STRUCTURAL STUDIES OF METAL HYDRIDES FOR BATTERY AND HYDROGEN STORAGE APPLICATIONS.

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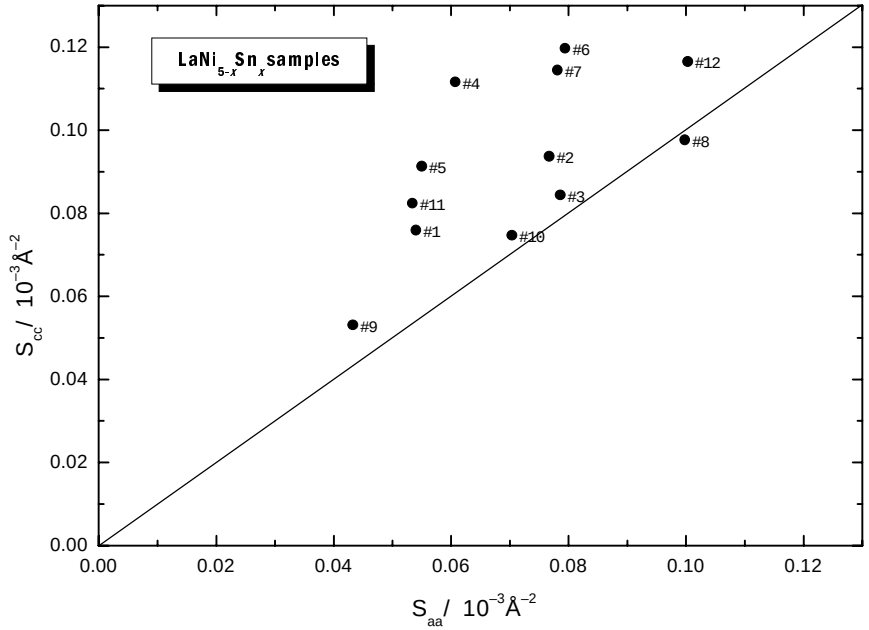
The project focuses on two important aspects of hydrogen storage in metal hydrides: (a) detailed studies of tin-substituted $LaNi_5$ compounds (both stoichiometric and non-stoichiometric alloys) for battery applications and (b) studies of metal hydrides based on light-weight elements.

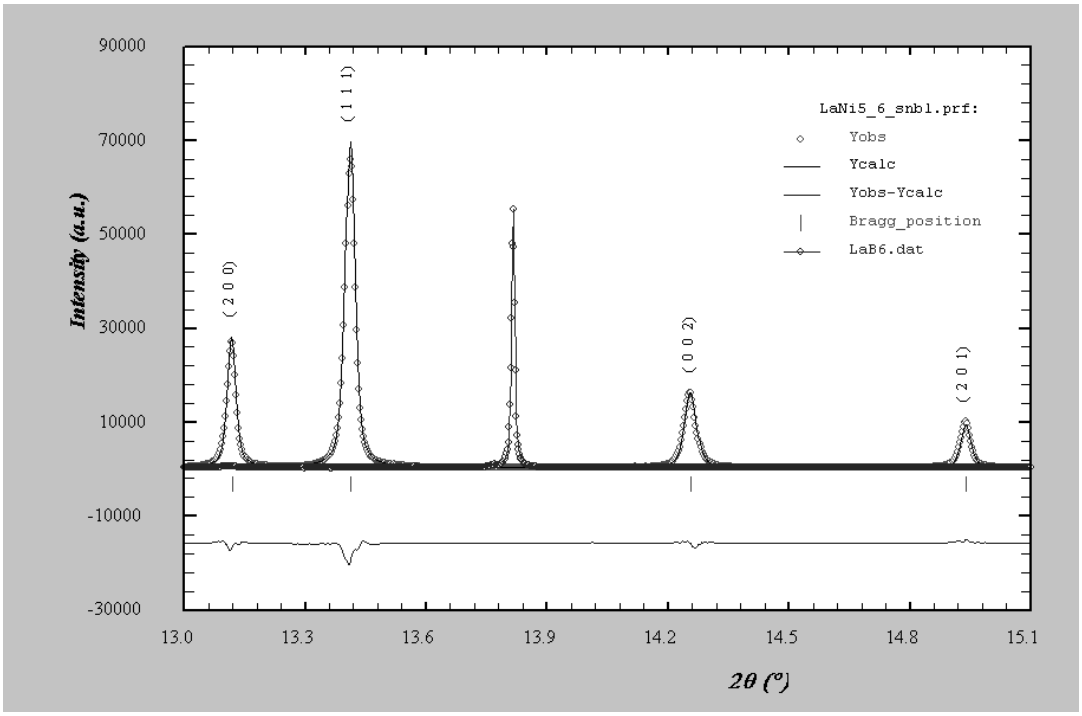
The $LaNi_5$ and $LiAlD_4$ samples were in 0.3 and 0.5 mm boron-glass capillaries, respectively. The wavelength was 0.49983 Å.

(a) Detailed structural studies of new tin-containing alloys for metal hydride battery applications.

$LaNi_5$ alloys substituted with tin improve corrosion resistivity and cycling stability and will be cheaper than commercial alloys for NiMH batteries. The major goal of the project was to study the microstructure for different samples in the $La(Ni,Sn)_{5\pm x}$-system prepared by different methods and to compare samples before and after reaction with hydrogen. This involves detailed analyses of the diffraction peak widths, and with a particular focus on anisotropic broadening. The effect of stoichiometry/non-stoichiometry was also a part of the experiment. This part of the proposal is in collaboration with Dr. R. C. Bowamn from Jet Propulsion Laboratory, NASA, USA.

In total 12 different samples were studied. The compositions were: $LaNi_{4.78}Sn_{0.22}$, $LaNi_{4.87}Sn_{0.22}$, $LaNi_{4.74}Sn_{0.22}$. The total measurement-times were 3-6 hours each. Data were collected with step-lengths 0.003-0.0035° in 2θ. LaB_6 was used as a standard. The data were analysed using a hexagonal strain model with strain along *a* (S_{aa}) and *c* (S_{cc}) and fully correlated strains (Cerny et al., J. Appl. Cryst. (2000), **33**, 997) using the FullProf software. The strain parameters for the different samples are shown in the figure below. Typical for the samples with largest anisotropic strain is $1.2 \cdot 10^{-4}$ Å^{-2}. Refined data for sample 6 together with a LaB_6 peak is shown in the figure. Notice that (002) is broader than (200). Further analysis of the data with individual profile fitting is in progress.





(b) Studies of metal hydrides based on light-weight elements.
During the last 4-5 years it has been an increased focus on the so-called alanates (e.g. $LiAlH_4$, $NaAlH_4$) as hydrogen storage materials. These compounds have been known for many years, their hydrogen gravimetric storage capacities are above 5-6 wt%, but the problem with respect to applications is very slow kinetics and lack of reversibility. However, by using catalysts (doping) and special sample treatments both the hydrogen release kinetics and possible reversibility can be improved. Still not very much is known about these compounds, even the structures of the different phases present in the desorption process. The $LiAlD_4$ system was studied in this experiment. During desorption both Li_3AlD_6, Al and LiD is formed in 2 steps. The process of desorption has been studied by thermal desorption spectroscopy. Both pure $LiAlD_4$ and 9 other samples prepared at different stages of the desorption process were studied in this experiment. Typical step-lengths for the data collection were 0.003° in 2θ. Le Bail-fit to sample prepared at 160 °C with mainly the Li_3AlD_6 phase is shown below. The refined parameters are: $a = 5.6373$, $b = 8.0708$, $c = 7.8698$ and $\beta = 92.0714^o$ in space group $P2_1/c$. Work is in progress on solving the unknown structure of Li_3AlD_6 using combined neutron (data from the PUS instrument at the JEEP II reactor at Kjeller, Norway) and the present synchrotron X-ray diffraction data.