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Understanding Complex Metal Hydrides via Synchrotron X-ray Studies

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Our research uses several specialized synchrotron X-ray techniques to elucidate the interactions between transition metal salts additives and hydride host structures. The presentation will describe those techniques and their use in describing the physical and chemical processes occurring between the hydrides and catalysts.

Summary

This research reports on results which seek to understand the role of catalysts in hydride structures. X-ray absorption spectroscopy (including EXAFS and XANES) combined with Ultrasmall Angle X-ray scattering (USAXS) reveal some catalyst induced phenomena occurs in host hydrides. EXAFS and XANES results show that TiCl_3 transforms first to metallic Ti—then reacts with Al^{3+} in NaAlH_4 to form TiAl_x complexes. X-ray scattering data (capable of measuring feature sizes of 6 micrometers to 2nm) and wide-angle X-ray scattering crystallographic data are attained providing a unique view of both morphological and microstructural changes during elevated temperature conditions. All NaAlH_4 samples were catalyzed using high energy ball milling of 3 mol% of each ScCl_3 , ZrCl_4 , and VCl_3 . For reference, a sample of NaAlH_4 was also ball milled at the same time as the other samples. X-ray scattering data were collected at temperatures ranging from near room temperature (30°C) to just below the desorption temperature of uncatalyzed NaAlH_4 (170°C). Isothermal measurements were performed at 30°C, 65°C, 100°C, 135°C, and 170°C. For isothermal studies, samples were taken from room temperature to the desired temperature and held for 60 minutes. Results show that the catalysts mitigate diffusion rates (and therefore dictate the kinetics of microstructural changes) in the hydrides.

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