

STRUCTURAL STUDIES OF MATERIALS FOR HYDROGEN STORAGE

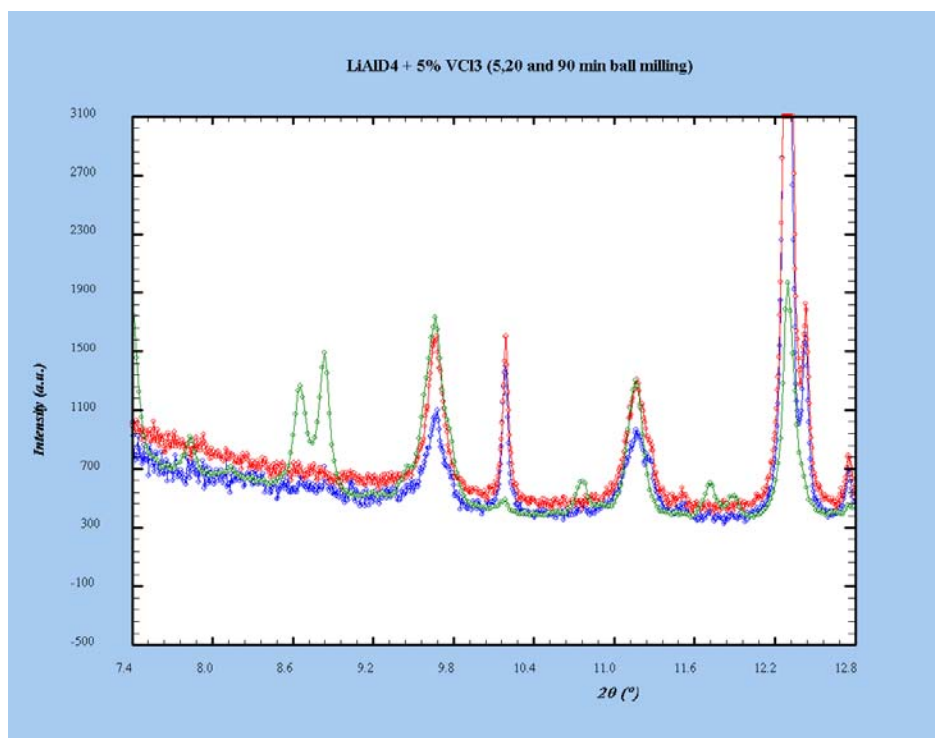
High Resolution measurement: 01-01-679 (May 2005) Beamline BM01B

D. Blanchard, M. Sørby, M. Pitt, H. W. Brinks, B. C. Hauback,

Department of physics, Institute for Energy Technology, P. O. Box 40, NO-2027 Kjeller, Norway.

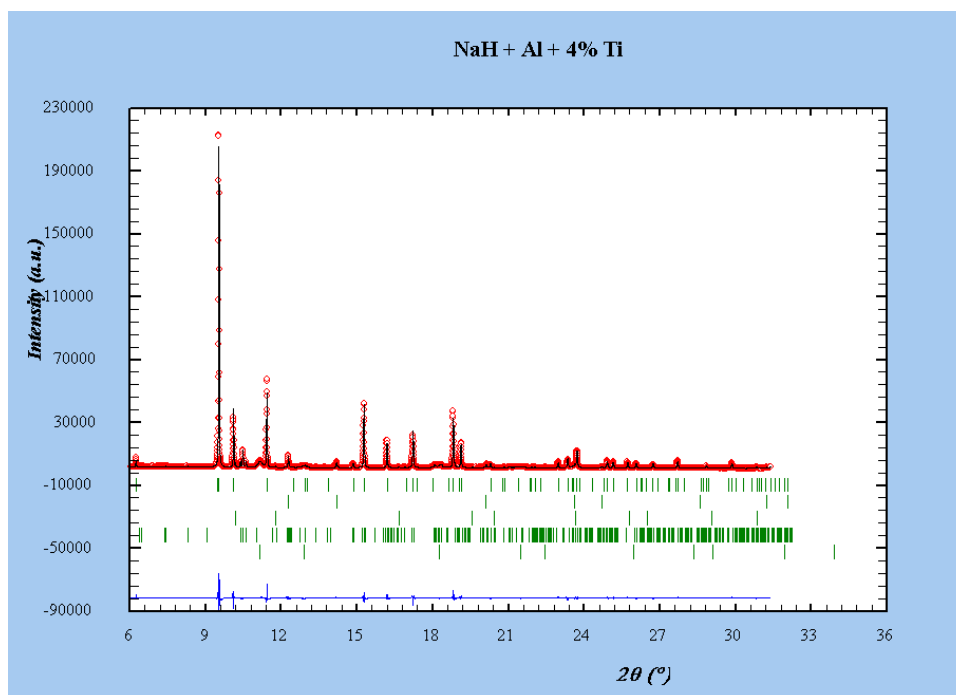
LiAlD₄ + 5% VCl₃

The samples were measured as a function of ball milling time, because of earlier results indicating LiCl formation under tough ball milling conditions, but formation of a Li-V-Cl phase under milder conditions. Since vanadium apparently acts as a catalyst for decomposition of LiAlD₄ all information of the state of vanadium is of interest. Our theory was confirmed at SNBL.



NaH + Al + Ti

It has earlier been found by two paper of Wang et al. that it is possible to add Ti catalyst as metal in a NaH + Al mixture, thus avoiding by-products like NaCl (with TiCl_3) and simplifying the production of reversible metal hydride. Ball milling of the mixture in H_2



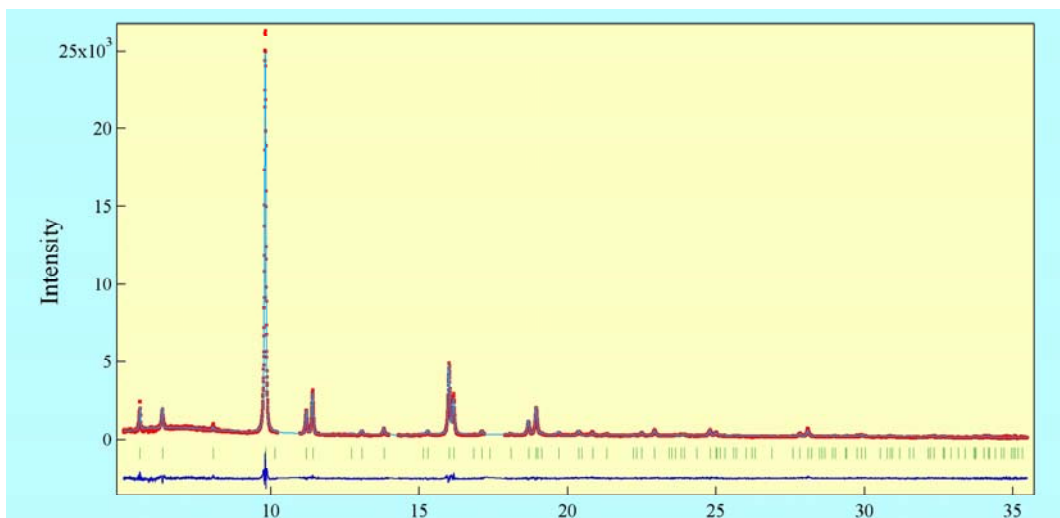
was found to give better kinetics than ball milling in Ar. Our idea was to try to find out why this difference occurs. Preliminary analyses have not given us this answer, but we have found that in this case the only crystalline Ti-species after cycling is different from the Ti-species we found after cycling TiCl_3 -doped NaAlH_4 samples at SNBL. This results is submitted in a paper (H.W. Brinks, M. Sulic, C.M. Jensen, B.C Hauback to J. Phys. Chem. B). The paper is a continuation of the work mainly based on SNBL data (2004): H.W. Brinks, S.S. Srinivasan, B.C. Hauback, C.M. Jensen, J. Phys. Chem. B 109 (2005) 15780.

Microstructural analysis of titanium in NaAlH_4

In order to get a more detailed understanding of the catalytically active phases in NaAlH_4 after addition of TiCl_3 , a series of samples were prepared and measured at SNBL. This time, freshly ball milled samples (to avoid relaxation of strains) as a function of additive amount was carried out, in order to determine the evolution of dislocations (types and densities) and size. The results are yet being analysed and will be compared with complementary techniques.

Structure of amides/imides

A promising group of hydrogen storage materials is the reversible reaction between amides (NH_2^-) and imides (NH^{2-}). Structural investigation of the materials is important, and was carried out at SNBL in combination with neutron diffraction data for Li_2ND , LiND_2 and MgND . Li_2ND appears to undergo structural transitions below room temperature (probably rotation of imide group) and will be studied in more detail in near future.



Rietveld refinement of LiND_2 data from SNBL

K_3AlD_6

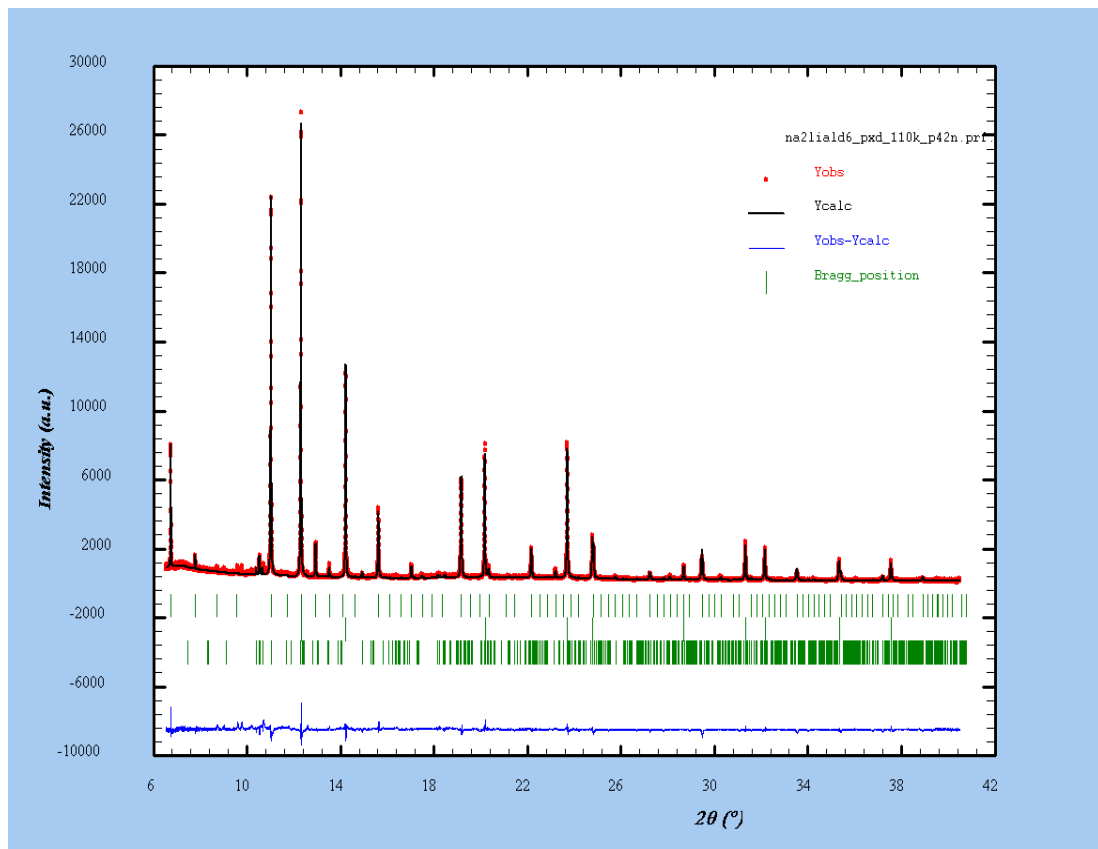
SNBL data of K_3AlD_6 has been obtained and the structure is soon going to be published. It is related to the $\text{Na}_2\text{LiAlH}_6$ structure.

Zr_2CuD_4

Structural determination of Zr_2CuD_4 and $\text{Zr}_2\text{CuD}_{4-x}$ has been attempted by SNBL data.

$\text{Na}_2\text{LiAlD}_6$, low temperature structure

$\text{Na}_2\text{LiAlD}_6$ has from neutron diffraction been determined take a lower symmetry at 8 K, compared to the Fm-3m structure at room temperature. Based on the PND data three alternative space groups were possible, and SR-PXD data at 110 K were obtained in an attempt to reveal the true symmetry. The splitting of the reflections turned out to be too small to distinguish between the different models. This work is also supported by state-of-the-art DFT calculations which show that all three structures are nearly equal in energy with the Fm-3m structure. Phonon calculations to find the temperature dependence of the structures are nearly completed.



LaPtInD_x

This is an analogue of LaNiInD_{1.333} which has extremely short D-D distance of about 1.6 Å, which breaks the well-established rule that H-H distance of fully occupied hydrogen positions should exceed 2-2.1 Å because of repulsion. Structural investigation of this compound has successfully been carried out.

