SNBL	**Experiment title:** Structural characterisation of intermetallic compounds and metal hydrides by powder diffraction.	**Experiment number**: 01-01-619
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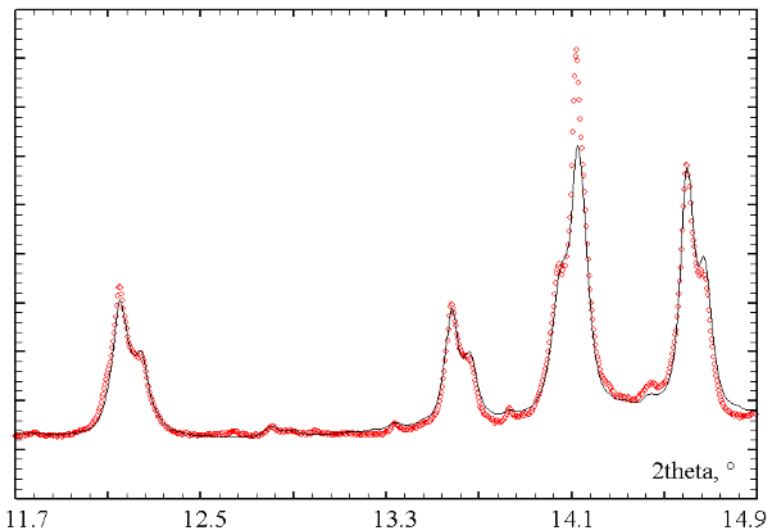
Several intermetallic compounds and their hydrides were studied according to the proposal. All high resolution powder patterns were obtained at the wavelength of 0.500140 Å.

$Ce_2Ni_7H_4$

High resolution synchrotron study (2-38 deg 2θ, step 0.004 deg 2θ, 1 sec/step) allowed us to identify a tiny orthorhombic distortion (see figure) of the parent hexagonal structure ($P6_3/mmc$) of the Ce_2Ni_7 alloy.



Refinement of the metallic substructure in the orthorhombic space group *Pmcn* (*Pmna*) allowed us to build an orthorhombic model for locating deuterium atoms using neutron powder diffraction data measured at the PSI. Neutron data, if used alone, do not allow to identify this tiny distortion (less than 1%). Cell parameters derived from the SNBL data: a = 4.87764 (14), b = 8.5315(2), c = 29.6215(9) Å.

$Ce_2Ni_5B_4H_{\sim 2}$ and **$Nd_2Ni_5B_4H_{\sim 2}$**

According to the neutron data measured at the PSI, monoclinic structures of $RE_2Ni_5B_4$ alloys undergo a symmetry change (from *C*2/*m* to *C*2 or *Cm* space group) upon hydrogenation. To refine the metallic substructure in the non-centrosymmetrical space group we have measured high-resolution synchrotron powder diffraction data (2-40 deg 2θ, step 0.004 deg 2θ, 1 sec/step). Preliminary refinement of the centrosymmetrical model in *C*2/*m* is satisfactory, for the Ce-containing alloy and hydride the cell parameters are: a = 9.75986(15), b = 5.13639(7), c = 8.08387(12) Å, β = 136.9701(9)°, V = 276.533(7) Å^3 – for the alloy; and a = 9.76472(11), b = 5.32035(6), c = 8.21818(9) Å, β = 137.5075(7)°, V = 288.402(6) Å^3 for the hydride. Joint structure refinement using the synchrotron and neutron data is planned.

$Er_3Ni_7B_2H_x$, high-pressure phase (~50 bar equilibrium hydrogen pressure).

The sample was taken from an autoclave and filled into a capillary immediately before the data collection. The powder pattern (4-38 deg 2θ, step 0.01 deg 2θ, 1 sec/step) presented no line asymmetry showing that the hydrogen release during the measurement was negligible. The powder pattern is indexed, but the structure is not yet fully refined.

$ErCo_3D_{1.3}$

The structure has been refined (anisotropically for all atoms) jointly using the synchrotron (3-38 deg 2θ, step 0.004 deg 2θ, 1 sec/step) and neutron (PSI) data. High resolution synchrotron data have proved that the parent rhombohedral symmetry is preserved, the structure is not distorted to monoclinic *C*2/*m*, as in the case of (La,Pr,Nd)Ni_3 hydrides (our recent results). Cell parameters: a = 4.98639(2), 26.0302(3) Å, R_p = 4.14%, R_{wp} = 5.50%, χ^2 = 3.46.

$La_3Pd_5SiH_x$

Immediately after opening an autoclave with La_3Pd_5Si hydride we have observed a previously unidentified (very) high pressure phase $La_3Pd_5SiH_{\sim 6}$. It disappears in 20 minutes from the diffraction pattern, therefore the data only from three detector channels (no. 4 to 6) were integrated. We succeeded to obtain good data (3-40 deg 2θ, step 0.004 deg 2θ, 1 sec/step) that allowed to determine the cell parameters of the orthorhombic cell: a = 13.185, b = 7.918, c = 8.236 Å, V = 859.8 Å^3. Having identified this new phase from the SNBL data we have performed a low temperature neutron powder diffraction experiment to solve the $La_3Pd_5SiD_{\sim 6}$ structure. The obtained neutron data fully confirm our findings made from the SNBL measurements.

Another high-pressure phase, $La_3Pd_5SiH_{\sim 4}$, was identified by fast synchrotron diffraction experiment. We have obtained good data (3-36 deg 2θ, step 0.004 deg 2θ, 1 sec/step) that

allowed us to refine the structure with a good precision: *Imma*, a = 12.9562(3), b = 7.74426(17), c = 8.32015(18) Å, V = 834.81(3) Å^3.

Third phase, $La_3Pd_5SiH_{\sim 3.5}$, identified in the synchrotron data seems to be an individual compound existing in equilibrium with $La_3Pd_5SiH_{\sim 4}$. *In-situ* diffraction study is planned.

$Ce_3Pd_5SiH_{\sim 3.5}$

A sample containing the compound isostructural to the lanthanum analogue ($La_3Pd_5SiH_{\sim 3.5}$.) was measured. The hydride was obtained at 75 bar of hydrogen pressure and observed immediately after releasing hydrogen pressure. That means that the plateau pressures for Ce-containing hydrides are higher than those for $La_3Pd_5SiH_x$. Due to this fact we decided to stop working on Ce_3Pd_5Si-H_2 system, despite very good quality of the data (2-38 deg 2θ, step 0.004 deg 2θ, 1 sec/step) and of the refinement achieved.

Zr_2AlD_x

One high resolution pattern was collected (2-36 deg 2θ, step 0.004 deg 2θ, 0.5 sec/step). The observed background is high (proximity of the Zr Kα absorption edge), however, the resolution of the data is higher than we were able to obtain with laboratory X-ray powder diffraction. These data, along with the neutron powder diffraction data (PSI) will allow us to index and solve the structure of the new hydride.