ESRF	**Experiment title:** Refinement of the melting diagnostic in laser-heated diamond anvil cells: lead as a case study	**Experiment number**: HS-3159
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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. 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 before and during heating. These changes are described below.
Most of the heating series led to the melting of the sample, evidenced by the x-ray pattern characteristic of scattering by a liquid (see Fig. 1 (a-2) and (b-2)). At temperatures much below melting, the lead sample recrystallized and single crystal spots, with temperature-diffuse scattering, could be seen on the detector (see

Fig. 1 (b-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. In several heating series, the appearance of liquid was even preceded by an episode of intense recrystallization of the sample (Fig. 1 (a)). These two observations can be interpreted as a liquid-solid equilibrium in the sample, the melt being detected only when its amount is sufficient to produce a clear scattered signal. For some heating series, the melt could be evidenced only during a few seconds. Even if the lasers power was increased during the observed liquid-solid equilibrium, the measured sample temperature remained stable (Fig. 1).

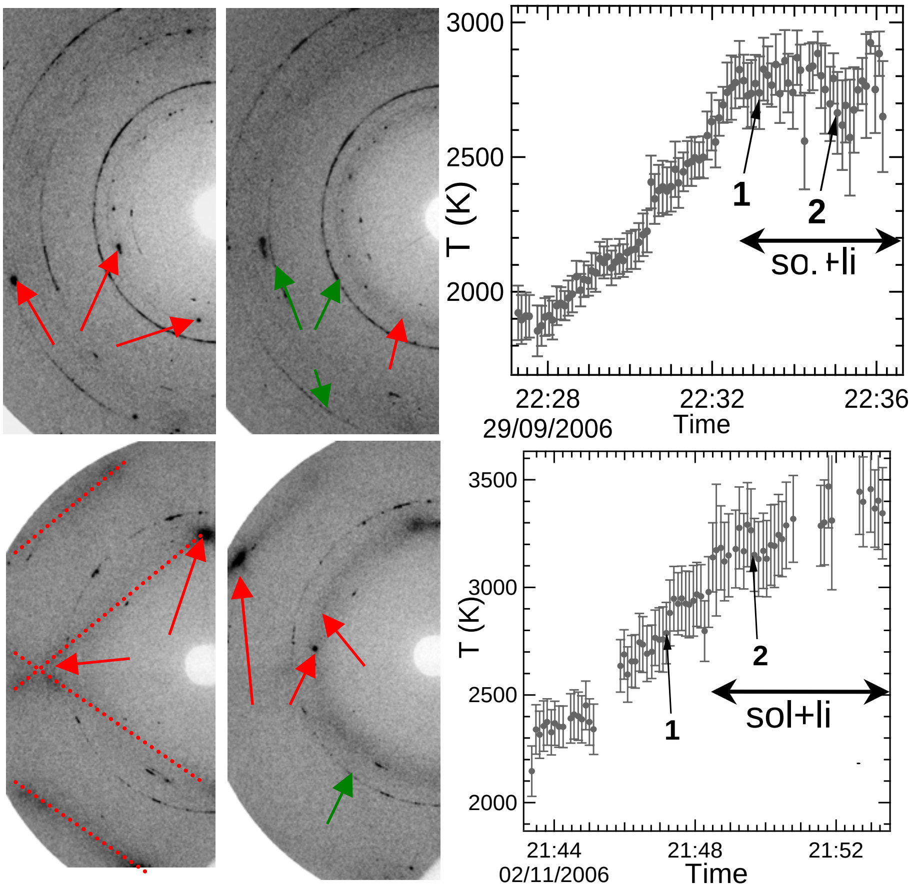

Fig. 1 (a) Two monochromatic X-ray diffraction patterns (λ=0.2647 Å) for a {Pb+NaCl} assembly, at P=50 GPa during a heating series. The corresponding temperature ramp is plotted on the left. On the first spectrum, x-ray spots corresponding to single crystals of Pb crystallized from the melt can be seen; on the second spectrum, the signal scattered by the melt can also be seen. The temperature ramp exhibits a plateau during the solid-liquid equilibrium of lead. (b) Similar patterns, at P=59 GPa and λ=0.3738 Å. On the first spectrum, temperature-diffuse scattering by a Pb single crystal can be seen; on the second spectrum, an additional signal scattered by the melt and x-ray spots of Pb crystallized from the melt can also be seen.

These experiments allowed us to measure the melting curve of lead up to 80 GPa, and to better characterize the hcp to bcc solid-solid phase transition that occurs in the 70 GPa range. An important result is that our measurements reconcile LHDAC and shock wave measurement of melting, in the case of lead. We are planning to submit a manuscript that reports these observations in the forthcoming month.

[1] Godwal et al., Science, **248**, 462, 1990
[2] Partouche-Sebban et al., J. Appl. Phys., **97**, 043521, 2005