	<b>Experiment title:</b> Structural determination of the high pressure solid phase of hydrogen- continuing work;	<b>Experiment number:</b> HS3266
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<b>Shifts:</b> 18	<b>Local contact(s):</b> M. Mezouar	
<b>Names and affiliations of applicants</b> (* indicates experimentalists):  *P. Loubeyre, CEA/DAM Bruyères-le-Châtel, France  *F. Occelli, CEA/DAM Bruyères-le-Châtel, France  *G.Weck, CEA/DAM Bruyères-le-Châtel, France		

## Report:

The search of metal hydrogen is probably the most challenging problem in high pressure physics. Three phases are known in the experimentally accessible range of pressure. The structural studies of phase I and phase II have been done at the ESRF[1,2]. Optical studies of solid hydrogen show that phase III is stable from 160 GPa up to at least 320 GPa and that its direct band gap is closing to below 2 eV[3]. The direct x-ray determination of the structure of phase III is certainly considered a central issue to progress in the understanding of the metallisation of hydrogen. The aim of this proposal was to continue our effort to characterize the structure of phase III by the single-crystal x-ray measurements. In a previous set of experiments, we believe that we had successfully obtained the diffraction of a single crystal just after its phase transition in phase III. But there was no direct evidence that the transition had been taking place. A spectroscopic proof, like the Raman signature of a strong vibron frequency discontinuity at the transition, was missing. In this proposal, the single crystal diffraction was coupled to in-situ Raman measurements.

## Experimental method

At least 10 single crystals were grown. Four single crystals were successfully measured under pressure. Membrane diamond anvil cells were equipped with diamond anvils with 70  $\mu\text{m}$  culet size. Experiments were performed at 30 K and 100K. The pressure was measured by the luminescence of  $\text{SrB}_4\text{O}_7:\text{Sm}^{2+}$  and also the  $\text{H}_2$  vibron frequency. Only for two out of those four crystals the maximum pressure achieved was sufficient to cross the transition line to phase III.

## Results

The d-spacings,  $d_{100}$  and  $d_{101}$ , that have been measured are plotted versus pressure in the figure below. There is not dramatic change at the transition. Also, since we were following the various d-spacings of the

100 class, we can rule out an orthorhombic distortion. Unfortunately, the 002 peak was not accessible within the x-ray aperture of the DACs for the orientation of the crystals that have been here characterized.

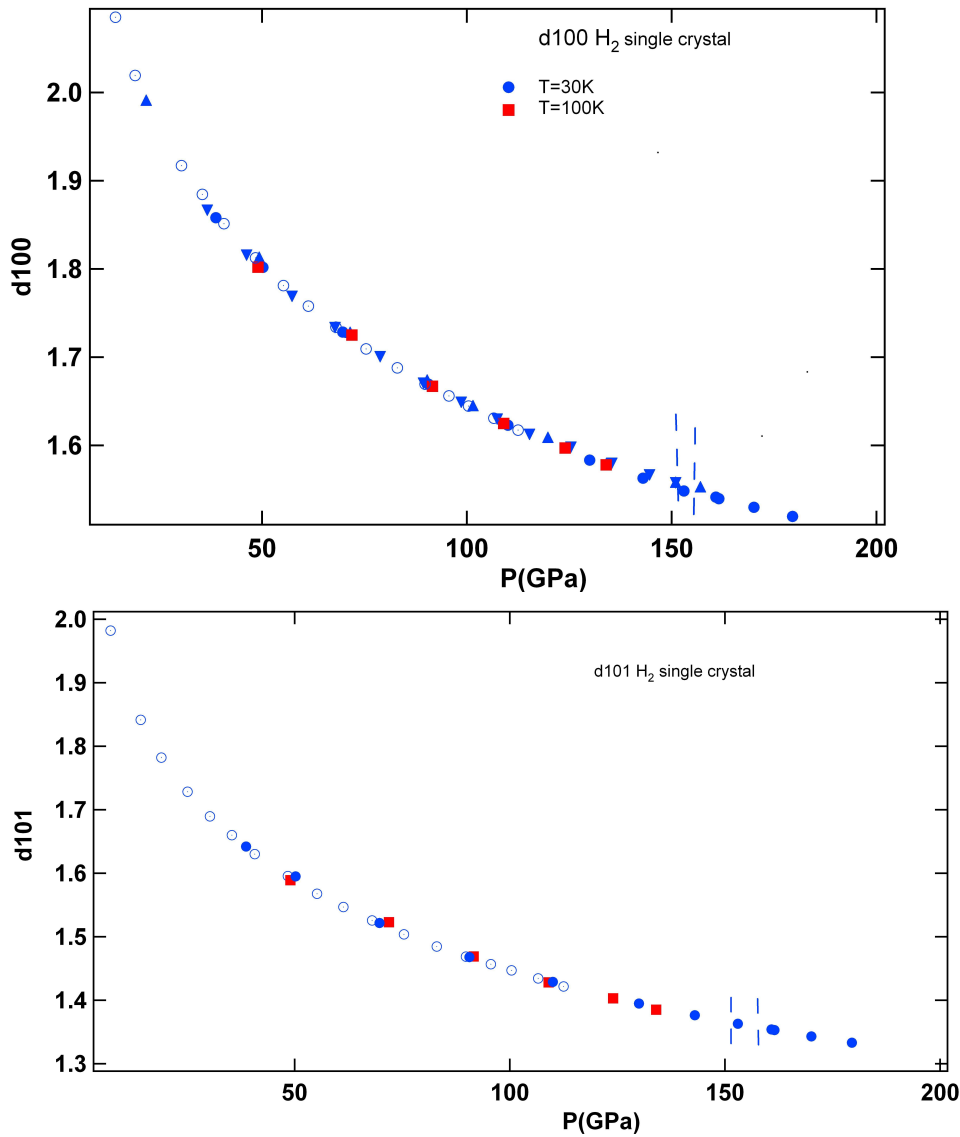


Figure 1: Evolution of the  $d_{100}$  and  $d_{101}$  lattice parameter versus pressure. Different symbols indicate different single crystals. Experiments have been performed at 30 K (bleu) and 100 K (red). The vertical dash line indicate the transition pressure range to phase III over which the vibron frequency discontinuity was observed. .

## References

- 1) Loubeyre, P. et al, **Nature** **383**, 702-704 (1996).
- 2) Goncharenko, I. and Loubeyre, P., **Nature** **435**, 1206 (2005).
- 3) Loubeyre, P., Occelli, F. and LeToullec, R., **Nature** **416**, 613-617 (2002).