

# A high-resolution inelastic x-ray scattering spectrometer at the Advanced Photon Source

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We have commissioned a new instrument for high-resolution inelastic x-ray scattering (HRIXS) on the inelastic scattering beamline of the Synchrotron Radiation Instrumentation Collaborative Access Team (SRI-CAT) on sector 3 of the Advanced Photon Source (APS). So far, the instrument is set up at 13.84 keV with a total energy resolution of 7.5 meV and a momentum resolution of  $\leq 0.1 \text{ \AA}^{-1}$ . We present technical details of the instrument, which includes an in-line monochromator, a focusing mirror, and a focusing analyzer. The performance of the instrument was demonstrated in studies of phonons in diamond and chromium.

The inelastic scattering beamline in the SRI-CAT of the APS has instead employed angle-tuned in-line monochromators to set a narrow bandpass [1, 2]. Changing the energy from an elastic peak to the desired phonon peak is no longer time consuming. The high-resolution monochromator follows a mirror, which focuses the x-ray beam horizontally and vertically onto the sample position of a five-circle diffractometer. A focal size of approximately  $0.5 \times 0.5 \text{ mm}^2$  can be achieved. The horizontal focal length is fixed due to a fixed sagittal curvature of the mirror. A vertical bending mechanism can focus the beam at different locations downstream. The photon flux at the sample position ranges between  $3$  and  $5 \times 10^9$  counts/s in a 5 meV bandpass. On the five-circle diffractometer, a high-resolution backscattering analyzer is mounted at a distance of 2.7 m. A schematic layout of the beamline is shown in Fig. 1.

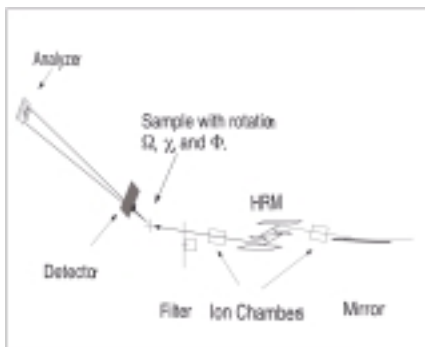


Figure 1: Instrument layout at the APS (HRM = high-resolution monochromator).

The primary high-heat-load (HHL) monochromator is a double-crystal monochromator in ( $\pm$ ) configuration. It

consists of two diamond crystals employing (111) reflections. The first crystal, which is exposed to the total radiation power emitted by the undulator insertion device, is water cooled. Details concerning the performance of the water-cooled diamond crystal are described elsewhere [3, 4]. A high-resolution monochromator limits the bandpass of the monochromatic photons from the HHL monochromator to 5.2 meV at 13.84 keV [5]. Its design and performance have already been detailed by Macrander *et al.* [6]. The four-reflection monochromator with one channel-cut crystal nested inside another uses asymmetric Si(422) and Si(884) reflections. Tuning of the incident energy is performed by rotating the inner crystal. A scan range of a few electron volts is achievable.

Perfect crystals are required to achieve an energy resolution approaching the intrinsic Darwin width. The current setup uses silicon wafers obtained from Virginia Semiconductors. Strain due to bending broadens the width of their reflections. Various methods have been developed to solve this problem [7, 8].

To avoid strain broadening in the bent crystal, Dorner *et al.* [9] had the silicon wafers diced into a pattern of  $0.8 \times 0.8 \text{ mm}^2$  blocks. The blocks were kept oriented by leaving a 200  $\mu\text{m}$ -thick backwall. Nevertheless, this kind of prepared wafers still shows significant strain propagating from the bent backwall into the remaining blocks. Furthermore, the reflected x-rays also probe the severely strained backwall exposed by the grooves. The current work was motivated by the need to relieve the residual strain and avoid scattering from within the grooves. The new approach is a silicon wafer bonded to a glass wafer using epoxy. After dicing through the silicon into the glass, a backwall is left in the glass only. A detailed description of fabricating such a “sandwich” analyzer is given elsewhere [10]. So far, analyzers have been made for bending radii of 2.6 m and 1.0 m, with the latter one intended for medium-energy resolution (a few 100 meV). The propagating strain from the backwall is reduced because glass is softer than silicon. Furthermore, the strained glass backwall does not contribute to the scattering.

We note that the magnitude of the intrinsic term depends on the material and on the order of the back reflection used. High-energy resolution ( $\sim 1 \text{ meV}$ ) requires high-order reflections (i.e., high-energy x-rays,  $> 20 \text{ keV}$ ). At these high energies, the penetration of the x-rays can exceed a few millimeters, in which case the x-rays become even more sensitive to strain.

The latest innovation in fabrication has yielded an analyzer with very good energy resolution and high reflectivity. To quantify the reflectivity, we have done measurements using the Si(777) reflection. The elastic scattering from plexiglass at 8.5 deg scattering angle, maximum of  $S(Q)$ , was measured using a strain-free flat crystal at a distance of 3 m. The detector was positioned 0.6 mrad away from backscattering, which implies an acceptance angle of 0.6 mrad for the Si(777) reflection. The detector aperture was  $2 \times 2 \text{ mm}^2$ . This setup yielded a count rate of 6 counts/s for a flat  $\langle 111 \rangle$  crystal positioned at 3 m distance (Fig. 2a). The acceptance angle of 0.6 mrad implies a reflecting area of  $1.8 \times 1.8 \text{ mm}^2$ . But, the source size and the small aperture of the detector only permit the detection of x-rays from a reduced area of  $1.4 \times 1.4 \text{ mm}^2$ . Instead, with a focusing analyzer placed at a distance of 2.7 m, this area reduces further to  $1.1 \times 1.1 \text{ mm}^2$ . The block sizes of current analyzers sitting at a distance of 2.7 m cover an area of ca.  $0.9 \times 0.9 \text{ mm}^2$  (i.e., *the whole area of each block contributes to the reflectivity*). However, one loses 20% reflectivity in the 0.1 mm-wide grooves. The exposed area of our analyzer is 83 mm in diameter, which encompasses 5411 blocks. Assuming all blocks are well aligned, we calculate an expected count rate with the focusing analyzer of  $21.7 \times 10^3$  counts/s, calculated by  $(0.9/1.1)^2 \times (6 \text{ counts/s}) \times 5411$ . In fact, we measure a count rate of  $14.3 \times 10^3$  counts/s (Fig. 2b), which represents an analyzer efficiency of 66%.

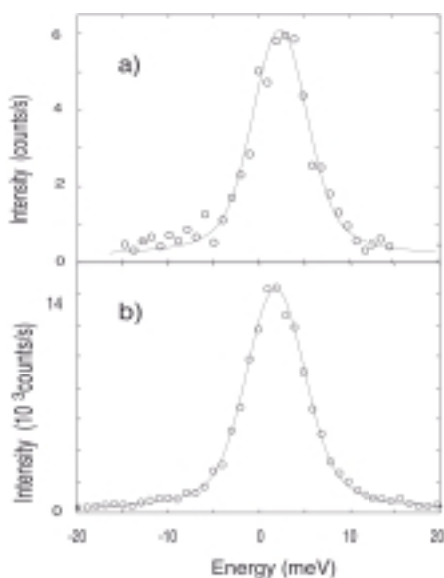


Figure 2: Total energy resolution function of the monochromator and the analyzer measured using a) a flat crystal, and b) a focusing analyzer. The elastic scattering is from a Plexiglass, and the employed reflection was the Si(777).

We note that this is a net gain provided by the analyzer of about 2400. The measured full width half maximum (FWHM) of the analyzer focus of 2.6 mm was larger than the detector aperture and indicates the presence of a slope

error of about  $150 \mu\text{rad}$  [11] (i.e., the mismatch of focus and detector aperture explains the 34% loss in the efficiency). Besides the high efficiency, the analyzer also performs with the same total energy resolution of 7.6 meV (FWHM) as was achieved with the flat crystal (Fig. 2). The profile of the energy resolution function is not purely Lorentzian. The line is the energy resolution calculated via ray tracing considering the intrinsic resolution of the Si(777) reflection, a block size of  $0.9 \text{ mm}^2$ , and a source size of  $0.5 \text{ mm}^2$ . During the reflectivity measurements, the flux of the monochromatized incident beam was  $3 \times 10^9$  counts/s. Finally, the use of a well-designed detector is essential to the efficiency and reliability of the instrument. The detector that we used is a CdZnTe detector specially adapted by Amptek Inc. The measured background was 0.03 count/s at 14 keV.

To demonstrate its feasibility for studying high-frequency phonons, we measured longitudinal acoustical and optical phonons in diamond (Fig. 3a) [12]. Our work on phonon scattering by chromium (a high-Z element) yields intensities of 15 counts/s comparable with the intensities in diamond (i.e., the higher electron density compensates the loss due to absorption, see Fig. 3b).

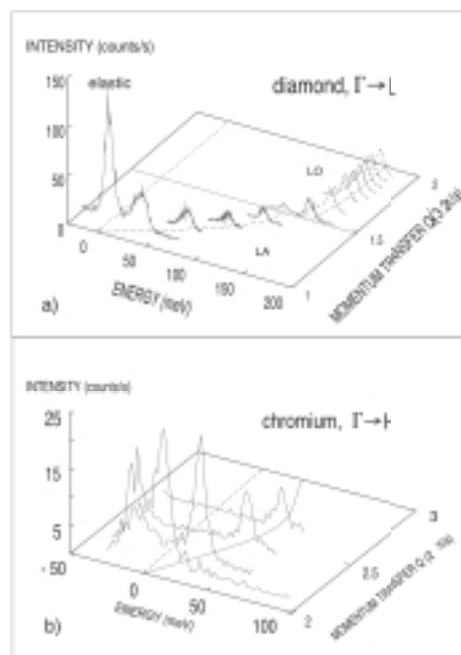


Figure 3: Spectra at different momentum transfers representing longitudinal modes in diamond (a) and chromium (b).

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## References

- [1] T.S. Toellner, T. Mooney, S. Shastri, and E.E. Alp, *SPIE Proceedings Series, Bellingham, Washington*, **1740**, 218 (1992).
- [2] W. Sturhahn, T.S. Toellner, E.E. Alp, X.W. Zhang, M. Ando, Y. Yoda, S. Kikuta, M. Seto, C.W. Kimball, and B. Dabrowski, *Phys. Rev. Lett.* **74**, 3832 (1995).
- [3] L. Assoufid, K.W. Quast, and H.T.L. Nain, *SPIE Proceedings Series, Bellingham, Washington*, **2855**, 250, (1996).
- [4] P. Fernandez T. Graber, W.-K. Lee, D.M. Mills, C.S. Rogers, and L. Assoufid, *Nucl. Instruments & Methods*, **A400**, 476 (1997).
- [5] T. Ishikawa, Y. Yoda, K. Izumi, C.K. Susuki, X.W. Zhang, M. Ando, and S. Kikuta, *Rev. Sci. Instrum.* **63**, 1015 (1992).
- [6] A.T. Macrander, M. Schwoerer-Böhning, P.M. Abbamonte, and M. Hu, *SPIE Proceedings Series, Bellingham, Washington*, **3151**, 271 (1997).
- [7] M. Schwoerer-Böhning, P.M. Abbamonte, A.T. Macrander, and V.I. Kushnir, *SRI-CAT Newsletter* **3**, 2 (1997), <http://www.aps.anl.gov/xfd/www/xfd/sri-cat/nl>).
- [8] E. Burkel, *Springer Tracts in Modern Physics Vol.125*, (Springer, Berlin, 1991).
- [9] B. Dorner, E. Burkel, and J. Peisl, *Nucl. Instrum. and Meth.* **A426**, 450 (1986).
- [10] M. Schwoerer-Böhning, P.M. Abbamonte, A.T. Macrander, and V.I. Kushnir, *SPIE Proceedings Series, Bellingham, Washington*, **3151**, 282 (1997).
- [11] R. Blasdel and A.T. Macrander, *Rev. Sci. Instrum.* **66**, 2075 (1995).
- [12] M. Schwoerer-Böhning, A.T. Macrander, and D.A. Arms, *Phys. Rev. Lett.* **80**, 5572 (1998).