EUROPEAN SYNCHROTRON RADIATION FACILITY

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Experiment Report Form

	Experiment title: XAFS study of nanoparticle catalysts in H-sorption kinetics of Mg based nanocomposites	**Experiment number**: ME-1291
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In Situ Energy-Dispersive XAS and XRD Study of the Superior Hydrogen Storage System MgH_2/Nb_2O_5

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By in situ energy-dispersive X-ray absorption spectroscopy (EDXAS) and X-ray diffraction (XRD), we analyzed the evolution of niobium in a MgH_2/Nb_2O_5 system based on high-energy ball milling during hydrogen cycling. The high time resolution of the EDXAS method allowed us to monitor fast sample changes during this process. Thereby, we demonstrated that the Nb_2O_5 is already partially reduced during the milling process with the MgH_2. Further reduction occurs during the heating and cycling processes, in which a lower limit of oxidation state is reached. Hereby, a reaction between the niobium oxide and the Mg/MgH_2 leads to a decrease of crystalline Nb_2O_5 and the formation of a ternary oxide phase Mg_xNb_yO. During the cycling processes a repetitive Nb oxidation−reduction process was observed, which may indicate hydrogen diffusion along the ternary oxide by the formation of metastable niobium hydrides. This points to a mechanism of kinetic sorption improvement by diffusion of hydrogen through pathways of ternary Mg−Nb oxides, which may also reduce the activation energy of the $Mg-MgH_2$ transition.