# STRUCTURAL STUDIES OF MATERIALS FOR HYDROGEN STORAGE

**In-situ SR-PXD measurement: 01-02-707 (May and September 2006) Beamline BM01A**

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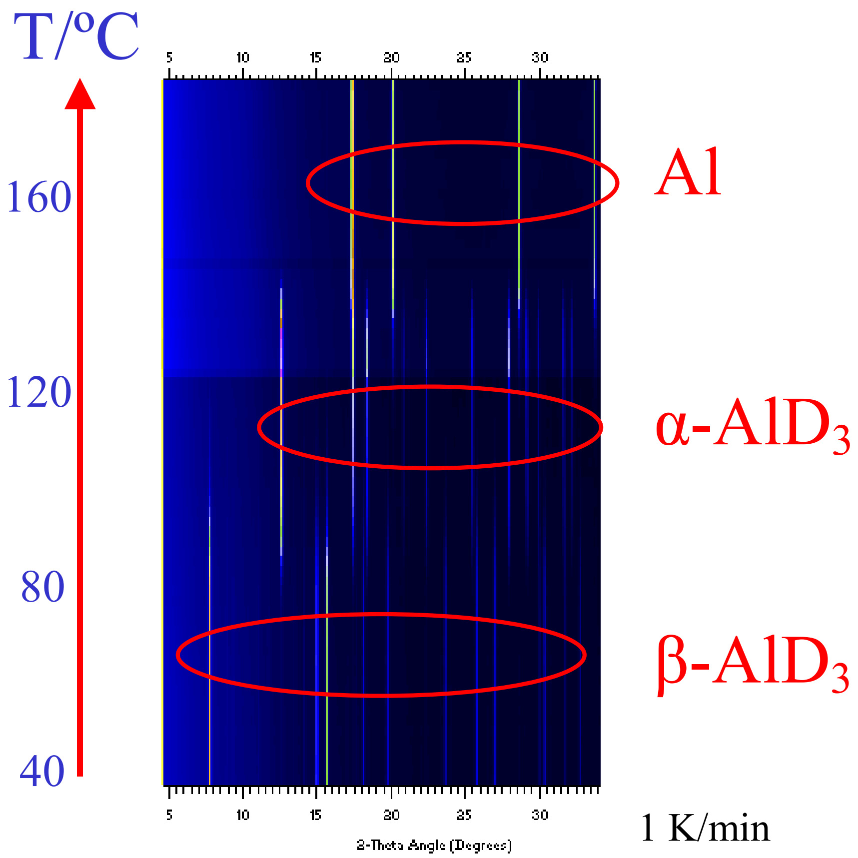
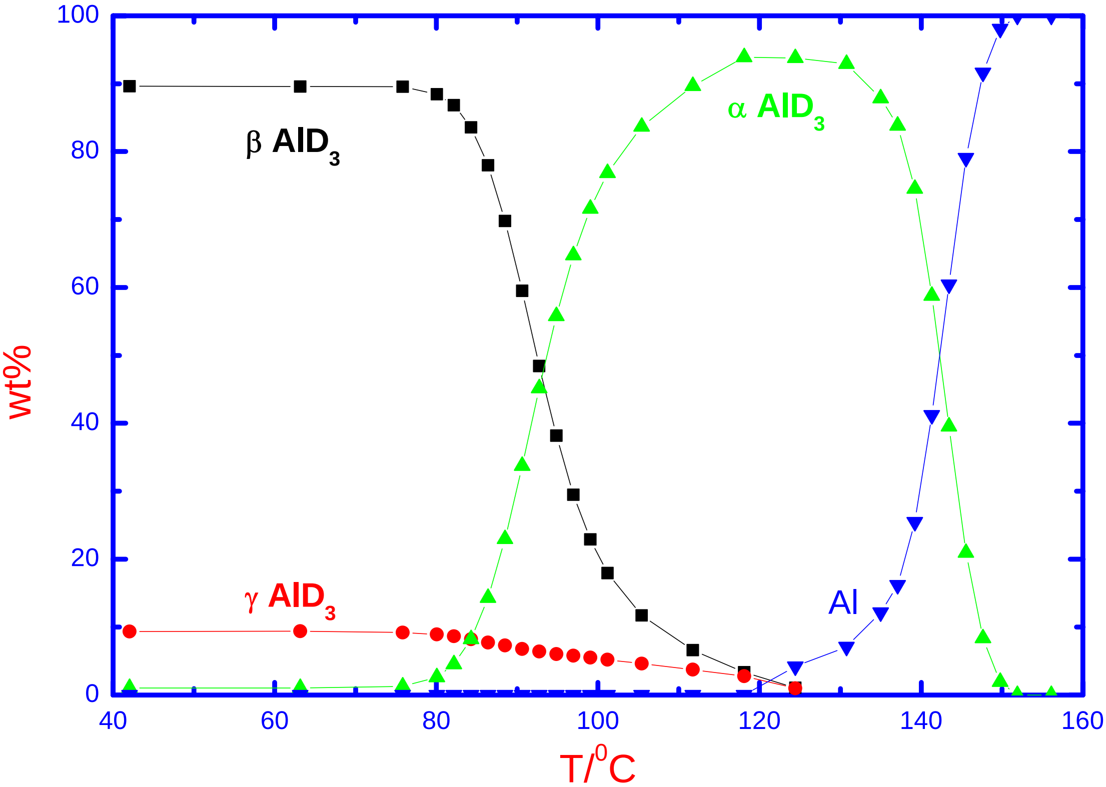
All experiments were performed under dynamical vacuum to investigate the thermal decomposition behaviour of hydrogen storage materials.

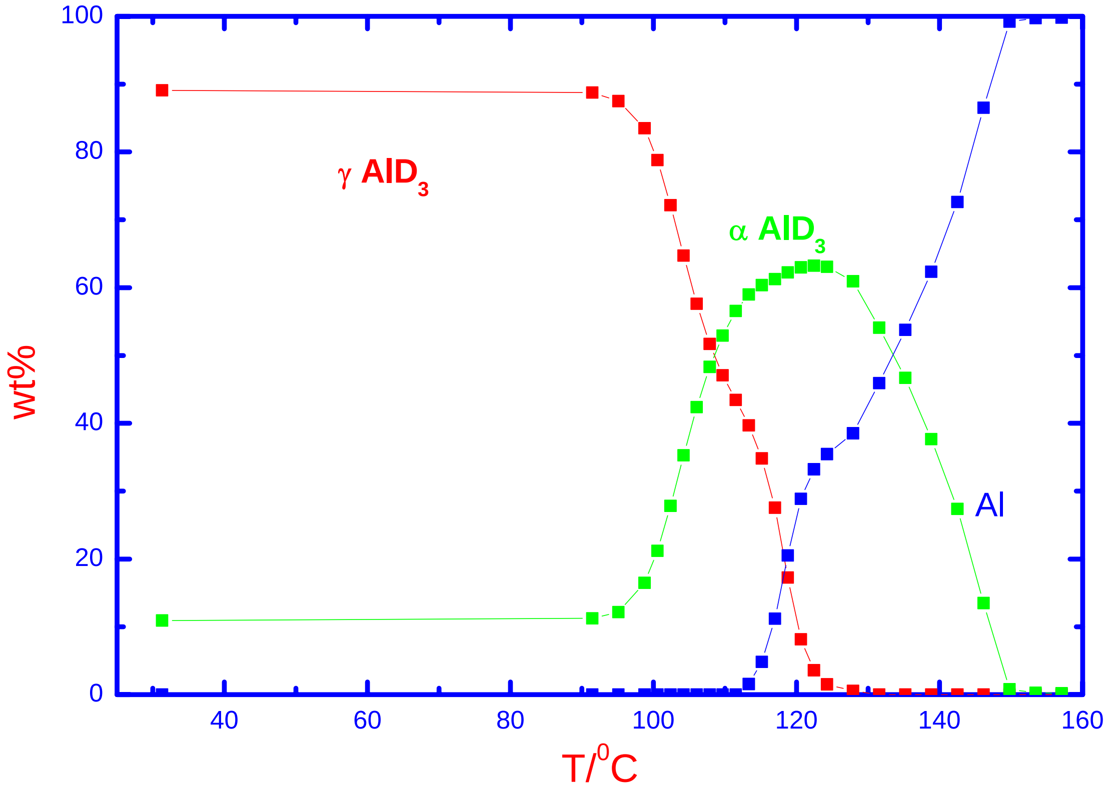
## $AlD_3$

$AlH_3$ ("alane") contains 10 w% hydrogen which is released at moderate conditions and is therefore a potential hydrogen storage material for use in vehicles.

The thermal decomposition of the two modifications β-$AlH_3$ and γ-$AlH_3$ were investigated using a heating rate of 1K/min. Accurate quantitative phase analysis could be performed thanks to the structure determinations done on these phases at BM01B. Both the β- and the γ-phase are transformed to α-$AlD_3$ prior to decomposition to Al and $H_2$ gas. The manuscript for these results is in the final stage of preparation.





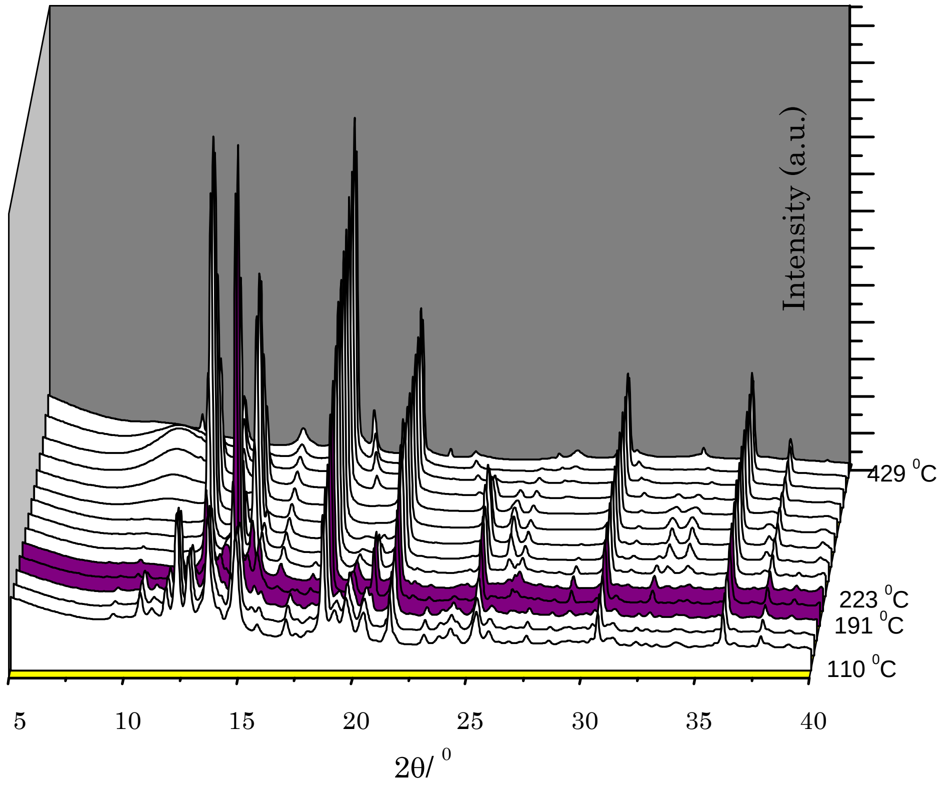




*Results from quantitative Rietveld phase analysis of thermal decomposition of samples containing (primarily) β-$AlD_3$ (top) and γ-$AlD_3$ (bottom).*

**$KAlH_4$**

$KAlH_4$ is stable up to 150 ˚C when it starts to decompose and a new phase appears. According to preliminary results, it comes out that $K_3AlH_6$ is formed. Since the structure of $K_3AlH_6$ is not yet solved, these measurements could contribute to its determination.

At the same time a minor quantity of an unidentified secondary phase is formed. At higher temperatures $K_3AlH_6$ decompose and KH is formed. After 325 ˚C, the decomposition of KH is complete, giving a final powder containing the only crystalline phase of LiAl, Al and the unidentified phase. The phases evolution is shown below.




*In situ PXD sample $KAlH_4$ + KH + LiH. The sample was heated under vacuum fro room temperature to 430 ˚C at a constant heating rate of 2 ˚C/min. In violet is evidenced the phase $K_3AlH_6$.*

**$LiMg(AlD_4)_3$**

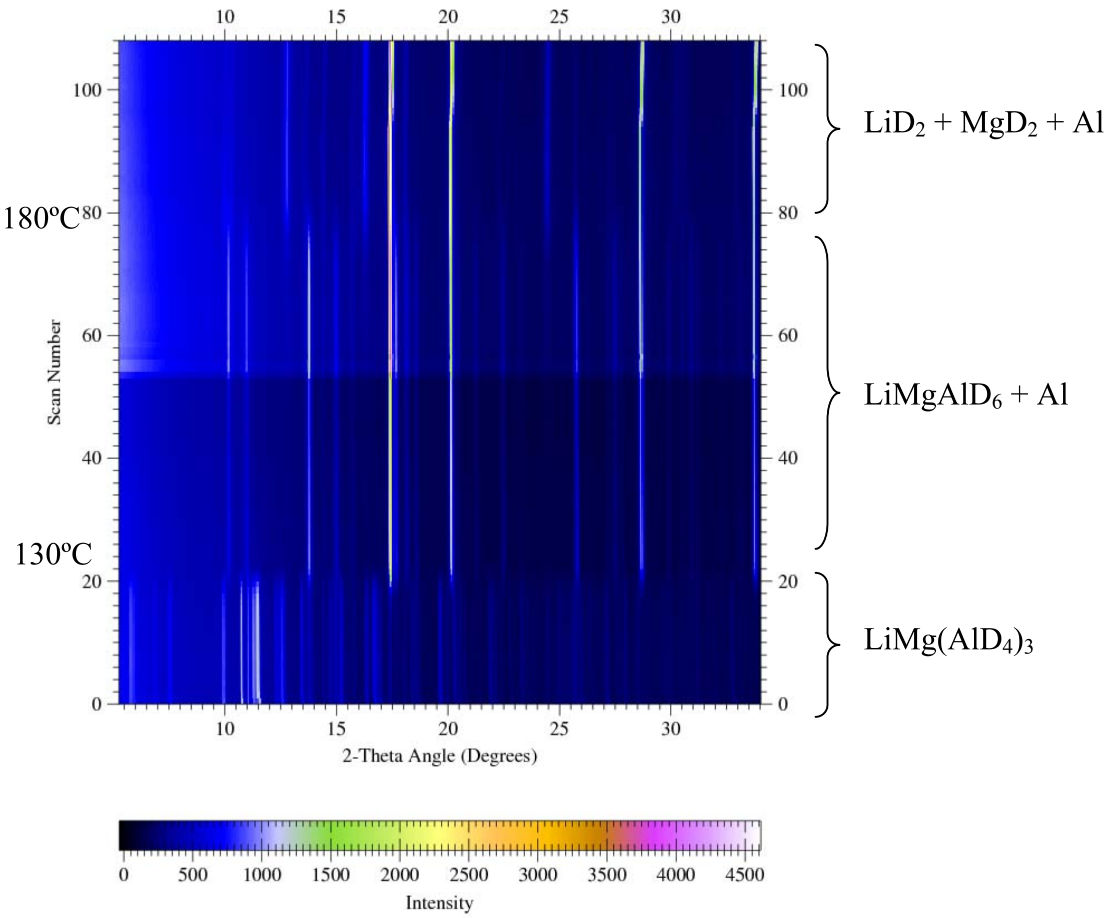
A highly crystalline sample of $LiMg(AlD_4)_3$ was heated at constant rate 0.5°C/minute from 100°C to 200°C under dynamic vacuum; synchrotron powder X-ray diffraction data was recorded every second minute. Analyzing the diffraction data gives that at about 130°C $LiMg(AlD_3)_4$ releases deuterium according to:

$LiMg(AlD_4)_3 \rightarrow LiMgAlD_6 + 2Al + 3D_2$

Further heating to about 180°C caused decomposition of $LiMgAlD_6$ according to:

$LiMgAlD_6 \rightarrow LiD + MgD_2 + Al + 3/2\ D_2$

This desorption experiment gave us information about the temperature at which the two desorption reactions occur and also confirmed the decomposition reactions above. The results will be published later.



**$Ca(AlH_4)_2$**

A sample with $Ca(AlH_4)_2$ was heated from 100°C to 250°C with a constant rate of 2°C/minute. $Ca(AlH_4)_2$ decompose according to:

$Ca(AlH_4)_2 \rightarrow CaAlH_5 + Al + 3/2\ H_2$

and

$CaAlH_5 \rightarrow CaH_2 + Al + 3/2\ H_2$.

The data are being analyzed and the results will be published later.

## General remarks

Right before the beginning of our run in September 2006, the monochromator lost its cooling and was seriously misaligned due to overheating. Thanks to the intensive effort from the SNBL staff, the beam was recovered but we lost some 32 hours out of our 48 hours of beam time.