Experiment title: High-resolution three-dimensional imaging of mineral - bacteria assemblages	Experiment number: ec-16
Beamline: ID-19	**Date of experiment**: from: 16. to: 18.06.2006	**Date of report**: 19.03.2008
Shifts: 9	**Local contact(s)**: Lukas Helfen	*Received at ESRF:*

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Report:

Aims and scientific background

An Fe(III) and sulfate containing mineral ($KFe_3(SO_4)_2(OH)_6$, jarosite) has been exposed to sulfate reducing bacteria (SRB) in laboratory experiments to investigate if bacteria are able to initiate the transformation of Fe(III) and sulfate from solid phases into iron sulfides. Such processes are discussed to take part in remediation measures for neutralizing acidic mining lakes at the water sediment interface where under certain conditions jarosite has been formed. Compared to control experiments the runs with bacteria show grey to black colored covers on the surfaces of the jarosite crystals. Iron sulfides have been identified by XPS and XAFS [1] measurements in the run products proving that bacteria could trigger the iron and sulfate reduction in an initial iron and sulfate free solution in the presence of an Fe(III) and sulfate containing mineral.

X-ray tomography - Experimental

X-ray tomography has been performed at ID-19 to uncover the 3D microstructure of the bacteria mineral assemblages. As energy of the synchrotron radiation 27 keV. The pixel size of the detector (based on the “Frelon 2k” camera) corresponds to 0.28 µm. Sample positioning turned out to be a challenge, because the extremly fragile aggregates were hardly possible to be fixed on any support. Several variants of mounting have been tried: Gluing on glass fibres, clamping in conical plastic tubes, gluing at a vertical metal plane or chucking between to metal plates (Fig. 1a,b,c1,c2,d). Additionally, flushing the sample with nitrogen gas shall avoid oxidation of the reduced parts of the samples during the measurement that were of several hours duration. But the gas flow have increased sample displacement. We decided to abstain from flushing assuming that oxidation might not be so severe for freeze dried substances. Nevertheless, sample drift reduced the usable sets of data. In a few cases tomograms have been recorded at different distances (6, 10, 16 and 22 mm)) for holography which is not completely evaluated yet but could yield additional information.

Fig 1a,b,c1: *Variants of mounting fragile mineral assemblages*

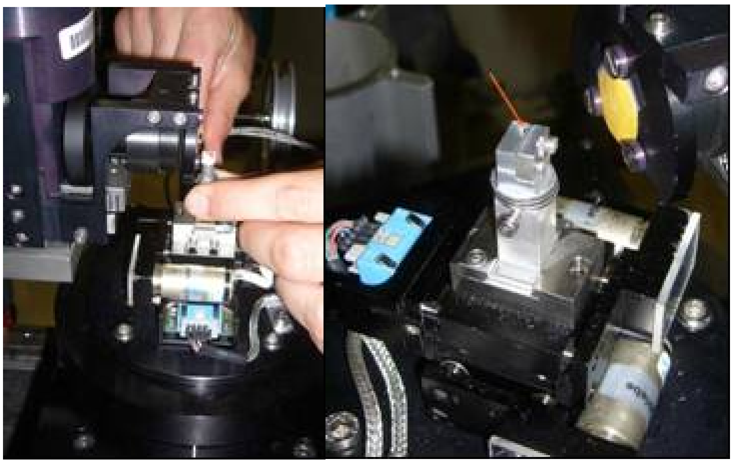

Figure 1c2,d*: (c2) Mounting the sample holder (c1) on the tomography station; (d) fragile crystal aggregate clamped between two metal blocks.*

X-ray tomography – Evaluation and Results

Tomograms have been calculated with the programme PyHST (developed at the ESRF). VGStudioMax was used to select representative slices for each of the evaluated samples to demonstrate differences in the microstructures (Fig. 2). Untreated jarosite and jarosite treated with the same solutions that have been used for the experiments with the bacteria, but here without bacteria, show relatively smooth/pristine slices (Fig. 2a,b) whereas the jarosite aggregates that was in contact with SRB show a microporous structure which may be due to dissolution processes triggered by bacteria (Fig. 2c,d). It is not yet clear why some parts of the jarosite seems not to be affected and other, even inner parts did. We were not able to resolve the bacteria which might be due to the sample drift which was in the order of 1 to 3 µm. A proper correction of a drift presumes a predictable drifting behavior which has not been observed.

Figure 2a: *Slice of a fast reconstructed tomogram of untreated jarosite ($KFe_3(SO_4)_2(OH)_6$); size of the slice 573 µm x 573 µm; samlpe: (ja200_1__slice.jpg*

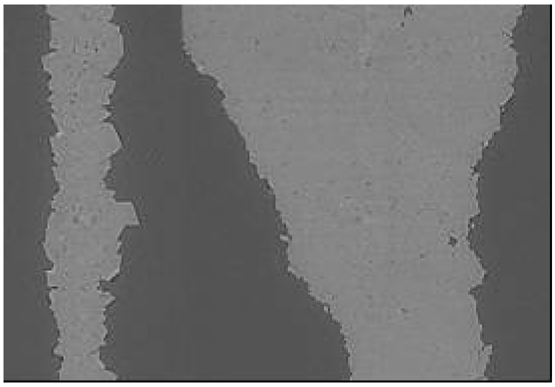

Figure 2b: *xz and yz slices of the reconstructed tomogram of jarosite ($KFe_3(SO_4)_2(OH)_6$) treated in the same manner as sample of Fig. 4 but without bacteria, control run; size of the combined slice 837 µm (hor) x 573 µm (vert); sample: probe4bA_2_xz1024yz470w2989h2048_c.jpg*

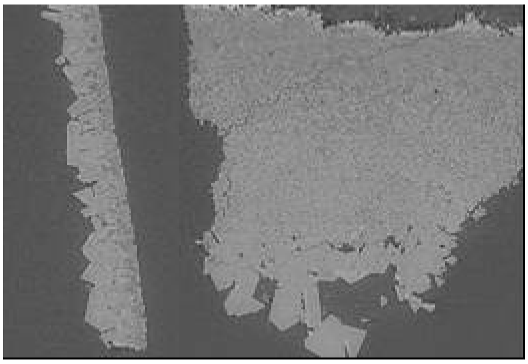

Figure 2c: *xz and yz slices of the reconstructed tomogram of jarosite ($KFe_3(SO_4)_2(OH)_6$) treated with sulfate reducing bacteria, nutrient was ethanol; size of the combined slice 846 µm(hor) x 573 µm (vert), sample: Probe7dC_6mm16mm_1_xz650yz550w3020h2048_c.jpg.*

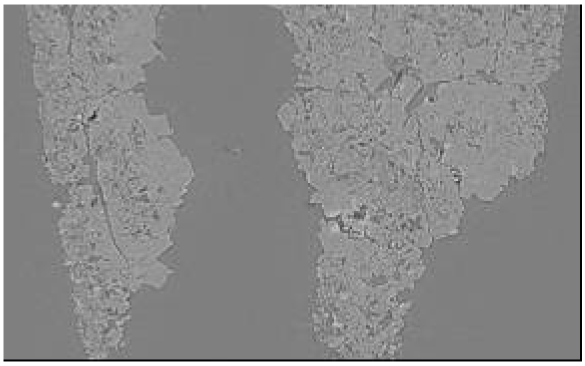

Figure 2d: *xz and yz slices of the reconstructed tomogram of jarosite ($KFe_3(SO_4)_2(OH)_6$) treated with sulfate reducing bacteria, nutrient was lactate; size of the combined slice 934 µm (hor) x 573 µm (vert), sample: probe8eA_1_xz400yz600w3335h2048_c.jpg.*

Outlook

To conclude, we have detected a microporous structure in the crystal assemblages of the jarosite that have been exposed to sulfate reducing bacteria. But, the sample drift prevented to achieve a spatial resolution sufficient to resolve the bacteria. Because of the plate like shape of the crystal assemblages an attempt should be made to record tomograms with high spatial resolution by the newly developed laminography method [2-4] on samples which are glued to a substrate.

Acknowledgements

We thank the ESRF for providing the beamtime and we thank the ID-19 team for their excellent support.

References

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