Bragg crystal polarimeter for the Spectrum-X-Gamma mission*

J. Holley, E. Silver, and K.P. Ziock Laboratory for Experimental Astrophysics Lawrence Livermore National Laboratory Livermore, California 94550

R. Novick and P. Kaaret Columbia Astrophysics Laboratory 538 West 120th Street New York, New York 10027

M. Weisskopf and R. Elsner NASA/Marshall Space Flight Center Huntsville, Alabama 35812

J. Beeman Lawrence Berkeley National Laboratory 1 Cyclotron Road Berkeley, California 94720

ABSTRACT

We are designing a Bragg crystal polarimeter for the focal plane of the SODART telescope on the Spectrum-X-Gamma mission. A mosaic graphite crystal will be oriented at 45 ° to the optic axis of the telescope, thereby preferentially reflecting those x-rays which satisfy the Bragg condition and have electric vectors that are perpendicular to the plane defined by the incident and reflected photons. The reflected x-rays will be detected by an imaging proportional counter with the image providing direct x-ray aspect information. The crystal will be ~50 μ m thick to allow x-rays with energies \geq 4 keV to be transmitted to a lithium block mounted below the graphite. The lithium is used to measure the polarization of these high energy x-rays by exploiting the polarization dependence of Thomson scattering. The development of thin mosaic graphite crystals is discussed and recent reflectivity, transmission, and uniformity measurements are presented.

1. INTRODUCTION

In a previous report ¹ we reviewed the theory of Bragg crystal diffraction as it applies to the design of a crystal polarimeter with optimum sensitivity for observing stellar x-ray sources above the energy of 1 keV. The Stellar X-Ray Polarimeter (SXRP), to be flown aboard the Soviet Spectrum-X-Gamma spacecraft, will incorporate a thin (=50 μ m) mosaic graphite crystal mounted above a target of metallic lithium, as shown in Fig. 1. The graphite crystal, mounted at 45°, acts as a polarization analyzer for photons with energies that satisfy the condition for Bragg reflection at this angle. The Bragg energy is given by the relation

$$E = \frac{nbc}{2d\sin 45^\circ}$$

where E is the energy of the photon, n is the diffraction order, h is Planck's constant, c is the speed of light, and d is the crystal lattice spacing. We use the (0,0,2) plane of graphite, with a 2 d spacing of 6.7 Å. This spacing gives

•This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract No. W-7405-ENG-48 with support from NASA Funding Order W-17,151, MOD.1.

UCRL-JC--104996

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first, second, and third order Bragg reflections at 2.6, 5.2, and 7.8 keV, respectively. The lithium block, mounted below the graphite, Thomson scatters x-rays in a polarization sensitive manner. It forms a second polarimeter which is effective in an energy band extending above 4 keV. The simultaneous use of the graphite crystal and lithium polarimeters will provide polarization data over the energy band from 2.6 keV to 12 keV.^{1,2,3} A third polarimeter, also shown in Fig. 1, is included in the SXRP. It is based on the recently reported vectorial photoelectric effect in CsI⁴ and may be operated by removing the graphite crystal and lithium block from the telescope focus. In this report we present recent results from our measurements of thin mosaic graphite crystals being developed for the SXRP.



Figure 1. Projection view of the polarimeter arrangement for the SXRP.

Most stellar x-ray sources emit a weak continuum flux over a broad energy range. Consequently, to achieve maximum sensitivity in a Bragg crystal polarimeter the crystal must have a large integrated reflectivity (sum of the reflection coefficient over all Bragg angles). Perfect crystals typically used for Bragg diffraction are unsuitable for the SXRP due to their narrow energy bandwidths. Instead, the SXRP will employ an imperfect crystal, consisting of a randomly oriented mosaic of small perfect crystal domains. A mosaic crystal has a much broader energy bandwidth than does a perfect crystal because the variation in the orientations of the crystalets allows Bragg reflection over a larger range of angles. This results in a greater integrated reflectivity and makes a mosaic crystal a superior detector for continuum x-ray sources. For a fixed photon energy, E_B , the integrated reflectivity, $\Delta \Theta(E_B)$, of a mosaic crystal is defined as

$$\Delta\Theta(E_B) = \int_{\theta_1}^{\theta_2} R(\theta, E_B) \, d\theta$$

(2)

where $R(\theta, E_B)$ defines the reflectivity of the crystal as a function of angle for energy E_B and is commonly called the "rocking curve" over the Bragg angle range from θ_1 to θ_2 . The mosaic spread, which is the angular width at half maximum of $R(\theta, E_B)$, provides a direct measure of the misorientation of the crystal domains of the mosaic crystal.

2. CRYSTAL MEASUREMENTS

In testing crystals for the SXRP we are interested in measuring the integrated reflectivity, the mosaic spread, the

transmission, and the uniformity of these parameters across the crystal surface. To measure these parameters, a wellcollimated, monoenergetic x-ray beam from a triple-axis spectrometer as described by Christensen *et al.*⁵ is used. The instrument consists of two identical, perfect crystals in the dispersive configuration which produce a monoenergetic ($\delta E \leq 1 \text{ eV}$) x-ray beam collimated to better than 1 arc min. This is ideal for measuring the relatively broad (~30 arc min) rocking curves of the mosaic graphite crystals.

The experimental setup is shown in Fig. 2. We use the (1,1,1) planes of silicon for the monochromator (M) and analyzer (A) crystals. An x-ray tube with a rhodium anode creates 2.70 keV (L α) x-rays which are Bragg reflected by the silicon crystals at a Bragg angle of ~47°. At this angle, the silicon crystals produce a beam for probing the graphite crystal that is 99% linearly polarized. Since we are interested in the integrated reflectivity of graphite for unpolarized x-rays, numerical results presented here have been corrected for the polarization of the probing beam. The graphite crystal is rocked about the Bragg angle of ~43° to obtain the reflectivity $R(\Theta, E_B)$. It may also be translated in order to check the crystal uniformity by measuring at different regions of the crystal. The reflected x-rays are detected with a gas proportional counter. The uncertainty of the integrated reflectivity measurements is *±1.5% and includes time variation of the x-ray source intensity and counting statistics.

To determine the x-ray transmission of the crystals, the full bremsstrahlung spectrum of the rhodium anode, up to ~15 keV, is used. Two spectra are accumulated; the first one with the graphite crystal between the source and the detector, the second with the crystal removed and the detector viewing the source directly. The ratio of the first spectrum to the second is used to generate the transmission efficiency of the crystal as a function of energy. The transmission measurements are repeated with the graphite oriented at various angles with respect to the x-ray beam. A Si(Li) detector with an energy resolution of 180 eV at 6 keV is employed for these measurements.



Figure 2. Schematic of the triple-axis spectrometer used to probe the mosaic graphite crystals.

We are currently applying the theory of thin crystal diffraction to the design of the SXRP. We have calculated^{1,6,7} that a 50 μ m thick graphite crystal will provide the asymptotic value of integrated reflectivity for Bragg reflection and still allow 66% transmission at 4 keV (and higher transmission at higher energies). To verify these calculations we have undertaken a comprehensive crystal measurement program on graphite from two different vendors, Union Carbide Corporation and Fanasonic Corporation.

Union Carbide, which supplied the graphite crystals for the OSO-8 polarimeter, has furnished us with relatively thick samples (3-5mm thick) of pyrolytic graphite from which we can produce wafers with thickness $\approx 50 \ \mu m$ by a technique of successive thinning.¹ The sample is first cleaved to produce a $\approx 250 \ \mu m$ thick wafer using a razor blade. The wafer is then fixed to a carbon block with melted wax and is successively reduced using adhesive tape to lift off

layers of the graphite. Using this technique, we have produced crystals 25 and 50 μ m thick, with lateral dimensions of 1 cm by 2.5 cm. We have also tested larger samples with lateral dimensions of 5 cm by 5 cm. In our first attempt at thinning such a large sample, we inadvertently produced a wafer that was too thin (~10 μ m). Although a wafer this thin does not give the desired integrated reflectivity, its structural integrity has encouraged us to believe that the 50 μ m thick, 4 cm by 6 cm piece necessary for the flight model will be self-supporting. The 10 μ m sample has recently survived vibration tests as prescribed by the Soviet spacecraft designers without any sign of damage. This finding is important since any beryllium foil required to support the crystal will further attenuate the x-rays reaching the lithium polarimeter.

We have also obtained samples from the Panasonic Corporation, which uses a unique manufacturing process to produce their graphite crystals. They have developed a proprietary method for converting a polycarbon material into one that is crystallographically similar to pyrolytic graphite. Their trade name is "Supergraphite". We have tested a sample supplied by Panasonic with dimensions 1.5 cm by 2 cm by 80-100 μ m.

3. RESULTS

3.1 Union Carbide crystals

From Union Carbide Corporation we have obtained three types of pyrolytic graphite, differing in the sizes of the crystalet domains. Smaller domains should produce a larger mosaic spread and also a larger integrated reflectivity. In addition to testing these samples, we also tested one of the 200 μ m thick flight spare crystals from the OSO-8 polarimeter experiment. Although the OSO-8 crystals are not large enough to be used on the SXRP, we performed these measurements as a check on our facility and to allow comparison with previous work.⁸ We present our results for the OSO-8 crystal in Figure 3, which shows an overlay of rocking curves measured at the three crystal locations indicated. The mosaic spreads and integrated reflectivities at these positions are listed in Table 1. The average integrated reflectivity of 1 x 10⁻³ rad is in agreement with measurements made by Kestenbaum⁸ but our average measured mosaic spread is 0.56° rather than his reported value of 0.80°. We attribute this difference to the fact that Kestenbaum's x-ray beam was not as monoenergetic as that produced by the dispersive double-crystal spectrometer we used. The measured integrated reflectivity is ~25-30% less than theoretical predictions^{1,6,7}. However, it is still the highest value measured for any crystal at this energy to date.

The measurements of the OSO-8 sample provide a reference for comparison to the measurements on samples recently supplied by Union Carbide. (Note that the OSO-8 crystals are not large enough for use in SXRP). Figure 4 shows the rocking curves and locations of the measurements for a 1.3 mm thick crystal of a type that Union Carbide calls "ZYB". The measured integrated reflectivity is $\sim 5.5 \times 10^{-4}$ rad and the mosaic spread varies greatly with position, as listed in Table 1. Figure 5 presents results for a "ZYC" type crystal 50 mm x 50 mm x 0.8 mm in size. The measured integrated reflectivity is $\sim 8 \times 10^{-4}$ rad, with a variation of $\pm 3\%$ in values measured at four points on the crystal surface (see Table 1). One of the four rocking curves is significantly skewed with respect to the other three, possibly indicating a bend in the crystal surface. We tested our technique for thinning on this sample, obtaining the 10 µm wafer described above. Not surprisingly, the measured integrated reflectivity for the rocking curves depicted in Fig. 6 is only 5-6 x 10⁻⁴ rad, as compared to the theoretical value for this thickness of 1.1 x 10⁻³ rad^{1.6.7}. We have consistently found measured integrated reflectivities for graphite to be lower than the theoretical values. In Fig. 7, we show the rocking curves for a "ZYA" type crystal that we thinned to 50 µm. Its measured integrated reflectivity is 8-9 x 10⁻⁴ rad and it is the most promising type of Union Carbide material we tested.

We have also performed transmission measurements on the thin samples mentioned above. In Fig. 8 the transmission of the 50 μ m ZYA oriented at 45° to the incident beam is plotted versus energy. From weighing bulk samples of the pyrolytic graphite we have estimated the density to be 2.2 g/cm³, in agreement with the standard range of densities for graphite. We find that the transmission is well described by conventional mass attenuation data for carbon using this value for the density. The theoretical transmission curve for 50 μ m of carbon is shown by the solid line. The spread of the points about the curve is due to the statistical fluctuations in the data. We have also measured the thickness of the 10 μ m ZYC crystal by assuming a density of 2.2 g/cm³ and finding a best fit theoretical curve through the transmission data. The uniformity of the crystal thickness may be measured by



Figure 3. Rocking curve measurements of one of the flight spare OSO-8 graphite crystals made with the triple-axis spectrometer depicted in Figure 2. The measurements were made at the crystal locations indicated.





Figure 4. Rocking curve measurements of a 1.3 mm Union Carbide sample designated as "ZYB."

ZYB 1.3 mm



Figure 5. Rocking curve measurements of a Union Carbide sample designated as "ZYC." The sample was 50 mm x 50 mm x 0.8 mm in size.



Figure 6. Rocking curve measurements of the same Union Carbide sample "ZYC" as depicted in Figure 5, but thinned to $10 \ \mu m$.



Figure 7. Rocking curve measurements of a Union Carbide sample termed "ZYA" thinned to 50 μ m.



Figure 8. The x-ray transmission of the 50 μ m thick "ZYA" sample oriented at 45° to the incident x-ray beam. This measurement was made with a Si(Li) detector with an energy resolution of 180 eV at 6 keV. The solid line represents the theoretical transmission curve for 50 μ m of graphite with a density of 2.2 g/cm³.

comparing results obtained at several different locations on the crystal. In Figs. 9 (a) and (b) we plot the transmission spectrum at two different locations, while Fig. 9 (c) shows a plot of the ratio of the two spectra. The peaks in the spectrum represent the emission lines of rhodium $L\alpha$ (2.70 keV) and zinc K α (8.64 keV) and K β (9.57 keV). Note that a drift in the electronic gain of about one channel results in a large difference in the ratio at these energies due to the large slope of the peaks. However, at other energies the RMS spread in the ratio is approximately 2.5%, in good agreement with fluctuations expected from counting statistics.

3.2 Panasonic Supergraphite

A small sample of Supergraphite measuring 1.5 cm by 2 cm by 80-100 μ m has given very good results for reflectivity and uniformity. An overlay of rocking curves from three points on the crystal is depicted in Fig. 10. The measured integrated reflectivity of 9 x 10⁻⁴ rad is comparable to the highest values for the current material supplied by Union Carbide. The variation of measured integrated reflectivities for three points on the crystal is ±2%, approaching the uncertainty of the measurement. Moreover, the individual rocking curves line up very closely, indicating a smooth, uniform crystal surface. Measurement of the transmission of the Panasonic crystal at the single energy of 2.70 keV yields an estimated thickness of 99 μ m, while weighing the crystal gives an estimate of 83 μ m. Both of these estimates assume a density of 2.2 g/cm³.

We have recently manufactured a chromium anode (K α 5.41 keV) and used it to make preliminary measurements of the second order Bragg reflection of the Panasonic crystal. Figure 11 shows three rocking curves obtained at positions 5 mm apart. The mosaic spread is 0.5° and the integrated reflectivity is 4 x 10⁻⁴ rad for the second order reflection.

4. FUTURE WORK

We are nearing completion of a vacuum facility capable of containing the complete SXRP flight unit for final calibration. The facility includes a 1.5 m diameter, 1 m high vacuum chamber. The calibration procedure will require both polarized and unpolarized x-ray beams. The polarized beam will be used to measure modulation of the polarimeters, and the unpolarized beam will calibrate the spurious instrumental modulation. A double-crystal spectrometer, similar to that shown in Fig. 1, will be used to provide the low divergence polarized beam. For the unpolarized beam, we will use an 8 m beam line to collimate the output from an unpolarized x-ray tube.

The new facility will allow us to improve and complete measurements on the graphite crystals. More of the thick samples must be thinned to 50 μ m to provide large area thin crystals for testing. Full transmission curves, with better counting statistics for improved precision, are needed for both the Union Carbide and the Panasonic crystals. Rocking curves in second order have been measured only for the Panasonic Supergraphite, and we need to measure the OSO-8 crystals in second order for comparison. We will also perform a more detailed study of the uniformity of the crystals, both in reflectivity and in thickness. To improve the precision of the rocking curve measurements, we plan to add a second proportional counter to monitor the time variation of the x-ray source intensity.

As the OSO-8 flight spares have given the best results of any of the Union Carbide crystals tested, we are currently working with the company to see if they can recreate the high quality of these old crystals in the larger pieces we require. We are also negotiating with Panasonic to obtain several more samples of Supergraphite for testing.

5. SUMMARY

Significant progress has been made in evaluating mosaic graphite crystals for the SXRP Bragg crystal polarimeter. Measurements of new pyrolytic graphite samples from the Union Carbide Corporation and Panasonic Corporation have shown integrated reflectivities approaching the highest values previously measured.⁸ In addition, we have found the Panasonic Supergraphite crystal to be quite uniform and smooth. We have successfully thinned a large area graphite crystal to a thickness less than required yet found that it survived vibration tests. An improved



Figure 9. A and B are x-ray spectra, emitted by the rhodium anode, which were measured in transmission at two different locations on the 10 μ m "ZYC" crystal. The peaks represent the emission lines of rhodium La (2.70 keV) and zinc Ka (8.64 keV) and K β (9.57 keV). C is the ratio of the spectra in A and B.



Figure 10. Three rocking curves of a small sample of 80-100 µm thick Panasonic "Supergraphite."



Figure 11. Rocking curves of the Panasonic sample measured in second order, at 5.41 keV.

and larger polarimeter calibration facility is nearly on-line and should yield improved graphite crystal measurements.

6. ACKNOWLEDGEMENTS

The authors wish to thank D. Van Lue for greatly assisting in the design and fabrication of the spectrometer and calibration facility that was used to perform these measurements and for his work in the design and construction of the new facility. We also wish to thank Arthur Moore of Union Carbide Corporation and Morie Kusunose of Panasonic Corporation for supplying the graphite crystals measured. Finally, our thanks to Richard Spalding and Robert Woods of Sandia National Laboratory, in Albuquerque, New Mexico, for performing the vibration tests of the large thin crystal on such short notice.

7. REFERENCES

1. E. Silver, A. Simionivici, S. Labov, R. Novick, P. Kaaret, C. Martin, T. Hamilton, M. Weisskopf, R. Elsner, J. Beeman, G. Chanan, G. Manzo, E. Costa, G. Perola, and G. Fraser, "Bragg Crystal Polarimeters," SPIE Vol. 1160 X-Ray/EUV Optics for Astronomy and Microscopy, 598, 1989.

2. P. Kaaret, R. Novick, C. Martin, T. Hamilton, R. Sunyaev, I. Lapshov, E. Silver, M. Weisskopf, R. Elsner, G. Chanan, G. Manzo, E. Costa, G. Fraser, and G. C. Perola, "SXRP: A Focal Plane X-Ray Polarimeter For The SPECTRUM-X-Gamma Mission," SPIE Vol. 1160 X-Ray/EUV Optics for Astronomy and Microscopy, 587, 1989.

3. M.C. Weisskopf, R.F. Elsner, R. Novick, P. Kaaret, and E. Silver, "On The Design Of Scattering Polarimeters At The Focus Of An X-Ray Telescope," SPIE Vol. 1160 X-Ray/EUV Optics for Astronomy and Microscopy, 610, 1989.

4. G.W. Fraser, J.E. Lees, J.F. Pearson, M.R. Sims, J.E. Spragg, and R. Willingale, "The Photoemission Polarimeter in Soft X-Ray Astronomy," SPIE Vol. 1160 X-Ray/EUV Optics for Astronomy and Microscopy, 568, 1989.

5. F.E. Christensen, H.W. Schnopper, E.H. Silver, and N.J. Westergaard, in *Proceedings of the ESA Workshop on a Cosmic X-Ray Spectroscopy Mission*, 24-26 June 1985, Lyngby, Denmark.

6. E. Silver, J. Holley, K.P. Zióck, R. Novick, P. Kaaret, M. Weisskopf, R. Elsner, and J. Beeman, "Bragg Crystal Polarimeters," to be published in *Optical Engineering*.

7. G.E. Bacon and R.D. Lowde, Acta Cryst., 1, 303, 1948.

8. H.L. Kestenbaum, Appl. Spectrosc., 27, 454, 1973.

TABLE 1

Reflection Measurements in First Order at 2.7 keV

		Integrated	
Sample	Position	Reflectivity*	Mosaic Spread
		(rad)	(deg)
		· · · · · · · ·	
Union Carbide OSO-8	Α	9.8 x 10-4	0.57
(200 µm)**	B	9.2 x 10-4	0.57
·	C	1.04 x 10-3	0.54
Union Carbide "ZYB"	Α	5.3 x 10-4	0.30
(1.3 mm)	В	4.8 x 10-4	non-Gaussian
	c	4.9 x 10-4	0.39
Union Corbido "7VC"	A	7.9 10.4	0.20
	A	7.8 X 10-7	0.39
(0.8 mm)	В	8.1 x 10-4	0.42
	C C	8.1 x 10-4	0.39
	D	7.6 x 10-4	0.36
Union Carbide "ZYC"	Α	5.8 x 10-4	0.51
(10 μm)	B	5.3 x 10-4	non-Gaussian
	C	0.3 x 10-4	non-Gaussian
Union Carbide "ZYA"	A	88 x 10-4	0 44
(50 μm)	B	8.1 x 10-4	non-Gaussian
Panasonic			
Supergraphite	Α	9.3 x 10-4	0.55
(80-100 µm)	В	9.0 x 10-4	0.54
	C	8.9 x 10-4	0.55

Reflection Measurements in Second Order at 5.4 keV

Panasonic			
Supergraphite	Α	4.2 x 10-4	0.50
(80-100 µm)	В	3.8 x 10-4	0.50
	С	3.7 x 10-4	0.50

*The uncertainty of these measurements is $\approx \pm 1.5\%$.

** The crystal thickness.



