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| | Experiment title: Local structure of Pd clusters decorating Mg nanoparticles for hydrogen storage | Experiment number: CH-2942 |
| Beamline: | Date of experiment: from: 01/10/2009 to: 06/10/2009 | Date of report: 29/02/2012 |
| Shifts: 15 | Local contact(s): Chiara Maurizio | <i>Received at ESRF:</i> |
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Report: XAFS measurements at the Pd K-edge were carried out on Mg nanoparticles with partial Pd coating, according to the submitted proposal. The beamline ran smoothly and the quality of EXAFS and XANES spectra was very good. The EXAFS spectra were analyzed with the ATHENA and ARTEMIS packages. EXAFS oscillations were extracted from the raw spectra using a low distance cut-off of 1 Å in the k^2 weighted Fourier transform and using a cubic spline. As a starting point for non linear fitting of the spectra, EXAFS signals were calculated using the FEFF 8.20 code using crystallographic structures as input. The figure below displays the EXAFS spectra for the the different samples that were studied. The results of data analysis allowed us to determine the local structure at the Mg/Pd interface, and to map its evolution in response to annealing and H-sorption. This task was accomplished by X-ray absorption spectroscopy, also including *in situ* experiments, with the support of crystallographic information from X-ray diffraction. We showed that the initial Pd surface layer reacts with Mg at relatively low temperatures, leading to irreversible formation of a Mg-rich intermetallic phase Mg₆Pd. Due to the high Mg-H binding energy, this phase reversibly transforms, upon H absorption, into a nanophase mixture of magnesium hydride and a Pd-rich intermetallic with H in solid solution, MgPdH_□. These reversible structural changes are understandable with reference to recent calculations that highlight their relevance to the thermodynamics of the metal-hydride transition. The picture derived from the experiment is relevant to other multiphase materials presently investigated in the field of hydrogen-related science and technology. The results have been fully published in:

L. Pasquini, F. Boscherini, E. Callini, C. Maurizio, L. Pasquali, M. Montecchi, E. Bonetti, *Local structure at interfaces between hydride-forming metals: A case study of Mg-Pd nanoparticles by x-ray spectroscopy*, Physical Review B **83**, 184111 (2011)

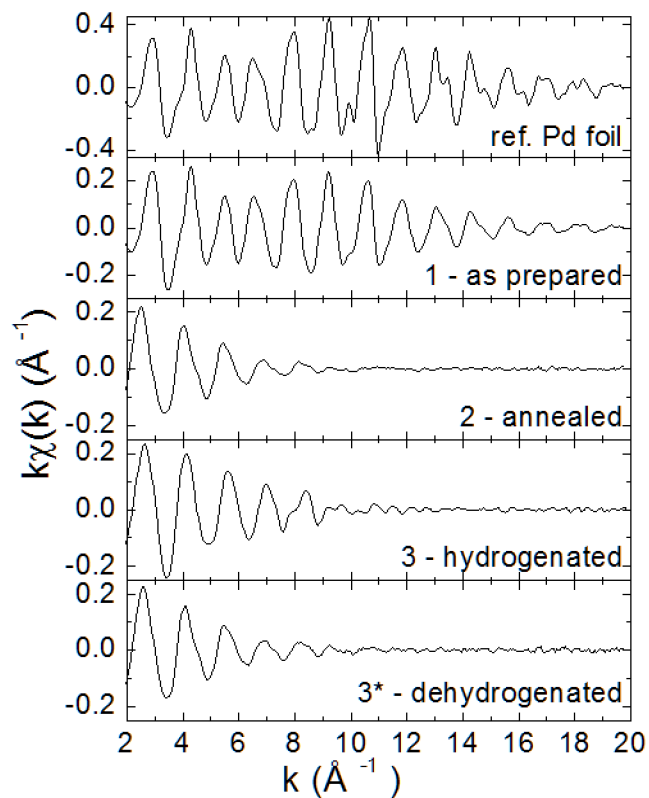


Figure 1: k -weighted raw EXAFS spectra after background subtraction, measured at 80 K in transmission mode for a reference Pd foil and the Mg-Pd nanoparticles. 1: as-prepared; 2: vacuum annealed; 3: after hydrogen absorption; 3*: after hydrogen desorption.