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:** High Pressure-High Temperature Studies and Synthesis of dense C-N-H and CxNy compounds from precursors	**Experiment number**: CH-2676
Beamline: ID27	**Date of experiment**: from: 8/5/08 to: 13/5/08	**Date of report**:
Shifts: 15	**Local contact(s)**: Jean-Philippe Perrillat	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Ashkan Salamat*, Katherine Woodhead*, Paul F. McMillan*, Raul Quesada Cabrera*, Jean-Philippe Perrillat*, Aisha Rahman, Davy Adriens, Furio Corà

Report:

This work carried out at the ESRF has been published in Physical Review B **80**, 104016 (2009).

X-ray diffraction and Raman scattering data supported by *ab initio* calculations are reported for carbon nitride imide, C_2N_3H with a defective wurtzite (dwur) structure, obtained by laser heating synthesis in a diamond anvil cell. Initial laser heating produces a number of metastable phases in the HPHT landscape, identified by Raman spectroscopy and synchrotron radiation work. Extended laser heating transforms this mixture of phases into a single stable phase, dwur-C_2N_3H. The new phase ($Cmc2_1$) is recoverable to ambient conditions and its decompression is followed from 45 GPa downwards. The ambient pressure volume (V_0 = 137.9 Å^3), bulk modulus (K_0 = 258 $\pm$ 21 GPa) and active Raman modes agree with the values predicted from density functional theoretical calculations (V_0 = 134.7 Å^3; K_0 = 270 GPa). The Raman mode associated with the hydrogen bound nitrogen shows peak broadening between a pressure range of 21 – 9 GPa and describes various "wagging" vibrational modes of the hydrogen. Density function theorem was then used to study the ordering pattern of the N-H groups within the structure and its effect on the density and elastic properties. All three methods confirm the formation of the highly incompressible single phase, dwur-C_2N_3H. Ammonia ($P2_12_12_1$) was formed and identified during synthesis as a product of the metathesis reaction taking place. The formation reaction of C_2N_3H indicates a possible synthesis route to the predicted superhard material C_3N_4.

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