The detection system shown in Fig. 1 allows measurements of the x-ray-diffracted intensity to be synchronized with the helicity modulation of a circularly polarized incoming x-ray beam, which alternates in the 1- to 50-Hz range. A square wave with half-duty cycle expands/contracts a piezoelectric actuator causing a phase-retarding optic to yield opposite helicities of circularly polarized x-rays. Upon helicity switching, a timing module triggers the incident and scattered intensity scalers of a dual photon counter for a time interval (gating) just below the half period of the square wave. This allows measurements of x-ray-incident and scattered intensities for opposite helicities to be performed over many helicity switchings in a short time, with the data for each type of helicity stored in even and odd addresses, respectively, of the photon counter's memory arrays. This detection scheme, coupled to a fast-counting avalanche photodiode detector, yields large improvements in signal-to-noise ratios and reduction of systematic errors over conventional detection, in which the helicity is switched only once.

The right panel of Fig. 1 compares data collected by using the conventional method (top) to that collected by using digital lock-in over 20 cycles of helicity switching (bottom). In addition to improvements in data quality, the lock-in measurement was done in half the time of the conventional measurement. This development extends the detectability of dichroic scattering/diffraction to 1 part in 10,000. It is especially suited for detection of dichroic diffraction from single crystals and small-angle dichroic reflectivity/scattering from layered nanostructures.

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AREA DETECTOR-BASED GIXD MEASUREMENTS FROM LIQUID SURFACE SAMPLES

Capabilities at CMC-CAT beamline 9-ID for investigating grazing incidence x-ray diffraction (GIXD) from liquid surfaces are now augmented by the use of a charge coupled device (CCD)-based area detector in conjunction with the liquid surface spectrometer (LSS). The CCD detector complements the two detectors routinely used with the LSS: a scintillation point detector used for reflectivity and a linear position-sensitive detector (PSD) equipped with Soller slits, normally used for GIXD. The area detector allows the GIXD pattern over a large range of reciprocal space to be captured simultaneously with exposures as brief as 10 s. This is especially advantageous for organic or biological specimens, which may be damaged by radiation during the longer exposures required to complete scans with the PSD. As an example, the new configuration was useful in observing the GIXD from a Langmuir monolayer composed of alpha-helical peptide bundles, which scatter weakly in the region near q_{xy}≈0.55 Å^{-1} (Fig. 1). At this value of the momentum transfer, the effects of the decreasing Δq_{xy} resolution with increasing scattering angle are not so severe. To scan the same region of reciprocal space with the PSD (10 s/point) would have required approximately 600 s exposure for a Δq_{xy} resolution of 0.012 Å^{-1}.

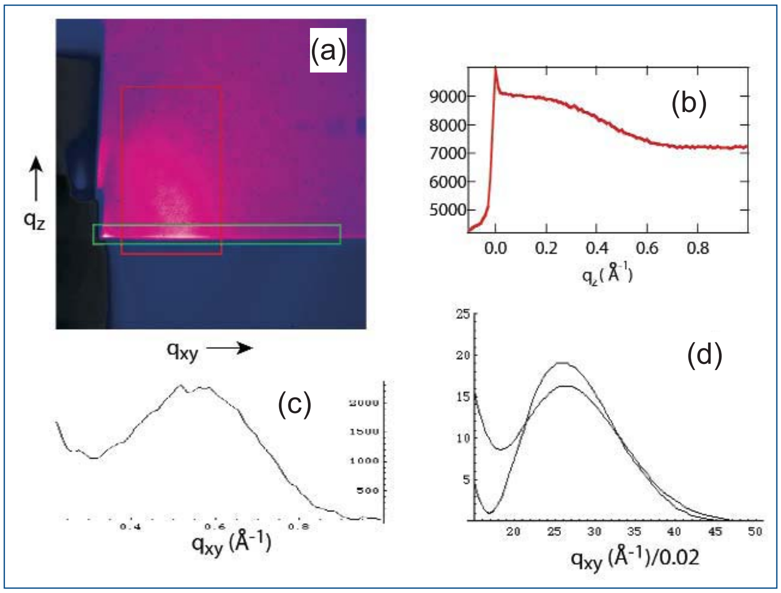

Fig. 1. Grazing incidence x-ray diffraction from peptide monolayers. (a) Two-dimensional CCD image of GIXD from a monolayer of a synthetic peptide designated AP2 at high surface pressure. Integration of the region bounded by the red rectangle along q_{xy} gives the intensity as a function of q_z (b), including background scattering. Integration of similar data from a monolayer of another peptide, designated AP0, along the q_z direction (as indicated by the green box), after background subtraction of otherwise equivalent GIXD from the pure subphase in the absence of the peptide monolayer, gives the background corrected GIXD as a function of q_{xy} (c). Model calculations for the q_{xy} dependence of the in-plane diffraction from a bundle of 4 cylinders show good agreement with the data in (c), especially once the experimental Δq_{xy} resolution is considered.

In the most recent configuration of CCD-based GIXD measurements, a Langmuir trough on the sample stage of the LSS contained the specimen. The CCD detector (Bruker SMART 1500) was

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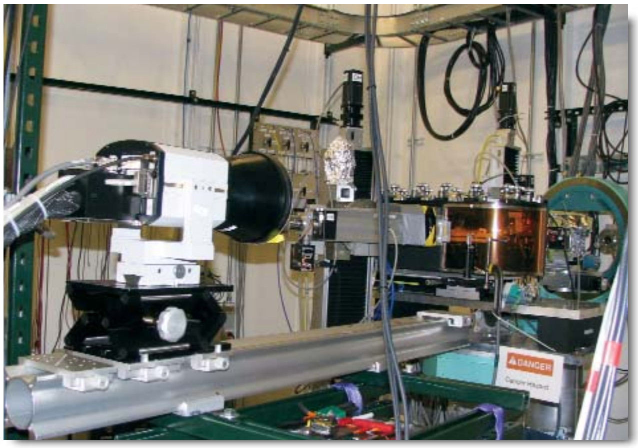

Fig. 2. Area detector-based GIXD setup. The CCD detector is shown in the foreground at the end of the X-95 rail, out of the way of the point detector. The beamstop is mounted against the kapton exit window of the canister enclosing the Langmuir trough on the sample stage of the liquid surface spectrometer.

mounted on an X-95 rail supported from the floor. This allows the specimen-detector distance to be varied from 26 to 120 cm; or, for x-ray energy of 13.474 keV, the corresponding q-ranges to be varied from more than 2 $Å^{-1}$ to about 0.5 $Å^{-1}$. However, the CCD detector cannot be scanned. The beamstop at the exit window of the trough canister limited the minimum q to about 0.3 $Å^{-1}$. Additional shielding upstream of the sample and a fixed aperture inside the entrance window of the canister reduced background. Alignment of the sample requires use of the point detector in reflectivity mode, with the CCD detector moved out of the way to the largest specimen-detector distance, so that the user must enter the hutch to return the CCD detector to the desired specimen-detector distance. The geometry prevents the PSD from being used when the rail is in position near the sample, but the point detector can still be scanned through angles up to approximately 15°, or q_{xy}~1.7 $Å^{-1}$.

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Silicon Sawtooth Refractive Lenses for High-Energy X-ray Optics

X-ray Operations and Research beamline 1-ID has been utilizing Si-based refractive lenses for vertical focusing of high-energy (50-100 keV) x-rays at both low and high source demagnification geometries (~1:0.6 and ~1:0.05, respectively). The high x-ray intensity from vertical focusing has enhanced a number of sector 1 programs, including time-resolved diffraction, small-angle scattering, and high-pressure studies. Such lenses can also be used for x-ray angular collimation in order to improve the throughput of subsequent high-energy-resolution optics, as has been demonstrated with cylindrical aluminum lenses [1].

The Si lenses are based on a sawtooth geometry, described elsewhere [2], and exhibit a number of desirable properties. First, their effective profile closely approximates a single parabolic lens, eliminating cylindrical aberration. Second, by adjusting the taper between the top and bottom sets of the teeth, the focal length can be varied. For a given position downstream of the lens, this allows for tunability in the focused beam

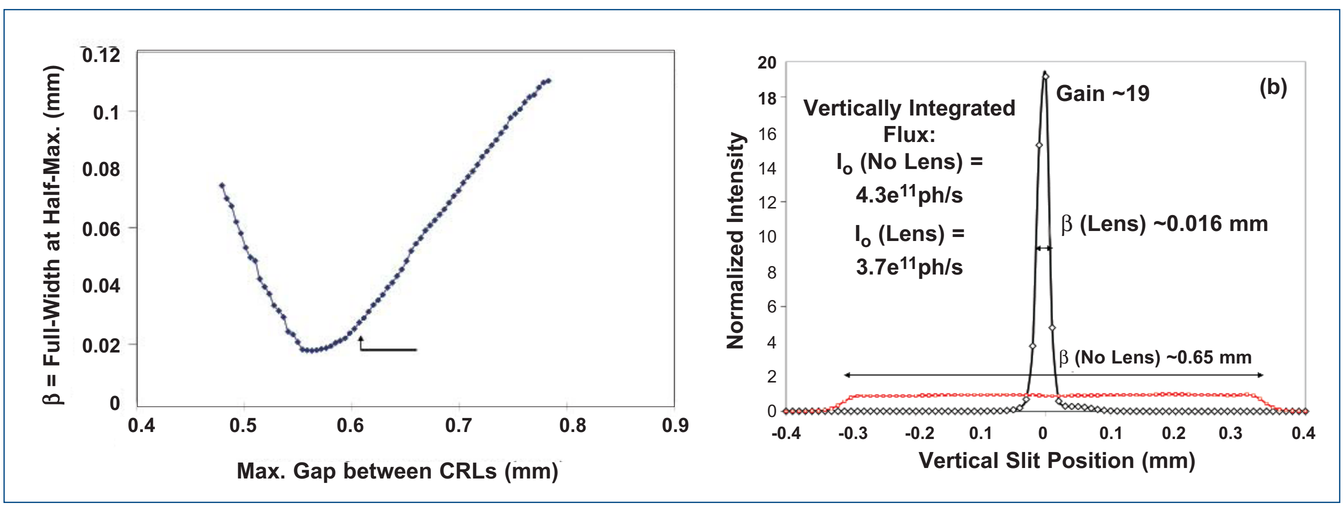

Fig. 1. (a) Variation in vertical beam size with taper end-gap between lens pieces and (b) beam profiles with and without the lens, as measured using a 0.005(v) × 1(h) mm^2 aperture and PIN diode at the entrance to the C-hutch.