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 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:** Structural changes of iron hydrides in the 100-200 GPa range	**Experiment number**: HC836
Beamline: ID27	**Date of experiment**: from: 05/01/2013 to: 05/06/2013	**Date of report**: 07/29/2013
Shifts: 12	**Local contact(s)**: Mohamed Mezouar	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Simone Anzellini*

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Report:

Scientific background

Iron hydride is of prior interest for geophysical applications as hydrogen is a possible light element in the Earth's core. The question of its stability, stoichiometry and density under high pressure and temperature needs to be addressed .

Iron hydride gives rise to a double-hcp structure, ε'-FeH_x with x~1 under pressure. A previous study showed that it is stable up to at least ~80 GPa at ambient temperature [1]. It has been predicted[2] that ε'-FeH undergoes a phase transition to an fcc structure above that pressure. Experimental studies in the Mbar range were thus needed to test this prediction.

Experimental technique

Iron samples have been loaded in the high pressure chamber of diamonds anvil cells with hydrogen as pressure medium; ε'-FeH_x formed spontaneously above 3.5 GPa. The conditions reached in 5 experimental runs are summarized in table 1. Diamond anvils with pits drilled by focused ion beam have been used in oder to ensure a better thermal insulation of the hydride samples during laser heating. X-Ray diffraction characterization was performed *in situ* with wavelength λ=0.3738Å.

Run	**Anvil culet size (µm)**	**Pits on the diamond anvils**	**P range (GPa)**	**Comment**
CDMX4	60*300	N	10 to 90	Cold compression, poor signal
CDMX7	100*300	N	10 to 45	Hydrogen leaked in the gasket
CDMX131	150*300	Y, filled with KCl	10 to 105	Laser heated at 27 GPa
CDMX17	200*300	N, LiF layer	10 to 41.5	Laser heated at 41.5 GPa.

		acting as insulator		Sample melted and lead to the loss of the diffraction signal
CDMX11	100*300	Y, filled with KCl	45 to 90	Laser heated at 68 and 90 GPa

Table 1 - Experimental conditions for HC-836

Results

Diffraction signal of ε'-FeHx with the dhcp structure was succesfully followed up to 105 GPa. The predicted phase transition to a fcc structure was not observed. The equation of state of ε'-FeH is reported in figure 1. The increase of bulk modulus above 40 GPa was not observed so that the change of stoichiometry proposed in Ref 1 can be ruled out.

By laser heating, we induced two phase transitions in the Fe-H system. Determination of the structures of these new phases is still ongoing. This very exciting observation needs to be reproduced in independent experimental runs; the electronic properties of these materials will be characterized with infrared spectroscopy which will complement the structural information provided by X-ray diffraction.

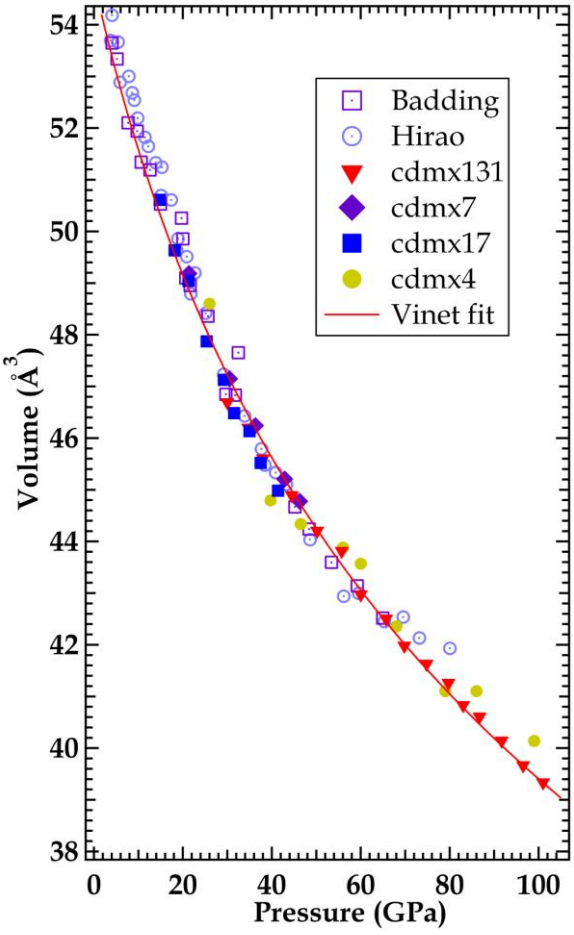

Figure 1 - Volume of dhcp-FeH as a function of pressure

References

[1] N. Hirao *et al*., Geophys. Res. Lett. 31, L06616

[2] E. Isaev *et al*., PNAS 94, 9168