

MAR08-2007-020301

Abstract for an Invited Paper
for the MAR08 Meeting of
the American Physical Society

New Possibilities for Understanding Complex Metal Hydrides via Synchrotron X-ray Studies¹

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Ultras-small-angle x-ray scattering (USAXS) and X-ray absorption spectroscopy (XAS) are used for the study of chemical and morphological changes in metal hydride powder (e.g. NaAlH₄) both before and after transition metal salt catalytic dopant additions by high energy ball milling. The variation in surface fractal dimension and particle size with milling time and dopant content were tracked. These studies show that dopant content level (e.g. 2 mol % and 4 mol %) and dopant type (i.e. TiCl₂, TiCl₃, VCl₃, and ZrCl₄) markedly affects NaAlH₄ powder particle surface area (determined using USAXS surface fractal dimension). As well, the chemical reaction between the catalyst and hydride powder was further elucidated using XAS. Ti-metal reacts with the Al desorption product (from NaAlH₄) to form TiAl_x product phases. These studies were able to link powder particle surface area to catalytic doping and were able to link dopant chemical state with dehydrogenation reactant and product phases.

¹Support from the Dept. of Energy, Basic Energy Sciences and the National Science Foundation, Division of Materials Research is gratefully acknowledged.