



	Experiment title: PDF analysis of liquid and amorphous complex hydrides	Experiment number: CH4986
Beamline: ID31	Date of experiment: from: 09/03/2017 to: 12/03/2017	Date of report: 28/04/2020
Shifts: 9	Local contact(s): Maria Valeria Blanco	<i>Received at ESRF:</i>
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Report:

Pure borohydrides, and mixture of them were analysed as a function of temperature by in-situ high energy synchrotron X-ray diffraction, collecting patterns at high resolution. In details, patterns were collected for: LiBH_4 , $\gamma\text{-Mg}(\text{BH}_4)_2$, $\text{LiBH}_4\text{-NaBH}_4$, $\text{LiBH}_4\text{-KBH}_4$, $\text{NaBH}_4\text{-KBH}_4$, $\text{LiBH}_4\text{-Mg}(\text{BH}_4)_2$ and $\text{LiBH}_4\text{-Ca}(\text{BH}_4)_2$.

We aimed at the investigation of a short order in the solid and liquid state by PDF analysis. The study was conducted using a custom cell available at ID31, which allowed to load backpressure of hydrogen in order to avoid the decomposition of the samples and any contact with air, since the materials are moisture sensitive.

The setup allowed to collect patterns of the empty cell loaded with hydrogen, to be used as background, and of the cell filled with above cited samples.

The samples were heated up to the desired temperature, in order to overcome the melting temperature, using a blower. While heating, patterns were collected to monitor phases evolution. Once the liquid phase was obtained, the collection of pattern continued, and a cooling ramp was performed afterward to check the reversible solidification from the liquid. We encounter some problems in managing the heating and data collection, because samples were often moving inside the capillary, making the pattern collection tricky, and no camera was available to check the state of the sample while measuring.

Unfortunately, up to now, it was not yet possible to process the collected data and obtain some evidence of the occurrence of short-range order of the liquid phase, because of the limited quality of the data collected, due to problems related to compound/phase stability and statistics.