The high-resolution powder diffraction, especially using synchrotron radiation, has become an irreplaceable tool in the characterization of new intermetallic compounds and metal hydrides. In the past 3 years we have solved and characterized around 20 new compounds using the high-resolution powder diffractometer of the SNBL. The examples presented here show a complexity of the crystal structure fully characterized (MgIr) and a tiny monoclinic distortion revealed (LaMg$_2$NiH$_7$) when high-resolution data are available.

The crystal structure of MgIr was solved and refined using the synchrotron powder diffraction data measured at the wavelength of $\lambda = 0.50012$ Å (Fig. 1). High-energy X-rays were chosen here to maximize diffracted intensities from this highly absorbing compound. The data were indexed with the orthorhombic cell $a = 18.46948(6)$ Å, $b = 16.17450(5)$ Å, $c = 16.82131(5)$ Å, and the space group $Cmca$ identified from the observed extinction conditions. The structure was solved by the global optimization of a structural model in direct space using the recently developed program Fox [1]. For more details on the optimal structure solution procedure see [2]. The global optimization method was successfully applied here to a 25-atoms structure with the close-packing and high symmetry. Alternative methods using integrated intensities extracted from the powder pattern (direct methods or Patterson synthesis) have failed, probably because of difficult recognition of a structural motif the methods using extracted integrated intensities from the powder pattern (direct methods or Patterson synthesis) have failed, probably because of difficult recognition of a structural motif either in E- or in Patterson maps.

LaMg$_2$NiH$_7$ is an interesting compound from both fundamental and technological points of view. The particularity of this hydride is the way of synthesis: the intermetallic compound LaMg$_2$Ni absorbs hydrogen near ambient conditions forming the non-metallic hydride LaMg$_2$NiH$_7$ which has a nearly unchanged metal host substructure (atom shifts < 0.7 Å) [3]. The transition is induced by a charge transfer of conduction electrons into tetrahedral [NiH$_4$]$^{4-}$ complexes having closed-shell electron configuration [4]. LaMg$_2$Ni is the first ternary compound known to absorb hydrogen without important rearrangement of the metal host substructure that leads to a metal-semiconductor transition. A complete structural characterisation of the hydride was of primary importance to elucidate the metal-semiconductor transition.
transition. Figure 2 shows the crystal structures, before (LaMg$_2$Ni) and after (LaMg$_2$NiH$_7$) hydrogenation. The structure of monoclinic LaMg$_2$NiH$_7$ (LaMg$_2$NiD$_7$) was solved and refined by using synchrotron and neutron powder diffraction ($P2_1/c$, $a = 14.0164(6)$ Å, $b = 4.7146(2)$ Å, $c = 16.0572(8)$ Å, $\beta = 125.222(2)^\circ$, $Z = 8$). Synchrotron powder diffraction data ($\lambda = 0.499490$ Å) was useful to elucidate the true symmetry and position of the metal atoms (8 sites), whereas neutron powder diffraction data (D2B at ILL, Grenoble, $\lambda = 1.594$ Å) was useful to locate deuterium atoms (14 sites).

Only the high-resolution synchrotron data (Figure 3) allowed us to observe a tiny monoclinic distortion and a doubling of the cell volume of the LaMg$_2$NiH$_7$ lattice that was previously indexed using the laboratory data in an orthorhombic cell corresponding to the expanded cell of the intermetallic compound LaMg$_2$Ni. Only the true monoclinic symmetry allowed us to identify the well-ordered hydrogen (deuterium) network.

**References**