	Experiment title: Refinement of the melting diagnostic in laser-heated diamond anvil cells: lead as a case study	**Experiment number**: HS-3159
Beamline: ID27	**Date of experiment**: 13-15/09/06; 27-30/09/06, 31/10-3/11/06	**Date of report**: 27/08/07
Shifts:23	**Local contact(s)**: M. Mezouar	*Received at ESRF:*

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Report:

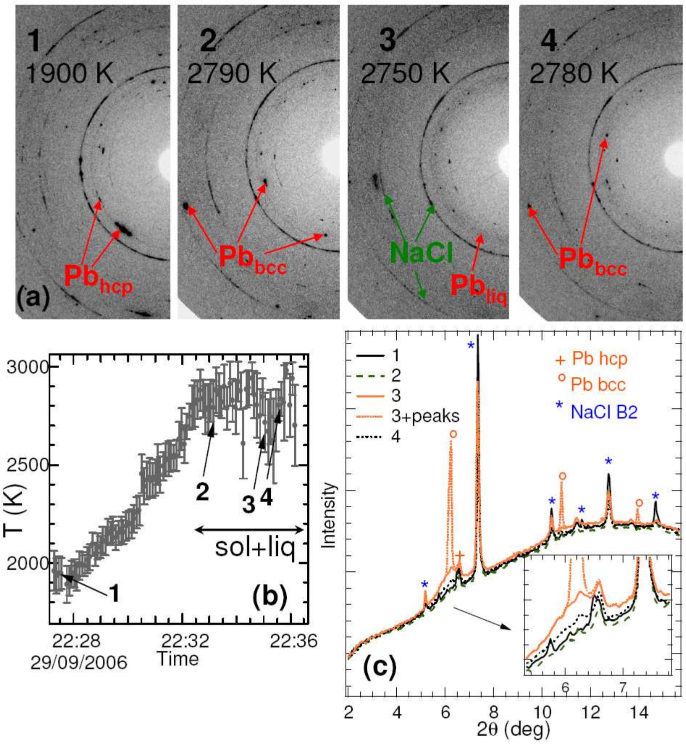
Accurate measurement of the melt line has been a long-standing and controversial problem in high pressure science, with highly-publicized inconsistencies between several experimental methods dominating the discussion. Lead was an example of these controversies, the melting curve measured by laser-heated diamond anvil cell (LHDAC) [1] being several hundreds of K under the melting curve measured by shock-wave compression [2]. So far, X-ray diffraction has been used only very scarcely to detect melting in LHDAC. The aim of this proposal was to measure the melting curve of lead, detected by X-ray diffraction in a DAC, to progress in the discussion of this controversy.

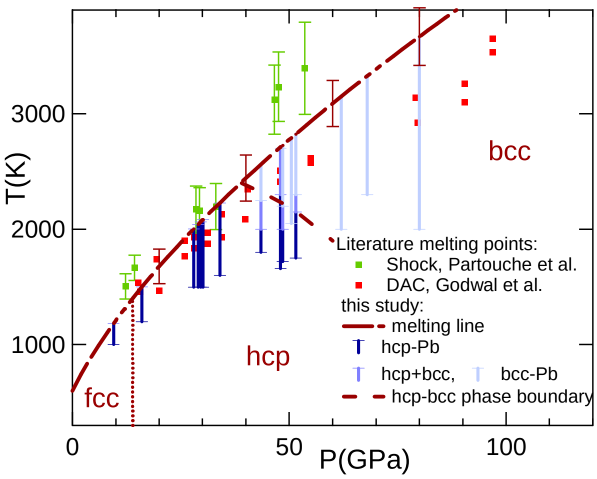
Four experimental runs have been carried out. The sample assembly was formed by a lead foil and a pressure transmitting medium which was also used as x-ray pressure gauge (NaCl). The sample was heated on both sides by defocused lasers (each 40 W) and x-ray beam was aligned on the laser-heated spot to record the structural changes in the sample during heating. Several heating series (gradual increase of the lasers power) were carried out on each sample at different pressures. The high x-ray flux on the ID27 beamline and the short response time of the MAR-CCD x-ray detector allowed us to follow, in real time – an x-ray diffraction spectrum was recorded every 6 s –, the microscopic structural changes occuring in the sample during heating.

Most of the heating series led to the melting of the sample, evidenced by the x-ray pattern characteristic of scattering by a liquid (**Figure 1**). In most of the cases, solid lead, either not molten or crystallized from the melt (single crystal spots appearing and disappearing on each new x-ray pattern), was observed at the same time as the liquid. When liquid and solid lead were observed, the measured sample temperature remained stable, even if the lasers power was increased. These observations can be interpreted as a liquid-solid equilibrium in the lead sample.

The measurement of the pressure and temperature conditions of liquid-solid equilibriums observed during several heating series lead to a melting curve that lies well above the melting points detected by optical means

in a LHDAC (see **Figure 2**). The use of X-ray diffraction to detect melting thus allowed to reconcile the LHDAC and shock melting curves in the case of lead, their difference being now within experimental error bars. A reliable hcp-bcc phase boundary of solid lead has also been measured, and the bcc-hcp-liquid triple point has been located at 39 GPa and 2400 K. The bcc phase of lead has been discovered several years ago, but metastability problems at ambient temperature prevented to accurately measure the hcp-bcc transition pressure [3], [4].
A report of this work is in press in Phys. Rev. B.





Left: Fig. 1 (a). X-ray diffraction patterns for a {Pb+NaCl} assembly, at P=52 GPa during a heating series. On the second spectrum, x-ray spots corresponding to single crystals of Pb crystallized from the melt can be seen (bcc phase); on the third and fourth spectrum, the signal scattered by the melt - a diffuse ring labelled Pb_{liq} - can also be seen. **(b)** Corresponding temperature ramp. Lasers power increases continuously with time. The temperature ramp exhibits a plateau during the solid-liquid equilibrium of lead. **(c)** Angularly integrated x-ray spectra. Solid x-ray diffracted spots have been hidden, except for the dashed curvefor that corresponds to the third spectrum. An inset is shown around the major diffuse ring.

Right: Fig 2. Melting points of lead found in the literature ([1], [2]) compared with the melting curve obtained in the current study. The P-T paths followed during different heating series are represented by the blue vertical lines; they also allowed to measure the hcp-bcc phase boundary of lead.

[1] Godwal et al., Science, **248**, 462, 1990
[2] Partouche-Sebban et al., J. Appl. Phys., **97**, 043521, 2005
[3] Vanderborgh et al., Phys. Rev. B, , 41, 73381990
[4] Mao et al., Solid State Comm, 74, 1027, 1990