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***In-situ* X-ray Studies of Ti-doped Sodium and Lithium Alanate.** S.A. Speakman¹, J.H. Schneibel¹, D.S. Easton¹, T.A. Dobbins², ¹Oak Ridge National Laboratory, M&C Div., Oak Ridge, TN 37831, ²Louisiana Tech Univ., Ruston, LA 71272.

PCI measurements and *in-situ* XRD are well suited for complementary studies, as PCI measures the gas phase changes during reaction while XRD measures the solid phase changes. These techniques were used to study reaction rates and thermal desorption spectra of Ti-catalyzed sodium and lithium alanates. Reaction rate constants derived from XRD data were slower, by an order of magnitude, than those derived from PCI data. These differences were attributed to experimental artifacts. The amount of Al that formed exceeded the amount expected from the observed decomposition of the alanates; consequently, rate constants calculated from Al were larger than those calculated from the hydride phases. Peak profiling and quantitative Rietveld analysis techniques yielded comparable results. Variation in reaction rates with milling history was recorded. XRD could not detect significant differences with variations in milling parameters. Therefore, XANES/EXAFS measurements were used to study the effect of milling on the Ti catalyst. *In-situ* XRD data indicated that Ti-catalyzed LiAlH₄ began to decompose at ~20 °C; therefore, the decomposition of LiAlH₄ sometimes observed during milling was due to thermal desorption rather than mechanochemical decomposition.