ESRF	**Experiment title:** Fe in silicate melts and its interaction with sulphur: a Fe K-edge XANES and EXAFS study.	**Experiment number**: EC-930
Beamline:	**Date of experiment**: from: 26 september 2012 to: 1 october	**Date of report**:
Shifts:	**Local contact(s)**: Angela Trapananti	*Received at ESRF:*

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Report:

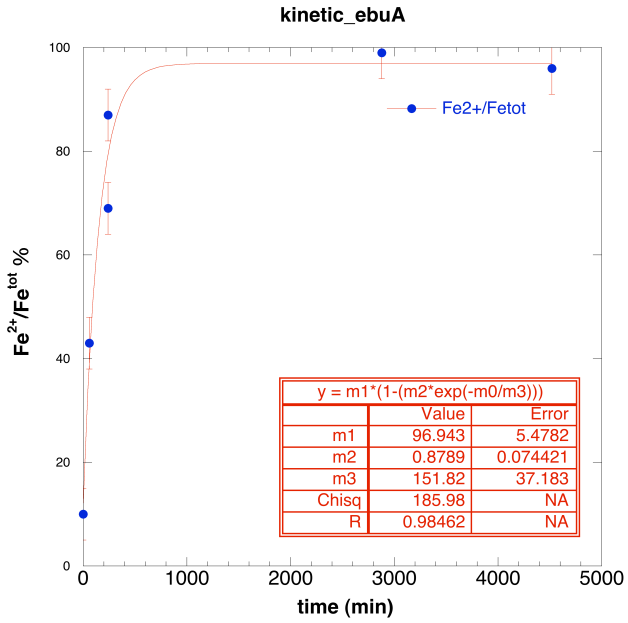
During this run we were able to collect XANES and EXAFS data of high quality at the Fe K-edge for a series of synthetic silicate glasses of pantelleritic composition prepared to study the Fe structural environment and oxidation state and its effect on the solubility of various S species in silicate glasses representative of natural magmas (explosive volcanism).
These data are important to understand the mechanism underlying a drop os sulphide solubility experimentaly observed at fugacity conditions near NNO buffer in samples of similar composition.

The compositions of the glasses has been chosen so as to study selectively the effect of alkalis on the Fe oxidation state. 3 different compositions have been prepared at a range of oxygen fugacity:

1) S, Cl and F bearing
2) High Na
3) High K.

All of these compositions have been prepared in air (water free) and the first two compositions have also been prepared hydrothermally to study the effect of water content (5 wt % in the present samples)
For all these compositions we studied the kinetic of Fe oxidaytion/reduction.
During the run we were able to collect good XANES data for 50 samples and EXAFS data for 5 samples.

The analysis of the pre-edge peak data and comparison with pre-edge peak of model compounds (See wilke et al., 2001, Giuli et al., 2002, 2011) allowed to determine the $Fe^{3+}/(Fe^{2+}+Fe^{3+})$ ratio on all the samples with an error of ± 0.05. Tesehe results have been used to derive information on the kinetics of Fe reduction.
In the figure below we show, as an example, the change in the Fe oxidation state as a function of time for an oxidised glass held at high temperature in a reducing atmosphere.



For all the water free compositions (glasses melted at room pressure in a gas mixing apparatus), fit of the experimental data allowed to ascertain that equilibration of the Fe oxidation state is achieved after 16 hours, whereas for the glasses produced hydrothermally (and containing ca. 5 wt % water) equilibration of the Fe oxidation state was achieved after 1 hour.
These data (which will be submitted as a paper) are the prerequisite in order to equilibrate Fe oxidation state and to prepare new samples at intermediate redox conditions, so as to study the effect of redox conditions on the Fe oxidation state in magmas. The new samples for the continuation proposal are being currently prepared and will be ready before February.
EXAFS data of both oxidised and reduced glasses are currently under study.