SNBL	**Experiment title:** Powder diffraction line broadening study of hydrogen cycled $LaNi_5$-type compounds and implications on the corrosion resistance behavior of metal hydride battery electrodes.	**Experiment number:** 01-01-161 01-01-186
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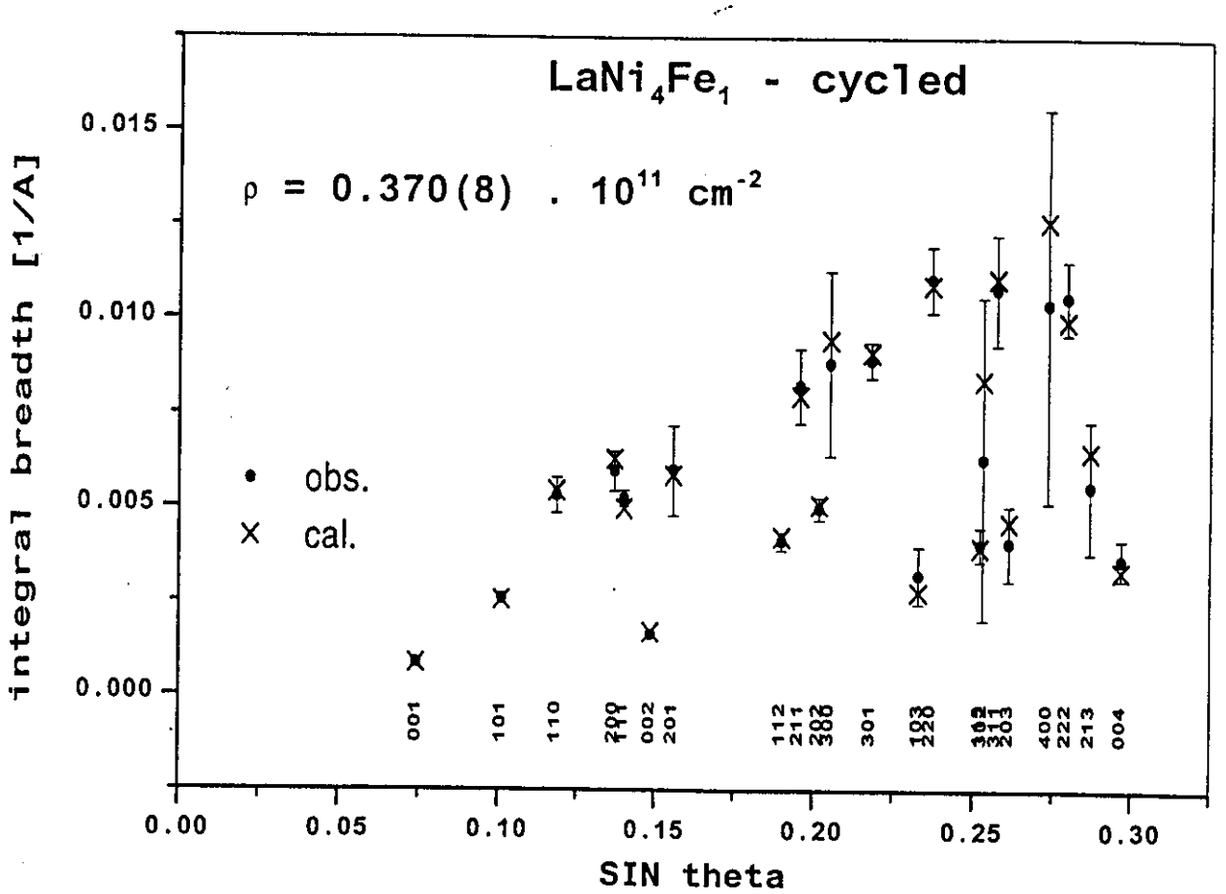
Hydrogen absorption-desorption cycling in $LaNi_5$ substituted compounds is known to produce diffraction line broadening which may be isotropic or anisotropic. Depending on the substitution nature and rate, the extent and character of the isotropic or anisotropic broadening can be modified. In order to establish correlations between the nature of crystal defects responsible for this broadening and the corrosion behaviour of those materials in nickel-metal hydride batteries, high resolution diffraction data are needed.

The present report is a final report on the long term project (see also report on 01-01-128) which was allocated 23.25 shifts in total (divided into three sessions). Sixteen alloys in non-cycled and cycled states have been studied : $LaNi_5$, $LaNi_{4.25}Co_{0.75}$, $LaNi_3Co_2$, $LaNi_{4.6}Mn_{0.4}$, $LaNi_4Mn_1$, $LaNi_{4.9}Al_{0.1}$, $LaNi_{4.7}Al_{0.3}$, $LaNi_{3.94}Mn_{0.4}Al_{0.3}Co_{0.36}$, $LaNi_{3.55}Mn_{0.4}Al_{0.3}Co_{0.75}$, $LaNi_{4.5}Sn_{0.5}$, $LaNi_4Cu_1$, $LaNi_4Fe_1$, $LaNi_{5.4}$, $La_{0.5}Ce_{0.5}Ni_5$, $LaNi_{3.85}Mn_{0.4}Co_{0.75}$ and $LaNi_{3.95}Al_{0.3}Co_{0.75}$. Powder samples were measured in the high-resolution Debye-Scherrer setting (0.3 mm capillary, crystal analyser) using wavelengths 0.65049, 0.50018 resp. 0.59716 Å. The NIST silicon powder (SRM 640b) was used for the wavelength calibration. All diffraction profiles were fitted individually with the symmetric Voigt function. The observed line breadths were corrected for the instrumental contribution

using the NIST LaB_6 powder (SRM 660) as the profile standard. Typical line breadths for LaB_6 were 0.01° 2 theta with a lorentzian shape. The line breadths of studied samples were in the range of 0.02 – 0.40° 2 theta which is 2 – 40 times greater than the instrumental broadening. At a laboratory source the sample broadening was similar to that of the diffractometer. The analysis of the line broadening was based on the theory developed by Krivoglaz [1]. The line broadening was supposed to be due to dislocations created during the absorption-desorption cycling. The line broadening anisotropy is described by orientation factors for different dislocation types and different reflections, and for hexagonal crystal system they were expressed analytically by Klimanek & Kuzel jr. in [2]. The edge dislocations of the type *a*/3 <-2110>{0-110} were found as dominating in most of the cycled alloys, especially $LaNi_5$, $LaNi_{5.4}$ and alloys substituted by Co and Fe. In the alloys substituted by Mn, Al, Sn, Cu and in $La_{0.5}Ce_{0.5}Ni_5$ also screw dislocations with Burgers vector *a*/3 <-2110> or *c* <0001> were observed. Observed dislocation densities ρ were between 0.01 and $1.9 \cdot 10^{11}$ cm^{-2}. Example of fitting the line broadening anisotropy is given for the sample $LaNi_4Fe_1$ in the figure below.



[1] Krivoglaz, M.A. (1969) *Theory of X-ray and Thermal Neutron Scattering by Real Crystals*. New York: Plenum.

[2] Klimanek P. & Kuzel R. jr. (1988) *J. Appl. Cryst.* **21**, 59-66.