	Experiment title: **XANES micro-mapping at the Fe *K*-edge: Application to a metamorphic rock thin section**	**Experiment number**: ME-1116
Beamline: ID 24	**Date of experiment**: from: 23/03/2005 to: 29/03/2005	**Date of report**: 05/09/2005
Shifts: 18	**Local contact(s)**: Sakura PASCARELLI	*Received at ESRF:*

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Report:

This experiment was originally planed to be performed later, however, for technical purposes (i.e., major incident in the electron storage ring of the ESRF), we decided to replace the experiment entitled "Dissolution-diffusion-crystallization kinetics of silicate glasses and minerals at hydrothermal P-T conditions" by the present one: "XANES micro-mapping at the Fe *K*-edge: Application to a metamorphic rock thin section".

The goal of the experiment consisted first of the analysis of a natural rock thin section by micro-XANES spectroscopy in order to highlight and characterize the "theoretical" variations of iron redox due to hydrothermal fluid alteration. However, to characterize such a complex geological sample, mapping analyses usually provide much more information compared to punctual analyses (e.g., diffusion processes, mineralogical reactions).

Consequently, micro-XANES mapping including a few thousands of XANES spectra has been performed for the first time, on the ID24 beamline of the ESRF. Such an experiment needs high technical specificities, and only the dispersive EXAFS beamline ID24 round up these conditions at the moment. First of all, a micro-focused, with a stable and high-flux, X-ray beam was required. Then, the fluorescence detection mode was needed because of the energy involved (i.e., 7112 eV) and the thickness of the so-standard type of rock sample (i.e., 30 μm on a 1 mm glass sample-holder). This type of

detection mode was possible thanks to the use of the so-called "Turbo-XAS" experimental setup. Finally, a very fast acquisition time was required to perform the experiment in rational allotted time, and only a dispersive beamline can provide this because all the optical elements remain static (including the bended-monochromator) during data collection.

The horizontal and vertical sampling was done every 5 μm with a beam size of 10 by 10 μm, so that the collected images could be deconvoluted. Around 3000 XANES have been recorded (see Figure 1a) on a 150 by 380 μm surface (see Figure 2a). One important part of this study was the automation of data reduction. Consequently, we developed a software in order to perform an automatic fitting procedure, and to normalize the XANES spectra (see Figure 1b). It was then possible to reconstruct the images based on the different characteristics of the fits and the normalized spectra (see Figure 2b, 2c and 2d). These results are currently submitted for publication.

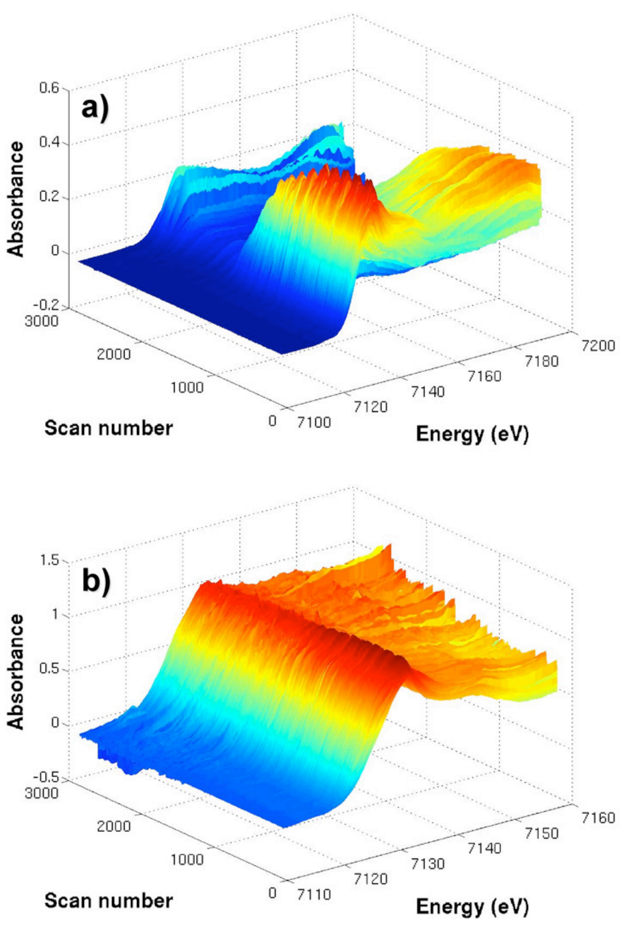

Figure 1: XANES spectra collected in the fluorescence mode at the iron K-edge for the mapping of a natural rock thin section. a) before normalization; b) after automatic normalization procedure.

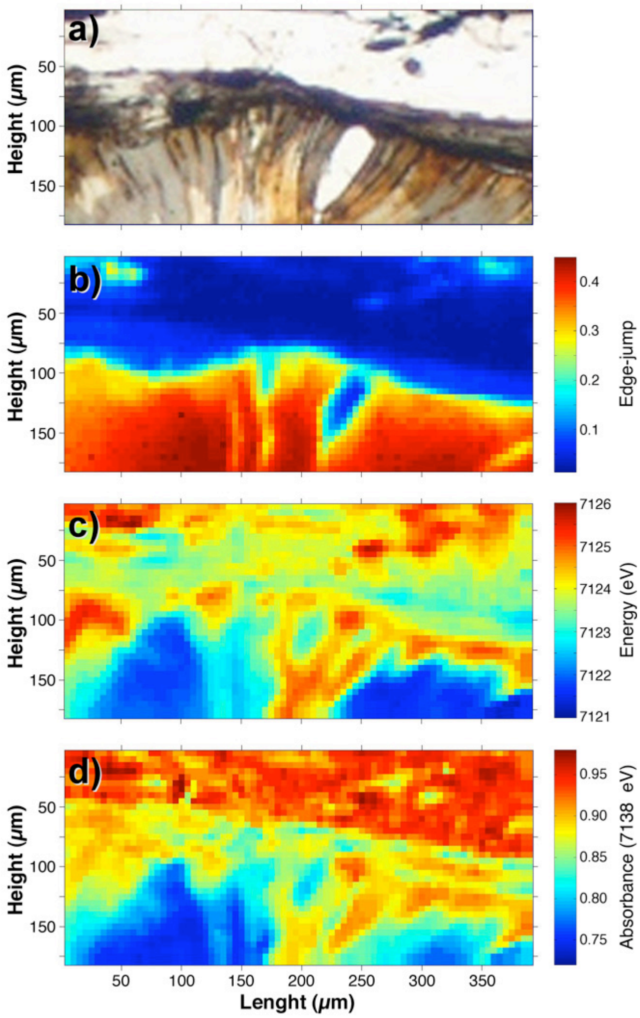

Figure 2: Imaging of the chlorite mineral of a natural metamorphic rock. a) optical image; b) iron content map based on the edge jump of the spectra; c) iron redox map based on the edeg position of the spectra; c) iron speciation map based on a specific feature of the spectra.