ALANATES FOR HYDROGEN STORAGE: TIME-RESOLVED AND HIGH RESOLUTION POWDER DIFFRACTION EXPERIMENTS.

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The project focuses on studies of metal hydrides based on light-weight elements. During the last few years different so-called alantes e.g. $LiAlH_4$, $NaAlH_4$ containing up to 10 wt% hydrogen have been intensively studied. Even though these materials have been known for a long time, details about structure of the starting material and desorption products are defective. For possible applications, doping/catalysts are needed, but the effect of the catalyst/dopants on the absorption / desorption process is not understood.

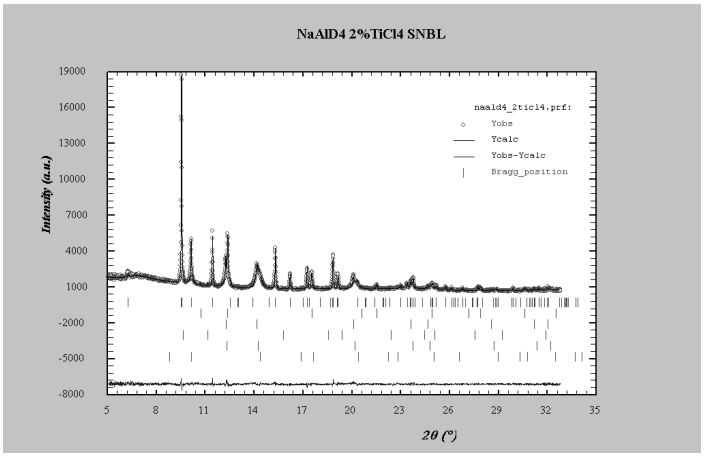
The high-resolution experiments were carried out on the following samples:

(a) $NaAlD_4$, pure and doped with $TiCl_4$ (3 samples).
(b) $LiAlD_4$ doped with VCl_3 and $(Ti,Al)Cl_3$ (4 different samples).
(c) $KAlD_4$.

All samples were in 0.5 mm boron-glass capillaries. The wavelength was 0.49972 Å.

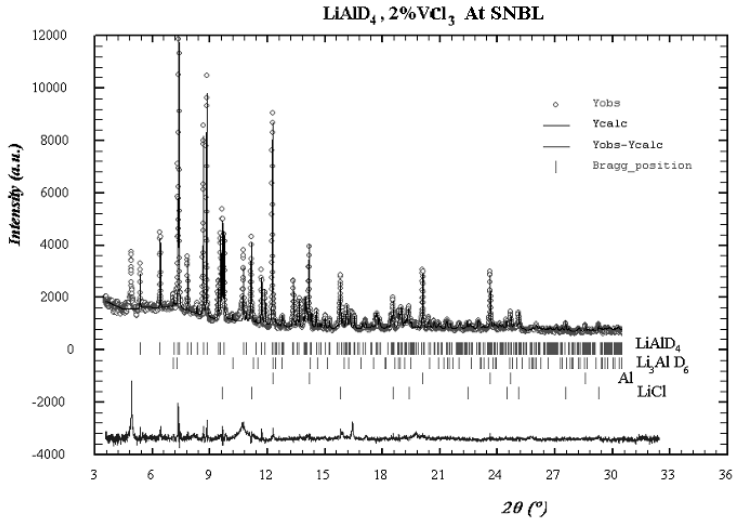
(a) $NaAlD_4$.

Ti additives gives a large reversible storage capacity, and it is very important to understand the nature of the Ti additives, which improves the desorption kinetics and makes reversibility possible. $NaAlD_4$ (pure, 2% $TiCl_4$, 6% $TiCl_4$ and 10% $TiCl_4$) was measured at room temperature. At the moment, data analysis to find the positions of Ti is being carried out. Preliminary analyses suggest a secondary cubic phase with a ≈ 5.6 Å.



(b) $LiAlD_4$.

$LiAlD_4$ doped with 2 and 5 mol% VCl_3 and $(Ti,Al)Cl_3$, respectively, were studied. The samples were prepared by ball-milling, and the motivation was to study how doping influences the structures. No changes in the $LiAlD_4$ unit cells were observed by doping. Determination of the phase compositions showed that the doping procedure induced a partial



decomposition of $LiAlD_4$ into Li_3AlD_6, LiD and Al. The VCl_3 gave the smallest degree of decomposition. Nevertheless some peaks still remain unexplained, and work is in progress.

(c) $KAlD_4$.

The structural arrangement of $KAlD_4$ has been suggested by Bastide *et al*. (Rev. Chim. Min. 24 (1987) 248) and Vajeeston *et al.* (to be published) based on density-functional total-energy calculations. A sample of $KAlD_4$ with small amounts of LiF was studied in the experiments on BM01B. The step-length for the data collection was 0.008° in 2θ. A Rietveld refinement is shown below. The refined parameters are: a = 8.8448, b = 5.8035, c = 7.3367 Å in space group *Pnma.* Detailed structural refinements are in progress using the combination of the present synchrotron X-ray diffraction data and neutron diffraction data from the PUS diffractometer at the JEEP II reactor at IFE, Kjeller in Norway.