

## 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://www.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.



	<b>Experiment title:</b> Stability and structural changes of the Boron/Hydrogen system under pressure	<b>Experiment number:</b> HC1339
<b>Beamline:</b> ID09a	<b>Date of experiment:</b> from: 07/27/2014 to: 07/30/2014	<b>Date of report:</b> 03/02/2014
<b>Shifts:</b> 9	<b>Local contact(s):</b> Michael Hanfland	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants</b> (* indicates experimentalists): <b>Charles Pépin*</b> <b>Thomas Plisson*</b>		

## Report:

### Scientific background

There is currently a great interest for the synthesis of novel hydrides, driven by their potential applications for hydrogen and energy storage and by their possible high-temperature superconductivity. Boron hydrides are part of these promising hydrides, with a predicted  $T_c$  of 100 K in its metallic phase. However large discrepancies exist between *ab initio* calculations concerning the stability, the structures and the stoichiometries of the compound under pressure<sup>1,2,3</sup>. Different pressure evolutions are obtained, hence showing possible metastable behaviors.

### Experimental technique

Boron grains powder has been loaded in the high pressure chamber of diamonds anvil cells with hydrogen as pressure medium and heated at several pressures by YAG-laser heating the sample. Synthesis of the hydride was characterized in our laboratory by the appearance of a characteristic new Raman spectrum. The conditions reached in 4 experimental runs are summarized in table 1. X-Ray diffraction characterization was performed *in situ* with wavelength  $\lambda=0.415\text{\AA}$ . Diamond anvils were thermally insulated using KCl or c-BN grains.

Run	Anvil culet size ( $\mu\text{m}$ )	Heating	P range (GPa)	Comment
CDMX21	300	YAG-laser	20	No signal
CDMX23	300	YAG-laser	40	No signal
CDMX18	150*300	YAG-laser	50	No signal
CDMX22	100*300	YAG-laser	80	Diamond failure

Table 1 - Experimental conditions for HC-1339

## Results

No diffraction signal could be obtained from our samples. Further investigation by Raman spectroscopy in our laboratory revealed that the crystalline synthesized sample progressively becomes amorphous over time and that after ~2 days it is completely amorphous, explaining the absence of X-ray diffraction signal.

## References

- [1] Y. Yao *et al.*, J. Am. Chem. Soc. 133, 21002 (2011)
- [2] K. Abe *et al.*, Phys. Rev. B, 84, 104118 (2011)
- [3] C-H. Hu *et al.*, Phys. Rev. Lett., 110, 165504 (2013)