EUROPEAN SYNCHROTRON RADIATION FACILITY
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ESRF

ESRF	**Experiment title:** **Redox and Speciation Mapping in the Laser Heated Diamond Anvil Cell**	**Experiment number**: HS3182
Beamline: ID24	**Date of experiment**: from: 15-3-06 to: 17-3-06	**Date of report**: 28/8/06
Shifts: 6	**Local contact(s)**: S. Pascarelli	*Received at ESRF:*

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Report:

In the past 2 years we have exploited the characteristics of an energy dispersive spectrometer, that features no movement of optics during acquisition leading to an enhanced stability of energy scale, spot size and position, combined with a micron sized spot and the option of fluorescence detection, to address 2D mapping with micron resolution on heterogeneous samples, providing full XAS information on each pixel [1]. It is worth noting that due to the absence of mechanical scanning of the monochromator, the spatial resolution is not affected by the energy scan and remains fixed to the dimensions of the probe. Moreover, the dwell time per pixel is short enough to make it practically possible to acquire 100 x 100 pixel images in a few hours.

A number of preliminary experiments were carried out to explore the practical feasibility of complete 2D mapping of natural samples, such as metamorphic rocks, in fluorescence mode [2,3].

In March 2006, during IHR beamtime and in preparation of experiment HS3182, we tested for the first time 2D mapping in transmission mode to perform "in-situ" investigations in the Diamond Anvil Cell. As test sample, we chose a major component of Earth's mantle, ringwoodite [γ-$(Mg,Fe)_2SiO_4$], which is thought to undergo a chemical decomposition, at around 660 km depth (approximately 23 GPa) and 1600ºC in correspondence to a strong seismic discontinuity, towards $(Mg,Fe)SiO_3$-perovskite and (Mg,Fe)O-magnetowustite. We aquired Fe K-edge XANES maps at different pressures, up to ~ 40 GPa, before and after laser heating, covering for each map an area of 200 x 200 μm^2 at 5 μm resolution. The recently implemented FReLoN camera allowed to record the 1600 spectra required for each map in only about 3 hrs.

We show in Figure 1 the normalized absorbance maps at E = 7125 eV recorded at 26 GPa, before (left) and after (right) laser heating. The hot spot is clearly visible Figure 2 as a dark spot of about ~ 30 µm in diameter. Figure 2 shows 13 Fe K-edge XANES recorded every 5 µm through the hot spot (line on the map).

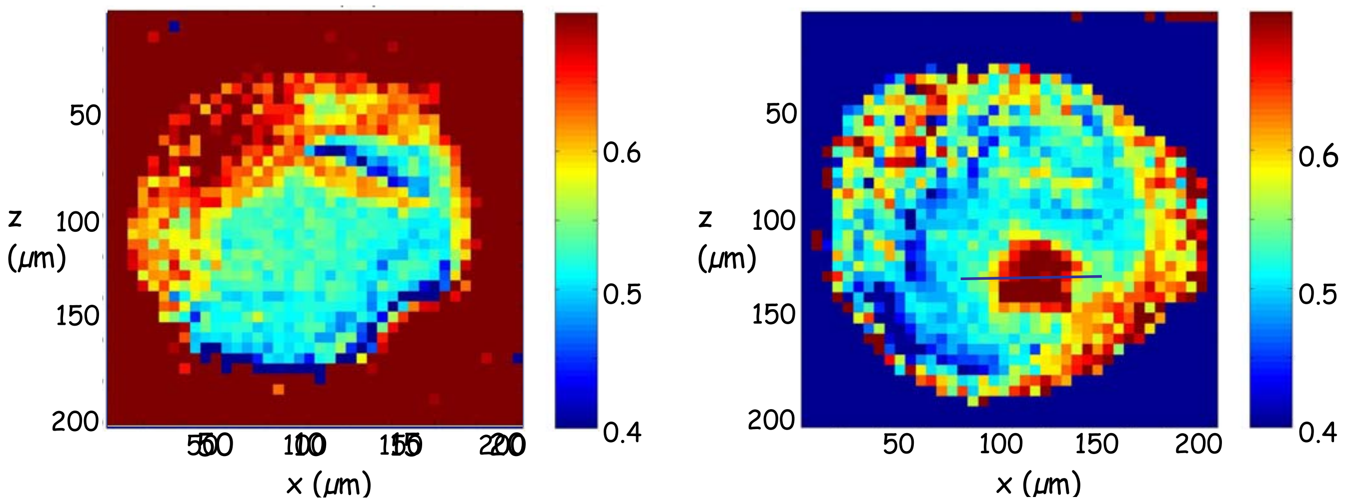
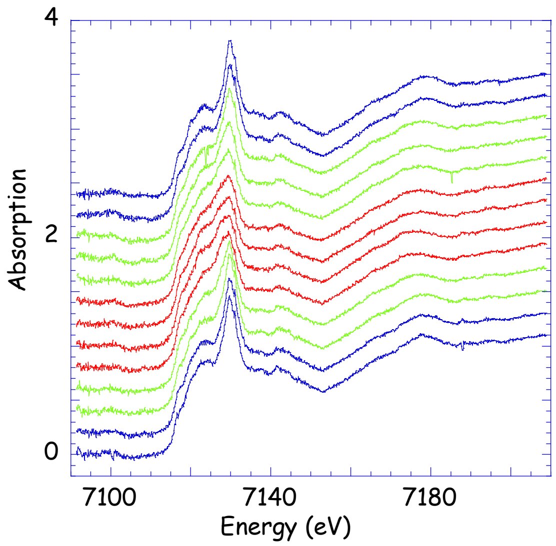


Figure 1: Fe K-edge 2D maps of normalized absorbance at E = 7125 eV, before (left) and after (right) laser heating.



The data shown in Figure 2 illustrates that Fe speciation is modified upon laser heating, i.e. with temperature, and that the spectrum of a homogeneous phase can be recorded in the central 20 µm of the spot, whereas a transition region is observed on the borders of the spot.

Up to now, information on oxidation state and speciation of Fe in geologically (or planetologically) relevant processes at high pressures and temperatures is based only on ex-situ (« quenched ») experiments. This newly tested method opens the way to « in-situ » quantitative mapping of Fe redox and speciation at extreme conditions, yielding precious information on possible phase transitions and/or chemical reactions that occur at P and T conditions in the interior of planets.

This method is still in a development phase, and there is a large margin for improvement. The next obvious step is to exploit the EXAFS region with quantitative mapping of local structural parameters, such as bond distances and coordination numbers. The implementation of simultaneous elemental mapping using analysis of fluorescence lines is also foreseen.

References

[1] S.Pascarelli, O. Mathon, M. Muñoz, T. Mairs and J. Susini, J. Synchr. Rad. 13, 351-358 (2006)

[2] O. Vidal, V. De Andrade, E. Kewin, M. Munoz, T. Parra, and S. Pascarelli, J. Metamorphic Geology (in press)

[3] M. Muñoz, V. De Andrade, O. Vidal, E. Lewin, S. Pascarelli, and J. Susini, G-cubed (in press)