ESRF	**Experiment title:** Speciation of light elements (C, N, S) in micro-glasses of geological interest using micro-XANES	**Experiment number:** MI-402
Beamline: Id 21	**Date of experiment:** from: 8/04/00 to: 11/04/00	**Date of report:** 28/02/01
Shifts: 12	**Local contact(s):** Jean Susini	*Received at ESRF:*

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Report:

We obtained 21 shifts for the period covering January to June 2000. The 21 shifts were divided in two periods, the first period (from 06 to 10/02/00) was dedicated to the study of sulphur, the ~~corresp~~onding report has already been sent in march 2000 (mi402). The second period (from 08 to 11/04/00) was dedicated to the feasibility of XANES at the iron K-edge in the same samples. The beamline was not yet optimized for this experiment. Only reference glasses and two analysis points on two glass inclusions with a microbeam were studied.

A paper is submitted to Spectrochimica Acta, part B

Abstract. Glass inclusions trapped in minerals of lava present a major geological interest to assess the chemistry occurring in deep magmas. In particular information about chemical states of elements such as sulfur and iron is of major importance to identify the physico-chemical conditions of magma evolution. Although X-ray absorption methods are best suited for such investigations, the size of those glass inclusions, ranging from a few micrometers to several tenths of micrometers, makes micro-spectroscopy necessary. This work present a combined micro-XANES analysis at both sulfur and iron K-edges with a sub-micrometer spatial resolution. In particular, an immiscible phase (sulfur-rich phase) in a glass inclusion was mapped, revealing heterogeneities in the local environment of sulfur. A semi-quantitative approach, based upon spectra calibration by use of reference glasses, is proposed to evaluate the fraction of iron and sulfur in various chemical states. These new data is then used to obtain more accurate information on oxygen and sulfur fugacities (partial pressures) within deep magmas prior inclusion trapping.

~~As the first report resumed the main results obtained with sulphur, we will give in this report,~~ **the main features obtained for iron.**

The used reference glasses cover the compositional range of the studied glass inclusions, they display basaltic chemical compositions with slightly variable amounts of alkalis and SiO_2/Al_2O_3 molar ratios. They were obtained by melting powdered natural lava samples from Stromboli (*Str85*), Etna (*Et83-08*) and MAPCO basalt (*Ch98Dr11*), in alumina crucibles, between 1260 and 1300°C, under gas fluxes (He, Ar + H_2, air), and quenched in water. The content homogeneity in major elements was tested with electron microprobe. The FeO content of the bulk glass fragments and the total Fe content were determined by volumetric method and by absorptiometry (wet chemistry), respectively. The Fe_2O_3 content is thus calculated by difference.

The glass inclusions selected for the XANES studies were collected from different volcanoes: Piton de la Fournaise in Reunion Island (Pf samples); Stromboli in Aeolian Islands (Str945 samples); sub-marine volcano from the FAMOUS zone (sample ARP 4). They have been widely analysed for major and volatile elements. The inclusions are hosted in olivine phenocrysts and did not evidence any significant post-entrapment evolution which could have strongly affected their original chemical composition. They are basaltic in composition with variable contents in alkalis and H_2O concentrations.

The variation of the pre-edge feature, whereas the $Fe^{3+}/\Sigma Fe$ atomic ratio increases from 0.098 to 0.85, is illustrated in the figure 1 for the Et83-08 reference glass. When reported together XANES spectra from references of the three geodynamical contexts (Stromboli, Etna, and the oceanic basalt), we are able to both discriminate without any problem very close $Fe^{3+}/\Sigma Fe$ ratios such as 0.049, 0.098 and 0.107 and also for Stromboli and Etna (0.32 to 0.48). From these data, we can emphasise that indifferently, Etna or Stromboli glasses can be used as references for glass inclusions of basaltic compositions. Following this point of view, we have processed pre-edge data in these glasses and reported significant parameters in order to evaluate the limits of this assumption.

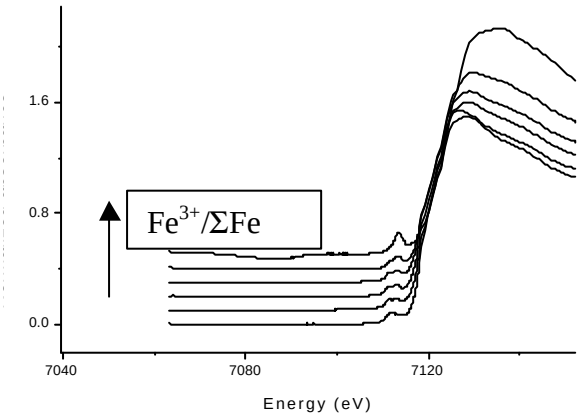

Figure 1 : Fe XANES spectra from Etna reference glasses, as a function of the $Fe^{3+}/\Sigma Fe$ variation

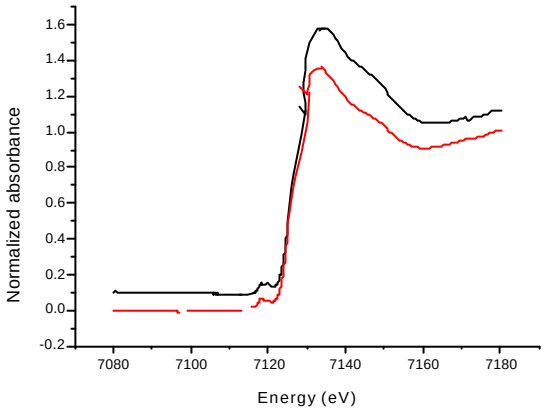

Figure 2 : XANES spectra of two glass inclusions from two origins.
Beam spot size: 2×2 µm^2
Stromboli: black – Arp4: red

Figure 2 reports the XANES spectra registered in the same conditions than for the references with a focused beam related to the dimension of the glass inclusions, that means around the µm^2 in order to reproduce the measurements several times. Only inclusions from Stromboli and the Famous zone (Arp4) has been investigated.

More results are needed with glass inclusions in order to give an interpretation both depending on the geological setting and inside the setting.

We measured 0.12 ± 0.01 $Fe^{3+}/\Sigma Fe$ in Arp4, and 0.24 ± 0.01 in Stromboli using the results from the references. These calculations are in good accordance with the estimated values.