EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

# Experiment Report Form

**The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF**.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

*http://193.49.43.2:8080/smis/servlet/UserUtils?start*

***Reports supporting requests for additional beam time***

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

***Reports on experiments relating to long term projects***

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

***Published papers***

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

## Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

## Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

| ESRF | **Experiment title:** Microbial transformation of Fe minerals | **Experiment number**: EC 145 |
|---|---|---|
| **Beamline**: ID21 | **Date of experiment**: from: 03.05.2007 to: 08.05.2007 | **Date of report**: 31.08.2007 |
| **Shifts:** 15 | **Local contact(s)**: Murielle Salomé | *Received at ESRF:* |

**Names and affiliations of applicants** (* indicates experimentalists):

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## Report:

Microbial iron reduction may cause the formation of Fe(II) containing minerals such as magnetite ($Fe_3O_4$), siderite ($FeCO_3$), ferrous hydroxy carbonates, vivianite ($Fe_3PO_4 \cdot 8H_2O$), or green rust type phases. Experiment EC 145 was accomplished to investigate these biotransformed Fe(II) minerals. A main objective was to check whether or not Fe(II)-rich coatings on the original Fe(III) minerals induce surface passivation at later stages of microbial reduction.

With that aim in view we examined different incubation experiments involving the dissimilatory iron-reducing bacterium *Shewanella oneidensis* and (i) well crystalline goethite (α-FeOOH), (ii) poorly crystalline goethite, (iii) ferrihydrite and (iv) and material from a rice field soil. Whereas literature suggests the formation of magnetite for the chosen experimental conditions, we found a couple of other Fe(II) phases in each sample. However, we could not identify these phases by comparison to our set of standards or to published spectra (Figure 1). In other words, the formation of new Fe(II) phases was evident, but must be explained by other minerals/solids than magnetite, siderite, vivianite, pyrite, FeS, marcasite or green rust.

To visualize the spatial Fe(II) distribution across several distinct particles we collected maps at different energies (pre-edge: 7110eV; high energy: 7200 eV; whiteline Fe(II): 7127.5 eV, whiteline Fe(III): 7131.5eV) and maps of XANES spectra. While very fine Fe(III) particles were not converted to Fe(II) phases, we observed that most of the larger particles (5-100µm) have been completely transformed or newly formed.

Within the spatial resolution of the instrument (~1µm) we found for all bioreduced Fe(II) particles a homogeneous distribution of XANES spectra and major elements (Figure 2).

From these preliminary results we conclude that

- the mineralogy of the microbially produced phases is more complex than usually acknowledged and needs further research
- *Shewanella oneidensis* does not produce Fe(II) crusts on Fe(III) oxides under the given experimental conditions. Therefore surface passivation seems not to be responsible for the ceasing reduction rates during the incubation experiments. However, we cannot exclude the formation of coatings thinner than 1 µm.

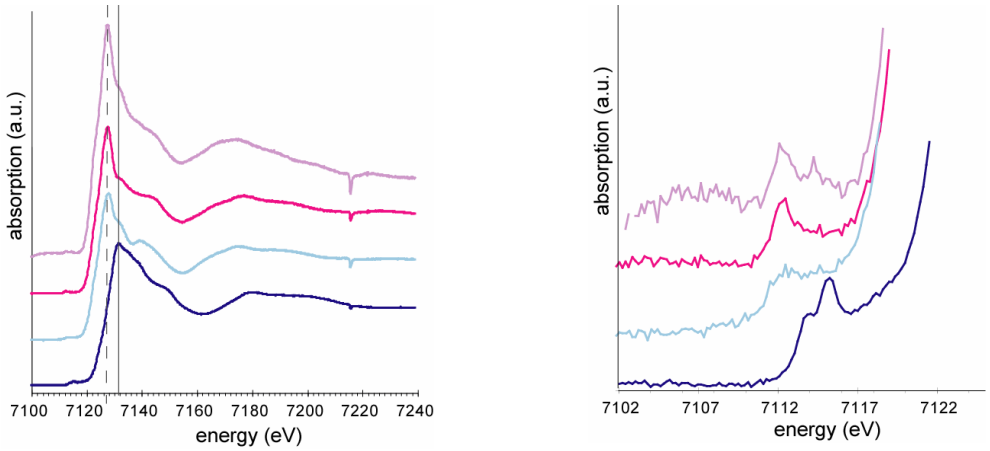
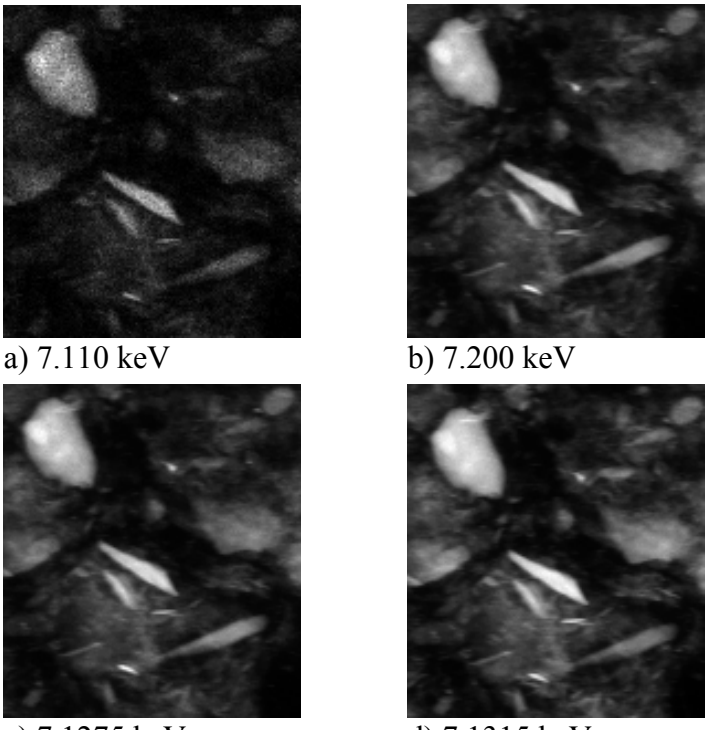
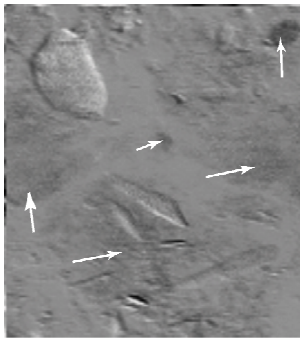



**Figure 1:** (left) Spectra obtained after incubation of well crystalline goethite with *Shewanella oneidensis*. The lowermost spectrum is very similar to the spectrum of goethite [Fe(III) OOH ], but the others could not be identified; (right) close up of pre-edge peak spectra.



a) 7.110 keV b) 7.200 keV



e) difference 7.1675-7.1715

c) 7.1275 keV d) 7.1315 keV

**Figure 2:** (a-d) Maps at different energies have been collected to visualize the Fe(II) distribution across several particles. Figure 2e displays the subtraction of picture c minus picture d. Here, Fe(III) rich particles are supposed to appear darker (arrows). Width × height = 80 × 100µm.