

The 11-BM instrument consists of two radiation enclosures. The first houses successively, a beryllium window, a beam mask, motor controlled slits, a platinum-stripped collimating mirror, a double-crystal Si(111) monochromator, where the second crystal has an adjustable sagittal bend. The final optical component is a vertically focusing mirror, which can be used with or without a platinum stripe, for energies above and below 15 keV, respectively. (Wang *et al.*, 2008)

The second hutch contains two sets of motor-driven slits with a beryllium window and evacuated flight tube between. The first of these slits is used for controlling beam size and the second for limiting scattered radiation. Incident flux is monitored by pair of ion chambers. The first ion chamber is placed just after the beam-size defining slits and a Be window. The second, smaller ion chamber, is placed after the final slit and just before the sample. A vertical Huber 480 goniometer, equipped with a Heidenhain encoder, positions the analyzer and a three-axis translation stage holds the sample mounting. The stage also allows the sample to be spun as high as 6000 RPM (100 Hz). Two of these translation directions are used to center the sample stage at the central axis of diffractometer rotation; the third can be used to translate along the sample with respect to the beam. A Mitsubishi robotic arm with a custom-designed finger set is used to mount and dismount samples on the diffractometer. (Preissner *et al.*, 2009) An Oxford Cryosystems Cryostream Plus (formerly model 700+) cooling device is integrated with the robot, so that sample temperatures can be controlled over the range 80-500 K. Motorized translations bring the Cryostream to the sample position or move it out of the way — allowing the sample position to be accessed by the robot. The diffractometer, final slit, ion chamber, robot and Cryostream are mounted on a stage with two translation directions, both perpendicular to the beam direction; this allows the centering of the diffractometer circle to the beam.

The instrument uses perfect Si(111) crystal analyzer detection, since this provides excellent collimation and thus low backgrounds and optimal resolution that is not limited by sample size. The analyzers also provide excellent energy discrimination, so that sample fluorescence and Compton scatter does not impact data quality. (Cox *et al.*, 1988) Since perfect-crystal analyzers are not efficient, the instrument incorporates a multiplexed set of twelve such detectors, with a spacing of $2^\circ 2\theta$ between adjacent detectors. Each of the dozen analyzer crystals are mounted on separate goniometers; motors provide coarse ω -angle adjustment, with a piezoelectric driver and a weak-link bearing to provide the fine ω adjustment. Alignment along the perpendicular axis, χ , is provided by additional flexures and a picomotor; in practice this is adjusted manually. Oxford-Danfysik LaCl₃ Cyberstar scintillators are used to observe photons diffracted from the analyzer crystals. (Lee *et al.*, 2008)

The diffractometer is controlled via EPICS (Dalesio *et al.*, 1994) running in a VME crate dedicated to each radiation enclosure and an EPICS brick (<http://www.aps.anl.gov/bcda/ebrick/>), serving as a dedicated interface to the robotic system. Most motors are controlled using Oregon Micro Systems (OMS) motor controllers with Advanced Control Systems Step-Pak motor drivers. A 32-channel Struck scaler is used for intensity measurement. Data are collected while continually scanning

the diffractometer 2θ arm and where pulses generated by the OMS motor controller advance the channel used in the Struck card.

Data are reduced by applying calibration corrections for detector sensitivity, 2θ offset, and small differences in wavelength between each of the twelve detectors (typically on the order of 1 eV) as well as fluctuations in the source intensity, as noted by the second ion chamber monitor. These corrections allow the data for each detector to be interpolated onto a regular grid; intensities are then averaged, with appropriate adjustment of standard uncertainties. Calibration is performed using a mixture of NIST standard reference materials, Si (SRM 640c) and Al_2O_3 (SRM 676). The GSAS software package is used to fit a model to the data, where the reference Si lattice constant determines the wavelength for each detector individually. (Larson & Von Dreele, 2000)

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