Europlanet TNA Report

PROJECT LEADER

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COLLABORATORS

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(MISS student)	
Date of TNA visit:	10-14 (11-13) September 2012
No. of access days:	3,5
No. of days stay:	5
Host laboratory:	The Cameca 1270 Ion Microprobe
	Centre de Recherches pétrographiques et Géochimiques
	(CRPG) Vandoeuvre - les - Nancy, France
Reimbursed	Yes

Project Title -

Quantitative determination of H_2O , CO_2 and trace element concentrations in melt inclusions in olivine from Shatsky Rise (IODP Expedition 324)

Scientific Report Summary.

(plain text, no figures, <u>maximum 250 words</u>, to be included in database)

Due to the analytical problems, the measurements of H₂O and CO₂ in melt inclusions hosted in olivines from the Shatsky Rise ocean Plateau have not been conducted successfully. See full report below.

Full Scientific Report on the outcome of your TNA visit

Approx. 1 page

In 2009, IODP Expedition 324 cored at 5 sites into igneous basement of the Shatsky Rise ocean plateau, Pacific Ocean. Cores from the site U1349 recovered an interval of picritic samples which contain olivines, survived nearly pervasive alteration. In few samples relatively fresh olivines with glassy and partly crystallized inclusions have been found (Almeev et al., 2011, Prague, Goldschmidt Conference). These melt inclusions in olivine are of particular interest, since they host ultra-depleted primitive mantle-derived

melts and can also provide information on pristine pre-eruptive volatile abundances in LIP basalts.

The Cameca 1270 Ion Microprobe (Nancy) was used to analyze carbon and water concentrations in basaltic glass inclusions in olivine crystals from the Shatsky Rise ocean plateau. Glass inclusions were homogenized at 1150°C and exposed to the surface of the olivine crystals by polishing. Olivine grains with the melt inclusions and pieces of basaltic glasses with known H_2O and CO_2 concentrations were mounted into an inch-size Al-ring using special epoxy (mixture of Araldite D and hardener HY956) and coated with Au. Prior to measurements on the Cameca 1270 Ion Microprobe, samples and standards were held in a vacuum gas chamber for degassing and the analyses were started when the vacuum on the level of 10^{-8} to 10^{-9} Torr was achieved. Cs⁺ beam was used to produce negatively charged secondary ions: C, OH and O. The glasses were sputtered with a 10 kV Cs⁺ primary beam of the 10-15 nA intensity with a size of approximately 20x20 µm. The duration of a single measurement with 16 cycles was about 15 minutes.

During analytical sessions a series of representative standards were measured for calibration of carbon and water concentrations (from 0.77 to 5.7 wt.% H_2O and from 0.0114 to 0.3277 wt.% CO_2 ; Shishkina et al., 2010).

The quantification of C and H concentrations was based on calibration lines that show linear correlation between known H_2O and CO_2 contents and OH/O and C/O ratios, respectively, for the whole range of analyzed concentrations in the standard glasses. It must be noted, however that the analytical technique was not completely successful, because the intensity for both OH/O and C/O showed continuous drift of the obtained signal even after 16 cycles of the analysis. The reason for that might be a contamination of the surface with organic material or continuous evaporation of epoxy in the sample chamber. Another indication for such contamination is the relatively high detection limit for both H and C, as evidenced by the intersection of the calibration curve with the concentration axis (at about 0.7 wt.% for H_2O and 195 ppm for CO_2). These observations indicate that improved analytical approaches should be used in future, e.g., organic-free matrix and "pre-cleaning" of the surface with large beam and long exposure time with consequent analysis using smaller beam size.

Unfortunately, due to the analytical problems, after 3 days of measurements on Cameca 1270 Ion Microprobe, we were able to measure only H_2O concentrations in 4 melt inclusions and 5 glass rinds, representing natural samples of the pillow lavas from the Shatsky Rise. The measured H_2O contents range from 0.3 to 0.7 wt% and from 1 to 1.8 wt% in melt inclusions and basaltic glasses respectively. However, the values of H_2O determined by SIMS in glasses are not in agreement with H_2O determinations conducted on the same samples by FTIR (determined in the Institute of Mineralogy, Hannover). Unfortunately melt inclusions and glass rinds were measured during different sessions (different days) and calibration line from one set of glass samples (with corrections after FTIR) can not be applied to the second set of glass inclusions. In addition, we failed to produce a reliable calibration line to determine CO_2 .

Thus, the results obtained so far are not satisfactory and new improvements of sample preparation and analytical approaches are required for precise and accurate quantitative determination of both H_2O and CO_2 concentrations in silicate glasses. We have an agreement with the Lab representatives (Dr. E.Deloule, DR-CNRS-CRPG) to perform additional analytical session in March 2013. The new set of olivine crystals and glass standards have been already prepared. All samples were mounted into the ring of indium, used as a carbon-free matrix to hold samples.

Please include:

- <u>Publications arising/planned</u> (include conference abstracts etc)

Almeev, R.R., Portnyagin, M.V., Wengorsch, T., Sano, T., Natland, J.H. and Garbe-Schönberg, D., 2011. Highly depleted melt inclusions in olivine from Shatsky Rise. (paper in preparation); results were partly presented at 2011 Goldschmidt Conference in Prague (abstracts published in Mineralogical Magazine, 75(3): 426.)

<u>Host approval</u> The host is required to approve the report agreeing it is an accurate account of the research performed.
Host : Etienne Deloule, DR-CNRS-CRPG