ESRF	**Experiment title:** Sulfur speciation in natural silicate glasses. Micro-X-ray Absorption Near Edge Structure (µXANES) studies	**Experiment number**: MI-353
Beamline: ID 21	**Date of experiment**: from: **03 September 1999** to: **07 September 1999**	**Date of report**: 29 October 1999
Shifts: 12 shifts	**Local contact(s)**: **Jean Susini**	*Received at ESRF:*

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Report:

The main objective of these preliminary experiments on sulfur has been reached. Sulfur K-edge spectra by micro-XANES are obtained for the first time in natural silicate glasses trapped as inclusions in minerals $SiO_4(Mg, Fe)_2$ and containing between 800 and 1700 ppm S, with a beam focussed at 0.5 µm and a resolution in energy of 0.2 eV.

During the first 3 shifts, the experimental conditions were accurately defined as follows:

-Energy calibration

-Selection of the mirrors (Si)

-Position of the detector

-Acquisitions of XANES reference spectra, in air, on minerals highly concentrated in sulfur: FeS_2, $CaSO_4$ and a S-bearing silicate mineral phase (Fig. 1), by scanning the energy between 2.45 and 2.5 keV.

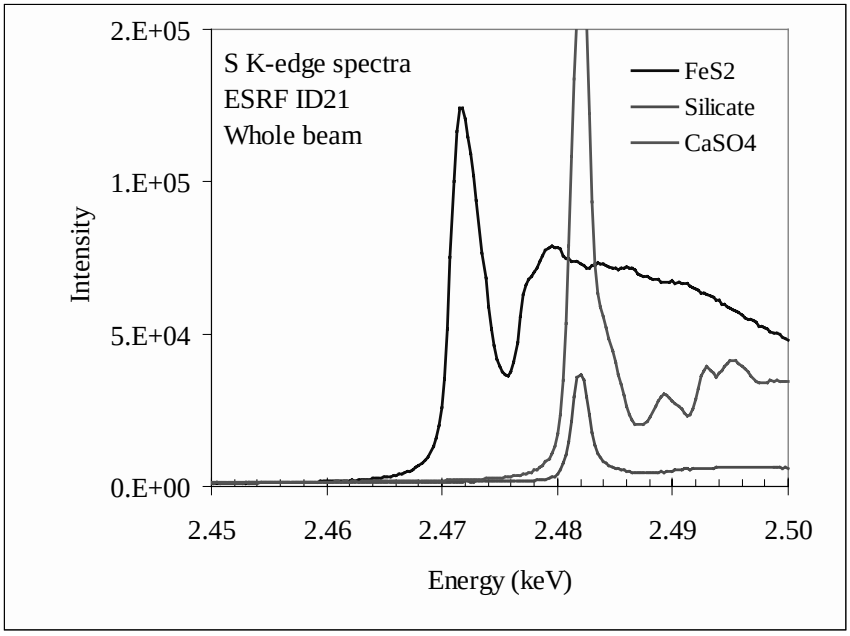

Figure 1. Sulfur K-edge spectra on S-rich minerals

Then, time was dedicated to get a small sized beam between 200 and 100 µm diameter, in air. XANES spectra were acquired on the reference minerals and compared to the previous ones. Many spectra were obtained on samples poorly concentrated in sulfur. Because air is an efficient absorber of 2.34 keV photons, the crystals and their glass inclusions were put under vacuum. The sample holder was specifically made to authorize 3 samples + 1 reference to be analyzed. The selected samples are silicate glass inclusions whose size varied between 80 et 200 µm. In order to minimize the contribution of the host crystal and get spectra from the silicate glass only, the size of the beam was reduced using pinholes of 50, 100 and 200 µm, depending on both the size and the S concentration of the sample.

About 80 XANES spectra were obtained on 7 samples, and on the reference minerals by scanning the energy between 2.46 and 2.5 keV. A specific attention has been paid to a 10µm Fe-sulfide globule present in one of the glass inclusions. Systematic images and XANES spectra were performed, with 50, 25 and 10 µm pinholes, in order to get the best information about the sulfur distribution within and around the sulfide globule. It has to be noted that, during these 6 shifts, time was spent to check the pinhole positions, to change and locate the samples, but we did not notice any shift in energy.

Because of the good results obtained, we tried to get a focussed beam by using a zone plate. The 3 last shifts were allocated to optimize the beam size and get some spectra. The experiment was successful. The beam was focussed at 0.5 µm with a flux high enough to get reliable XANES spectra on both the 10 µm sulfide globule (Fig. 2a) and the associated silicate glass containing 1000 ppm S (Fig. 2b).

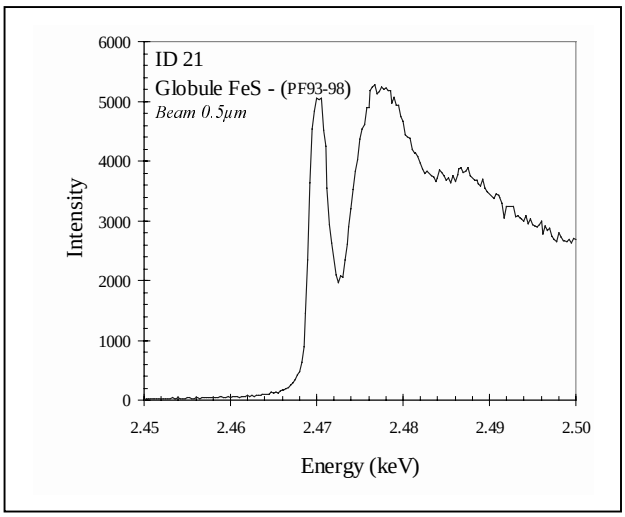

Figure 2a: S K-edge spectrum of pyrrhotite globule associated with glass.

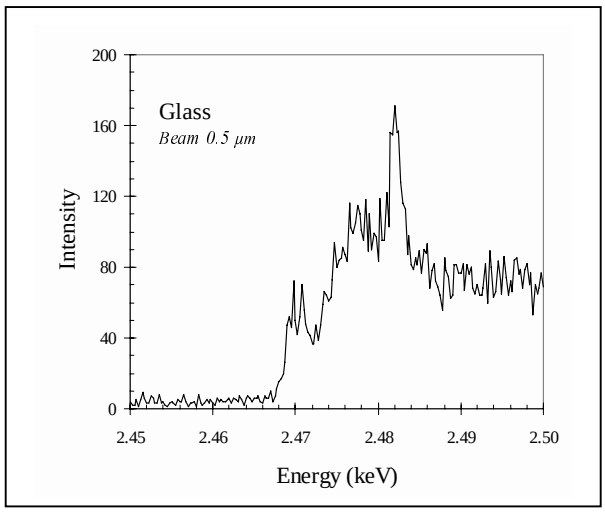

Figure 2b. S K-edge spectrum of S-bearing glass associated with the FeS

Although the present results are still preliminary, they demonstrate that sulfur in silicate volcanic glasses have variable speciation and chemical environment. By simulating the experimental S K-edge XANES spectra, it becomes possible to identify chemical environment of sulfur in natural silicate glass inclusions and identify the structural disorder in theses glasses at the interface glass-FeS phase or glass-host mineral.