

Supporting Information

Reversibility and Improved Hydrogen Release of Magnesium Borohydride

Rebecca J. Newhouse^{1,2}, Vitalie Stavila^{2*}, Son-Jong Hwang³ and Jin Z. Zhang¹

¹ *Department of Chemistry and Biochemistry, University of California, Santa Cruz, CA 95064 USA*

² *Sandia National Laboratories, Livermore, CA 94551 USA*

³ *Division of Chemistry and Chemical Engineering, California Institute of Technology, Pasadena, CA 91125 USA*

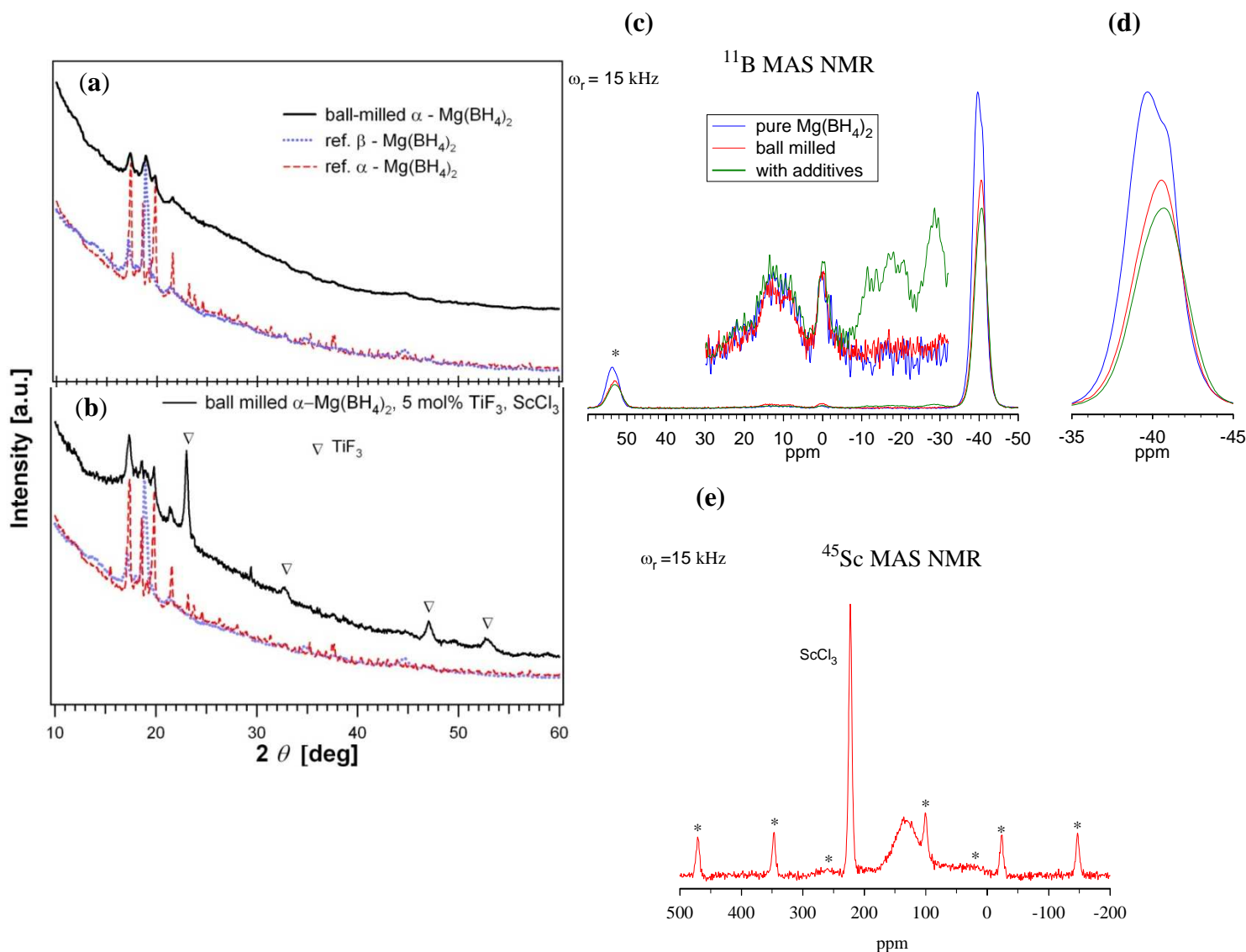


Figure S1. pXRD patterns (a and b), ^{11}B MAS NMR spectra (c and d) of α - $\text{Mg}(\text{BH}_4)_2$ ball-milled for 30 minutes and α - $\text{Mg}(\text{BH}_4)_2$ ball-milled for 30 minutes with 5 mol % TiF_3 and ScCl_3 added, and ^{45}Sc MAS NMR spectrum (e) for the sample with additives. For pXRD, as-made α - $\text{Mg}(\text{BH}_4)_2$ (---), as-made β - $\text{Mg}(\text{BH}_4)_2$ (....) and TiF_3 peaks (∇) are shown for comparison. For NMR, the spectrum of as-synthesized pure $\text{Mg}(\text{BH}_4)_2$ sample is included. The spectral range between -30 and 30 ppm was scaled up by 15 times to show the presence of boron oxide contaminants (peaks between -5 to 20 ppm) and boron species formed after the ball milling in the presence of additives (c). The position shift of the major $\text{Mg}(\text{BH}_4)_2$ peak from the α -phase to the β -phase is presented in the extended view (d). Spinning sidebands are marked with an asterisk (*).

* To whom correspondence should be addressed. E-mail: vnstavi@sandia.gov; Phone: 925-294-3059

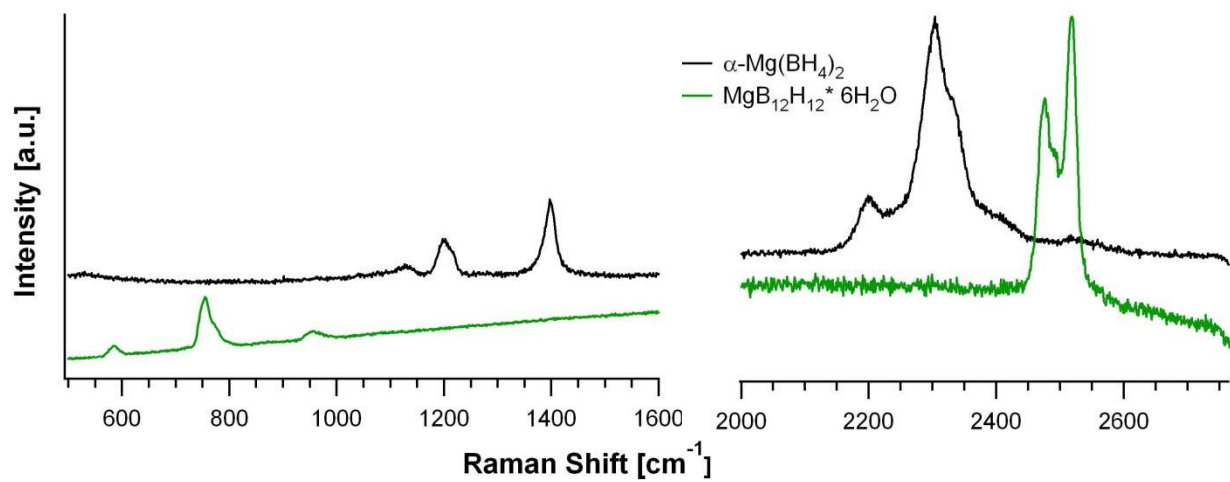


Figure S2. Raman spectra for hydrated $\text{MgB}_{12}\text{H}_{12} \cdot 6\text{H}_2\text{O}$ (—) and desolvated $\alpha\text{-Mg(BH}_4)_2$ (—).

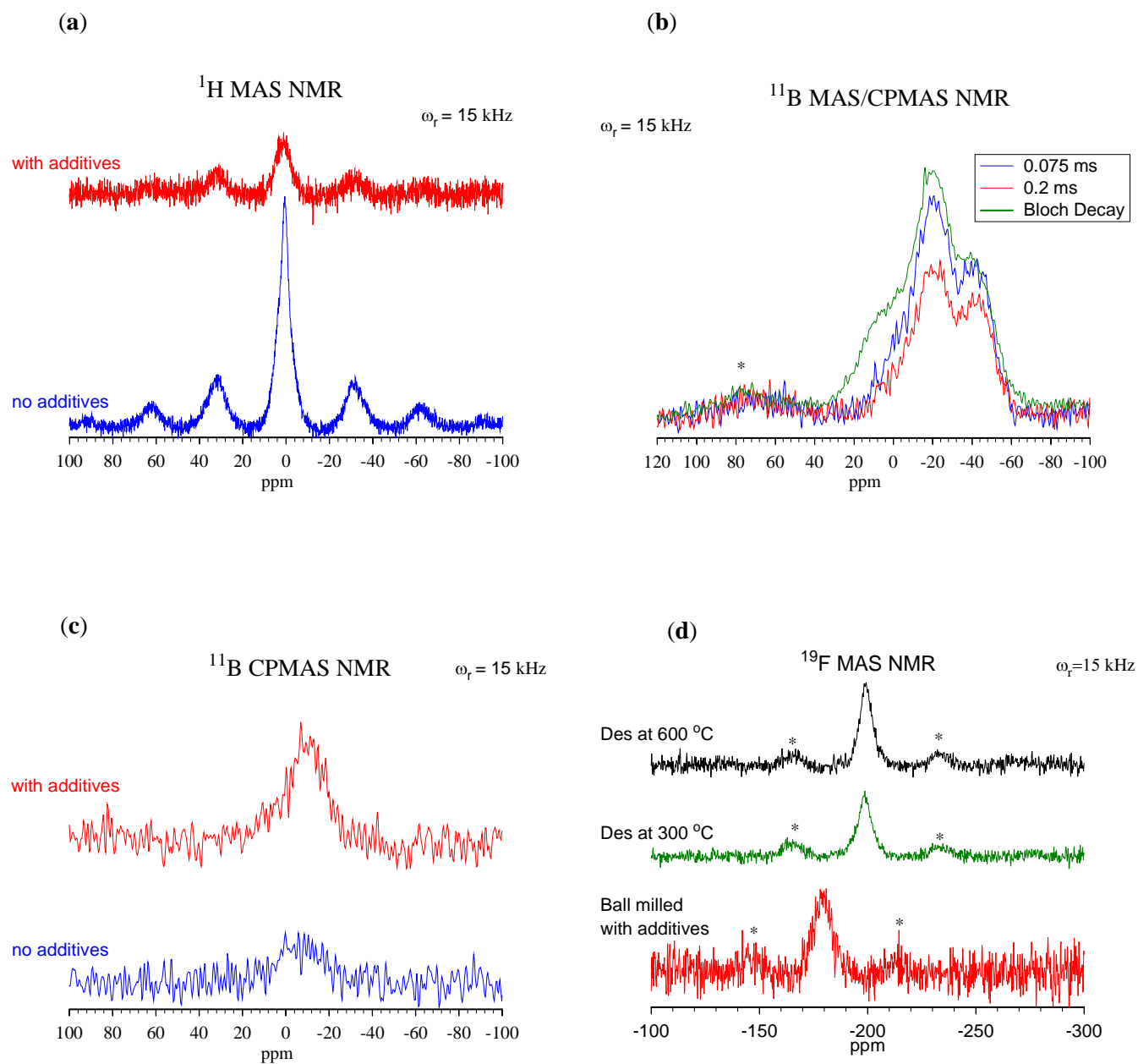


Figure S3. NMR spectra measured for samples with and without additives after desorption reactions. a) ^1H MAS NMR spectra after desorption at low temperature (300 °C). b) ^{11}B CPMAS NMR and MAS (Bloch decay) NMR spectra of sample with additives after desorption at low temperature (300 °C). Broad peak at 40-80 ppm range is a spinning sideband from the major peak in the upfield. c) ^{11}B CPMAS NMR spectra of both samples after desorption reaction at 600 C. d) ^{19}F MAS NMR spectra before and after desorption reactions showing the formation of MgF_2 .

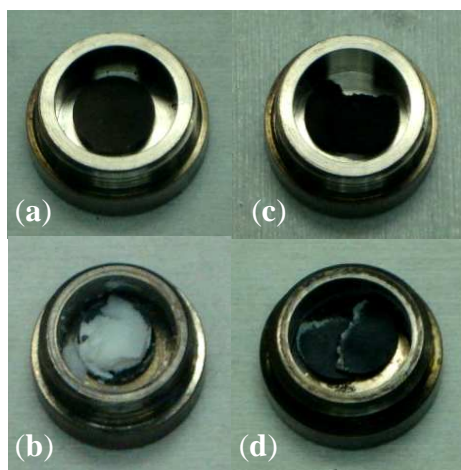


Figure S4. MgB₂ with and without additives before the high pressure experiment (a) and (c), respectively, and after exposure to 900 bar H₂ at 390 °C for 72 hours ((b) and (d), respectively).