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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 via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can 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: Investigation of phase relations and electronic state of iron in Fe-O compounds at high P-T by means of energy domain synchrotron Mössbauer spectroscopy**	**Experiment number**: HC-825
Beamline: ID18	**Date of experiment**: from: 19.06.2013 to: 26.06.2013	**Date of report**: 01.09.2014
Shifts: 18	**Local contact(s)**: Chumekov Aleksander	*Received at ESRF:*

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Report:

Iron has the ability to adopt different electronic configurations, and transitions and its spin state in the different iron compounds in the lower mantle can significantly influence mantle properties and dynamics. The aim of the project is to investigate phase relations and electronic state of iron in Fe-O compounds at high pressures and temperatures by means of energy domain Mössbauer spectroscopy. Energy domain ^{57}Fe Mössbauer spectroscopy generally enables an unambiguous resolution of all hyperfine parameters which can be used to infer spin states; however high pressure measurements using conventional radioactive point sources require extremely long counting times (generally more than one week per spectrum). Third generation synchrotron sources offer a solution in the form of time-domain Mössbauer spectroscopy (i.e., nuclear forward scattering) (Rüffer, 2008); however this method is not well suited to materials with a large number of components due to the non-uniqueness of fitting models. To solve this problem, we have to use an energy-domain synchrotron Mössbauer source (SMS) developed at ID18 (Potapkin et al., 2012). We studied different Fe-O compounds in a wide range of pressures and temperatures, coupled SMS with portable laser-heating system for diamond anvil cells (Kupenko et al., 2012).

SMS spectra of Fe_2O_3 in hematite structure show all Fe atoms to be in the high spin state up to ~ 45 GPa (Fig. 1a). Upon further compression a new nonmagnetic component appears (Fig. 1b) in agreement with previous study (Pasternak et al., 1999). Further compression leads to a decrease of the magnetic component structure (Fig. 1c), which disappears entirely above ~ 70 GPa (Fig. 1d). The spectra at 75 GPa has only one non-magnetic component with a centre shift corresponding to low spin state of iron. After heating of the sample above ~ 1600 K a transformation to a new phase occurs (Fig. 1e). The Mössbauer spectrum of the new phase has two components corresponding to a high spin iron, while one of the positions is magnetically ordered and the other is not. Note that Shim et al. (2009) has also reported magnetic ordering in the high pressure phase of Fe_2O_3 after laser heating based on nuclear forward scattering measurements, and one of the magnetic sites authors reported has hyperfine parameters close to that we observed, but second non-magnetic component in the spectra has not been identified.

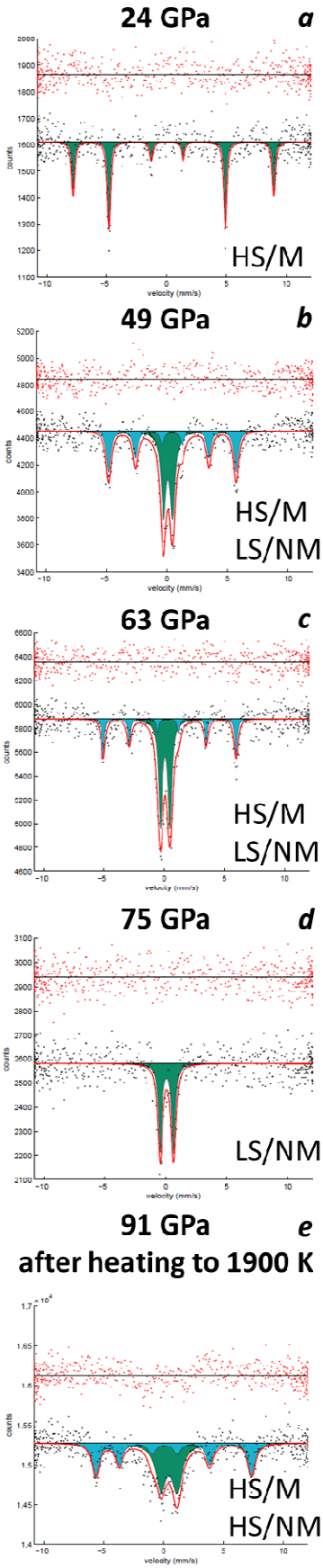

Figure 1. Evolution of SMS spectra of Fe_2O_3 during compression (*a* – *d*) and after heating (*e*).

We collected SMS spectra of Fe_4O_5 at ambient conditions with and without exposure to a weak magnetic field (~0.36 T). These measurements allowed us to identify the spectra as a single eight-line pattern, arising due to the relaxation of the transition selection rules to allow spin-forbidden transitions, which occurs when the orientation of the nuclear magnetic moment is not parallel to the magnetic field of the nucleus.

We collected SMS spectra of (FeMg)O compound with various Fe-Mg content (namely $Fe_{0.12}Mg_{0.88}O$, $Fe_{0.2}Mg_{0.8}O$, $Fe_{0.72}Mg_{0.28}O$) up to 59 GPa at room temperature. 14 spectra of (FeMg)O were collected in situ during laser heating in 30-55 GPa pressure range. Although we made an effort to prepare samples that were thin (about 5 to 7 µm thick), the high temperature SMS spectra show a superposition of components that can be modeled as absorption arising from hotter and cooler material (Kupenko et al., 2012). The detailed anali is of the (FeMg)O data is underway.

References:

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