SNBL	**Experiment title:** Structural characterisation of intermetallic compounds and metal hydrides by powder diffraction.	**Experiment number**: 01-01-619
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$Mg(BH_4)_2$

Low (tetragonal) and high temperature (cubic above 459 K) phases were reported [1, 2] for this compound, however, the crystal structures are unknown. Powder patterns of the $Mg(BH_4)_2$ compound have been collected at room temperature and at 523 K (glass capillary and hot air blower). The pattern recorded at room temperature shows the presence of a new phase (indexation not yet successful), probably the $Mg(BH_4)_2$ phase. Observed broadening of diffraction profiles, coming from a lower crystallinity of the main phase and presence of impurities due to the way of synthesis, makes the analysis difficult. The high temperature patterns have shown the decomposition of the $Mg(BH_4)_2$ phase. The analysis of the data is in progress.

Mg_2Ir_3

In the course of the studies of the Ir-rich part of the Mg-Ir phase diagram we have discovered two new intermetallic phases: orthorhombic $Mg_{1+x}Ir_{1-x}$ (x = 0 - 0.054) with 25 atoms in the asymmetric unit, which was recently characterized with the SNBL powder data [3], and monoclinic Mg_2Ir_3. Here we report on the crystal structure of the second phase.

For the Mg_2Ir_3 sample preparation, the mixture of Mg and Ir powders (0.5 g) was pressed into pallet using the 4 mm steel die mold. Then the pallet was sealed in quartz ampoule under 0.3 bar of Argon, heat treated at 600°C during 1 week and quenched in cold water.

High resolution synchrotron powder diffraction pattern (λ = 0.49969 Å, sample in a glass capillary, six analyser crystals detector) shows the presence of a phase with unknown structure and of iridium metal impurity. The pattern of the unknown phase was successfully indexed with a monoclinic cell by using the program DICVOL91: a = 18.5700(2), b = 5.18716(3), c = 8.49240(6) Å, ß = 97.2211(5)°. The crystal structure was solved by global optimization in direct space by using the program FOX [4]. Structural model (space groups $C2/m$) contains 6 iridium and 7 magnesium atoms in the asymmetric unit, and was refined using the program FullProf.2k (83 free parameters, R_{wp} = 0.068, χ^2 = 1.45). Rietveld plot is shown in Figure 1.

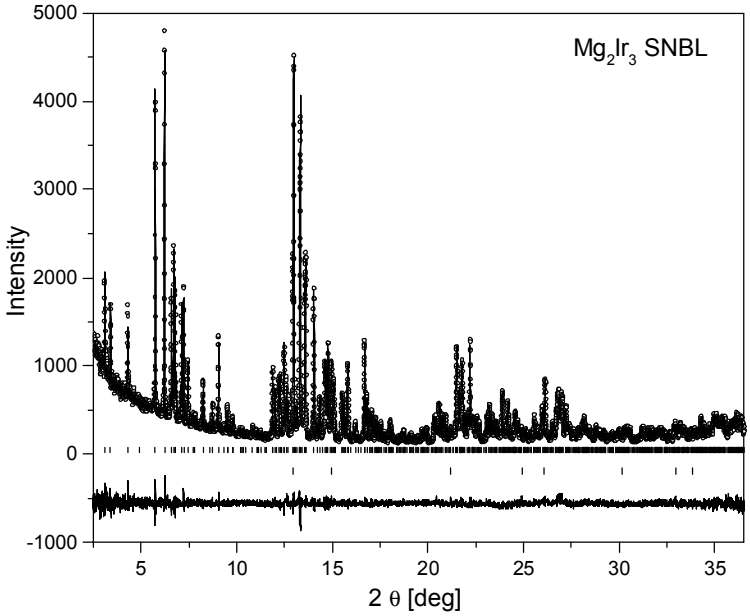

Figure 1. Rietveld plot of Mg_2Ir_3 (R_{wp} = 0.068, χ^2 = 1.45). Observed (dots) and calculated (solid line) synchrotron (SNBL) powder diffraction patterns (λ = 0.49969 Å) are shown with difference curve below. Ticks indicate the line positions of the main phase Mg_2Ir_3 (upper, R_B = 0.054) and the impurity phase Ir (lower).

Ce_2Co_7

The powder pattern (1-43 deg 2θ, step 0.0032 deg 2θ, time 3 s/step, λ= 0.49969 Å) obtained on a single phase Ce_2Co_7 sample, annealed at 800°C, shows intrinsic peaks from both hexagonal (Ce_2Ni_7 structure type) and rhombohedral (Gd_2Co_7 structure type) phases, that are basic polytypes of essentially the same structure. A fixed ratio of diffraction intensities (2:1 for hex./rhomb.) was observed in our preliminary X-ray diffraction experiments.

Constraints on the cell dimensions $\boldsymbol{a}$(hex) = $\boldsymbol{a}$(rhomb) and 3$\boldsymbol{c}$(hex) = 2$\boldsymbol{c}$(rhomb) are strictly fulfilled, the observed peaks are not split. The *h0l* reflections show severe peak broadening that can be explained by frequent 2H/3R stacking faults. The structural models for 2H and 3R Ce_2Co_7 polytypes with only scale factors refined resulted in R_F = 20%. Further refinement will be performed with a special modelling of the diffraction profile.

[1] Konoplev V.N.: *Russian J. Inorganic Chemistry,* **25**(7) (1980) 964-966.
[2] Konoplev V.N. and Bakulina V.M.: *Izv. Akad. Nauk SSSR, Ser. Khim.* **1** (1971) 159-161.
[3] Cerny R., Renaudin G., Favre-Nicolin V., Hlukhyy and Pöttgen R.: *Acta Cryst.* B, subm.
[4] Favre-Nicolin V. and Cerny R.: *J. Applied Crystallography* 35(2002)734-743.