



Experiment title:

Fe and S species in subglacial calcites from Antarctica: investigating the contribution of subglacial processes on Southern Ocean productivity during glacial maxima

Experiment number:

ES-169

Beamline:

ID-21

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08/07/2014

Date of report:

Shifts:

12

Local contact(s):

Camille Rivard, Giulia Veronesi

Received at ESRF:

Names and affiliations of applicants (* indicates experimentalists):

BORSATO Andrea(*), Museo Tridentino di Scienze Naturali, TRENTO ITALY; FRISIA Silvia(*), University of Newcastle, AUSTRALIA; PRETO Nereo(*), University of Padova ITALY.

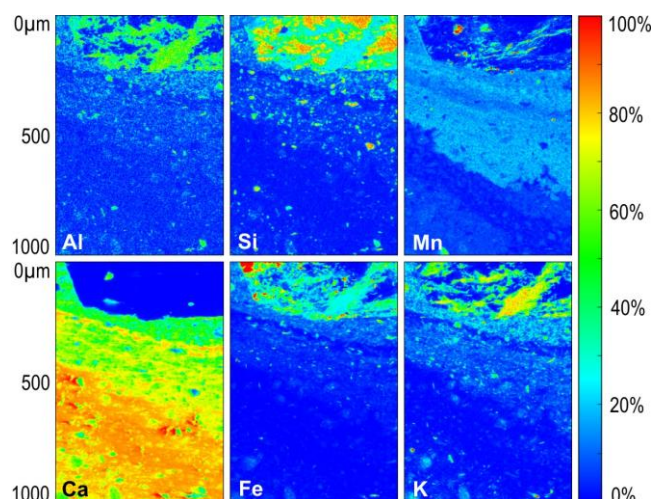
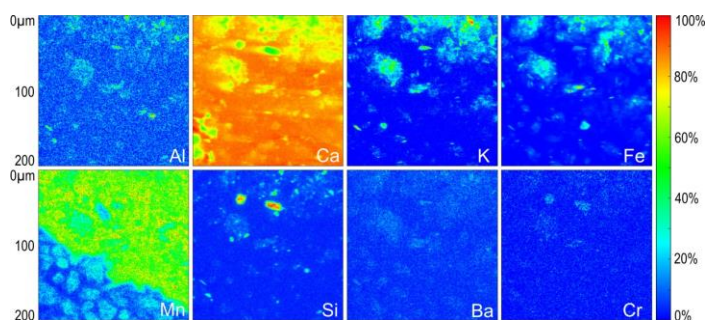
Report:

The first part of the experiment was made with the energy tuned at 7.5 keV at the Fe K-edge.

Sample BV11 Map BV11-3mu -1.0 x 0.7 mm pixel 3µm.

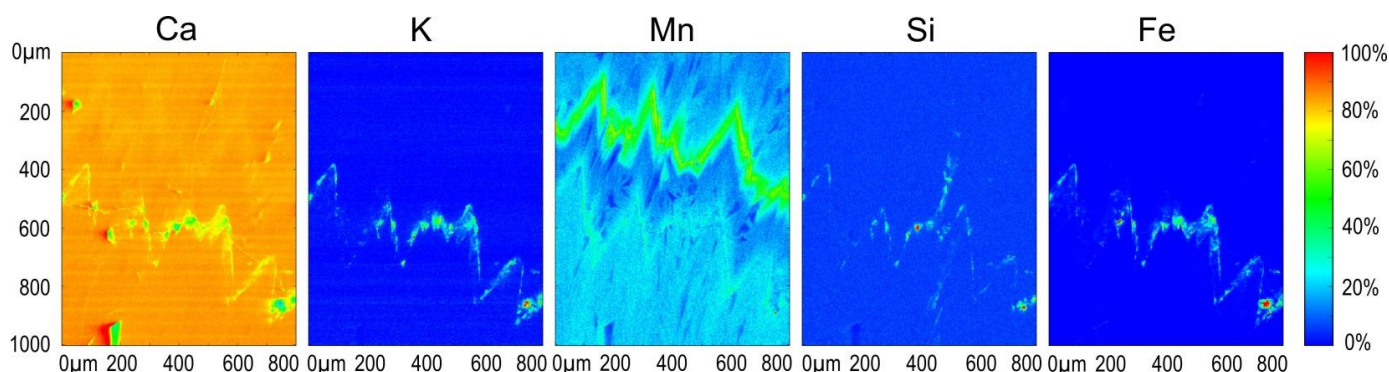
Sample BV11 Map BV11-1mu -0.2 x 0.2 mm pixel 1µm.

The maps were generated across an Al-Si granule and its microbial mat coating. The maps revealed a variable CaCO_3 content in the microbial mats and that most of the Fe is indeed concentrated in the granules and not in the calcite.



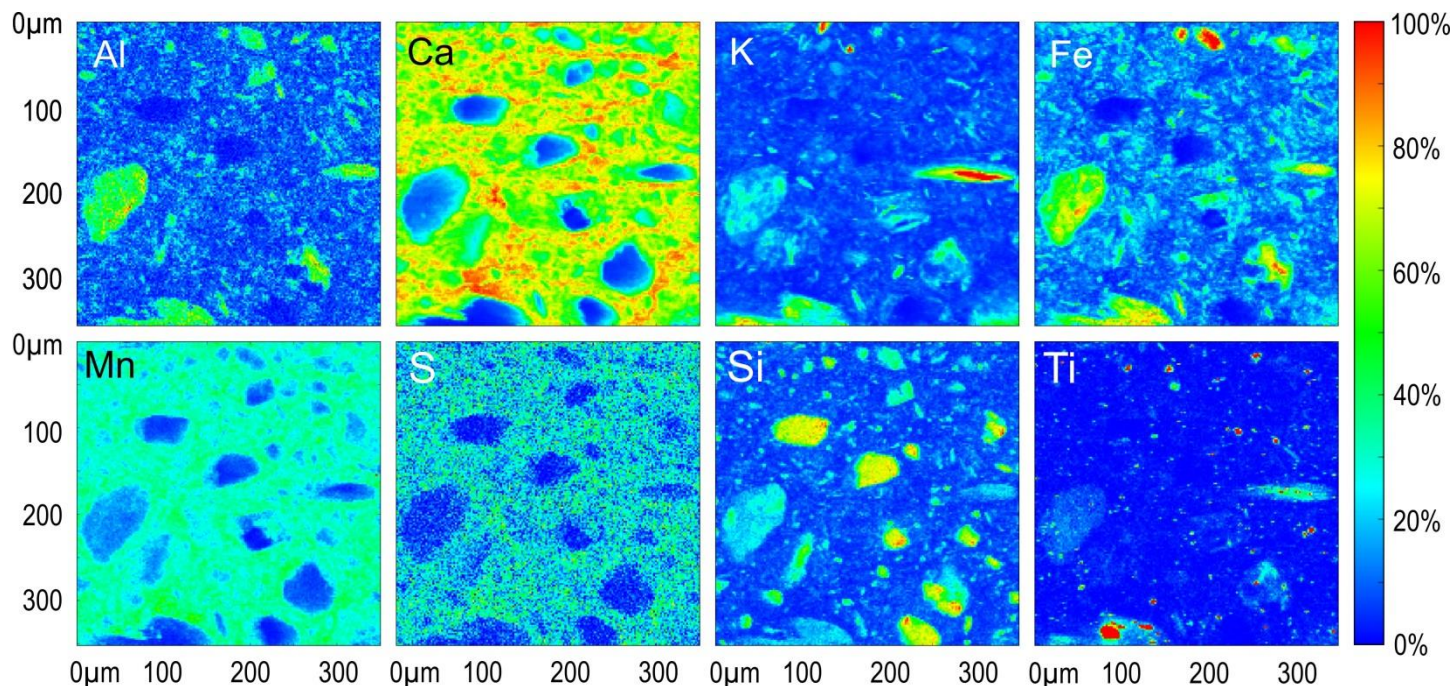
Sample BV8 Map BV8HR -0.8 x 1.0 mm pixel 2µm.

The map was generated across a discontinuity in the calcite crystal. It revealed the concentration of Fe, K and Si as discrete granules along the discontinuity.



Sample BV11 Map B11-HR -0.35 x 0.35 mm pixel 2 μ m.

The map was generated across a discontinuity in the calcite crystal. The map revealed a variable CaCO_3 content in the carbonate cement and that most of the Fe is indeed concentrated in the granules and not in the cement.



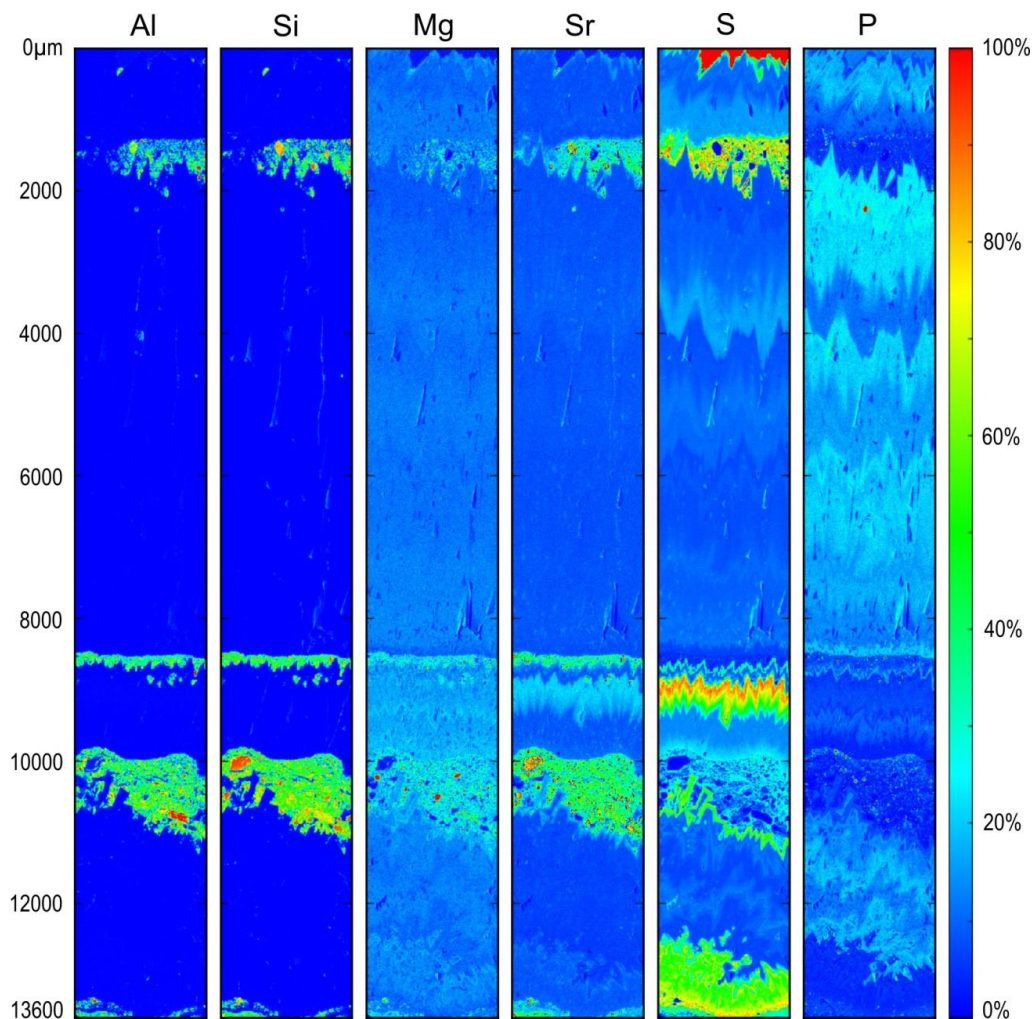
Sample BV9 Map BV9-XXL -1.8 x 13.6 mm pixel 8 μ m.

The map was generated across a discontinuity in the calcite crystal. The map revealed a variable CaCO_3

Fe-XAXES: Calibration done with pure standard Fe foil (K-edge 7112 keV).

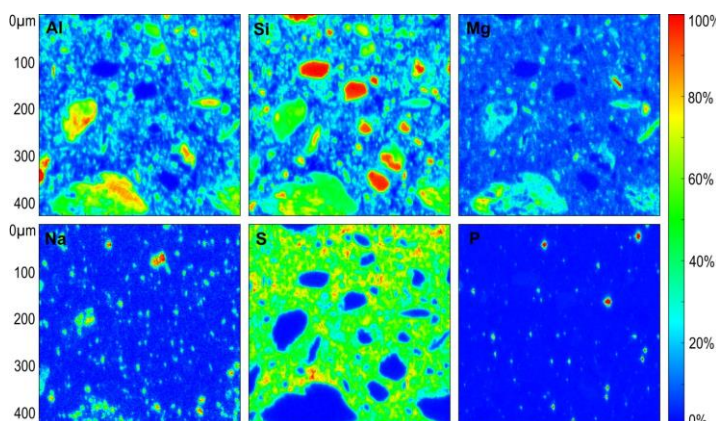
The XANES spectra in sample BV7 and BV9 revealed a progressive shift from a $\text{Fe}^{(3+)}$ in a 6-coordinated sites (peak at 7320), possibly hematite or goethite towards a $\text{Fe}^{(2+)}$ (peak at 7270 eV) possibly almandine.

No Fe signal was detected in pure calcite parts of the samples, confirming that the Fe-rich areas are within the detrital particles and in the microbial mats.

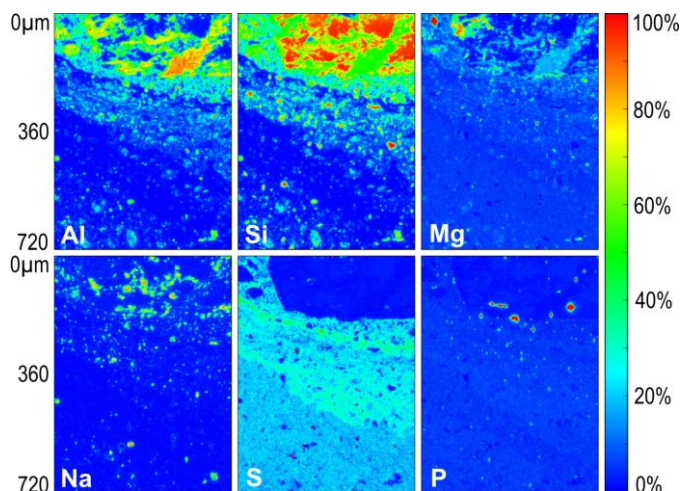


The second part of the experiment was made with the energy tuned at 2.55 keV at the S K-edge.

Map BV11-002HR - 0.45 x 0.45 mm pixel 2 μm

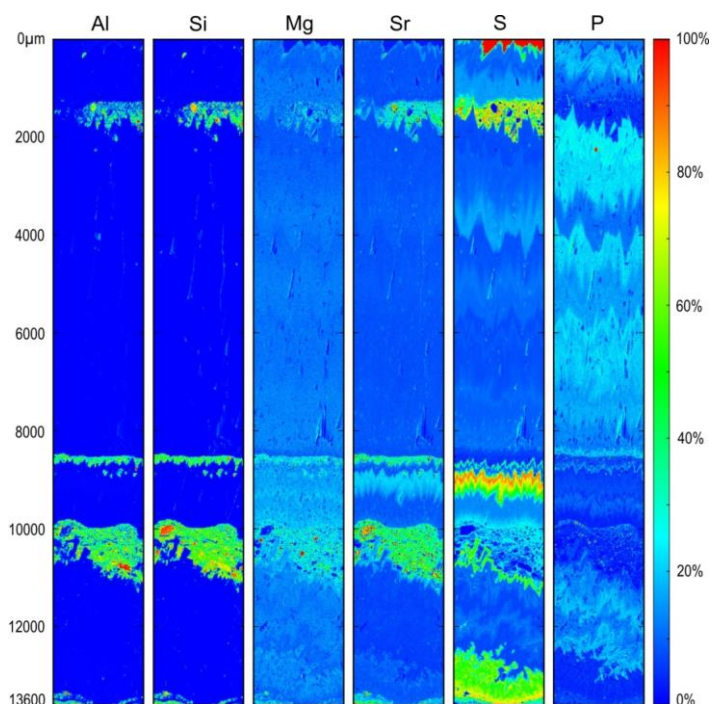


Map BV7.5 - 0.5 x 0.7 mm pixel 2 μm

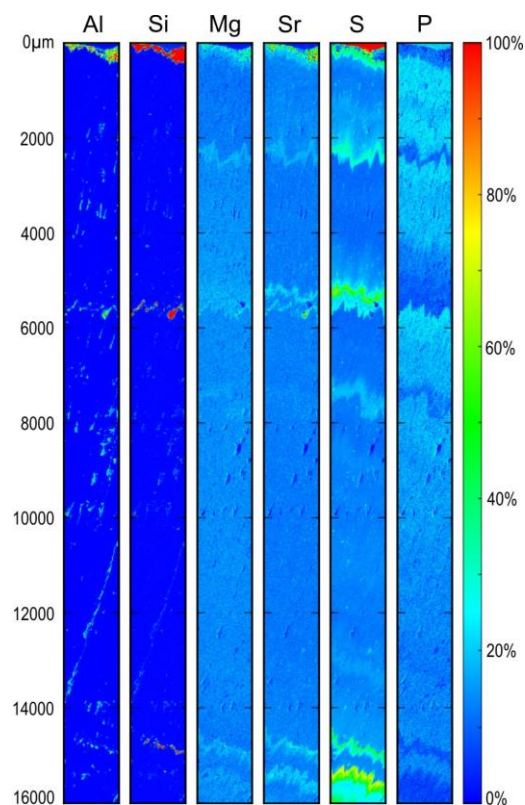


BV9-XXL - 1.8 x 13.6 mm pixel 8 μm .

From this samples 2 medium resolution (pixel 5 μm) maps were also made from the bottom and the top parts.



BV8S - 1.0 x 16.0 mm pixel 8 μm



S-XANES were made on samples BV7, BV8, BV9 and BV11. All samples display a strong peak at 2482.5 eV characteristic of oxidised sulfate phases (Ref. $\text{Ca}(\text{SO}_4) \times 2\text{H}_2\text{O}$). Sample BV11S-7 show an evident pre-edge peak at 2473.4 related to organic compounds, possibly aminoacids (cysteine?). The pre-edge peaks at ~ 2478 eV are instead due to fotoreduction as tested for few samples.

