



	<b>Experiment title:</b> EXAFS on Nb-doped magnesium and magnesium hydride: how the Nb-Mg correlation acts on the hydrogen absorption/desorption process.	<b>Experiment number:</b>
	<b>Beamline:</b>	<b>Date of experiment:</b> from: 25-4-2007 to: 30-4-2007
<b>Shifts:</b>	<b>Local contact(s):</b> C. Maurizio	<i>Received at ESRF:</i>
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## Report:

Nb-doped Mg samples (Nb concentration = 1 at.% and 3 at.%) were deposited by r.f. magnetron sputtering in form of films on polished graphite wafers. To prevent the surface oxidation and stimulate the H<sub>2</sub> dissociation at the Mg surface, samples were coated with a 15-nm-thick Pd capping layer without interrupting the vacuum conditions in the deposition chamber. After deposition, Mg samples peeled off from the substrate: the self-supporting samples were introduced in a Sievert-type apparatus to study the hydrogen sorption kinetics. Samples were submitted to several H<sub>2</sub> sorption cycles at 623 K, each one consisting of two steps: i) the sample is exposed to 1 MPa H<sub>2</sub> gas for 20 h to induce the H<sub>2</sub> absorption (absorption step) and then ii) to vacuum to induce H<sub>2</sub> desorption (desorption step). The EXAFS experiment was performed at the Italian beamline GILDA of the ESRF (F) on Nb K-edge. The monochromator was equipped with two Si (311) crystals and a couple of Pt-coated mirrors working at an incidence angle of 2.7 mrad were used to reject higher harmonics. Nb EXAFS spectra were collected in fluorescence mode by a 13-elements high-purity Ge detector; samples were cooled at 80 K to limit thermal vibrations; The spectrum of a Nb foil was also Measured in transmission mode as standard reference. Data analysis was based on FEFF8-FEFFT code.

The EXAFS spectra of the 3 and 1 at. % Nb-doped Mg films measured after H<sub>2</sub> desorption are shown in the figure: spectra are pertinent to the as-deposited layer and to layers submitted to 1, 3, 5 and 8 H<sub>2</sub>-cycles. The spectrum of the as-deposited film changes upon consecutive H<sub>2</sub>-cycles and becomes progressively similar (after 5 and 8 cycles) to that of metallic Nb. This fact is more evident for Nb(3%):Mg film than for the Nb(1%):Mg one, suggesting a slower Nb aggregation at low Nb concentration.

The results of the EXAFS analysis indicates that:

- in the first hydrogenation/dehydrogenation cycles, small non-bcc Nb clusters form, while part of Nb is bonded to Mg atoms; the fraction of metallic Nb and dispersed fraction depends on the Nb concentration.
  - after many (about 10) cycles all Nb is in form of bcc clusters.
- These results shed light on the rapid hydrogen kinetics observed during the first cycles; a paper is in preparation.

EXAFS spectra on Nb-doped Mg layers after subsequent hydrogenation/dehydrogenation cycles (the cycle number is indicated): (a) Nb concentration = 3 at%, (b) Nb concentration = 1 at. %.

