SNBL	**Experiment title:** Structural characterisation of intermetallic compounds and metal hydrides by powder diffraction.	**Experiment number**: 01-01-619
Beamline: BM01B	**Date of experiment**: from: 11-april-03 to: 14-april-03	**Date of report**: 28-march-03
Shifts: 9	**Local contact(s)**: Hermann EMERICH	*Received at UNIL:*

Names and affiliations of applicants (* indicates experimentalists):

*Radovan Černý
Klaus Yvon
*Yaroslav Filinchuk
*Yaroslav Tokaychuk

Laboratoire de Cristallographie, Université de Genève, 24, quai Ernest-Ansermet
CH-1211 Genève 4, Suisse

1. Alloy Ce_2NiSi with an own structure type

0-43 deg / 0.004 deg / 1 sec. $P6_3/m$, a = 15.99152(19), c = 4.27183(8) Å, V = 946.07(3), single phase. Ordering of Si and Ni atoms over four sites was detected.

2. Deuteride Ce_2NiSiD_x

0.6-37 deg / 0.008 deg / 3 sec. The parent alloy structure is preserved. Lattice expands anisotropically: a = 17.248(2), c = 4.0814(4) Å, V = 1051.5(2) Å^3. Single phase sample, strong anisotropic peak broadening.

3. Alloy $LaNi_3B$ with a new structure type

3-40 deg / 0.004 deg / 1 sec. *Imma*, a = 4.97675(3), b = 7.14918(4), c = 8.30753(6) Å, V = 295.579(3) Å^3. Synchrotron data allowed to localize and refine oxygen impurity which presumably stabilize this new phase. Refinement results are of single crystal experiment quality. Pronounced anisotropic peak broadening was modelled with a general orthorhombic model for anisotropic strain broadening. Sample contains few percents of unavoidable $La_2Ni_5B_4$ ($Nd_2Ni_5B_4$ str. type) and $LaNi_4B$ ($CeCo_4B$ str. type) phases. The structural information for the alloy will be coupled with those for its deuteride (No. 4).

4. Deuteride $LaNi_3BD_x$

3-40 deg / 0.004 deg / 2 sec. The parent alloy structure is preserved. Lattice expands anisotropically: a = 5.37472(11), b = 7.63953(18), c = 8.0698(2) Å, V = 331.347(14) Å^3. Pronounced anisotropic peak broadening. Sample contains few percents of unavoidable $La_2Ni_5B_4D_x$ and $LaNi_4BD_x$ phases.

5. Deuteride $CeCo_3D_4$

0.6-37 deg / 0.006 deg / 2.3 sec. Derivative of the $CeCo_3$ alloy structure, with enormous unique-axis expansion of the unit cell (30% along *c*-direction) upon deuteration. The deuteride partially decomposes to an amorphous $CeCo_2$ deuteride and crystalline $Ce_2Co_7D_6$ (see below a compound No. 6). High resolution synchrotron data allowed to reveal and to resolve fine structural details for both $CeCo_3D_4$ and $Ce_2Co_7D_6$ in their mixture.

6. Deuteride $Ce_2Co_7D_6$

0.6-37 deg / 0.006 deg / 2.3 sec. Derivative of the Ce_2Co_7 alloy structure, with enormous unique-axis expansion of the unit cell (30% along *c*-direction) upon deuteration. The deuteride partially decomposes to an amorphous $CeCo_2$ deuteride and crystalline $Ce_5Co_{19}D_x$. High resolution synchrotron data allowed to reveal and to resolve fine structural details for both $Ce_2Co_7D_6$ and $Ce_5Co_{19}D_x$ in their mixture.

7-8. Deuterides of $ErCo_3$ and $ErNi_3$ alloys

4-35 deg / 0.005 deg / 2.3 sec and 0.6-37 deg / 0.005 deg / 2 sec. AB_3 compounds, which are rhombohedral stacking variants of AB_2 and AB_5 structures. They demonstrate anisotropic cell expansion due to D-atoms and sharp plateau on pressure-composition isotherm. Both are expected to exhibit a local complex formation for transition metal atoms.

9-13. Deuterides of $Er_3Ni_7B_2$, $Ce_2Ni_5B_4$, $Nd_2Ni_5B_4$, YNi_2B_2, $HoNi_2B_2$ alloys

3-35 deg / 0.01 deg / 3.5 sec for **$Er_3Ni_7B_2$**, 4-37 deg / 0.005 deg / 1 sec for **$Ce_2Ni_5B_4$**
4-16 deg / 0.005 deg / 1 sec and 16-40 deg / 0.005 deg / 2 sec for **$Nd_2Ni_5B_4$**
0.6-33 deg / 0.005 deg / 1 sec for **YNi_2B_2**
5.6-17 deg / 0.005 deg / 2 sec and 17-32.5 deg / 0.005 deg / 1 sec for **$HoNi_2B_2$**

A few deuterides of intermetallic borides that absorb only a little amount of hydrogen (cell expansion typically 4% in volume). These new compounds are expected to form an ordered deuterium coordination of a single Ni-position. High resolution synchrotron data will allow localization of the voids occupied by D-atoms without performing expensive neutron diffraction study. Data treatment is still in progress.

14. Deuteride $Zr_3Al_2D_x$

3-35 deg / 0.008 deg / 3.5 sec. $P4_2/mnm$, a = 7.5960(3) Å, c = 7.2011(3) Å, V = 415.50(3) Å^3. Metallic substructure was determined and refined.

15. Alloy $Yb_{12.5}Fe_{67.5}Ga_{20}$

4-16 deg / 0.008 deg / 2 sec, 16-37 deg / 0.008 deg / 4 sec. $PrFe_7$(A)-type, R-3m: a = 8.6733(4) Å, c = 12.5677(6) Å, V = 818.75(6) Å^3. Single phase sample with the rhombohedral structure (difficult in the Yb-Fe-Ga system).

16. Alloy $Yb_{15}Fe_{65}Ga_{20}$

4-16 deg / 0.005 deg / 1.2 sec, 16-37 deg / 0.005 deg / 2.5 sec.
Main phase: $LuFe_{9.5}$-type, $P6_3/mmc$: a = 8.5379(3) Å, c = 8.3770(4) Å, V = 528.84(3) Å^3
Secondary: $PrFe_7$(A)-type, R-3m: a = 8.5823(6) Å, c = 12.5713(19) Å, V = 801.90(14) Å^3
and $Fe_{1-x}Ga_x$, Im-3m: a = 2.89386(7) Å, V = 24.234(1) Å^3
Strong anisotropic peak broadening in samples 15 and 16 was successfully modelled.

17. Alloy $Yb_{15}Fe_{65}Ga_{20}$

4-37 deg / 0.005 deg / 1.1 sec. Data analysis in progress.

The data of samples 5-8 and 14 are ready for joint refinement with neutron data.