The redesigned optics front-end also employs separate ultra-high-vacuum compatible monochromator and mirror enclosures that reduce thermal cross-talk between crucial optical components. These improvements have resulted in more stable x-ray beams into A1, F1 and F2 experimental stations. For example, the angular drift on the second monochromator crystal at F2 during a normal fill is dramatically reduced, by about a factor of five [1], resulting in a more stable beam and more reproducible beam energy for crystallography experiments.

We thank Ernie Fontes, Dana Richter, Alan Pauling, Tom Krawczyk, Bob Seeley, Karl Smolenski, and Jim Savino for leading the various projects in the high-heat-load optics area. This work is supported by NSF and NIGMS through CHESS.

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Multilayer Optics at CHESS

Alexander Kazimirov

Cornell High Energy Synchrotron Source, Cornell University

Silicon crystal optics provide the simplest way to produce monochromatic and collimated x-ray beams. However, for those experiments where narrow energy bandwidth of the order 10^{-4} is not required, x-ray optics based on multilayers (ML), with much wider bandpass, can deliver a factor of 10^2 more photons. Examples of experimental techniques used at CHESS that take full advantage of multilayer optics include time-resolved SAXS, radiography, x-ray scattering during crystal growth, and microbeam fluorescence analysis. During the last few years we have designed, fabricated, and tested ML optics for both bending magnet and wiggler beamlines. At present, 5 of 12 stations are based on ML optics. Below we describe some of our recent accomplishments [1].

Internally cooled optics: Operating at 300 mA the new A/G line 49 pole wiggler delivers a beam of about 3.0 kW to the A2 mono after the low energy part of the spectrum is removed by a Be window and Carbon filter. Internally water cooled Si substrates for MLs were designed and built [2] using our experience in internally water cooled Si optics [3] to handle this heat load. The internally cooled ML consists of two parts (Fig. 1); the top piece has 1 mm wide water channels and 1 mm wide fins between the channels, and the bottom part contains a water manifold. Their parts are bonded together at CHESS using a silver brazing technique [4], polished at Wave Precision (formerly General Optics), and coated together with matched flat uncooled substrates at the APS (group of Al Macrander) and Osmic. These multilayers show average reflectivity of about 62% and bandwidth of 2.5%. Fig.2 shows the assembled water-cooled ML inside the A2 monobox.

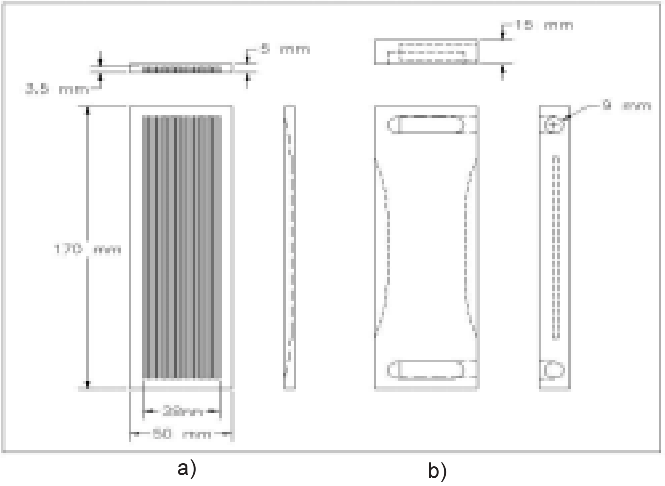

Fig. 1 Water cooled silicon multilayer substrate. (a) The top part contains the parallel cooling grooves, (b) the bottom part contains the manifold connected to the heat exchanger.

Fig. 2 Assembled ML in A2 mono (dark block) and surrounding cooling mount made of Cu.

Sagittal horizontal focusing optics: Due to the small incident ML Bragg angle, a sagittal focusing ML requires a very small radius of curvature of order 0.2-0.4 m. As a first step, a fixed radius sagittal ML was designed and built (Fig. 3). The radius of 225 mm was selected corresponding to the energy of 11keV at A2 and 8.0 keV at F3 beamlines. The cylindrical silicon substrate was fabricated by SESO and coating with d-spacing of 27Å to match the upstream, flat cooled ML made at the APS. The sagittal ML showed average reflectivity of 45%, lower than the flat MLs due to increased surface figure errors. An x-ray flux of 8×10^{13} photons/s/mm^2 within a spot size 2.8 mm wide was produced at A2 beamline by using this sagittal ML in combination with the first water-cooled ML.

Fig. 3 Sagittal focusing ML with a radius of 225 mm on a silicon substrate. The ML is 150 mm long, 50 mm wide, and 20 mm thick.

High-resolution optics: To bridge the gap between traditional Si and wide bandpass ML optics, high-resolution multilayers with an energy bandwidth in the range of 10^{-3} have been proposed [5]. They are based on a large number of bi-layers (up to several hundred) and use low-contrast low-Z layer materials. The scattering from such a multilayer becomes more "dynamical", leading to a much more narrow rocking curve. At present, the most promising layer combination is Al_2O_3/B_4C. The first samples produced by OSMIC have been tested recently at CHESS using high-resolution optics based on Si(111) double crystal mono and Si(400) channel cut crystals in a dispersive arrangement. Fig. 4 shows the resulting rocking curve for the multilayer with 500 bi-layers. The width of the curve is 14.6 arc sec and the bandwidth $\Delta E/E=0.29\%$ with reflectivity of 50%. Such high-resolution multilayers would be very useful for crystallography of small and medium size unit cell proteins.

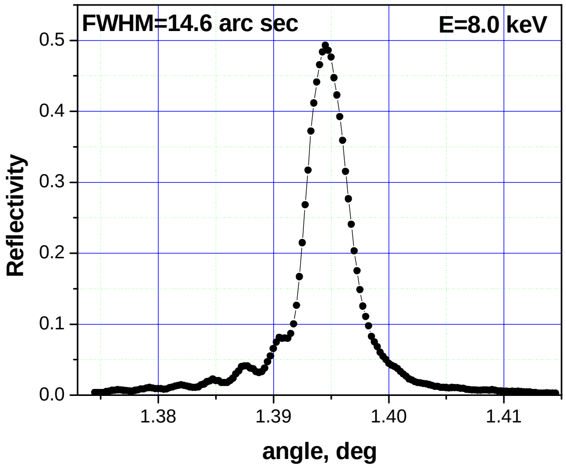

Fig. 4 Reflectivity curve from the OSMIC low-contrast multilayer with 500 bi-layers and d=31.5 Å.

These accomplishments were made possible by the collective efforts of CHESS staff (Randy Headrick, Karl Smolenski, Qun Shen, Ernie Fontes, Jim Savino, Tom Krawczyk, Walt Protas), our collaborators at the APS (Albert Macrander, Chian Liu, Ali Khounsary) and at OSMIC (Vladimir Martynov, Yuriy Platonov, Jim Wood).

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Microfabrication and Application of Novel X-ray Optics

Ken Finkelstein

Cornell High Energy Synchrotron Source, Cornell University

New Sagittal Focusing Crystals: Most synchrotron radiation experiments benefit from increased intensity, and one means to boost intensity is to collect x-rays diverging from a source and focus them on the sample or detector. The sagittal focusing crystal illustrated in Figure 1 is the downstream element in an energy tunable, double crystal monochromator used to collect and concentrate several horizontal milliradians of bend magnet or wiggler radiation. The crystal is bent tighter as the source and/or focus distances decrease and with decreasing Bragg angle (increasing energy), so dynamic (adjustable) bending is required. These crystals become slightly saddle shaped (anticlastic curvature) when bent and this broadens the rocking curve, significantly diminishing x-ray throughput. We counteract this effect with an array of stiffening ribs oriented parallel to the surface and along the incident beam.

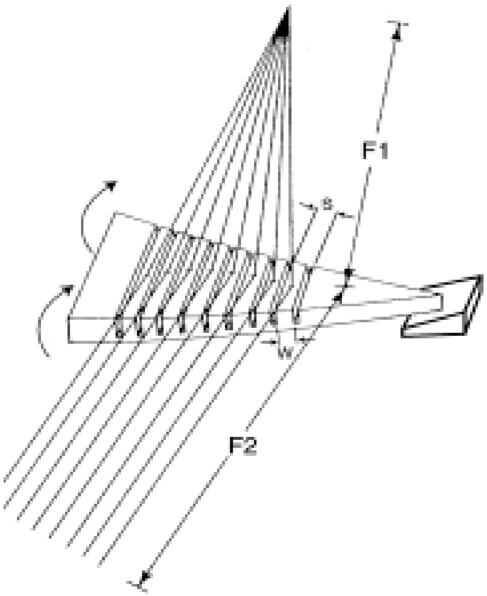


Fig. 1 Illustration of horizontal focusing of x-rays by a triangle shaped, segmented sagittal focusing crystal. The triangle bends in a cylinder when a moment is applied at the base with the apex contacting an inclined surface. Bending is confined to thin regions at the bottom of trenches. Ribs of width W stiffen the crystal against anticlastic deformation. S is the trench period.[1,2,3]

The crystal curvature is segmented by the ribs so the focus cannot be smaller than the projection of the x-ray source by one segment. If W (and S) are reduced the focus is made smaller, but diamond tool cutting and surface damage limit W to about 1 mm.

A new technology for x-ray optics, available at the Cornell Nanofabrication Facility, is used to reduce W and S [4]. Figure 2 shows what has become the standard sagittal crystal design for C1. The crystals are made in four steps from 100 mm diameter by 900 micron thick silicon: 1) the pattern is transferred by photolithography to photoresist coating a silicon dioxide etch mask covering the wafer, 2) this pattern is transferred to SiO_2 by reactive ion etching (RIE), 3) deep-RIE, using the “Bosch process”, etches trenches in silicon, 4) when pattern depth is achieved the optic is released from the wafer by laser cutting. Deep-RIE replaces diamond tool cutting, produces straight walled trenches independent of crystal orientation, etches up to 2.5 microns/min and eliminates need for chemical etching that is required after cutting.

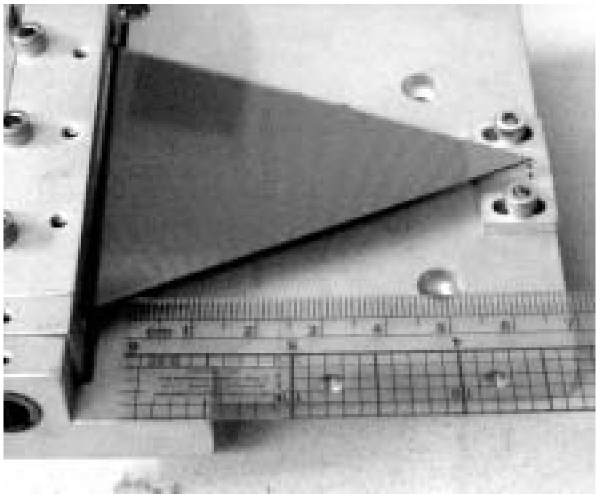
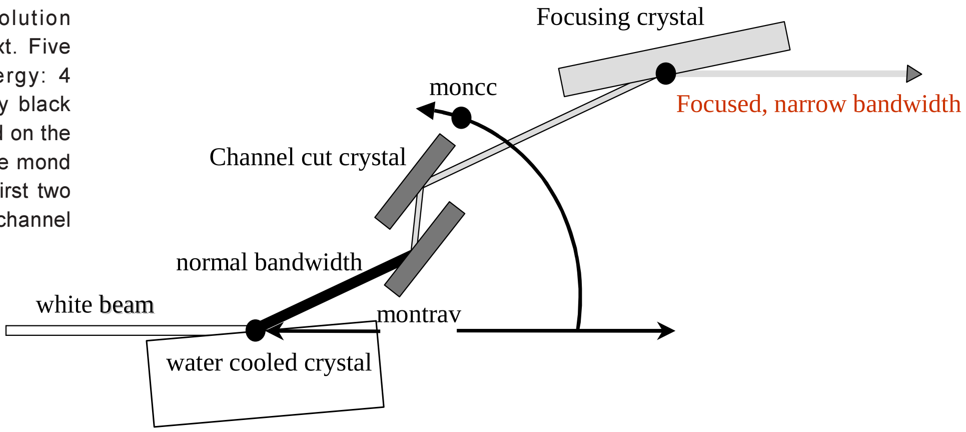


Fig. 2 Deep-RIE fabricated sagittal focusing crystal is mounted in a standard CHESS bender. Fabrication is outlined in the text. The diffracting surface, defined by 118 trenches, each 250 microns wide, has 500 micron period.

Novel Monochromator for High Resolution Energy Scanning: We have incorporated the new sagittal focusing crystal in a novel high flux, narrow bandwidth, energy scanning, monochromator for radiation between 7 and 16 KeV [5]. X-rays bounce 4 times through the device (illustrated in Figure 3) when a channel cut crystal (Figure 4) is “nested” between crystals of the 2-bounce mono. The energy width is reduced when the channel cut is dispersive (i.e. energy selective) relative to the other crystals. For high flux, the crystals are built to pass up to 5 milliradians (400 Watts incident power when CESR operates at 250 mA, 5.3 GeV) of radiation. The first crystal contacts a cooled copper mounting block via liquid indium-gallium. Bandwidth can be changed in a straightforward manner by: 1) selection of the channel cut reflection and 2) using an asymmetric first crystal reflection. 2-bounce operation is reestablished by lowering the nested crystal.

Fig. 3 Illustrates the high resolution monochromator discussed in the text. Five motions are required to scan energy: 4 rotations about axes are indicated by black dots (moncc travels on an arc centered on the first crystal axis), plus translation of the mond axis parallel to the white beam. The first two reflections set bandwidth. The crystal channel passes a wide horizontal beam



At 8KeV, the 2-bounce mono will focus 1x10^{12} photons/sec in a 1 mm by ½ mm spot. In 4-bounce configuration, using a (400) channel cut, 3x10^{10} photons/sec are focused to 2 mm by 1 mm and the beam energy width is less than 0.28 eV. Based on bandwidth reduction, we expect to approximately double the flux recorded at high resolution; this discrepancy is not yet fully understood.

The 4-bounce monochromator has recently been used for inelastic x-ray scattering (IXS) to investigate retinal isomerization in bacteriorhodopsin, a photosynthetic bacterium. IXS records the spectrum of electronic excitations

Fig. 4 Silicon channel cut crystals for the 4-bounce monochromator provide narrow bandwidth and high flux. These crystals utilize (111) reflections; the left (right) crystals operate between 7 and 10 (12 and 19)KeV. High order reflections are used for higher energy resolution.

at atomic length scales. Figure 5 shows the resolution obtainable when energy is scanned and signal collected by a 3" diameter germanium (444) backscattering analyzer.

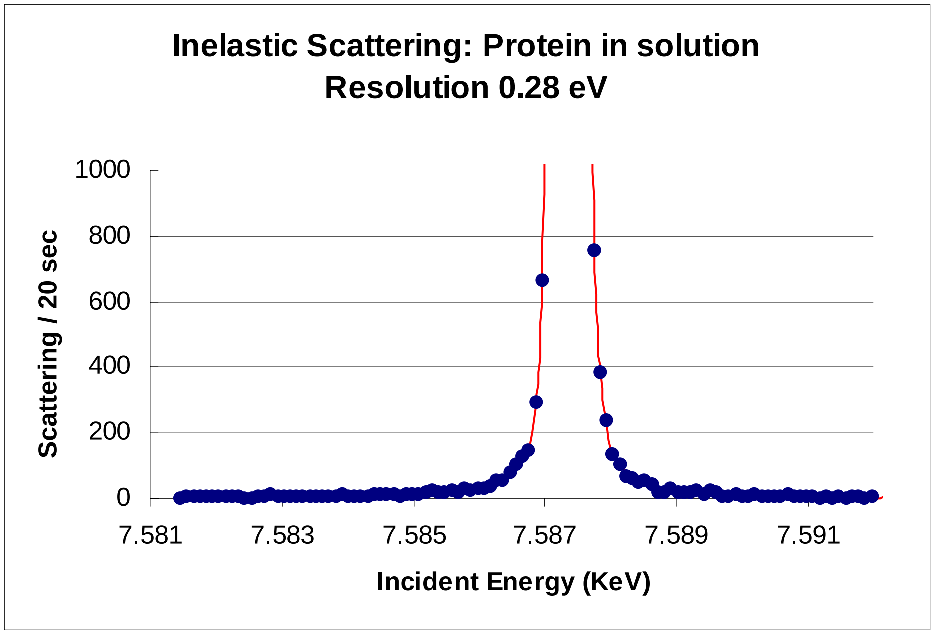


Fig. 5 The spectrum of x-ray scattering from bacteriorhodopsin in solution with 10% protein. Photons are collected at 20º scattering angle with a spherically bent germanium (444) analyzer as incident energy is scanned. The width of the elastic scattering peak, 0.28eV, measures the energy resolution of mono + analyzer.

Acknowledgements

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Microfocusing Monocapillary Developments and X-ray Applications

Don Bilderback[1], Rong Huang[1], Chang-Sheng Zha[1], Alex Kazimirov[1], Ernie Fontes[1], and A.A. Sirenko[2]

[1]Cornell High Energy Synchrotron Source, Cornell University, [2]Agere Systems

Introduction: Tapered hollow glass capillaries based on total reflection from the inside smooth glass wall can make beams on the micron scale and significantly enhance their beam intensity (flux/μm^2). Below we describe capillary applications to x-ray fluorescence on thin semiconductor films and to high-pressure experiments.

X-ray Fluorescence Study of Thin Films: Condensing capillary CHI015 with 0.8 micron exit tip size has been used to measure the thickness and uniformity of a 0.3 μm thick $In_{0.47}Ga_{0.53}As$ thin film grown by selective area growth (SAG) technique on an InP(100) wafer at D-line. The films are grown by metal organic vapor phase epitaxy in the vicinity of SiO_2 masks spaced 20 μm apart. These striped regions are important building blocks for optoelectronics. We observed that the presence of these masks induces thickness variations (compared to the open field) of 10 to 300% - a most interesting result as one might naively think that the deposited layer thickness should not depend on the presence or absence of silicon oxide layers nearby. We have not seen any changes in the arsenic concentration, but up to 7 ± 0.5% variations in the gallium concentration [1]; see figure 1. These findings have direct implications for future device construction.

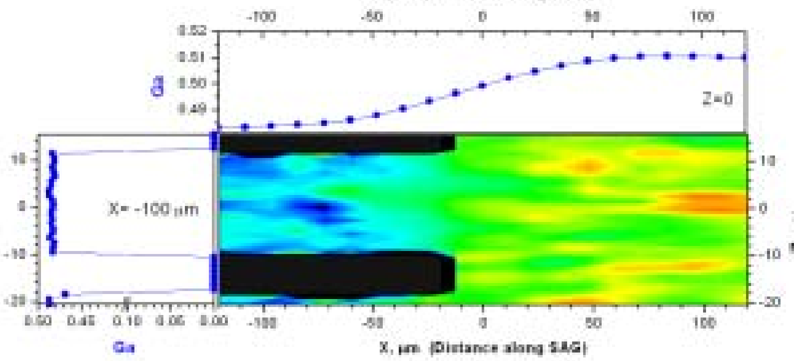
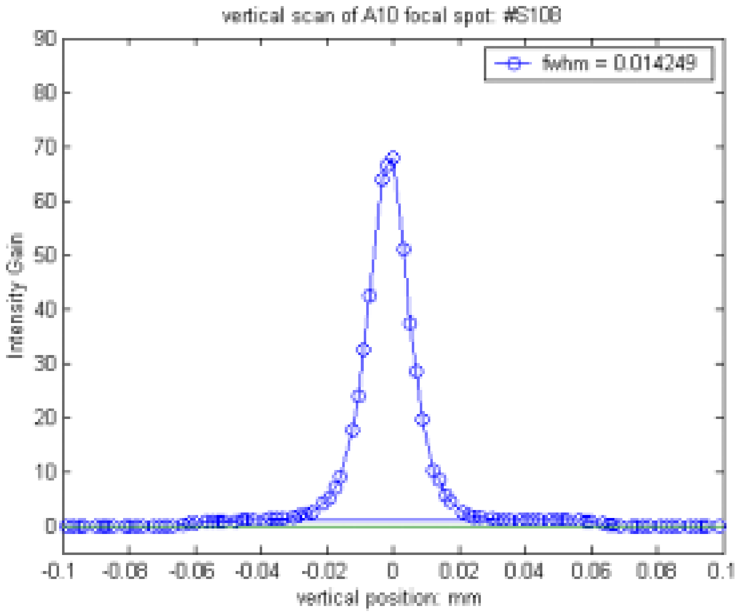


Fig. 1 Gallium concentration determined by x-ray fluorescence as observed between SiO_2 masks (black areas). The gallium concentration is significantly lower between the islands of SiO_2 on the left and the "open field" on the right side.

High-Pressure Diffraction in Diamond Anvil Cells: We have recently made a single-bounce tapered capillary to focus the beam at the CHESS A2 station. The capillary is 10.5 cm long and elliptically tapers from 211 to 123 mm from the base to the tip end. The distance from the capillary tip to the focus is 55 mm for this optic and the design divergence is 2 milliradians. From the optical metrology analysis, the observed departure from the ideal mathematical figure is only 1.7 microns (rms) and the slope errors measured with x-rays are 70 microradians. To date, this is the most accurately made capillary on the in-house capillary puller - not bad for a freely drawn shape from glass tubing!



At 15 keV at the A2 station, the x-ray properties were measured. Figure 2 shows the intensity gain as measured by flux through a 10 micron pinhole scan. This capillary is now available for users to use in future high pressure experiments.

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Fig. 2 Capillary A10 shows a 70 fold intensity gain on the A2 station compared to the no capillary situation. The base pedestal line comes from the direct straight through beam and is used to normalize the gain scale to 1. This capillary will be ideal for increasing the intensity of powder x-ray experiments in diamond anvil cells. With the angles of incidence being less than 1 mr on the glass, the capillary should focus fine to at least 30 keV.