EUROPEAN SYNCHROTRON RADIATION FACILITY

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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:** **An x-ray investigation under high pressure of the predicted LiBe and LiBe2 stoichiometric compounds**	**Experiment number**: HS4072
Beamline:	**Date of experiment**: from: 16/7/2010 to: 18/7/2010	**Date of report**:
Shifts:	**Local contact(s)**: Gaston GARBARINO	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Lazicki Amy*, Dewaele Agnès* (CEA-DAM)

Report:

We attemted to form intriguing theoretically predicted high pressure Li-Be phases in a diamond anvil cell. [1,2] The predicted phases are characterized by layers of Li and Be, from which valence electrons are expelled to form 2D delocalized sheets of charge between the layers. The most probable phase – $LiBe_2$ – should be thermodynamically stable by 20 GPa and dynamically stable by 80 GPa.

A total of 7 samples were prepared from mixtures of Li metal and Be powder pressed into thin pellets and loaded in diamond cells either between plates of NaCl or of pressed Be power, or surrounded by neon gas. In one of the neon cells the diamonds were coated with Al2O3. In one cell we found accidental contamination from tungsten (from the needle used for sample loading). In one cell no diffraction from Li could be detected and the starting material was assumed contaminated. Samples were pressurized up to ~80 GPa and laser heated at ~20 GPa or ~80 GPa. Pressure was determined from equation of state of either NaCl or neon, or from ruby fluorescence (and in one case from rhenium equation of state when other methods failed). Some problems identified: Li appears to react with NaCl above 15 GPa, a reaction which is accelerated by laser heating, Be reacts with diamond to form Be2C when laser heated in contact with the anvils at high pressure , and Be may also react with Al2O3 to form BeO when laser heated at high pressure.

In spite of spurious chemical reactions and extreme weakness of x-ray scattering, we were able to identify (in some of the samples), diffraction from beryllium up to 80 GPa and of lithium up to 25 GPa, and possibly 40 GPa (Figure 1). Laser heating at 20 GPa resulted in loss of lithium diffraction signal and melting and recrystallizing of beryllium but no evidence of reaction between the metals. Complete disappearance of lithium diffraction above 40 GPa is attributed to melting of Li near 300 K, recently reported [3].

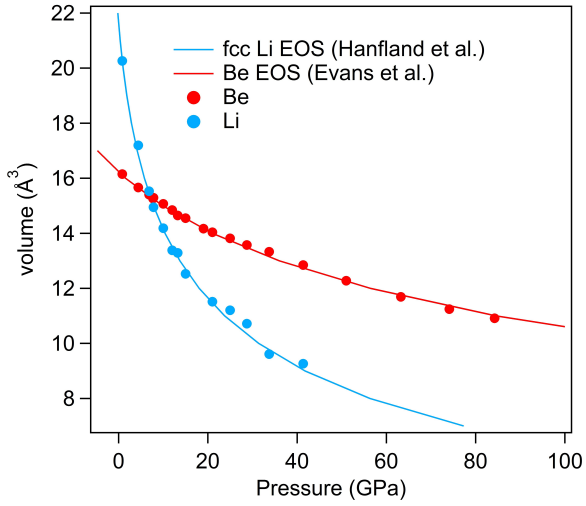

Figure 1. Equation of state of Li and Be together in the diamond cell prior to phase transitions. Experimental data points are compared to reported equation of state of Li (Hanfland et al. [4]) and Be (Evans et al. [5]).

The predicted Li-Be compounds could not be detected at 40 GPa, or on further compression up to 80 GPa. Laser heating at 80 GPa produced new phases which are not yet understood (Figure 2), but are inconsistent with predicted Li-Be, and, from the strength of the x-ray scattering, unlikely to come from any Li-Be phase.

Further work should be done to eliminate all sources of unwanted chemical reactions. However, our results suggest that, at very least, the pressure range over which the new Li-Be phases can be formed was incorrectly predicted and has not yet been reached.

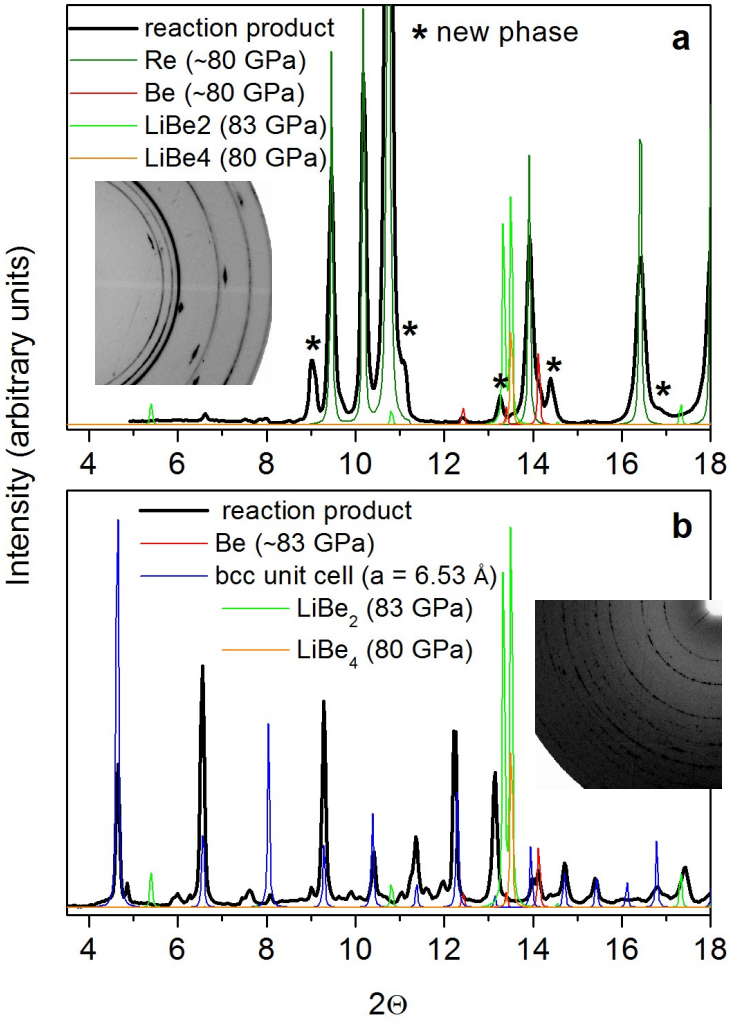

Figure 2. Li-Be samples after laser-heating at ~80 GPa. Inset in each plot is a portion of the image plate showing the distinct difference in character of reaction products in each case. The symmetry for the new phase in (a) was not identified. The new phase shown in (b) is reasonably consistent with a large unit-cell bcc lattice. The positions of the most intense peaks for the predicted high pressure $LiBe_2$ and $LiBe_4$ compounds are shown.

References

[1] J. Feng, R. G. Hennig, N. W. Ashcroft and R. Hoffmann, Nature **451**, 445 (2008).
[2] I. Errea, M. Martinez-Canales and A. Bergara, Physical Review B **78**, 172501 (2008).
[3] M. McMahon et al., personal communication.
[4] M. Hanfland, I. Loa, K. Syassen, U. Schwarz and K. Takemura, Solid State Communications **112**, 123 (1999).
[5] W. J. Evans, M. J. Lipp, H. Cynn, C. S. Yoo, M. Somayazulu, D. Hausermann, G. Shen and V. Prakapenka, Physical Review B 72, 094113 (2005).