Realizing an Analyzer Instrument for Medium-energy Sub-meV IXS

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Abstract. For the past few years, silicon (800) asymmetric back-diffraction at 9.13 keV has been investigated for use in inelastic x-ray scattering (IXS) analyzer with < 1 meV resolution and > 100 μrad acceptance. While the basic principles have been described and proven, attractive results have become consistently achievable only recently, with particular attention paid to key execution details, including crystal quality (substrate purity, orientation, surface flatness and strain), positioning (resolution, repeatability, and stability of critical axes), thermal environment stability and control, and x-ray diagnostics. While methods for positioning, diagnostics, and thermal stabilization have been refined in our work, crystal quality remains a critical limitation of ultimate performance. An overview of the implementation details is provided in the context of prototype x-ray beam test results collected in preparation for final design of the analyzer instrument to be incorporated into the IXS beamline of NSLS-II.

1. Introduction
A medium-energy x-ray energy analyzer with less than 1 meV resolution has been described [1]. This novel silicon crystal arrangement combines the wide angular acceptance (more than 100 μrad) of a collimating “C” crystal with the large dispersion (8.4 μrad/meV) of a highly asymmetric backscatter “D” crystal and the narrow acceptance (less than 5 μrad) of the combined transmission and wavelength selection of the “C” and “W” crystals. In this collimation-dispersion-transmission-selection (CDTS) scheme, the inherently narrow back-diffraction bandwidth (26.4 meV) is further reduced to below 1 meV, while maintaining high angular acceptance as needed for practical application in the spectrometer planned for use in the inelastic x-ray scattering (IXS) beamline of NSLS-II. Choice of the highly symmetric silicon (800) backscatter reflection at an energy of 9.13 keV makes nearby (440) reflections also available such that multiple wave effects must be explicitly considered for optimal design and operation [2].

Realization of this concept necessitates comprehensive control of the incident beam energy and precise two-axis angular alignment of the dispersion element, as well as stability and uniformity of temperature, angular positioning and crystal lattice. Methods for verifying the performance of these key features have been established in support of development of the IXS sub-meV analyzer.

2. Implementation
A prototype CDTS instrument was designed and constructed at BNL, making use of established technology in high-resolution angular positioning, temperature control, and crystal fabrication.
Figure 1 illustrates the mechanical assembly including crystal mounts and nominal beam locations. Crystal optic design parameters include 19 degree miscut for C and W (220) crystals (for 1.7 and 39.7 degree incidence angles, respectively), 300 µm C crystal thickness, and 88.5 degree miscut for D (800) crystal (for 1.5 degree incidence angle). High-performance circular flexures were implemented for precision angular positioning of D and W theta (rotation in the vertical dispersion plane) \[3\]. Translations effected with linear and stick-slip piezo actuators (both with encoders) are converted to rotations about central beam interaction pivot locations with sub-μrad control resolution (less than 50 nm at ~110 mm arm length). Other rotations utilize flex pivots or pin bearings, with closed or open-loop stick-slip piezo actuators or stepper motors. Translational alignments among the crystal (with respect to beam) were accomplished using conventional slide stages.

Figure 1. Prototype CDTS optical arrangement.

Silicon PIN photodiodes of 300 µm depletion layer thickness were used as x-ray intensity monitors and for establishment of crystal alignment. These are particularly convenient due to their small size, flux linearity and readily estimated responsivity \[4\]. Temperature controls were achieved utilizing Pt-100 sensors and Lakeshore 340 temperature controller / readout system. Crystals were fabricated commercially to specification by Crystal Scientific (UK) Limited and Sharan Instruments Corporation. X-ray beam tests were performed at NSLS, SPring-8 and Petra-III.

2.1. Energy Selection
Preliminary work with the CDTS spectrometer immediately demanded methodology development in monochromator energy selection. Although many monochromators can be absolutely energy-calibrated to within a few eV using reference absorption foils, the intrinsically narrow bandwidth of the CDTS instrument requires a more accurate refinement of the monochromator energy setting. Initial attempts to image or otherwise detect the (800) reflection near backscatter also proved difficult and inaccurate. However, since the (800) back-reflection is near-simultaneous with (440) reflections, for thin crystals it is possible to satisfy the Bragg condition of two (440) reflections (one up-going, one down-going) simultaneously at the same energy, as illustrated in figure 2; in prototype testing, the C crystal with (100) side-plane, set at 26 degree incident angle, has proven useful here. For energies away from exact backscattering the two reflections occur at different theta angles. Example experimental theta rocking curves confirming the correct monochromator energy are compared with full dynamical simulation \[5\] in figure 3.

2.2. Two-Dimensional Alignment
High resolution (< 1 meV) selection of photon energy by the D crystal to exact backscatter requires sub-μrad alignment in both vertical and horizontal planes. While the vertical reference is provided by design by C crystal transmission and W crystal wavelength selection, a horizontal reference is not provided by the CDTS scheme as originally conceived. However, again recognizing the near-
simultaneity of (440) reflections at the backscatter energy, we have specifically chosen the azimuthal orientation of the D crystal such that the surface normal is 1.5° from [011], thus providing two reflections, (440) and (404), each inclined 45° to the dispersion plane (inboard and outboard) and orthogonal to the beam incident upon D (1.5° incidence). These reflections occur at the same D theta (vertical angle) when the D phi (horizontal angle) is centered. This condition, verifiable by method of two overhead silicon photodiodes (as shown in figures 4 and 5), establishes energy and angle reference critical to the operational optimization of the CDTS analyzer [2].

2.3. Positioning, Temperature and Crystal Quality
The stability and repeatability of mechanisms responsible for crystal angles must be maintained to below 1 μrad in order to attain reliable 1 meV resolution and stability of the CDTS instrument. In an effort to demonstrate this capability, the D theta fine (linear piezo) angular motion was calibrated and verified to within 3% using multi-beam laser interferometry.

The crystal temperature stability and uniformity must be within 40 mK in order to reach the 1 meV level, based on the known linear coefficient of thermal expansion for silicon of 3×10⁻⁶/K. Practically this requirement has proven to be feasible with state-of-the art hutch controls and simple thermal enclosures; stability can be improved further using temperature controls (active crystal heating).
Lattice uniformity deviations in excess of $1 \times 10^{-7}$ arising from impurities, defects and strain can likewise degrade resolution beyond 1 meV. Ultra-high purity silicon (20-30 kohm-cm) substrate is used, and topography rules out gross localized crystal deformations. Strain from mounting is mitigated by use of polished steel mating surfaces, flat to 1 µm. Strain from polishing, however, is more difficult to control, in particular for the thin C crystal which is polished on both sides. Best known methods for mitigation of polishing strain include use of chemical-mechanical processes which simultaneously polish and etch.

Ultimately the test of performance of crystal quality (as well as positioning and thermal stability) to the 1 meV level is diffractometry in the form of integration into the CDTS instrument. A D crystal theta rocking curve width of $4.2 \mu$rad or less is indicative of sub-meV resolution performance. Reliable operation of the CDTS analyzer is demonstrated by repeatability over time, via data such as shown in figure 6 (under illumination by another CDTS unit), which indicate 0.5 µrad shift in peak position and width over several hours under otherwise constant conditions.

![Figure 6. D crystal theta repeatability data.](image)

### 3. Summary

Establishment of key operating parameters of CDTS sub-meV IXS analyzer has been described. Exploitation of multiple wave effects provides convenient energy and alignment reference for monochromator and analyzer. Precision control of angular position, temperature and crystal quality is required for reliable operation at bandwidth below 1 meV.

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### 5. References

[1] Shvyd’ko, Y V, Lerche M, Kuhtgens U, Rüter, H D, Alatas A and Zhao J 2006 Phys. Rev. Lett. 97 235502
[2] Stetsko Y, Keister J, Coburn D S, Kodituwakku C N, Cunsolo A and Cai Y Q 2011 Phys. Rev. Lett. 107 155503
[3] Shu D, Toellner TS and Alp EE 2001 Nucl. Instrum. Meth. A 467–468 771
[4] Owen R L, Holton J M, Schulze-Briese C and Garman E F 2009 J. Synchrotron Radiat. 16 143
[5] Stetsko Y P, Chang S L 1997 Acta Crystallogr A 53 28