INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON



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 magnetism and structure of the Fe ₂ O ₃ polymorphs at megabar pressures and high temperatures	Experiment number: HC-1901
Beamline:	Date of experiment:	Date of report:
ID18	from: 09.06.2015 to: 16.06.2015	20.09.2016
Shifts: 18	Local contact(s): Chumakov Aleksander	Received at ESRF:
Names and affiliations of applicants (* indicates experimentalists):		
Ilya Kupenko*: European Synchrotron Radiation Facility, CS 40220, 38043, Grenoble, Cedex 9, France		
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Report:

Fe₂O₃ is an important magnetic carrier mineral for deciphering planetary magnetism and a proxy for Fe in the planetary interiors. High pressure and temperature behaviour of Fe₂O₃ is important for analysis of the behaviour of banded iron formations in subducting slabs (Dobson and Brodholt, 2005) It is an end member in ferrites systems and is a model for studying of physical properties of M_2O_3 compounds. One of the fundamental characteristics of magnetic materials is their critical temperature (i.e. the temperature above which a magnetic material becomes paramagnetic) and its pressure dependence. The aim of the project is to investigate pressure dependence of Curie (Néel) temperatures of different Fe₂O₃ phases in the megabar pressure range by means of energy domain Mössbauer spectroscopy. Energy domain 57Fe Mössbauer spectroscopy generally enables an unambiguous resolution of all hyperfine parameters which can be used to study electrical and magnetic properties of iron; however high pressure measurements using conventional radioactive point sources require extremely long counting times (generally more than one week per spectrum). The recently developed Synchrotron Mössbauer Source (Potapkin et al., 2012) allows measurements of high quality and

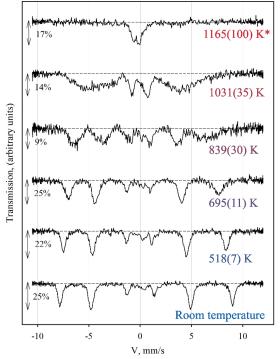


Figure 1. Temperature evolution of Mössbauer spectra of Fe_2O_3 at 19 GPa. Temperatures are determined from the change of the center shift except marked with * which was measured by common spectroradiometry method.

well resolved energy-domain spectra in the timescales of several hours that allows its coupling with the laser heating in the diamond anvil cell technique for *in situ* investigations at extreme conditions. We studied magnetic ordering in different phases of Fe₂O₃ at various pressures and temperatures employing SMS with portable laser-heating system for diamond anvil cells (Kupenko et al., 2012).

We measured temperature dependence of SMS spectra and derived hyperfine parameters of different phases of Fe₂O₃, namely α -Fe₂O₃ (0-26 GPa pressure range, Fig. 1), ι -Fe₂O₃ (~26-46 GPa pressure range), and η -Fe₂O₃ (~50-120 GPa pressure range). In order to determine critical temperature at each pressure we used empirical formula $H = H_0(1 - \frac{T}{T_c})^{\beta}$ (Eibschütz, 1970), where *H* is a measured hyperfine magnetic field, H_0 is a hyperfine field at zero temperature, $\beta \approx \frac{1}{3}$, *T* is temperature, and T_c is a fitted critical temperature. For α -Fe₂O₃ we were able also to trace a pressure dependence of Morin transition temperature i.e. the transition from antiferromagnetic to weak ferromagnetic ordering due to spin canting. Our results on Morin transition are in good agreement with neutron diffraction data (Klotz et al., 2013). Preliminary results are summarized on a Fig. 2. The detailed analysis is underway.

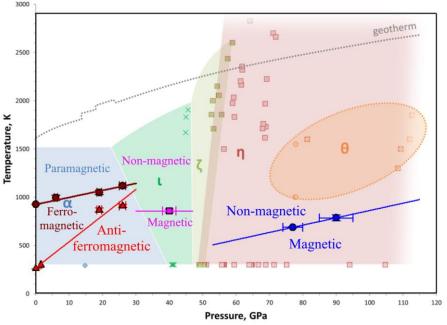


Figure 2. Magnetic phase diagram of Fe₂O₃. Modified after Bykova et al. (2016).

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