ESRF	**Experiment title:** Micro-EXAFS experiments: tests on heterogeneous fluid inclusions at 300K	**Experiment number**: 30-02-748
Beamline: BM30B	**Date of experiment**: from: 13 March 2006 to: 22 March 2006	**Date of report**: 26 July 2007
Shifts: 24	**Local contact(s)**: Dr. Vivian NASSIF	*Received at ESRF:*

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Aim of the experiment

On a technical point of view, the aim of this experiment was to test in user mode the new micro-focus set-up of the FAME beamline. The set-up had not been fully commissioned before the experiment due to unexpected depays in hardware delivery. One mirror focusing only had been tested (Palancher *et al.* 2007).

The scientific aim of the experiment was to characterize the local order around copper in the heterogeneous fluid inclusions. Fluid inclusion were chosen because their size is similar to the expected beam size in the focal plane. Moreover, fluid inclusions are complex chemical systems requiring more information than available from XANES spectra. EXAFS data could help to unambiguoulsy precise the nature of the copper ligands (Cu-Cl or Cu-S bonds) in the fluid inclusions. The knowledge of the copper ligands is of critical importance to understand the ore deposition processes as chlorine and sulfur compounds will not interact similarly with the host rock other fluids encoutered during the hydrothermal fluids motion. At room temperature, a better understanding of the Cu local environment is required as XANES spectra obtained in previous studies are similar to a hydrated Cu2+ species whereas fluorescence imaging data demonstrated that most of the Cu is concentrated in a Cu-Fe-As-S-rich mineral. A thorough understanding of Cu behaviour in hydrothermal fluids requires temperature dependant analyses, which was the aim of other experiments (experiment 30-02-751 and 30-02-812).

Results

This experiments enabled testing the Kirkpatrick-Baez mirrors system. The smallest beam obtained during the experiment was below the expected $10*10\mu m^2$ (HxV, FWHM) spot size in the focal plane (Figure 1). A first set of tests was also performed at the Cu K-edge on vapor-phase fluid inclusions at room temperature (Figure 2). These experiments have allowed i) to precise the design of our new set-up (mainly the best way to define the secondary source by the actual optic elements), ii) to measure the size of the micro-focused beam, iii) to gather valuable experience on the visualisation requirements of the sample that have to be achieved and finally iv) to check if the stability of the optic elements allows to maintain the position of this micro-focused spot constant when the energy changes.

Beam stability in position and flux was not satisfactory enough to perform an EXAFS scan (1keV range) in the energy range targetted (4 to 22 keV). The main source for this is the temporary table on which the micro-focus set-up was installed until the new granite table will be received. This has been solved since the experiment was performed (see experiment reports 30-02-751 and 30-02-812).

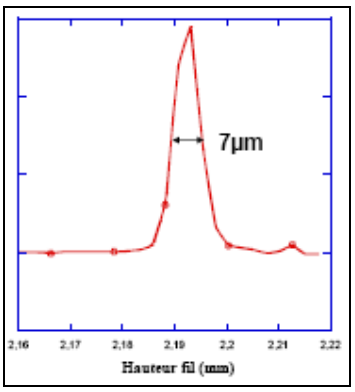
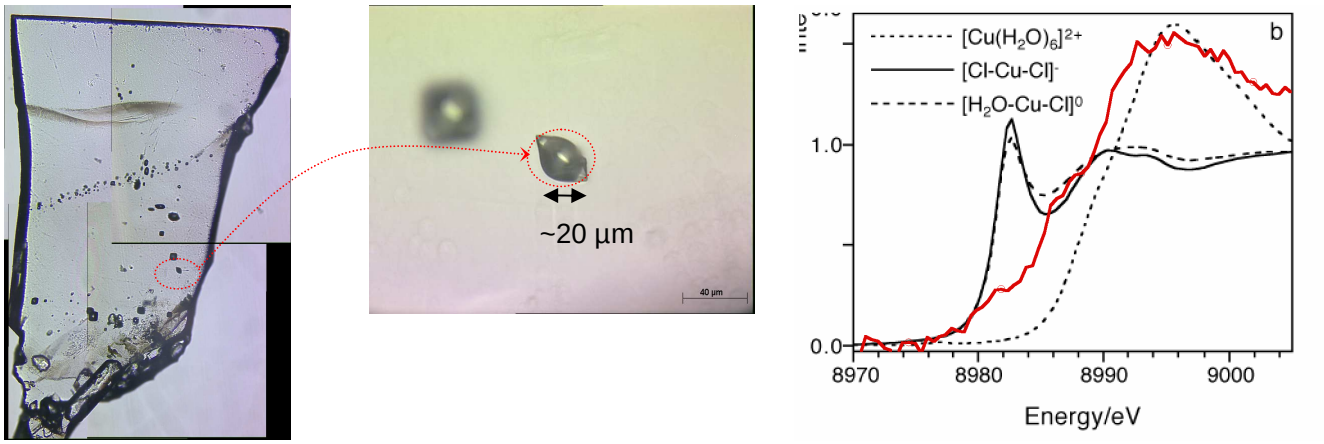


Figure 1 (left): spot size defined as the FWHM of the fluorescence signal given by scanning a Cu wire (

Figure 2 (below): first XANES spectrum obtained at the Cu edge on a vapour inclusion (middle) in a quartz crystal (left). Inclusion is 20 µm in size and hosts various phases: vapour, liquid and a daughter crystal. The obtained spectrum (right, red) is compared to spectra obtained on similar inclusions at various temperatures (Mavrogenes *et al*., 2002).



As the beam flux and position were not optimal and the EXAFS could not be reached, the scientific aim could not be attained. More data was gather in the following experiment (30-02-751).

References resulting from this experiment

Proux O., Nassif V., Palancher H. Lahera E., Prat A., Braillard A., and Hazemann J.-L., "New research improvements on the CRG-FAME XAS beamline at ESRF", *5th Synchrotron Radiation in Materials Science* (SRMS5), Chicago (30 Juillet - 2 Août 2006)

Palancher H., Proux O., Nassif V., Lahera E., Prat A., Braillard A., Cauzid J., Del Net W. et Hazemann J.-L., "Nouveaux développements instrumentaux sur la ligne CRG-FAME" *21ème Réunion des Sciences de la Terre*, Dijon (4-8 décembre 2006)

References cited in this report

Mavrogenes, J.A., Berry, A.J., Newville, M. and Sutton, S.R., "Copper speciation in vapor-phase inclusions from the Mole Granite, Australia", *American Mineralogist*, **87** (2002) 1360-1364.

Palancher H., Martin P., Nassif V., Tucoulou R., Proux O., Hazemann J.-L., Tougait O., Lahéra E., Mazaudier F., Valot C. and Dubois S., "Evidence for the presence of UMoAl ternary compounds in UMo/Al interaction grown by thermal annealing: a coupled µ-XRD/µ-XAS study", *Journal of Applied Cristallography* (2007) accepted