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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:** Speciation of arsenic and composition of fluid inclusions from gold deposits	**Experiment number**: 30-02-812
Beamline: BM30B	**Date of experiment**: from: 18 April 2007 to: 01 May 2007	**Date of report**: 26 July 2007
Shifts: 18	**Local contact(s)**: Dr. Denis TESTEMALE	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Aim of the experiment

The chemical composition of fluid inclusions found within gold lodes provides a unique window into the mineralization processes. In order to relate the elemental concentrations measured in fluid inclusions with the mineral assemblages (i.e. mineral solubility) and to imply the mineralizing process (e.g. phase separation, fluid-fluid and fluid-rock interaction), knowledge of the element speciation is essential at elevated temperatures. The aim of this study was to obtain in-situ X-ray absorption spectroscopy (XANES and EXAFS) data at the As K edge (11867 eV) in order to determine As speciation at ambient to homogenisation temperatures advancing previous experimental techniques which quantified the chemical composition of fluid inclusions using SR-XRF(ME824, 30-02-748, 30-02-751).

Experimental method

Doubly polished quartz crystals hosting fluid inclusions were held in Linkam THMSG600 heating-freezing stage installed vertically on the new microfocus end-station on BM30B (see experiments 30-02-748 and 30-02-751). At each temperature step, As speciation in target fluid inclusions was measured to check speciation changes in the fluid incluison. XANES and EXAFS spectra were collected for 18 fluid inclusions (5 – 35 wt.% equivalent NaCl) from three gold deposits (Australia, Madagascar, Italy). Arsenic concentrations of fluid inclusions measured by PIXE, SR-XRF or crush leach method ranged from 147 ppm to above 1 wt%. Data was recorded in the fluorescence mode using a Vortex Si-drift detector from 11500 eV to 12500 eV with an X-ray flux of 10^{10} photons/second.

Results

We reached a beam size of 15 x 15 µm, ideal for in situ XAFS analysis of fluid inclusions and succeeded in collecting spectra up to 235˚C, above this temperature inclusions tended to decrepitate.

It was possible to collect good quality EXAFS data (up to k ~ 10 Å-1) on the As-rich inclusions. Preliminary analysis of the XANES data indicates that:

- Photo-oxidation of As(III) to As(V) is a major problem, that can be easily monitored by XANES spectroscopy. All inclusions were totally transformed to As(V) after ~1 hour beam exposure. By reducing the flux to ~10^9, it was possible to collect EXAFS data on the Madagascar sample with little As(V). In dilute samples (e.g. typical low salinity, <100 ppm As), oxidation was extremely rapid.
- The As chemistry in the Australian sample is very complex. At room-T, the brine contains both As(III) and As(V), as well as inclusions of a mineral containing reduced As (most probably an As-sulfide).

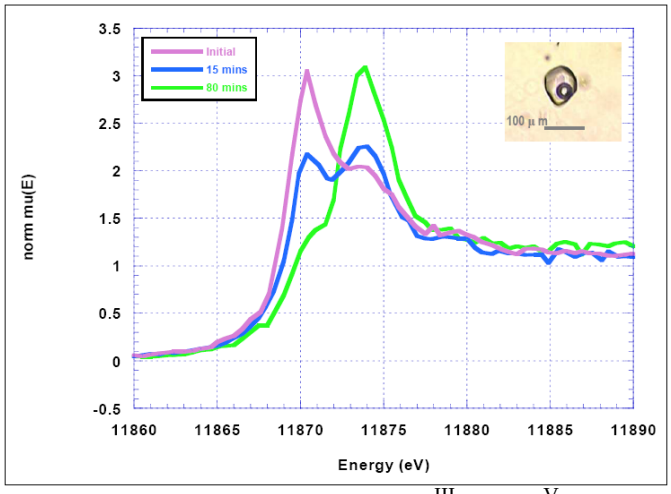

Figure 1: photo-oxidation of As^{III} to As^{V} in As-rich fluid inclusions from Madagascar (>1%wt As)

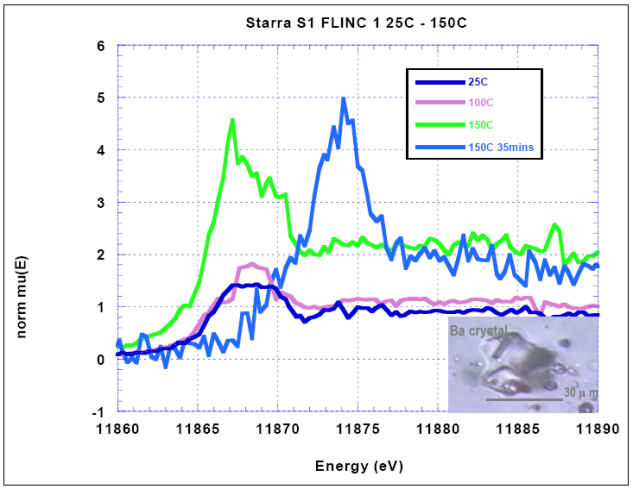

Figure 2: As is found as reduced species at room T in the Australian fluid inclusions .(~600ppm As) and changes to As^{V} at 150 °C, probably through photo-oxidation

From the EXAFS data analysed, we aim to determine the local structure of arsenic complexes in these fluid inclusions and compare them directly with the speciation of As from synthetic experiments at high T-P.

References resulting from this experiment

James-Smith, J., Brugger, J., Cauzid, J., Hazemann, J.-L., Liu, W., Proux, O., Testemale, D., Philippot, P. and Williams, P. 2007. Arsenic Speciation in fluid inclusions from Gold Deposits using X-ray Absorption Spectroscopy from ambient to homogenisation temperatures. European Current Research on Fluid Inclusions (ECROFI-XIX). University of Bern, Switzerland, 17–20 July, 2007. Abstract Volume, p.62

Cauzid, J., Brugger, J., Hazemann, J.-L., James-Smith, J., Liu, W., Philippot, P., Proux, O., Testemale, D., 2007. In-situ determination of arsenic speciation in natural fluid inclusion from Au-rich quartz veins. Goldschmidt Conference 2007. Cologne, Germany, 19-24 August 2007