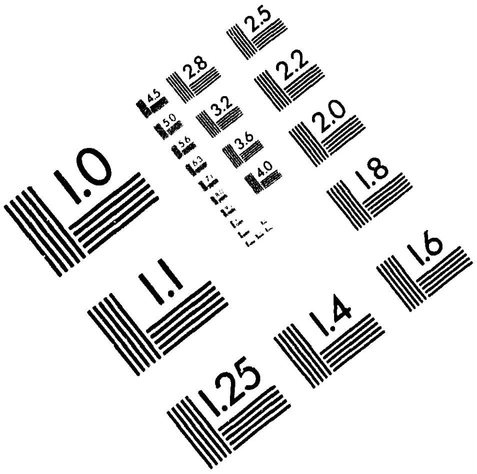

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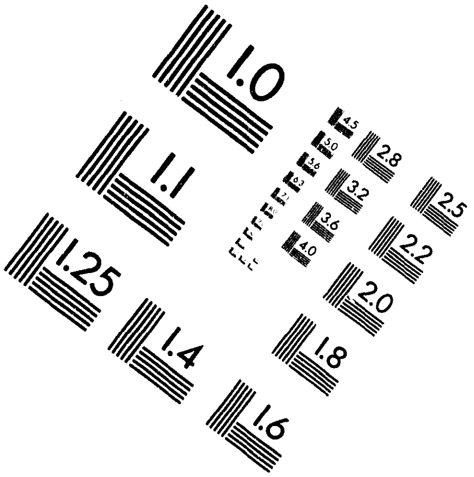

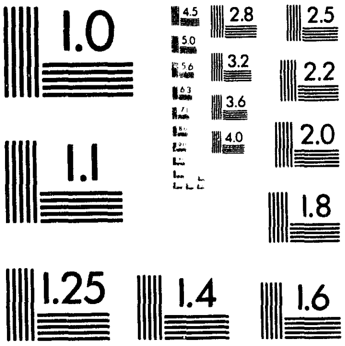

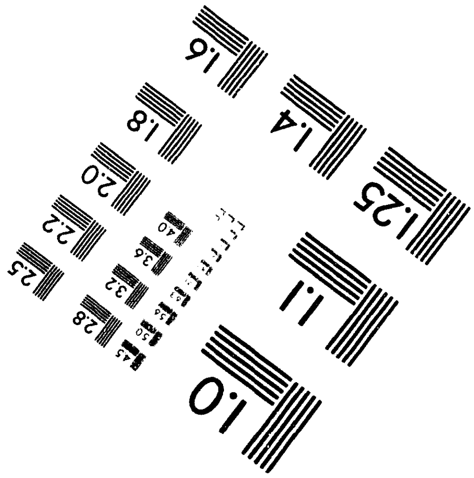

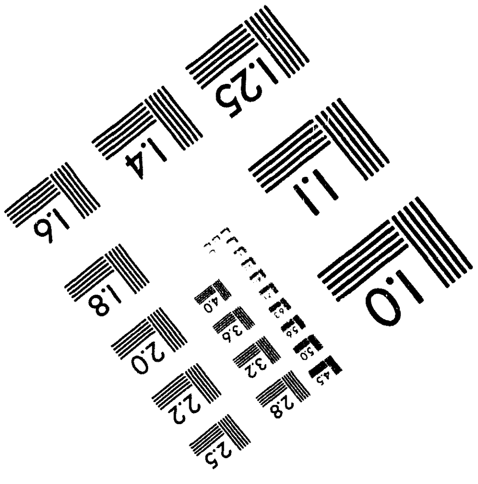

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Time-Resolved X-Ray Scattering Program At the Advanced Photon Source

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The Time-Resolved Scattering Program's goal is the development of instruments and techniques for time-resolved studies. This entails the development of wide bandpass and focusing optics, high-speed detectors, mechanical choppers, and components for the measurement and creation of changes in samples. Techniques being developed are pump-probe experiments, single-bunch scattering experiments, high-speed white and pink beam Laue scattering, and nanosecond to microsecond synchronization of instruments. This program will be carried out primarily from a white-beam, bend-magnet source, experimental station, 1-BM-B, that immediately follows the first optics enclosure (1-BM-A). This paper will describe the experimental station and instruments under development to carry out the program.

I. Introduction

The 7-GeV Advanced Photon Source (APS) synchrotron x-ray source is under construction at Argonne National Laboratory. Universities, industry, and national laboratories have joined together as Collaborative Access Teams (CATs) to run and operate sectors at the APS[1], where each sector comprises a bend magnet and insertion device source. The Synchrotron Radiation Instrumentation Collaborative Access Team (SRI CAT) will operate three sectors at the APS. Of these three, the Sector 1 bending magnet source has three experimental stations, namely 1-BM-A, 1-BM-B, and 1-BM-C. The first experimental station 1BM-A is also the first optics enclosure. It contains the double crystal monochromator, a vertically collimating mirror, filter box, beam stop, slits, shutters, etc. Station 1-BM-B is assigned for techniques and instrumentation related to time-resolved x-rays scattering, and station 1-BM-C is designated for polarization studies.

Components under development are focusing and wide bandpass optics, high-speed one- and two-dimensional detectors, and mechanical choppers for single-bunch x-ray scattering. Besides these components that are special to the program, the experimental station will be equipped with a four-circle 5020 Huber diffractometer, NaI scintillation detectors, ionizing chambers for beam normalization, video monitoring equipment, and high speed oscilloscopes (20-GHz equivalent time analog oscilloscope and a 500-MHz digital oscilloscope).

II. Focusing and Wide Bandpass Optics

One of the many factors limiting time-resolved x-ray scattering studies is the flux incident on the sample.[2] For some experiments, a highly monochromatic beam is not required. In which case, wide bandpass monochromators can be used to increase the flux. There are a number of ways flux and/or bandpass on the sample can be enhanced; mosaic crystals, layered synthetic multilayers, curved crystals, mirrors, etc., can be utilized (one could, of course, use an undulator source too). But this program is being designed to develop techniques in time-resolved x-ray scattering including the development of several focusing- and flux-enhancement techniques. A bend magnet beamline gives us the most flexibility to develop such techniques and, hence, was chosen over an insertion-device-based beamline. There are many time-resolved techniques that are brilliance limited (for example, coherent x-ray scattering). In this case, the related experiments will be carried out on an insertion device beamline.

As our first optical system to be incorporated in this program, we have chosen a curved crystal for horizontal focusing of the beam. The beam transport to this station is designed to accept 3.7 mrads of the bend magnet source. At a distance of 31 meters from the source, where the experimental station 1-BM-B begins, this swath translates into a beam that is approximately 11 cm wide. We would like to capture that entire swath of the beam for experiments. A curved crystal in horizontal geometry allows us to tune the bandpass besides allowing us to change the energy. By satisfying the Rowland condition, one can achieve a focused monochromatic beam and otherwise obtain a focused polychromatic beam whose polychromaticity depends on the curvature of the crystal. Thus, this geometry also allows us to set up for time-resolved dispersive diffraction or transmission EXAFS too. The drawback to such a system is that the monochromator is not a fixed-exit device, and so the diffractometer has to be moved around in the experimental station as the energy and/or focus is changed. To facilitate this, the table on which the diffractormeter will sit on will be on air pads. The motion of this table will be under computer control and tied to the curvature and orientation of the crystal.

Table 1 displays some characteristics of the APS that are important to this program. The optical element that will first be installed in 1-BM-B will be a Si(111) curved-crystal monochromator with a fixed curvature. Because the vertically collimating mirror has a cutoff at 24 keV, the calculations were performed for a crystal that is to be used from 6 to 24 keV. The total power in the bend magnet is 500 watts, and cooling the crystal is much easier if it does not have to be dynamically bent. The energy variation is then obtained by rotating the crystal about the scattering vector. The best energy resolution is obtained when the Rowland condition is satisfied.[3,4] Namely,

$$1/2R = \sin(\theta + \alpha)/p = \sin(\theta - \alpha)/p' = 1/b.$$

Where R is the radius of the Rowland circle, p is the source to crystal distance, p' is crystal to focus distance, and b is the bend radius.

The factors that affect the energy resolution of such a monochromator are

i) Horizontal source extension, h, which gives rise to

$$a1 = h/p$$

ii) The deviation of the crystal from the Rowland circle, giving

$$a2 = l/2(\sin(\theta - \alpha)/p - \sin(\theta - \alpha)/p'),$$

where l is the length of the crystal.

The energy resolution is given by

$$\Delta E/E = \cos\theta(\omega^2_{acc} + (a1+a2)^2)^{1/2},$$

where ω_{acc} is the acceptance of the asymmetrically cut crystal.

By deviating from the Rowland condition, a similar setup can be used for focusing quasi-parallel polychromatic radiation.[5] This energy-dispersive beam converges to a focus at the sample position; the beam transmitted/reflected from the sample then diverges towards a position-sensitive detector.

The total energy range obtainable by such a setup is given by

$$\Delta E = E_o l(l/R - \sin\theta/p)\cot\theta,$$

where E_o is the energy of the central beam, and l is the length of the crystal. A typical energy range achievable is from 100 eV to 1 keV.

Figure 1 shows the geometrical setup for the two focusing optical elements, namely, the monochromatic and polychromatic focusing. The polychromatic focusing element will be incorporated after the vertically collimating mirror is installed on the beamline. The bend magnet power is 86.7 W/mrad. The mirror, set for a cutoff of 24 keV reflects 59% of this power.[6] This will enable us to better manage the thermal heating of the optical element that needs to be dynamically bent.

Figure 2 displays a footprint of the white-beam station with the different focal points being plotted as a function of position in the experimental station. The polychromatic focusing is plotted for a Si(220) crystal whereas, the monochromatic focusing is plotted for a Si(111) crystal. Figure 3 displays the energy resolution and focal spot size achievable for a perfectly bent Si(111) crystal having an asymmetry of 15.13 degrees.

III. Detector Development

Another important factor limiting time-resolved studies is detectors. Depending on the experiment, time-resolution requirements can range from picoseconds to seconds, and spatial size ranges from a point detector to large area detectors. A few of the new detectors under development are described below.

During normal operation, the ring will be filled with 20 bunches of positrons with an interbunch spacing of 184 ns and a bunch width of 72.5 ps. To perform experiments with x-rays generated by positrons on these time scales necessitates that one have extremely high-speed detectors. To achieve the necessary high-speed, we are developing MBE-grown, CdTe-based photoconductive, position-sensitive array detectors. The arrays fabricated have 64 pixels with a gap of 100 μm between pixels. For test purposes, arrays have been fabricated with different pitch. X-ray static measurements were performed using a x-ray tube and synchrotron radiation to study the response of the array to flux and wavelength changes. A temporal resolution of 34 psecs was achieved using a short pulse laser.[7]

Charge coupled devices (CCDs) have been used successfully as x-ray detectors. Unfortunately, they are inherently slow because of the serial readout. EEV, a subsidiary of General Electric Company of England, has developed a CCD that has eight channels of parallel readout, thus increasing the speed eight fold. Using state-of-the-art VXI electronics, we have developed a readout system[8] that could read the entire array in 2.5 ms using a 20-MHz readout clock. For testing and characterization, the device was clocked at a significantly slower speed of 30 kHz. The data are preamplified, and all eight channels of output are simultaneously digitized to 12 bits and stored in buffer memory. The system is controlled by a 486-based PC through an MXI bus and VXI controller using commercially available software. The system is also capable of real-time image display and manipulation.

CCDs are high resolution, small area detectors. For large area applications, several groups modify the frontends using phosphor screens coupled to demagnifying tapers.[9] Our large area device consists of four CCDs, 2x2 matrixed, coupled to a fiber optic taper[9]. The front end of this device consists of a scintillator coupled to a fiber-optic taper. The fiber-optic taper consists of four smaller (70 mm x 70 mm) tapers fused together in a square matrix giving an active area of 140 mm x 140 mm. Each taper has a demagnification of 5.5 resulting in four small ends that are 12 mm diagonally across. The small ends of each taper are coupled to four microchannel-plate-based image intensifiers. The output from each image intensifier is focused onto a charge coupled device (CCD) detector. The four CCDs are read out in parallel and are independently controlled. The image intensifiers also act as fast (20 ns) electronic shutters. Additionally, with independent control on the readout of each row of data from the CCD, the system is capable of performing high speed imaging through novel readout manipulation.

IV Discussion

The comissioning of the APS storage ring is scheduled to begin early 1995. 1-BM-A and 1-BM-B will be the first two experimental stations built. The first experiment to be performed shall be on 1-BM-B. Towards our goal of developing a station for time-resolved experimental techniques, we have to yet design the mechanical chopper. Other components that are being addressed are elements for heating and monitoring samples at a rapid rate. Techniques that are being developed are pump-probe and single bunch scattering. We hope that in a few years, all it will take is for an experimenter to be able to bring just a sample and be able to perform a successful experiment.

Acknowledgments

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References

[1] See *Guidelines and Forms for Proposals to Establish Collaborative Access Teams at the **Advanced Photon Source**, November 1990*, Argonne National Laboratory publication.

[2] See *Time-Resolved Studies and Ultrafast Detectors: Workshop Report*, ANL/APS-TM-2 (February 1988).

[3] M. Lemonnier, R. Froume, F. Rousseaux and R. Kahn, Nucl. Instrum. and Meth. 152(1978) 173-177.

[4] W. Schildkamp, Nucl. Instrum. and Meth. A266 (1988), 479-483.

[5] H. Tolentino, F. Baudelet, E. Dartyge, A. Fontaine, A. Lena and G. Tourillon, Nucl. Instrum. and Meth. A289 (1990) 307-316.

[6] Private Communication with Lahsen Assoufid, SHADOW calculations for 1-BM.

Martin Stanton, Walter C. Phillips, Daniel O;Mara, Istvan Naday, and Edwin Westbrook, Nucl. Instrum. and Meth. A325 (1993), 558-567.

[7] Sung Shik Yoo, Kemei Wang, Pedro A. Montano, Jean-Pierre faurie, Qiang Huang and Brian Rodricks, Nucl. Instrum. and Meth. in press.

[8] Qiang Huang, Ron Hopf and Brian Rodricks Nucl. Instrum. and Meth. in press.

[9] Brian Rodricks, Qiang Huang, Ron Hopf and Kemei Wang, Nucl. Instrum. and Meth. in press.

Table 1. Important parameters of the APS bending magnet source

Energy (GeV)	7.0
Current (mA)	100.0
Bend Radius (m)	38.96
Peak Magnetic Field (T)	0.6
Critical Energy (keV)	19.5
Flux at Critical Energy	1.12×10^{13} ph/s/0.1%BW/mradH
Horizontal Size - σ_x (mm)	0.11
Vertical Size - σ_y (mm)	0.11
Vertical Divergence (mrad)	0.073

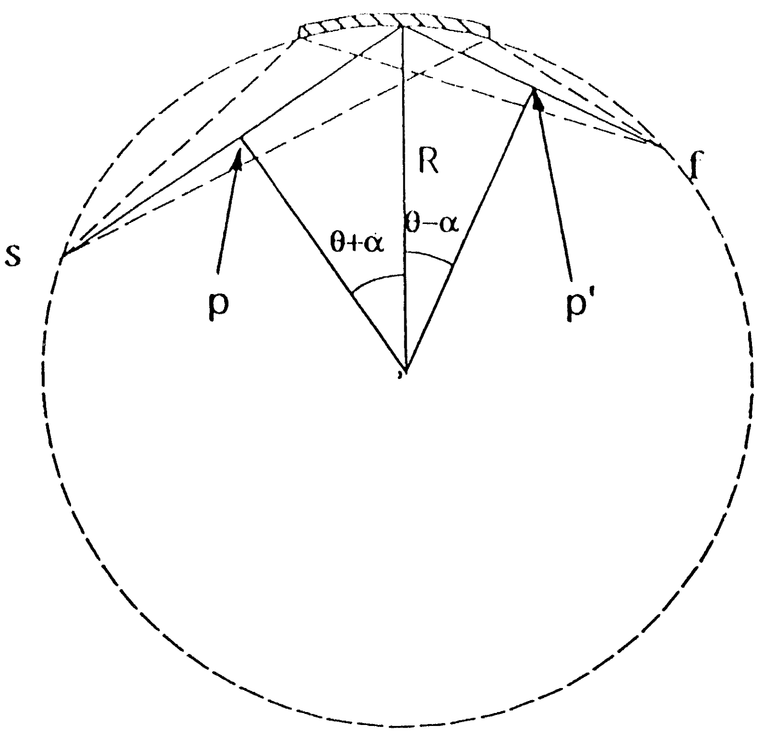
Figure Captions

Fig. 1 Focusing by an ideal cylindrically bent crystal. Here a) Bragg reflection for monochromatic focusing, and b) polychromatic focusing.

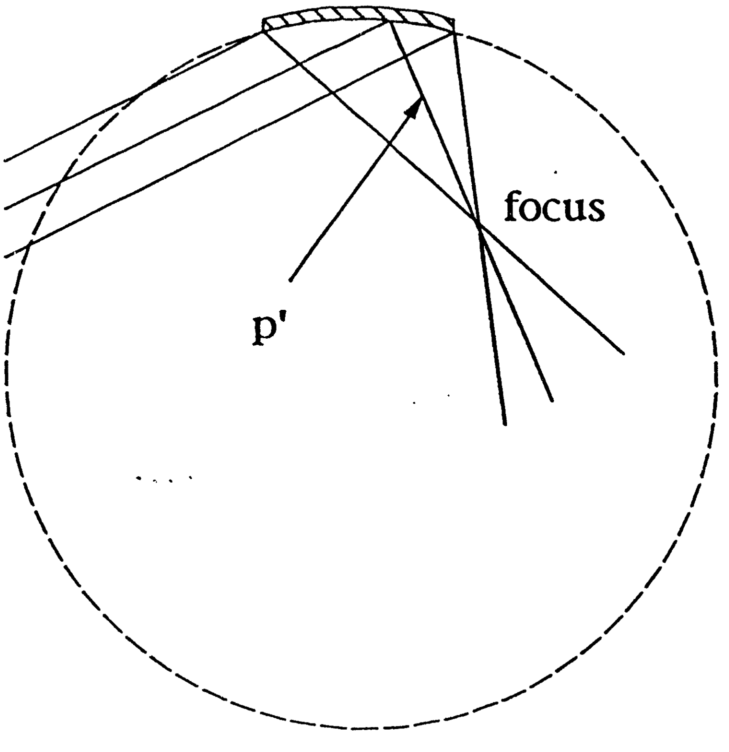
Fig. 2 Station 1-BM-B is 6.8 m long and 5 m wide. The locations of various focal point for both monochromatic and polychromatic radiation are shown in the experimental station. The monochromator location is at (.5,.5).

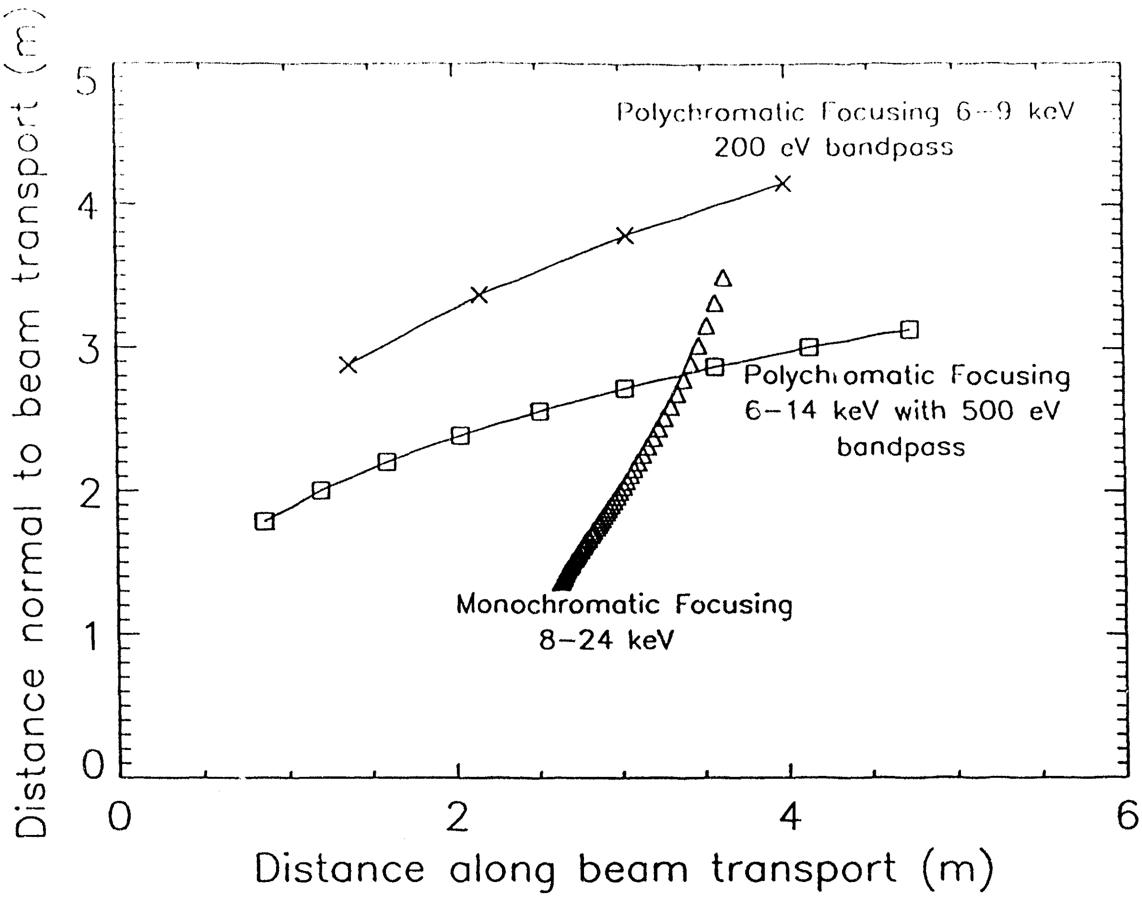
Fig.3 Energy resolution and focal point size for a perfectly bent Si(111) crystal with an asymmetry of 15.13 degrees.

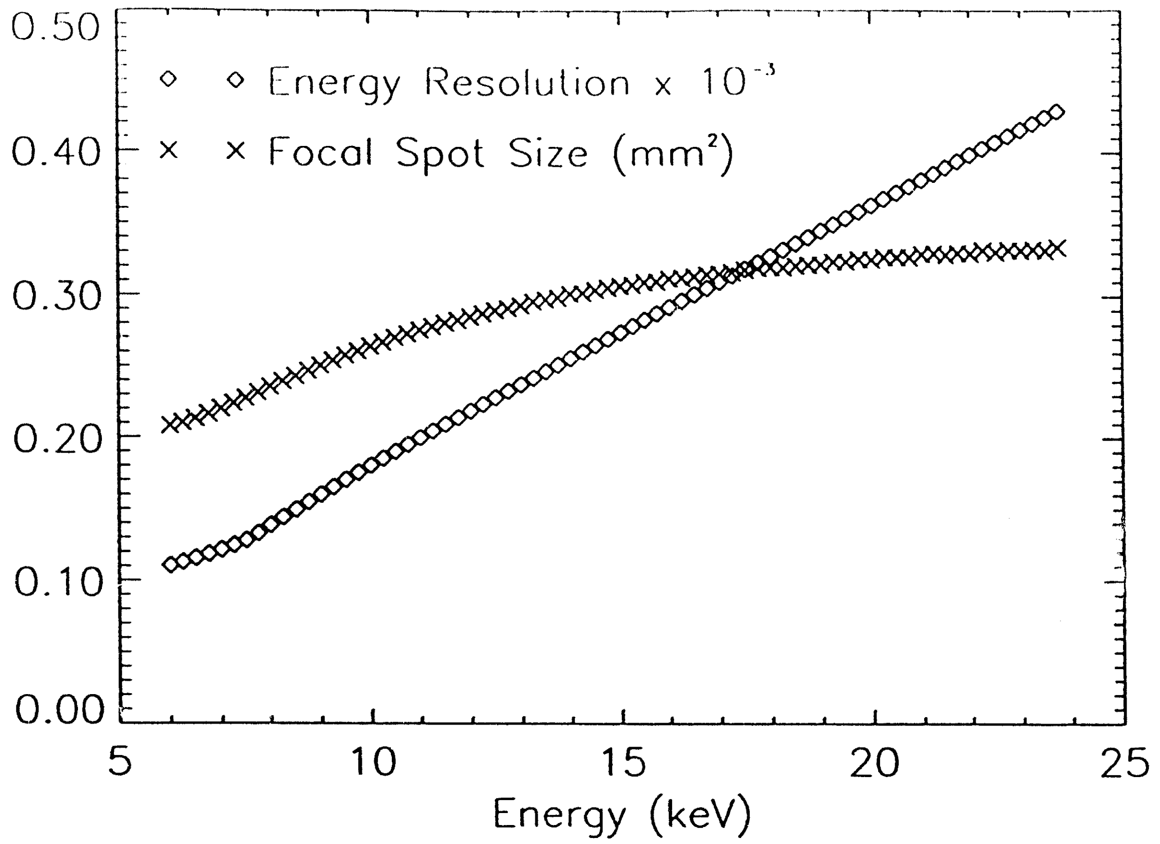
a)



b)




Polychromatic Focusing 6–9 keV
200 eV bandpass
Polychromatic Focusing
6–14 keV with 500 eV
bandpass
Monochromatic Focusing
8–24 keV
Distance normal to beam transport (m)
Distance along beam transport (m)
0
1
2
3
4
5
0
2
4
6



◇ Energy Resolution x 10^{-3}
× Focal Spot Size (mm^2)
0.50
0.40
0.30
0.20
0.10
0.00
5
10
15
20
25
Energy (keV)

DATE
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