ESRF	**Experiment title: Structural determination of the High pressure solid phases of Hydrogen.**	**Experiment number**: HS- 3018
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Report:

The aim of this proposal was to measure the structural properties of solid H_2 in between 100 GPa and 200 GPa below 100K. Consequently, structural data on phase II , above 110 GPa and on phase III, above 150 GPa could be obtained.

The size of H2 crystals under pressure over 1 Megabar is very small (less than 10 µm), hence with a very small scattering power. Consequently, the prerequisite condition for the success of x-ray diffraction experiment is to be able to keep a single crystal of hydrogen up to above 150 GPa. If the crystal is too much broken, its X-ray scattering signal merges into the background. Also, all the parasitic diffraction, coming from the gasket or from the cryostat, has to be removed. If not, it can overwhelm the H_2 crystal signal. For that, the configuration of the beamline and of the diffraction setup (focus of the beam, cleaning up with pinholes, extra beamstops to remove parasitic diffraction from the windows of the cryostat, lead shield) has been optimized. Also, to safely reach pressures greater than 150 GPa, culets of less than 75 µm diameter have to be used.

To avoid the breakage of the crystal due to the decrease in thickness of the gasket, we had proposed to use diamond anvil with a pit . The H_2 crystals could then be confined in a spherical shell of helium pressure transmitting medium. Unfortunately, the making of a pit in the culet then favored the diffusion of helium in the anvil under pressure that breaks the diamond. All our attempts failed. Not using helium pressure transmitting medium, even with a ring of gold was not good enough to keep the diffraction from the H_2 broken single crystal out of the noise.

We summarize in the table below, the various attempts and the measurements achieved. Out of five samples, only one could be brought up to 160 GPa. This single crystal of H_2 was embedded in helium.The crystal was oriented with a c-axis almost parallel to the axis of the diamond. Consequently, within the x-ray aperture of

the DAC only the 3 diffraction peaks, 100, 1-10, 010 were accessible. So, no information on the c-axis could obtained. The pressure was measured by the $SrB_4O_7:Sm^{2+}$ luminescence gauge.

Sample	**Anvils**	**Measurements**	**Maximum pressure**
Pure H2	75 µm culets with pits	100 and 101 peaks	80 GPa @25 K.
H2 in gold ring	75 µm culets.with pit. ring of gold to improve hydrostaticity.		Instability
H2 in helium	75 µm culets with pits.		Breakage 60 GPa..
H2 in gold ring	100 µm culet	100, 101 and 002 peaks	110 GPa @ 25 K
H2 in helium	75 µm culet	100, 1-10 and 010 peaks	160 GPa @ 40 K

The evolution of the 100 d-spacing measured on the last sample is shown in the figure below. This measurement demonstrates the feasibility of obtaining diffraction data in phase III. It is seen that the discontinuity of d_{100} at the II-III transition is small. At least an other orientation, which can give access to peaks along the c-axis is required to obtain the unit cell of phase III.

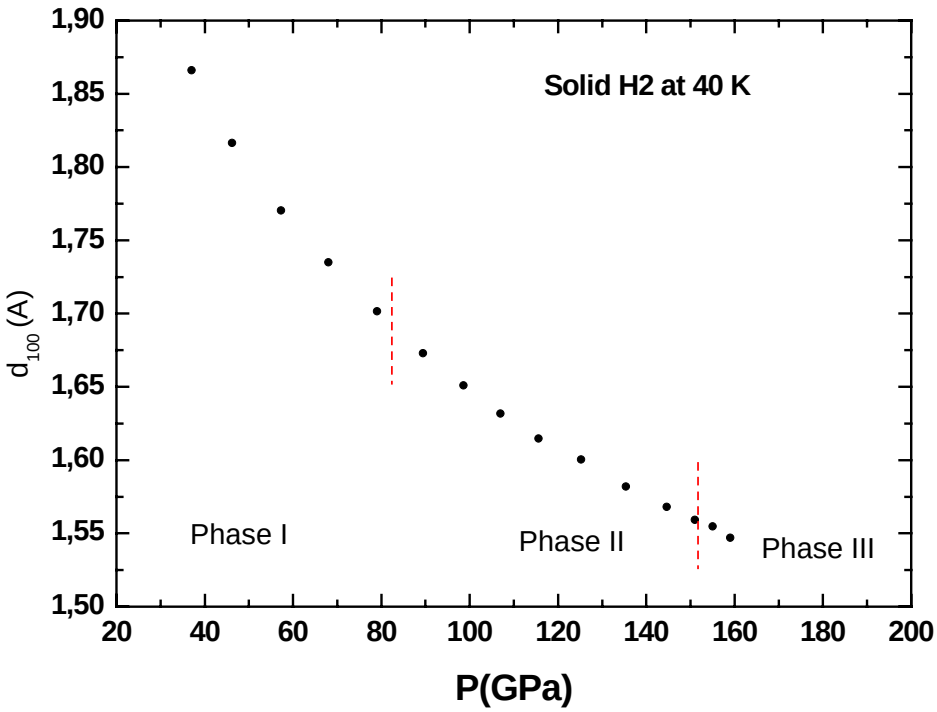

Figure 1 : Evolution of d_{100} of solid H_2 at 40 K up to phase III.