In-situ Neutron Powder Diffraction Measurement of Hydrogen Storage Properties of Mg_6Pd Alloys

Jacques Huot[1], Julien Lang[1], Helmut Fritzsche[2] and Roxana Flacau[2]

[1] Department of Physics, Université du Québec à Trois-Rivières, Trois-Rivières, QC, Canada G9A 5H7
[2] NRC Canadian Neutron Beam Centre, Chalk River Laboratories, Chalk River, ON, Canada K0J 1J0

In-situ neutron powder diffraction (NPD) in conjunction with pressure-composition-temperature (PcT) measurements is a powerful technique to monitor the hydrogenation/dehydrogenation reactions in potential hydrogen storage materials. The present experiment was a premier at CNBC, being the first one to use our recently developed *in-situ* top-loading furnace system which allows us to perform PcT and NPD measurements simultaneously at deuterium pressures up to 40 bar and temperatures up to 400 ^{0}C.

NPD patterns were continuously collected during D_2 absorption process in Mg_6Pd at 350 ^{0}C, in conjunction with PcT measurements. The pressure-composition isotherm recorded at C2 beamline and the one measured in the laboratory are in good agreement (Figure 1).

The diffraction patterns collected *in situ* during the hydrogenation process are shown in Figure 2. The three-step reaction process proposed earlier was confirmed by the present measurements and more in-depth information about the hydrogen absorption process was revealed. The diffraction patterns could be correlated with the PcT measurements and the exact onset point, in terms of absorbed deuterium (wt%), for each reaction was determined. Even though a steel sample holder had to be used due to the high temperature conditions required by the experiment, good quality NPD were collected and Rietveld analysis of patterns characteristic to each reaction step could be performed. Quantitative analysis results and information about particle size and strain were extracted as a result of the Rietveld analysis.

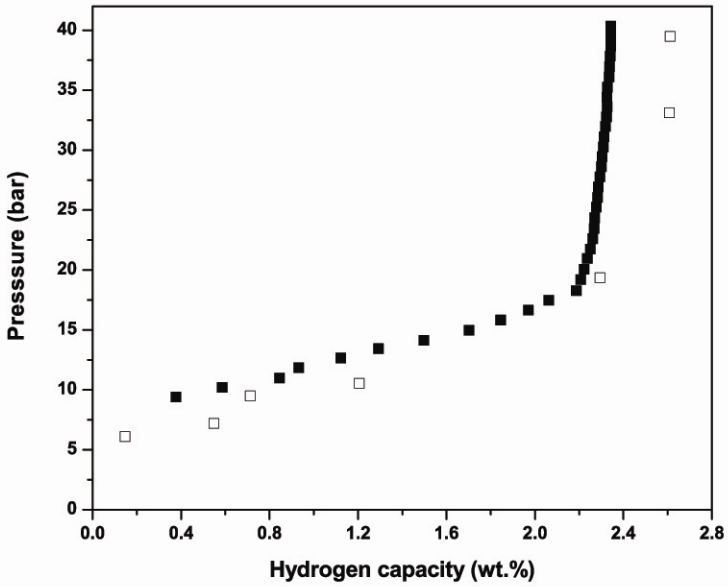

Fig. 1 Pressure-composition absorption isotherms of activated Mg_6Pd at 350 ^{0}C. The solid symbol are PcT points collected in the laboratory; open symbols are PcT points collected during at the same time with the PND measurements.

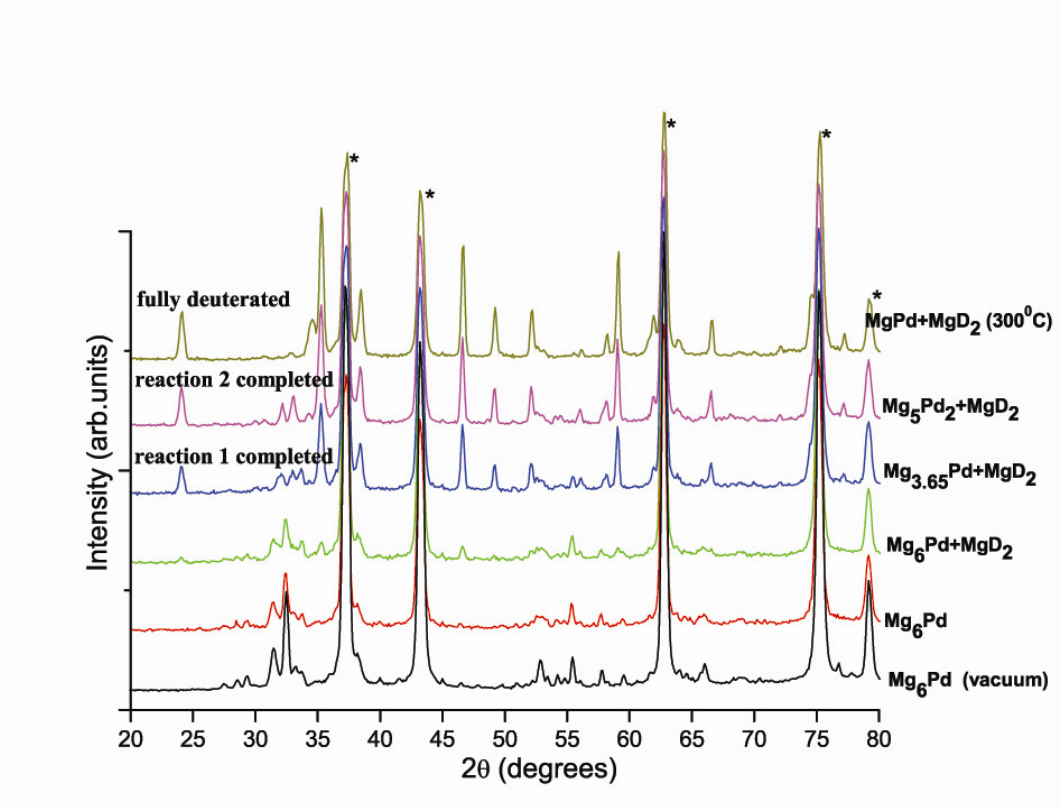

Fig. 2 Pressure NPD patterns collected during Mg_6Pd hydrogenation. The * symbol marks the peaks arising from the steel sample holder.