



	Experiment title: Complex mixtures of borohydrides	Experiment number: CH5590
Beamline: ID11	Date of experiment: from: 23/11/2018 to: 27/11/2018	Date of report: 28/04/2020
Shifts: 12	Local contact(s): Carlotta Giacobbe	<i>Received at ESRF:</i>
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Report:

Mixtures of borohydrides or borohydrides with halides were investigated as a function of temperature by in-situ high energy synchrotron X-ray diffraction, collecting patterns at high resolution. Collected data are experimentally supporting the analysis and assessment of binary and ternary phase diagrams of borohydride by the CALPHAD method for the full description of thermodynamic properties of these compounds as a function of composition, temperature and pressure. 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.

Biphasic and monophasic mixtures in the $\text{LiBH}_4\text{-LiBr-LiCl}$ system were investigated applying thermal ramps (heating and cooling from room temperature up to approx. 500 °C) for defining the solubility limits of the $\text{Li}(\text{BH}_4)_{1-x-y}(\text{Br})_x(\text{Cl})_y$ solid solution as a function of temperature. This data will use for the assessment of the ternary diagram with the CALPHAD method which is still under study.

Another investigated system was the binary $\text{Mg}(\text{BH}_4)_2\text{-Ca}(\text{BH}_4)_2$, which presents many polymorphs and also some unknown decomposition products. Obtained diffraction data (**Figure 1**) will be directly correlated with DSC measurements acquired at UNITO. In this way, possible phase transition or decomposition can be highlighted.

No particular difficulties were encountered, however the data analysis required long time for the huge amount of data collected, and we expect many experimental data information that will fully support our thermodynamic analysis and assessment.

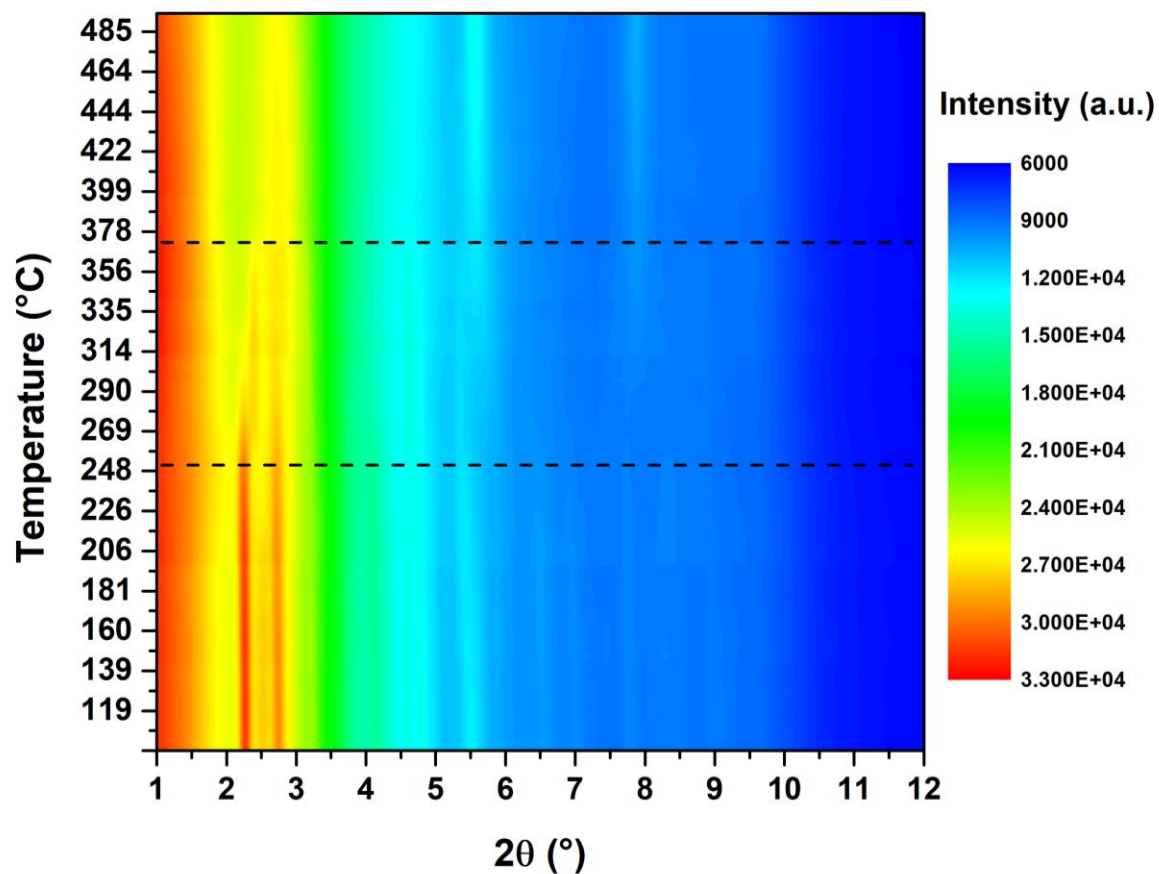


Figure 1. *In situ* SR-PXD of $\text{Mg}(\text{BH}_4)_2\text{-Ca}(\text{BH}_4)_2$ 1:1 sample heated from room temperature to 500 °C. A first decomposition process is observed from $T \sim 250$ °C to 370°C highlighted by the black dashed lines.