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 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:** Structural properties of H_2 and He up to 200 GPa at very low temperature by angle-dispersive single-crystal X-ray	**Experiment number**: HS-1527
Beamline:	**Date of experiment**: from: 22/03 and 24/05 to: 25/03 and 27/05	**Date of report**: 29/08/01
Shifts:	**Local contact(s)**: M. Mezouar	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Paul Loubeyre, DPTA, CEA. France.

*Florent Occelli, DPTA, CEA. France.

*René LeToullec, Université Paris 6, France.

Report:

We have performed single-crystal x-ray diffraction with a monochromatic beam (0.3738 Å) on solid ^4He and solid ^3He at low temperature up to the Mbar range. The equation of state of ^4He was measured to 122 GPa at 6K. The equation of state of ^3He was measured to 65 GPa at 300K and at 65K on the same sample.
The single crystals were grown in a membrane diamond anvil cell equipped with boron seats to offer an x-ray aperture of ± 35° . In the angle dispersive geometry, the direct beam should not touched the seats and therefore, horizontal slits were made in the boron seats. We could then rotate the cell by ±20°. The loading of the DAC were done in a high pressure vessel. Due to the high cost of ^3He, the loading density could not be very high and therefore the sample was quite small (30 µm diameter).
The technique has been validated previously on ID30 (report HS-1208). It is based on the use of a focussed monochromatic beam (about 15 µm square) and a fast readout on-line image plate detector (MAR) . Observed reflection were correlated by the Single program. That works reasonably well for a simple hcp structure like He in which the index of the diffraction peaks can be easily guessed and the orientation matrix calculated. In the present stage, neither the determination of the rocking curves nor the direct analysis of the pattern of the image plate as a single crystal information are possible. A CCD detector is now being commissioned that is

associated with a single-crystal software package. This new facility will certainly help to perform fine single crystal structural analysis under pressure.
A dedicated cryostat was developed for this project. It has a large X-ray viewing, it is very compact and flows directly inside the diamond anvil. Therefore the cooling is very fast (2 or 3 hours) and the rotation of the cryostat very precise. Unfortunately, the making and position of the seals is very delicate and not always reproducible. That is the reason why sometimes a small leak of helium gas occurs that breaks the vacuum and limit the lowest temperature that could be reached. That is why the EOS of ^{3}He could be measured only at 65 K. We are currently changing the design of the cryostat to remove this problem.
The aim of the experiment was to measure the isotopic shift between the EOS of ^{3}He and ^{4}He at low temperature. As seen in the figure below, the correct isotopic shift, as expected from a zero point effect, is measured: the EOS of ^{3}He is above the one of ^{4}He. But at 300K, the two EOS are crossing in an anomalous way. Further analysis and calculations are being performed to understand this problem.
18 shifts were given to the project. These were fully occupied to perform these difficult data acquisition. The results are very encouraging and we are confident that the same measurements could be performed in hydrogen that is the aim of the continuation proposal.

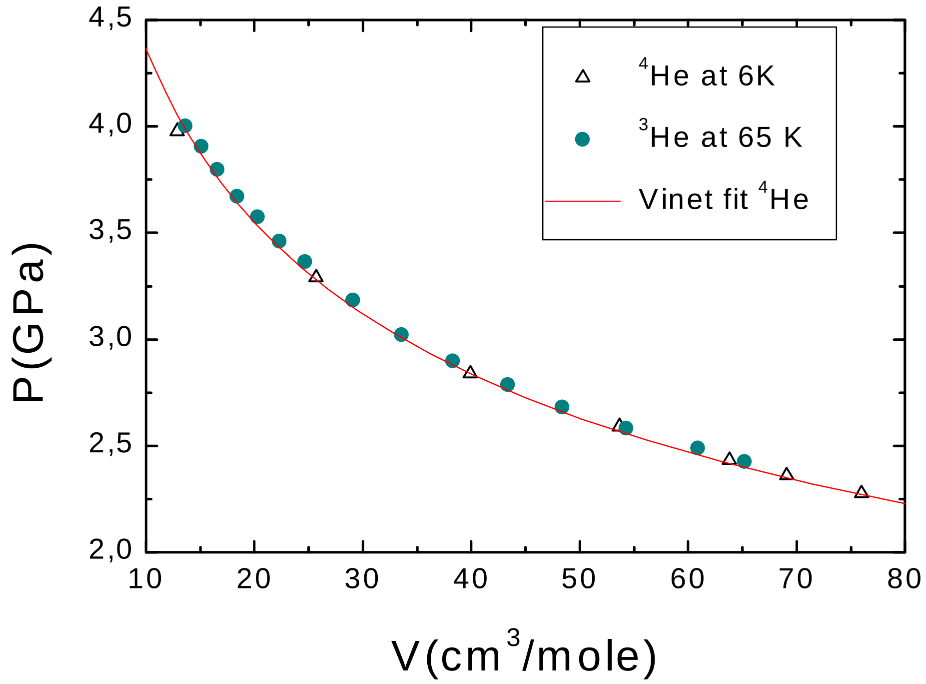

Figure 1. Measurement of the equation of state of ^{4}He at 6K and ^{3}He at 65K. At a given pressure, the volume of ^{3}He is larger than the one of ^{4}He as expected from the zero point motion.