

High resolution synchrotron powder diffraction data were collected using beamline 11-BM at the Advanced Photon Source (APS), Argonne National Laboratory using an average wavelength of \_\_\_\_\_ Å. Discrete detectors covering an angular range from -6 to 16° 2θ are scanned over a 34° 2θ range, with data points collected every 0.001° 2θ and scan speed of 0.01°/s.

The 11-BM instrument uses x-ray optics with two platinum-stripped mirrors and a double-crystal Si(111) monochromator, where the second crystal has an adjustable sagittal bend. (Wang *et al.*, 2008) Ion chambers monitor incident flux. A vertical Huber 480 goniometer, equipped with a Heidenhain encoder, positions an analyzer system comprised of twelve perfect Si(111) analyzers and twelve Oxford-Danfysik LaCl<sub>3</sub> scintillators, with a spacing of 2° 2θ. (Lee *et al.*, 2008) Analyzer orientation can be adjusted individually on two axes. A three-axis translation stage holds the sample mounting and allows it to be spun, typically at ~5400 RPM (90 Hz). A Mitsubishi robotic arm is used to mount and dismount samples on the diffractometer. (Preissner *et al.*, 2009) An Oxford Cryosystems Cryostream Plus device allows sample temperatures to be controlled over the range 80-500 K when the robot is used.

The diffractometer is controlled via EPICS (Dalesio *et al.*, 1994). Data are collected while continually scanning the diffractometer 2θ arm. A mixture of NIST standard reference materials, Si (SRM 640c) and Al<sub>2</sub>O<sub>3</sub> (SRM 676) is used to calibrate the instrument, where the Si lattice constant determines the wavelength for each detector. Corrections are applied for detector sensitivity, 2θ offset, small differences in wavelength between detectors, and the source intensity, as noted by the ion chamber before merging the data into a single set of intensities evenly spaced in 2θ.

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