STRUCTURAL STUDIES OF MATERIALS FOR HYDROGEN STORAGE – high resolution experiments - 01-01-618 (November 2004). Beamline BM01B

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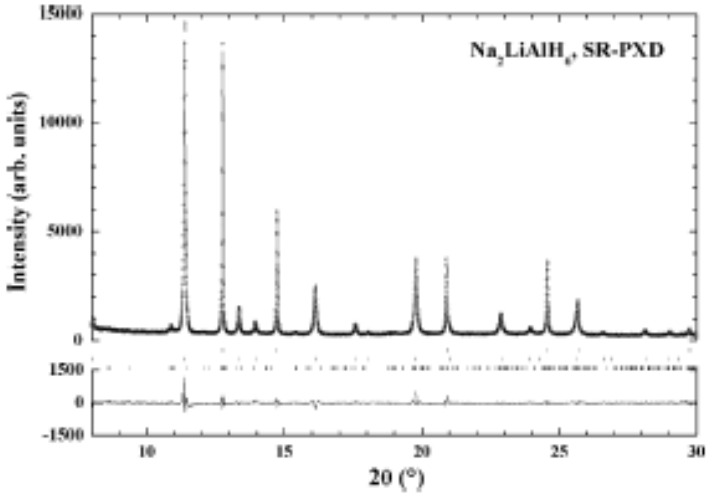
In a three-days measurement period 26th – 29th November 2004, 24 samples were measured and will probably result in 6-8 refereed papers. One is already accepted in J. of Alloys and Compounds. The focus of the measurements were on Al-based complex hydrides, and some highlights are:

Na_2LiAlH_6 + 2% TiF_3

As-prepared Na_2LiAlH_6 was compared with Na_2LiAlH_6 + 2% TiF_3 after 6 weeks of Pressure-Composition Isotherm (PCI) measurements at 170-250ºC. The unit-cell dimensions of Na_2LiAlH_6 were found to be the same, and there is hence no solid solution of the additives in any of the positions in the metal hydride.* But at least part of the catalysing Ti was found to be present in an $Al_{1-x}Ti_x$ phase. This phase is different from the $Al_{1-x}Ti_x$ phase found for $NaAlH_4$ with different Ti-based additives. This will be discussed in a forthcoming paper.

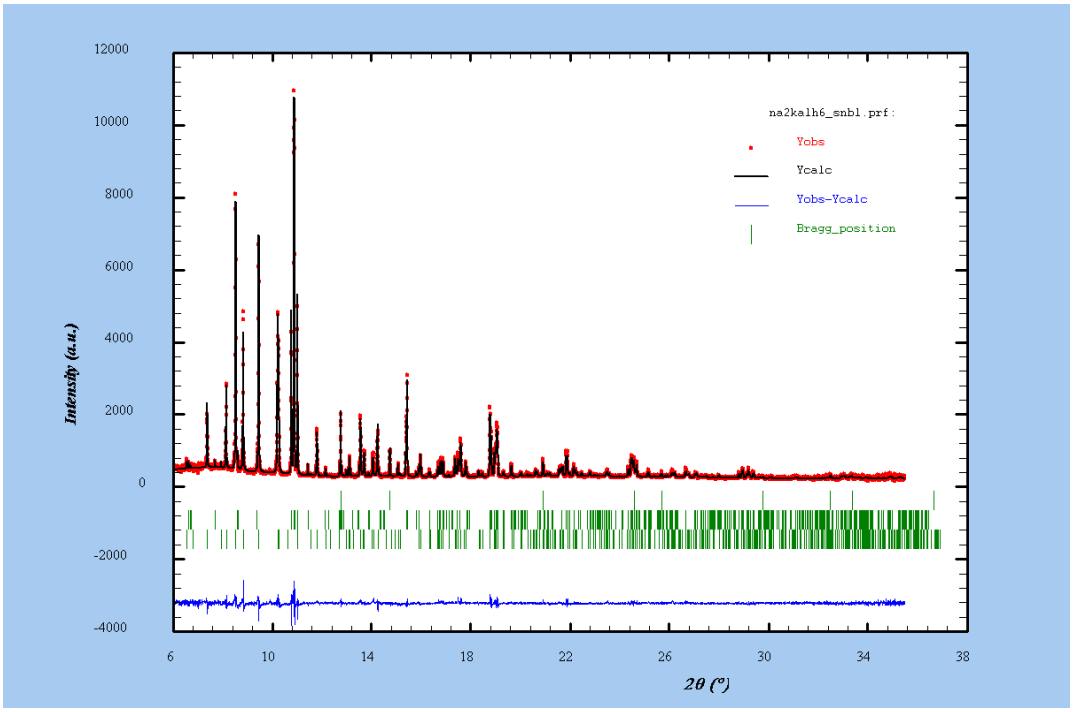


*Figur 1: BM01B data of Na_2LiAlH_6 before PCI measurements**

* A. Fossdal, H.W. Brinks, B.C. Hauback. J. Alloys Comp. 2005, In press

New Alanates

Samples of K_2LiAlD_6 and Na_2KAlH_6 have been synthesized. Na_2KAlH_6 was found to be phase separated into Na_3AlH_6 and K_2NaAlH_6, and the latter is now determined to be isostructural with Na_2LiAlH_6. A publication covering this is now in preparation. The structural determination of K_2LiAlD_6 is at present found from combined PXD synchrotron and neutron diffraction data.



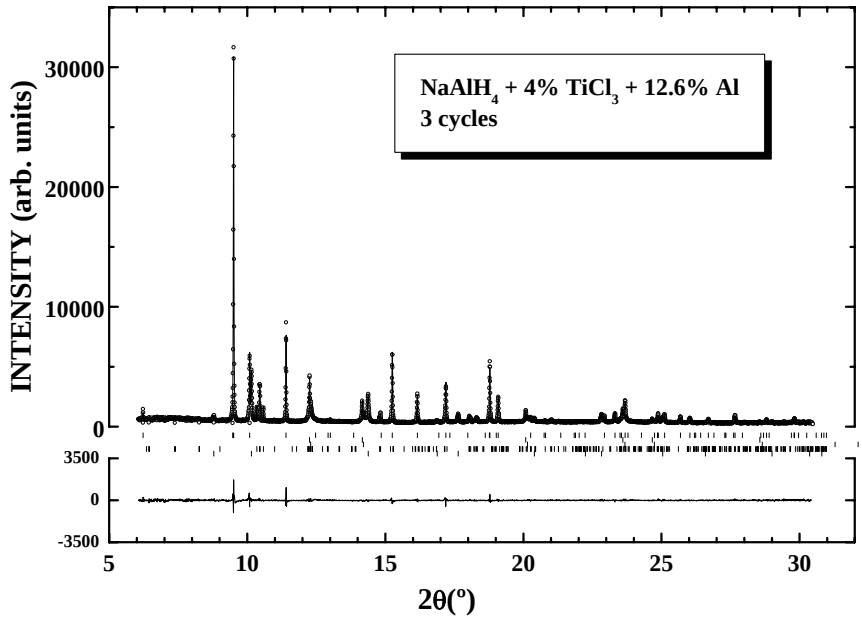
Figur 2: PXD diagram showing that Na_2KAlH_6 is not thermodynamically stable and that synthesis of this compound result in phase separation to K_2NaAlH_6 and Na_3AlH_6.

Microstructural investigation of Ti-enhanced $NaAlH_4$

In order to completely understand the mystery of the role of Ti in enhancing the kinetics in Al-based complex hydrides, a thorough understanding of the microstructure appears to be important. This may be done by analysing the broadening of high-resolution PXD diagrams. 5 samples specially made for this purpose by varying the synthesis conditions were measured, and the data is now being analysed for particle size and dislocation type and density. Based on the results from the analyses of these data, further data will be collected at the experiment in May 2005.

$NaAlH_4$ + $TiCl_3$ with excess Al

Because of diffusion problems of Al and the fact (earlier determined at SNBL), that $Al_{1-x}Ti_x$ formation prevents Al from reacting with Na_3AlH_6 to form $NaAlH_4$, a selection of samples with excess Al was synthesized and measured. These indicated that excess Al indeed could increase the storage capacity and improve the kinetics of the Ti-enchanced $NaAlH_4$ system.



Figur 3: Rietveld refinements of $NaAlH_4$ with 4% $TiCl_3$ and 12.6% Al after 3 cycles.

Structural determination of Ni-based complex hydrides.

$Na_2Mg_2NiD_8$ and $Li_2Mg_2NiD_8$ were synthesized by high-pressure techniques and were, in addition to powder neutron diffraction also measured at SNBL for accurate determination of the metal positions in the structural determination. The paper is in preparation.