Report of the experiment 08-01-679: EXAFS on Nb-doped magnesium (h-Mg) and magnesium hydride (b-MgH2): the role of Nb as catalyst of the hydrogen absorption and desorption kinetiks.

1 wt. % and 5 wt. % Nb-doped Mg films are prepared by rf-sputtering on a graphite substrate. To prevent surface oxidation and stimulate the H_2 molecule dissociation, the samples were coated with a 15-nm-thick Pd capping layer. The samples are then submitted to several cycles at T=625K in 0.8 MPa (H_2-absorption) or in 0.0015 MPa (H_2-desorption) of H_2 atmosphere. The EXAFS experiment (GILDA beamline, ESRF) was performed by measuring the x-ray absorption coefficient at the Nb K-edge in fluorescence mode. For the data analysis the FEFF8-FEFFIT was used.
The most significant EXAFS spectra colleced are shown in Fig. 1, while in Fig. 2 the corresponding Fourier Transform moduli and fits are reported. The spectra for the samples are compared to that one recorded for a metallic Nb foil. The EXAFS results for the first coordination shells (Nb-Mg and Nb-Nb ones) are reported in Table 1.

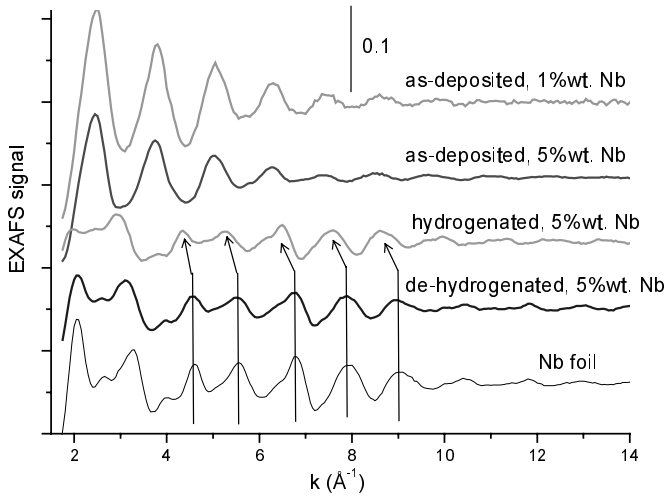

Fig. 1. EXAFS spectra.

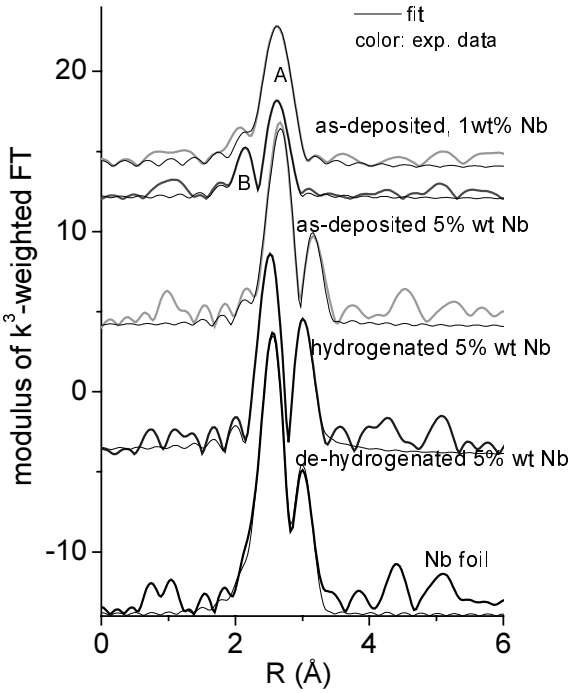

Fig. 2. Fourier transform moduli of the recorded EXAFS spectra: the corresponding fits are shown.

	Nb-Nb			Nb-Mg		
Sample	N (±20%)	R (Å) (±0.01)	σ^2 (10^{-4}Å^2) (±20%)	N (±20%)	R (Å) (±0.01)	σ^2 (10^{-4}Å^2) (±20%)
As-deposited 1at.% Nb				8.1	3.09	55
As-deposited 5at% Nb	1.2	2.74	55	8.5	3.08	95
Hydrogenated 5at% Nb	8.0 6.0	3.00 3.48	63 74			
De-hydrogenated 5at% Nb	3.9 2.9	2.87 3.32	46 38			
Nb crystal	8 6	2.86 3.31				
Mg crystal				6 6	3.20 3.21	

Table 1. Results of the first shell EXAFS analysis.

The main results are:
- As-deposited samples: part of Nb atoms coordinate just Mg atoms (peakA in Fig. 2). Small Nb aggregates are present for higher Nb concentration (peak B in Fig. 2).
- Hydrogenated sample: presence of Nb aggregates, bcc short-range order. The interatomic distance is 4.8% larger than the corresponding bulk phase and is in agreement with the presence of H atoms in interstitial position.

- De-hydrogenated sample: presence of Nb aggregates with a bcc short-range order. The interatomic distance is in agreement with the Nb bcc bulk phase.

So, it has been shown that the Nb (1-5wt%)-Mg deposition by sputtering is suitable to disperse Nb into the Mg So, lattice, the hydrogenation promote the formation of Nb-H aggregates (H in tetrahedral interstitial position?); the de-hydrogenation process (by heating in vacuum) restore the Nb aggregates: the Nb-Nb distance is in agreement with the corresponding bulk phase.

Work is in progress to investigate the presence of H atoms into the Nb nanocluster lattice and to model the Nb catalyst effect in increasing the H-absorption/desorption rate.