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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 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:** Structure and elasticity of Fe-Ni alloys above 330 GPa and 3000 K	**Experiment number**:
Beamline: ID27	**Date of experiment**: from: to:	**Date of report**: 28/09/2007
Shifts: 18	**Local contact(s)**: Guillaume Morard	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Nicolas Guignot*, Synchrotron Soleil, Saint-Aubin
Agnès Dewaele*, CEA DAM, Bruyère-le-Châtel
Paul Loubeyre*, CEA DAM, Bruyère-le-Châtel
Mathieu Roskosz*, LSPES, Université Lille 1

Report:

The aim of this experiment was to determine Fe and Fe-Ni alloys structures and elastic properties at P-T conditions as close as possible to real inner core conditions. Our first concern was to define a strong experimental procedure. We had to set a sample loading geometry optimized for laser heating in a diamond anvil cell (DAC), i.e. a good thermal insulation of the sample and a low reactivity. This is crucial since to actually measure thermoelastic properties of Fe and FeNi, a pressure scale is needed to quantify the thermal pressure produced during laser heating. A very good candidate for that purpose is MgO: its cubic B1 structure is stable up to around 500 GPa, the cubic structure is ideal to estimate non-hydrostatic stresses during the experiment and its PVT equation of state is one of the most documented in the literature. The problem is that this adds oxygen to the system, and we were wondering if that could be a source of oxidation for Fe compounds. The reactivity of a metal depends also on the particles size: even gold is highly oxidizable at room pressure and 300 K when the particle size is low (around 1 micron and less).

The sample loading geometry represented in Figure 1 takes into account all these issues: NaCl layers are used as thermal insulators and pressure transmitting medium. The metal sample consists of one piece of metal foil initially flattened between a diamond and the rhenium gasket. The typical size of the sample was 10 or 20 µm in diameter, 5-7 µm in thickness. A thin layer of MgO powder is loaded in contact with the sample. This way, the metal particle size is maximized, the contact surface between MgO and the metal is minimized and thermal insulation is insured.

We started our first run following that procedure. We loaded the DAC with all the materials initially stored in an air oven. Before heating, MgO indicated a pressure equals to 20.2 GPa, compatible with NaCl and Fe cell parameters. After heating, MgO unit cell volume was much bigger and indicated 8.9 GPa, while NaCl and Fe cell parameters barely didn't change. This can be easily explained by a solid solution of FeO in MgO. If we follow **[1]**, this change of unit cell volume corresponds to $Fe_{0.6}Mg_{0.4}O$. Thus a huge amount of iron has been oxidized, and we immediately suspected water to be the responsible.

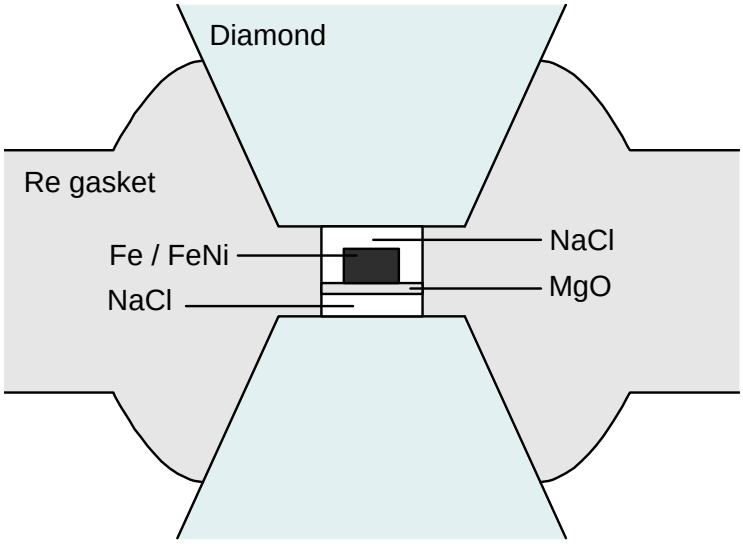

Figure 1. Schematic view of the loading geometry used in this study

We decided to dry NaCl and MgO powders during 12 hours at 625 °C and 1000 °C, respectively. They were then stored in a vacuum desiccator. All the loadings were done in a glove bag under Ar. Following this new procedure, the results were totally different: there were no more undesirable changes in MgO unit cell volume before and after heating. A very strict control of the hydration of the samples is thus mandatory for this experiment, increasing dramatically the loading difficulty (with diamond culet sizes down to 75 µm, gasket holes down to 20 µm).

In order to compare and understand the experimental results, it was very important to start with pure Fe. We collected approximately 200 data points between 55 and 165 GPa and up to 3000 K. In this P-T range, the stable Fe phase is hcp. The whole dataset is represented in Figure 2. A good P-V-T resolution, comparable to the one already achieved in **[2]** was observed and will definitely lead to a detailed PVT equation of state for hcp iron. This is also encouraging for our future works. No dramatic change of the cullet sizes are needed to reach 200 GPa, and this should be easily done. Reaching 300 GPa is a much more ambitious step: specific tests are currently done by our groups to make it possible.

We have also conducted runs with $Fe_{0.8}Ni_{0.2}$ samples, but this is only preliminary work, up to 90 GPa and 2500 K.

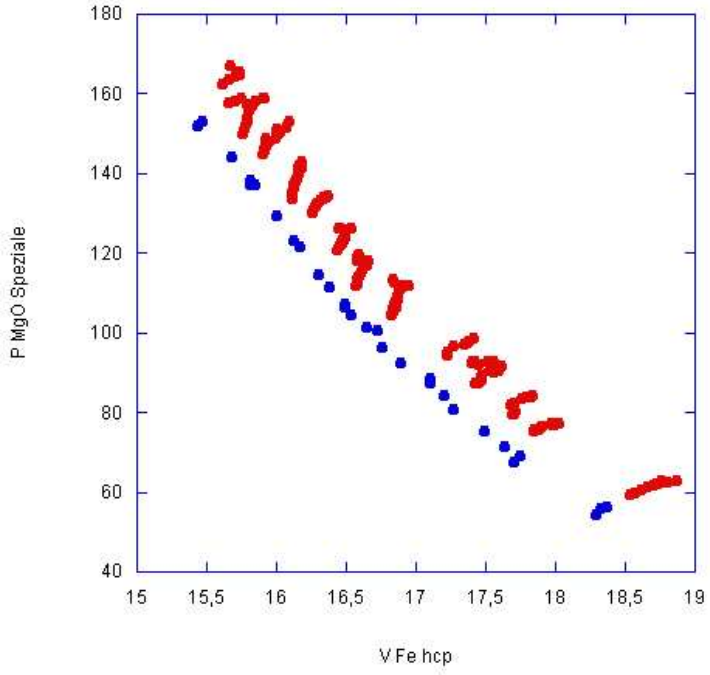

Figure 2. PVT dataset for hcp Fe (blue dots: 300 K data; red dots: high temperature data)

[1] D. Andrault (2001), Evaluation of (Mg,Fe) partitioning between silicate perovskite and magnesiowustite up to 120 GPa and 2300 K, JGR 106 (B2), 2079-2087.
[2] N. Guignot et al. (2007), Thermoelastic properties of post-perovskite phase $MgSiO_3$ determined experimentally at core-mantle boundary P-T conditions, EPSL 256, 162-168.