EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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:** Helical X-ray fluorescence tomography for the determination of the 3Dinternal elemental distribution inthe micron-scale	**Experiment number**: MI713
Beamline: ID22	**Date of experiment**: from: 30/06/2004 to: 06/07/2004	**Date of report**: 24/07/2007 *Received at ESRF:*
Shifts: 12	**Local contact(s)**: Andrea SOMOGYI	
Names and affiliations of applicants (* indicates experimentalists): **Dr Bruno GOLOSIO* Dr Antonio BRUNETTI, Dr Jean CAUZID*, Dr. Pascal PHILIPPOT, Dr Alexandre SIMIONOVICI*, Dr Andrea SOMOGYI***		

Report:

Chosen sample: fluid inclusions

Fluid inclusions are small quantities of fluids trapped in minerals. A single inclusion traps a few cubic micrometers of fluid, which migrates in km^3 volumes through the Earth's crust. Usually, variability between inclusions must therefore be smoothed out to allow extraction of information about past fluid-rock interactions from such samples. This is fulfilled through analysis of statistically relevant series of fluid inclusions. Quantifying series of fluid inclusions using Synchrotron Radiation –induced X-Ray Fluorescence (SR-XRF) was the aim of other experiments (ME401, ME824), which led to a series of publications (Cauzid et al., 2004; Cauzid et al., 2006; Cauzid et al., submitted; Foriel et al., 2004; Thébaud et al., 2006). In some specific cases, fluid inclusion generations are well constrained and no systematics are required on them, but some unknowns remain on the chemical speciation in the fluid, which can be accessed directly through spectroscopy (experiments ME824, 30-02-748, 30-02-751, 30-02-812) or inderectly through the spatial repartition of selected elements inside individual fluid inclusions.

This spatial repartition has been approached through the SR-XRF mapping of inclusions. However, SR-XRF mapping integrates the fluorescence signal over the sample thickness and some uncertainty may remain (Figure 1). Performing X-ray Fluorescence Computed Tomography (XFCT) with an helical mode enables reconstructing the 3D distribution of elemental concentrations at a micron scale in selected minerals hosting fluid inclusions. Thus uncertainties linked with SR-XRF mapping are solved. The chosen samples were extracted from three locations: the Creede epithermal deposit (Colorado), a pegmatite from Madagascar and the Yankee Lode of the Mole Granite (Australia). Quartz crystals from these three locations were prepared as needle of ~150*150 μm^2 section hosting one to ten fluid inclusion.

Results

Amongst the three samples two could not provide elemental reconstructions due to the following: in the Madagascar sample, the fluid inclusion hosted CO_2 liquid and vapour that were heated by the beam. The instability of the CO_2-rich phases led to fluid motion during the data acquisition, which is incompatible with a tomographic reconstruction. In the sample from Creede (Colorado), most concentrations in metals were too low to be resconstructed. The last sample from the Mole Granite provided exceptionnaly interesting results.

Elemental concentrations were high enough for 3D reconstruction. Cu, Fe and As distributions are presented in Figure 2. Cu concentrates in 3 inclusions identified as “vapour”, Fe concentrated in the inclusion identified as “liquid” and As has an intermediate behaviour. This confirms result obtained using destructives methods (Laser-Ablation Iductively Coupled Plasma Mass Spectrometry) and ensures that the Cu-rich crystal associated with “vapour”inclusions is really included in those inclusions, thus has to be taken into account for calculating the initia vapour composition.

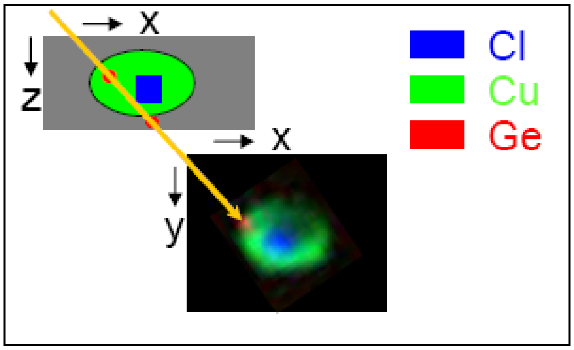

Figure 1:
2D mapping integrates the signal over the entire thickness of a sample. From the Cl, Cu and Ge maps of a fluid inclusion hosted in quartz (bottom) it cannot be known if the Ge signal is coming out of a concentrated point situated inside or below the fluid inclusion (top). The Ge signal could be generated in any point along the beam path represented by the gold arrow.

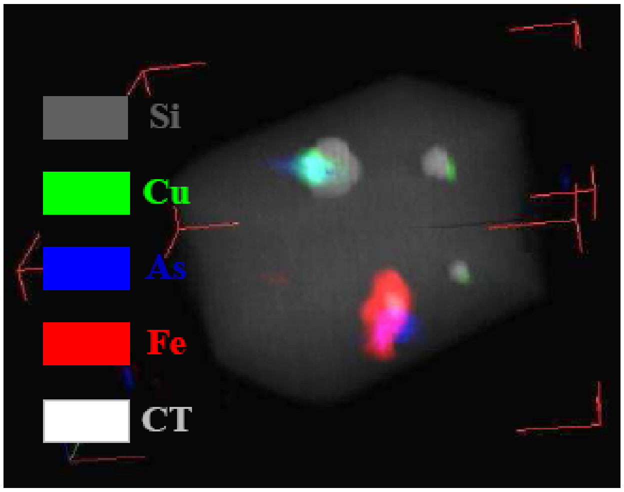

Figure 2: From a 3D reconstruction, there is no doubt that the Cu signals emerge from inside the 4 inclusions found into this quartz crystal.

Reference resulting from this experiment

Cauzid, J., Philippot, P., Bleuet, P., Simionovici, A., Golosio, B., Somogyi, A. 3D imaging of vapour and liquid fluid inclusions from the Yankee Lode deposit, Mole Granite, Australia using helical fluorescence tomography. Spectrochimica Acta part B, accepted

Related references from experiment performed at the ESRF

Cauzid, J., Philippot, P., Somogyi, A., Simionovici, A. et Bleuet, P., 2004. Quantification of single fluid inclusions by combining Synchrotron Radiation-Induced μ-X-ray Fluorescence and Transmission. Analytical Chemistry, 76: 3988-3994.

Cauzid, J., Philippot, P., Somogyi, A., Ménez, B., Simionovici, A. et Bleuet, P., 2006. Standardless quantification of single fluid inclusions using synchrotron radiation induced X-ray fluorescence. Chemical Geology, 227: 165-183.

Cauzid, J., Philippot, P., Martinez-Criado, G., Ménez, B., Labouré, S. Contrasting Cu-complexing behaviour in vapour and liquid fluid inclusions from the Yankee Lode deposit, Mole Granite, Australia. Submitted to Chemical Geology

Foriel, J., Philippot, P., Rey, P., Somogyi, A., Banks, D.A. et Ménez, B., 2004. Biological control of Cl/Br and low sulfate concentration in a 3.5-Gyr-old seawater from North Pole, Western Australia. Earth and Planetary Science Letters, 228: 451– 463.

Thébaud, N., Philippot, P., Rey, P. et Cauzid, J., 2006 Composition and origin of fluids associated with lode gold deposits in a Mesoarchean greenstone belt (Warrawoona Syncline, Pilbara Craton, Western Australia) using synchrotron radiation X-ray fluorescence. Contributions to Mineralogy and Petrololgy, 152: 485-503.