Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics

Supplementary Information

Remarkable hydrogen storage properties for nanocrystalline MgH_2 synthesised by the hydrogenolysis of Grignard reagents

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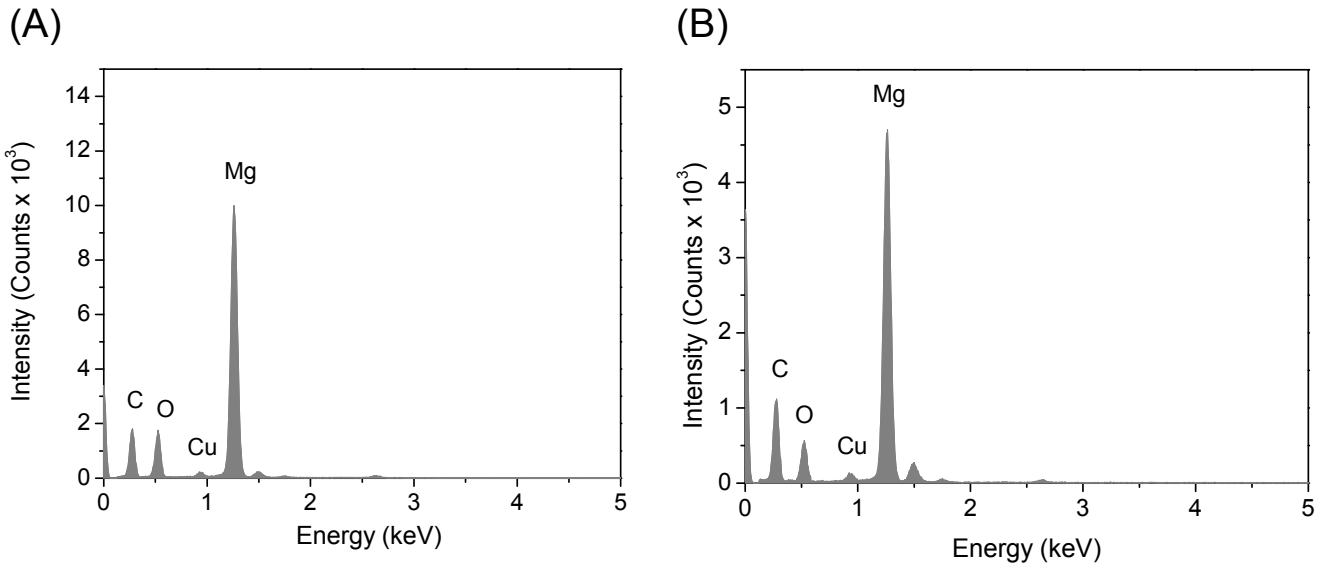

Figure S1. EDX analysis of H_2-DibutylMg (A) after hydrogenolysis - corresponding to Figure 5A and (B) after cycling - corresponding to Figure 8D.

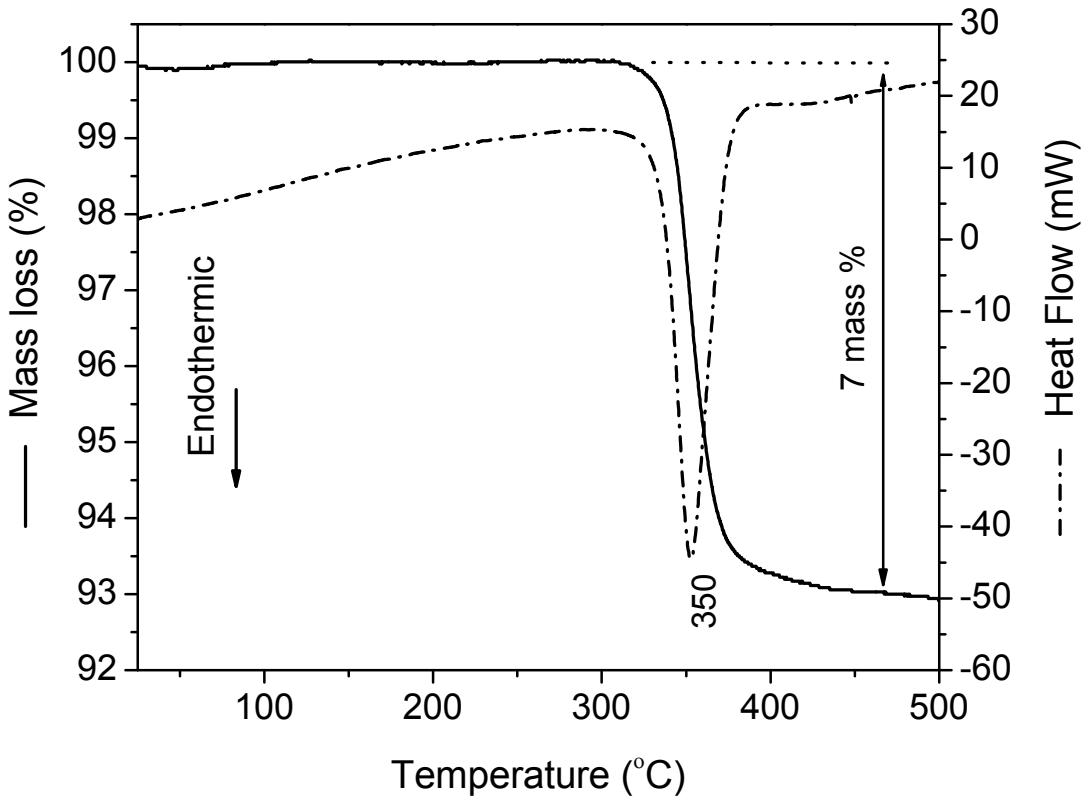

Figure S2. TGA/DSC of H_2-DibutylMg after the first hydrogen cycle. Measurement done with a heating rate of 10 °C.min^{-1} and under a 25 ml.min^{-1} Ar flow.

H_2-DibutylMg released 7 mass % of hydrogen in agreement with the hydrogen storage capacity measured during cycling, i.e. 6.8 mass %.

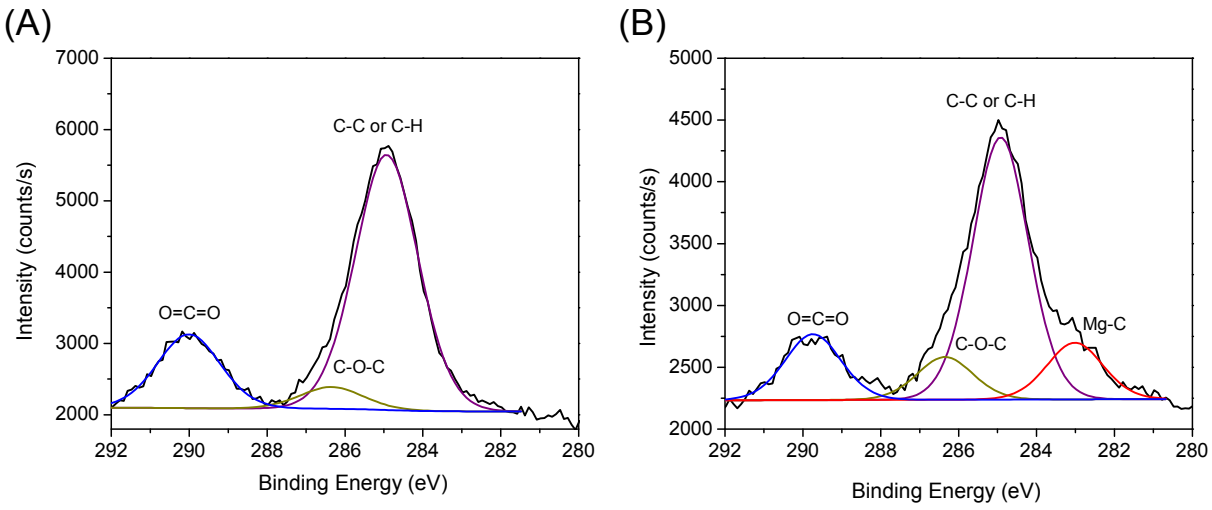

Figure S3. XPS narrow-scan of C1s for H_2-DibutylMg (A) resulting from the hydrogenolysis of di-*n*-butylmagnesium and (B) after the first hydrogen absorption/desorption cycle.

The peak at 289.7 eV was assigned to CO_2 adsorption at the magnesium surface,[1] and the peak at 286.3 eV to C-O-C bond and thus hydrocarbon contamination[2, 3] (Such hydrocarbon contamination may come from THF vapor to be found in our glove box). The peak at 284.9 eV may correspond to C-C or C-H,[2, 3] and the peak at 289.7 eV was assigned to Mg-C bond.[4]

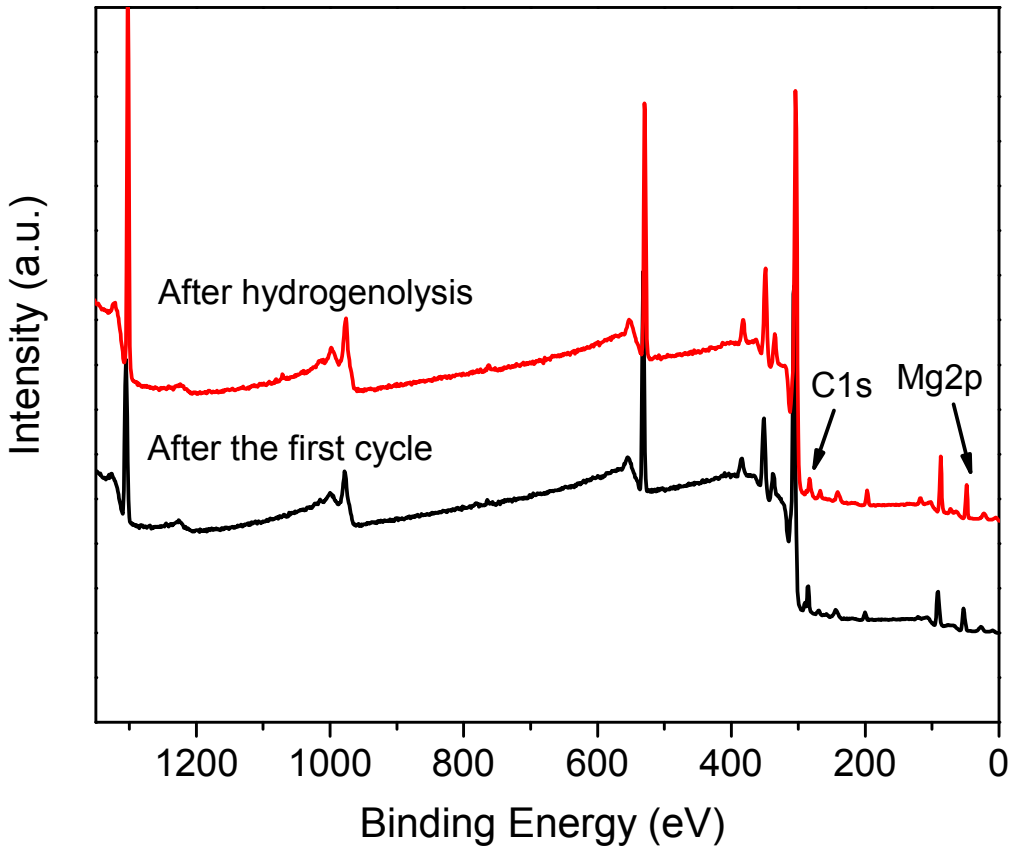

Figure S4. XPS wide-scan for H_2-DibutylMg (A) after hydrogenolysis and (B) after the first hydrogen absorption/desorption cycle.

1. H. Onishi, C. Egawa, T. Aruga and Y. Iwasawa, *Surf. Sci.*, 1987, **191**, 479-491.
2. C. D. Wagner, W. M. Riggs, L. E. Davis and J. F. Moulder, *Handbook of X-Ray Photoelectron Spectroscopy*, Perkin & Elmer Cooperation, 1997.
3. C. Fotea, J. Callaway and M. R. Alexander, *Surf. Interface Anal.*, 2006, **38**, 1363-1371.
4. A. S. Ingason, A. K. Eriksson, E. Lewin, J. Jensen and S. Olafsson, *Thin Solid Films*, 2010, **518**, 4225-4230.