EUROPEAN SYNCHROTRON RADIATION FACILITY

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:** Synthesis of new iron hydrides with unusual stoichiometries at pressures relevant for the Earth's core	**Experiment number**: HC1914
Beamline: ID27	**Date of experiment**: from: 23/04/2015 to: 28/04/2015	**Date of report**: 24/02/2016 *Received at ESRF:*
Shifts: 12	**Local contact(s)**: Gaston Garbarino	

Names and affiliations of applicants (* indicates experimentalists):

Charles Pépin*

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Report:

Scientific background

Because of the high cosmic abundance of iron and hydrogen, the properties of Fe-H system at very high pressure are very important for the understanding of planetary interiors. In particular, hydrogen is considered as a possible light element in the earth core [1,2] (130<P<360 GPa, T>1500K).

During a previous experiment (HC-836), we showed that under mild laser heating two new compounds with stoichiometries FeH2 and FeH3 were formed under high pressure, respectively at 67 and 86 GPa [3].

This sequence of iron hydrides was the first clear experimental validation of a rough rule emerging from a large amount of ab-initio calculations on many metals, namely that the hydrogen:metal ratio of hydrides should increase under pressure. Furthermore, it supports the expectations of new iron hydrides with higher H-stoichiometries at higher pressures, such as FeH4 predicted to be stable above 200 GPa and at the Earth's inner core conditions [1], which was the aim of this proposal.

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 4 experimental runs are summarized in table 1. Thermal insulation of the hydride samples during laser heating was ensured by the presence of c-BN grains, lying inbetween the sample and the diamond anvil. 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
CDMX23	100*300 (11°)	N	150 - 60 GPa	Laser heating first performed in our lab to form FeH3
CDMX22	70*300 (AB)	N	150	Hydrogen leaked in the gasket
CDMX9	150*300	N	70 to 135	
CDMX7	50*300	N	100 to 150 GPa	Laser heated at 100, 115, 125, 144 and 150 GPa

Table 1 - Experimental conditions for HC-1914

Results

By laser heating Fe in hydrogen medium above 140 GPa, we induced the synthesis of a new FeHx phase. Complete determination of the structure of this new phase is still ongoing but our first results, shown on fig: 1, indicate that it has a tetragonal symmetry (space group I4/mmm). This first result shows that this new phase is different from the one predicted in [1]. This new phase could be succesfully reproduced in two independent experimental runs. Ab-initio calculations, based on our experimental results, are now being performed in order to refine the positions of the H atoms in this phase and consequently to determine its stoichiometry.

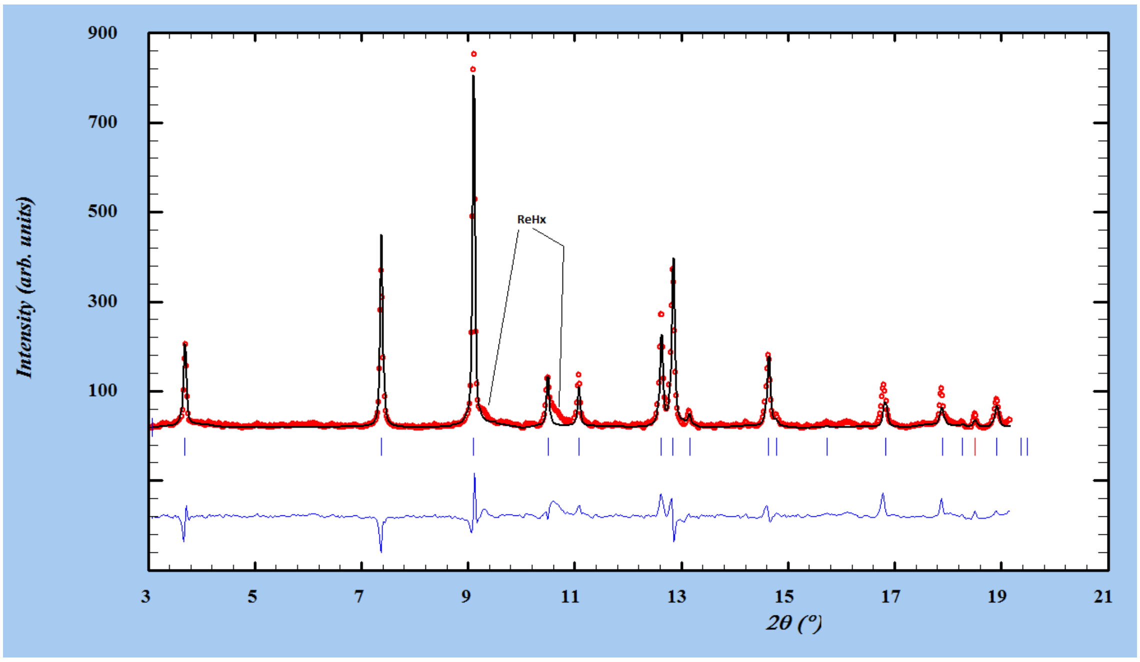

Figure 1 - Rietvield refinement of the new FeHx phase. Red: observed diffraction pattern, black: calculated diffraction pattern.

References

[1]Bazhanova et al., Fe-C and Fe-H systems at pressures of the Earth's inner core, Phys. Usp., 55, 489, 2013
[2] D. Stevenson, Nature 268, 130 (1977)
[3] C. Pépin et al., PRL 113, 265504 (2014)