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Ultrasmall Angle X-ray Scattering (USAXS) and Wide Angle X-ray Scattering (WAXS) Studies on the Complex Metal Hydride NaAlH₄
TABBETHA DOBBINS, CHRISTOPHER BENNETT¹, JAMES TORRES, Rowan University, Dept. of Physics Astronomy, JAN ILAVSKY, Argonne National Laboratory, Advanced Photon Source — This research seeks to understand the role of ScCl₃, ZrCl₄, and VCl₄ catalysts in NaAlH₄. We have examined these hydrides at multiple length scales using an X-ray scattering instrument which is capable of measuring scattering wave vector, Q , of 0.0001 Å⁻¹ to 6.0 Å⁻¹. The ultrasmall angle X-ray scattering (USAXS) instrument sector 9ID-D of the Advanced Photon Source (APS) simultaneously collects using USAXS, SAXS, and WAXS detectors. Studies were performed during in-situ heating up to 170C (just below the H desorption temperature for uncatalyzed NaAlH₄). Results showed that NaAlH₄ has a surface fractal (highly porous) morphology. Isothermal studies performed at 30C, 65C, 100C, 135C, and 170C reveals changes at low Q ($Q \sim 0.001$ Å⁻¹ to 0.01 Å⁻¹) associated with highly interconnected intraparticle porosity which is suitably described by a power-law slope for a Gaussian polymer chain structure of $p \sim 2$. At high scattering wave vector, $Q \sim 0.03$ Å⁻¹, the presence of a pore population which obeys Porod scattering and appears to have a size at 21nm is present. These fine pores increase in their population density as temperature is elevated. The WAXS data reveals thermal expansion to occur, but no solid state phase transformation to the product phase.

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