



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.



Experiment title: "High-pressure structural properties of MFe_2O_4 ($M=Fe, Co, Mg, Zn$) ferrite spinels".

Experiment number:
HS-3681

Beamline: ID27	Date of experiment: from: 27.11.2008 to: 30.11.08	Date of report: 23.08.12
Shifts: 9	Local contact(s): Dr. Gaston GARBARINO	<i>Received at ESRF:</i>

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Report:

E. Greenberg, G.Kh. Rozenberg, W. Xu, R. Arielly, M.P. Pasternak, A. Melchior, G. Garbarino, L.S. Dubrovinsky, *On the compressibility of ferrite spinels: A high-pressure x-ray diffraction study of MFe_2O_4 ($M=Mg, Co, Zn$)*, High Pressure Research, Vol. 29, No. 4, December 2009, 764–779.

ABSTRACT

High-pressure synchrotron X-ray diffraction studies were carried out on a series of ferrite spinels MFe_2O_4 ($M=Mg, Co, Zn$) up to ~ 70 GPa using diamond anvil cells. He and Ne, used as pressure media, provided quasi-hydrostatic conditions, resulting in a high-quality fit to both second- and third-order equations of state (EOSs). A quality fit to the second-order EOS allows a comparison between the compressibility of these spinels and that of other spinels found in the literature. Fitting to the second-order Birch–Murnaghan EOS results in the values of $K_0 = 170.5(8)$, $176.1(6)$ and $174(2)$ GPa for $M = Mg, Co$ and Zn , respectively. A

linear dependence of K_0 is obtained as a function of the normalized average cationic-sphere volume S_N of each spinel with a slope of $490(15) \text{ GPa}/\text{\AA}^3$. A number of previous studies of the same and similar spinels exhibit a strong deviation from $K_0(S_N)$, which can be attributed to a lack of hydrostatic conditions.

Based on the obtained XRD data we also have tried to identify the structure of HP post-spinel polymorph for the $M^{2+}\text{Fe}_2\text{O}_4$ ($M^{2+}=\text{Fe, Co, Zn, Mg}$) ferrite spinels taking into account the recently obtained Mössbauer data. The only possible structure from the earlier proposed models is the CaFe_2O_4 ($Pnma$) type characterized by two different Fe^{3+} sites [1]. Consistent with Mössbauer spectroscopy, XRD studies show irreversible first-order structural transitions at the range 25-40 GPa and a pure post-spinel structure above 40 GPa. However, fitting using this structural model gives rather good results in the case of F(calc) Weighted (Model biased) refinement but poor results in the case of full-profile Rietveld refinement. Further studies within the post-spinel pressure range (30 – 60 GPa) using **single crystal** XRD and He or Ne as a pressure medium are necessary to clear the dilemma of the post-spinel structure of ferrite spinels and, most importantly of magnetite.

1. N. Funamori, R. Jeanloz, H. Nguyen et al. *J Geophys Res* **103**, 20813 (1998).