment number**: HS 4216
Beamline: ID27	**Date of experiment**: from: 02/12/2010 to: 07/12/2010 from: 26/01/2011 to: 28/01/2011	**Date of report**: 28th Feb. 2011
Shifts: 21	**Local contact(s)**: Sylvain Petitgirard	*Received at ESRF:*

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Report:

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) rhyolite and basalt liquids for the pressure-temperature (P-T) range related to mid-ocean ridges, subduction zones and crustal settings using X-ray radiography. During the allocated beamtime at ESRF ID27, experiments were performed using a panoramic Paris-Edinburgh Press (PE Press) and we were able to systematically measure the density of dry and hydrous rhyolite melts as a function of pressure (1 to 2.5 GPa) , temperature (1400 to 2000 K) and water content (0, 5 and 10 wt% water). All samples had been synthesized in a piston-cylinder apparatus and characterized (compositional analysis, density measurement and FTIR analysis) at ETH Zurich prior to the beamtime. Offline experiments were performed at ID27 prior scheduled beamtime to verify that no volatiles are lost during the experiments and to investigate the possible contamination of the sample through reaction with the diamond capusle. The run products of these test experiments have been analysed for water and carbon content by Secondary Ion Mass Spectrometry (SIMS) at ISEI, Okayama University (coll. Eizo Nakamura). The SIMS and electron microprobe analysis of the recovered run products of the online experiments are currently underway.

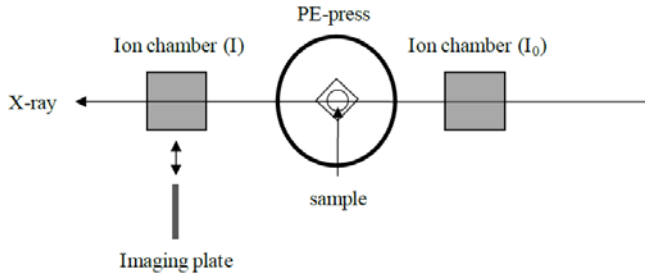


Fig.1: Experimental setup at ID27 using two ionization chambers, PE Press and an imaging plate (MAR345). The X-rays are detected by the second ionization chamber or the imaging plate for the absorption or diffraction measurements respectively.

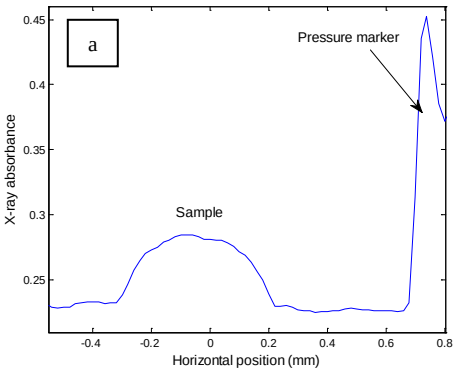
Setup and measurement at each PT condition:

The experimental setup for our experiments is illustrated in Figure 1. 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 lids and enclosed in an hBN cylinder and two hBN caps were placed on both ends acting as P- transmitting media. 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 frome the respective equations of state. At the beginning of the beamtime, we also investigated the possibility of X-ray diffraction on the silicate liquid, at high energy to extend the Q range, to obtain data on the melt structure, but this was not successful due to the low signal intensity, related to the weak interaction of the X-rays with the sample at this energy (Sm edge, 46.834 keV).

Experimental details and problems:

After verifying the liquid state of the sample by X-ray diffraction, absorption scans of the assembly (Fig. 2.a and b) were collected. The density was determined from the X-ray absorption contrast between the sample and the diamond capsule (Figure 3.b). The fitting procedure to extract the X-ray absorptivity and density of the sample from the absorption scan has been developed and we are currently in the process of fitting all of the collected scans.



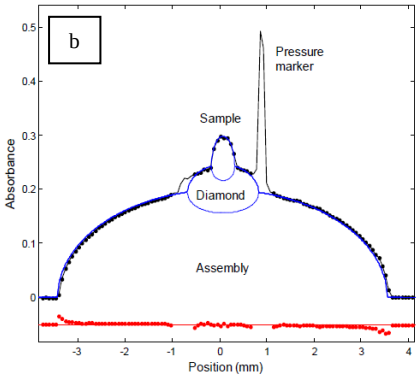


Fig. 2 a) Absorption profile of a dry rhyolite at 1.1 GPa and 1558 K. PT marker is Pt and hBN. b) Fit of an absorption profile.

We performed a total 22 experiments on volatile-free and hydrous rhyolites and basalts, 13 runs in December 2010 and 9 runs in January 2011. We successfully and systematically determined the density of dry and hydrous rhyolite melts in the PT range from 1-2.5 GPa and 1400-2000 K (Fig. 3). The measurements for the basalt melts, which have a higher liquidus temperature and lower viscosity, had a higher proportion of failed experiments (>50%), compared to the rhyolite melts. As a result, no systematic density data could be collected for the basalt composition. The exact reason for the failed experiments, during which the gasket breaks away to one side, is unclear at the moment, but may be related to the high temperature and/or the melt escaping from the capsule. With the goal of increasing the success rate, we modified the sample assembly by adding a second Pt disk to improve the seal and prevent the melt from escaping. Unfortunately this did not work as intended. A second problem was the crystallization of the melt during heating and the high temperatures needed to remelt the sample completely. This was particularly difficult for the high viscosity, dry rhyolite samples at pressures above 1.5 GPa, because the required high temperatures (≥ 2000 K) resulted in the melting of the platinum PT marker/lids. Therefore, data on the dry rhyolite melts have only been collected up to 1.5 GPa.

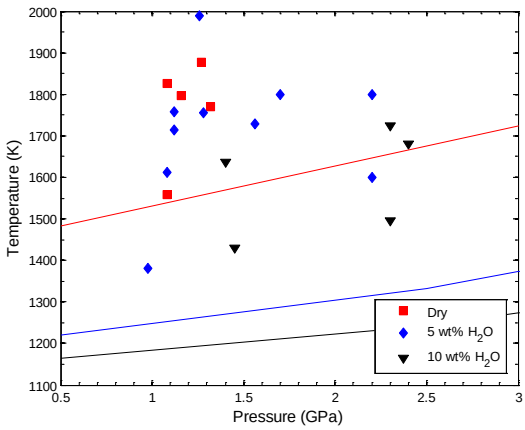


Fig. 3: PT diagram of experimental data for dry and water-bearing rhyolite melts in the range of 1-2.5GPa and 1400-2000K. Solid lines indicate the liquidus T for each composition.

Results expexted and reached:

The performed experiments will provide the first experimentally derived equation of state of dry and H_2O-bearing rhyolitic liquids. With this equation of state, we can predict the density of dry and hydrous rhyolite melts from the surface down to lower crustal PT conditions. The results of this study will be presented at the Goldschmidt conference 2011 in Praque, Czech Republic and a manuscript is being prepared for publication. However, in order to construct a general model of volatile-bearing magma density applicable to a wide range of magma compositions, additional density data is required. Thus, encouraged by the succesful application of the X-ray absorption measurements to rhyolite magmas, we request a continuation of HS-4216 to investigate the partial molar volume of water and CO_2 for different magma compositions (nephelinites and phonolites).