CONF-870610--3

6/16/87

THE ORNL BEAMLINE AT THE NATIONAL SYNCHROTRON LIGHT SOURCE*

Anton Habenschuss

University Programs Division
Oak Ridge Associated Universities
Oak Ridge, Tennessee 37831

CONF-870610--3

DE87 011676

Gene E. Ice and Cullie J. Sparks

Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Richard A. Neiser

Department of Materials Engineering
Virginia Polytechnic Institute and State University
Blacksburg, Virginia 24061

Submitted for Publication in

Nuclear Instruments and Methods
5th National Conference on
Synchrotron Radiation Instrumentation

University of Wisconsin-Madison
Madison, Wisconsin

June 21–25, 1987

*Research performed in part at the Oak Ridge National Laboratory Beamline X-14 at the National Synchrotron Light Source, Brookhaven National Laboratory, sponsored by the Division of Materials Sciences and Division of Chemical Sciences, U.S. Department of Energy, under contract DE-AC05-84OR21400 with the Martin Marietta Energy Systems, Inc.

THE ORNL BEAMLINE AT THE NATIONAL SYNCHROTRON LIGHT SOURCE*

ANTON HABENSCHUSS
University Programs Division, Oak Ridge Associated Universities, Oak Ridge, Tennessee 37831
GENE E. ICE AND CULLIE J. SPARKS†
Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37831
RICHARD A. NEISER
Department of Materials Engineering, Virginia Polytechnic Institute and State University, Blacksburg, Virginia 24061

ABSTRACT

The Oak Ridge National Laboratory's (ORNL) beamline at the National Synchrotron Light Source (NSLS) incorporates several novel features including X-ray optics based on sagittal focusing with crystals and a cantilevered mirror whose center becomes the pivot for all downstream optical elements. Crystal focusing accepts a much larger horizontal divergence of radiation than a mirror while maintaining excellent momentum transfer and energy resolution [1]. This sagittally bent crystal serves as the second element of a two-crystal, nondispersive monochromator. The cantilevered mirror provides a simple design for vertical focusing of the radiation. The beamline is suitable for both X-ray scattering and spectroscopy experiments requiring good energy resolution and high intensity in the energy range from 2.5 to 40 keV. This paper describes the optics of the ORNL beamline and reports their performance to date.

---

*Research performed in part at the Oak Ridge National Laboratory Beamline X-14 at the National Synchrotron Light Source, Brookhaven National Laboratory, sponsored by the Division of Materials Sciences and Division of Chemical Sciences, U.S. Department of Energy, under contract DE-AC05-84OR21400 with the Martin Marietta Energy Systems, Inc.

†Contact for proofs.

1. Introduction

The optical design for our beamline grew from the research needs of a broad users group interested in research on crystalline solid solutions, defect structures, thin films, surface layers and catalysts, liquids and amorphous solids phase transformations, and metallic phases. A flexible optical system was desired to meet the needs of such a diverse program in materials science. Our early criteria included maximizing the number of useful photons on the sample, a fixed exit beam, a simplified energy change to take advantage of anomalous dispersion variations in scattering cross sections for diffraction, small spot size at the sample, and good q-space and energy resolution. An upper energy limit of 40 keV was desirable to reach the K edges of those elements whose L_{III} edges at 3 to 5 keV are too low in energy to cover a sufficient momentum range to characterize the sample by diffraction.

Early calculations showed that a sagittally bent crystal would focus much more horizontal divergence than a specularly reflecting mirror [1]. Since the horizontal acceptance of sagittally focusing optics varies with the sine of the incident angle, a bent Si(111) crystal collects about 5 times more radiation at 10 keV and 30 times more at 40 keV than a platinum-coated focusing mirror. Ray tracing programs were used to study possible optical designs. These studies showed that a cylindrical curvature to the sagittally focusing crystal would have its maximum acceptance at a magnification of one-third. However, focusing aberrations could be reduced, and high through-put achieved with magnifications between 0.3 and 2 by using a conically bent crystal instead of a cylindrically bent crystal [2]. Of all the calculated designs, the conically bent focusing-monochromating optics yielded the highest flux while maintaining the good momentum transfer and energy resolution inherent with synchrotron radiation.

These advantages were considered sufficient to justify the reduction of rapid energy changing capability to achieve the complexity of a conical shape with the crystal bending mechanism. The successful test of a 3 mrad acceptance prototype bender at CHESS [1] provided the incentive to construct a crystal bender witn 15 mrad acceptance.

Since the sagittal crystal focused the horizontal divergence, the mirror need have only a simple cylindrical curvature to focus the vertical divergence and to provide harmonic energy reduction. Thus the mirror would be flat with a simple cantilevered arrangement for bending to a cylindrical curvature. All of the optics, including slits, would be mounted on a single beam pivoted about the center of the mirror surface. A change in the mirror reflectance angle would require only that the beam be rotated in the vertical plane by twice the mirror angle change without having to reposition any of the optical elements. The performance of this X-ray optical system is reported here.

2. Optical Design

2.1 Grazing Incidence Mirror

The overall ORNL optical design is shown in fig. 1. The two most important optical elements on the beamline are the glancing incidence mirror and the double crystal monochromator. A variable curvature, cantilevered mirror located 7.5 m from the bending magnet source point focuses the vertical beam divergence. As shown in fig. 2, the monochromator and all remaining beamline elements mount on a rail which pivots about the mirror center. This design allows rapid realignment of all the optical components with changes in the mirror's reflecting angle. The angle incident onto the mirror can be varied from 1 to 10 mrad, letting it act as a low-pass energy filter. The mirror's reflecting surface is a 20 nm (200Å) thick platinum coating on top of a polished electroless nickel-plated disk grade (5086) aluminum substrate. The mirror is 68 cm × 12 cm with a cube-root dependent thickness which allows it to be bent to a cylinder by cantilevered forces. Ray tracings show that a cylindrical curvature should focus the X-ray source's vertical divergence with negligible aberrations. The mirror does not modify the horizontal divergence. Under normal conditions, the mirror's radius of curvature is extremely large, being on the order of kilometers. The radius is given by

$$R_m = 2\, f_1 f_2/[(f_1 + f_2)\, \sin\alpha]$$

where α is the mirror's incident angle, f_1 is the source-to-mirror distance and f_2 is the mirror-to-image plane distance. The onset of plastic deformation in the aluminum limits the minimum radius of curvature to about 500 m. A magnification of 1.5 ($M = f_2/f_1$) focuses the beam at the sample. The mirror can act as a parabolic collimator (f_2 = infinity) removing the vertical divergence and optimizing the energy resolution of the dispersionless monochromator, or the focus can be brought from infinity to any point in front or behind the sample position for a variety of geometries.

The optical performance of the mirror can be degraded for a variety of reasons. Because of its large radius of curvature, the displacement of the mirror, due to its own weight, can represent a sizable fraction of the total displacement. Also, for this cantilevered system only the central 40 cm conform closely to a cylinder. To alleviate the sag problem, the mirror holder employs springs to lift the mirror at two places along its length while slits in front of the mirror can mask its poorly figured ends. These effects are discussed in more detail elsewhere [3]. If the mirror is improperly polished, both the figure error and surface roughness degrades both the reflectivity and the focused image. A root-mean-square (RMS) surface roughness less than 10Å below 1 mm spatial frequencies is calculated to give good performance.

Deterioration of the mirror surface is decreased if it is kept cool and deposit free. For this reason the mirror is kept in the ring UHV and is separated by a 0.015 in.-thick, water-cooled beryllium window from the beamline's rough vacuum (see fig. 1). The tank housing the monochromator is evacuated only to about $1 \times 10E^{-5}$ Torr because of the many motors and other non-UHV compatible components. As intense synchrotron radiation can damage a mirror's reflective surface, water cooling channels in the mirror were included to control thermal cycling. Typically, 30% of the incident beam's power (~180 W or 0.2 W/sq cm) is absorbed as heat by the mirror. At power loadings up to ~200 W, the water-cooled stainless steel flange to which the mirror is bolted has provided adequate cooling.

2.2 Sagittally Focusing Double Crystal Monochromator

The heart of the optical system is a two-crystal, fixed exit monochromator located 10 m from the source, fig. 3. The first crystal is a water-cooled Si(111) flat. The second crystal is a Si(111) crystal dynamically bent to a conical shape which focuses horizontally divergent rays but acts as a flat crystal to vertically divergent rays. A crystal intercepting 4.5 mrad of horizontal divergence has been used for the work described here, but the bender can accept a 15 mrad crystal in the energy range from 2.5 to 20 keV and 12 mrad at energies up to 40 keV with efficient through-put.

Like the variable curvature mirror, the crystal's bending radius can be changed to move the focal spot from well in front of the sample to well behind it, depending on the needs of the experiment. The source-to-focusing crystal distance (f_1) is 9.2 m while the crystal-to-sample distance (f_2) is 9.8 m. The useful magnification range of the monochromator is from one-third to two. Various experiments can benefit from having the focus at the detector or at the sample. Alternately, the horizontal width of the beam can be matched to the size of the sample if this is important.

The radius of curvature to which the crystal is bent is given by

$$R_S = 2f_1 f_2 \sin\theta/(f_1 + f_2)$$

where R_S is the sagittal radius and θ the incident angle. The sagittal radius is inversely proportional to photon energy for a fixed magnification. The minimum radius of curvature to which the crystal may be bent is 25 cm. This corresponds to about one-half the fracture stress of the 0.5 mm-thick curved silicon crystal.

The vertical ribs on the crystal seen in fig. 3 stiffen the crystal against anticlastic bending in the meridian plane (scattering plane), while minimizing its rigidity to bending in the sagittal plane (orthogonal to the scattering plane) [1,4,5]. The focusing crystal on the ORNL beamline is bent to a cone whose vertex lies near the synchrotron source point [2]. Thus, the radius R_S is slightly smaller at the end of the crystal closer to the source. The conically tapered crystal matches the Bragg condition of a flat crystal over a range of magnification values, as compared to its cylindrical counterpart which is constrained to operate at a magnification near 1/3 [6].

2.3 Other Components

There are other important components along the beamline in addition to the mirror and monochromator. The beryllium window reduces the thermal load on the first crystal by absorbing most of the low energy radiation present in the white beam and isolates the mirror tank vacuum from the monochromator vacuum. The two ion chambers shown in fig. 1 monitor the intensity and position of the beam. A third ion chamber used as an incident beam monitor is located just before the sample. A slit set immediately upstream of this third ion chamber defines the beam's dimensions at the sample. Several other sets of slits along the beamline's length each have independent horizontal and vertical apertures. Those in the white beam are water-cooled, UHV compatible slits which limit the width and height of the radiation swath admitted to the beamline. Slits in the monochromator beam reduce the intensity of stray radiation.

2.4 Harmonic Control

Harmonic control is an important consideration in many low-intensity scattering measurements. The ORNL beamline has several means by which we can reduce the harmonic content of the monochromated beam. One technique is to increase the mirror's incident angle beyond the harmonic's critical angle. Another effective approach is to slightly rotate the second crystal away from parallelism with the first. It is possible to detune the second crystal sufficiently to exceed the rocking width of the more narrow harmonic line, but yet stay within the rocking width of the desired line [7]. If more harmonic suppression is needed, a set of Ross balanced filters can be inserted into the beam, see fig. 1. Each pair of filters has an appropriate energy range. Finally, the electronics of the detection assembly can reduce harmonic contamination by discriminating photon energy.

3. Experimental Equipment

A variety of experimental equipment is available for conducting X-ray scattering and spectroscopy measurements. A four circle Huber diffractometer can place the sample at any orientation relative to the incident beam. This diffractometer sits on a motorized table having two translational and three rotational degrees of freedom. As the mirror reflection angle is changed, the table will track the vertical and angular motion of the beam. Helium-filled beam paths bring the incident beam to the sample and the scattered beam to the crystal analyzer and/or detector. A selection of analyzer crystals include Si(111), Ge(111), LiF(200), and graphite(002). Detectors include a Si(Li) solid state detector, a xenon-CO_2 linear position sensitive detector (~60 μm resolution), scintillation and proportional detectors, flow ion chambers, and PIN diodes. Various sample holders are available for single crystals, polycrystalline, and liquid and gaseous materials. A sample chamber with a hemispherical Be dome provides for specimen temperatures ranging from −190° to about 500°C. Software exists for many scattering experiments, including diffuse scattering measurements, powder and single crystal diffraction, and liquid and amorphous scattering.

4. Beamline Performance

Initial tests were made with a crystal focusing 4.5 mrad of horizontal divergence. Figure 4 shows the vertical and horizontal intensity profiles of the focus for a beam of 8 keV photons.

The values of the 4σ widths are 1.45 mm horizontally and 0.96 mm vertically. The theoretical limits based on the predicted electron source size are 1.1 mm and 0.61 mm, respectively, for perfect imaging. Studies of the mirror reflecting properties revealed topographic features a few millimeters apart that could account for the observed low angle scattering about the direct beam [9].

Measurements of the absolute photon flux at 8.38 keV and 14 keV were made with the storage ring operating at 2.4 GeV and 50 mA stored current. A flux of 3×10^{11} photons/sec was measured at an energy of 8.333 keV. When corrected for window absorptions and for mirror and crystal reflectance, this number compares well with the theoretical value of 6.6×10^{11} photons/sec for 100% transmission. Under similar conditions, a doubly focused beam of 14 keV X rays gave 5×10^{10} photons/sec and compared favorably with the predicted flux.

The energy spread in the monochromatic beam varies with the vertical opening angle of the radiation and can be controlled with the X-ray mirror. The mirror can operate in a focusing mode or a collimating mode. When focused at the sample, the beam is slightly convergent onto the monochromator's crystals, and a FWHM resolution of 1.3×10^{-3} was measured at 8.333 keV. This compares with a theoretical resolution of 1.1×10^{-3}. In the high resolution collimating mode, the energy resolution is 7.6×10^{-4} at the same photon energy. This value is a factor of three above the source size limited theoretical energy resolution of 2.7×10^{-4} for ideal collimation. Imperfections in the mirror surface account for the relatively poor collimation performance. Nevertheless, in the collimated mode, the energy resolution is improved by a factor of two, but the vertical size of the focal spot increases by a factor of three.

Studies of the reflectivity showed the mirror to be 67% efficient in reflection at a glancing angle of 3 mrad and a photon energy of 8.333 keV. At shallow incidence angles the focused image showed vertical tails several centimeters long; these features indicate a surface roughness of approximately 20Å. A visible light technique was used to estimate the figure error of the mirror [8]. These tests showed that the slope error of the mirror was less than 2.5 arc sec and that the two lifting springs to reduce the mirror sag improved the figure accuracy of the mirror.

Despite its complex design, the monochromator has not been difficult to align and focus. Computer routines for driving the eight motors which focus the beam and change the energy have performed satisfactorily, though fine tuning of the focus is required when energy changes exceeding 2 kV are made. A feedback system uses a lock-in amplifier, an ion chamber, and a dithered PZT stack to lock onto the monochromator output to keep the two crystals parallel. This circuitry ensures that the beam is not lost during an energy move and provides for stability intensities under changing thermal loads.

5. Conclusions

The components of the ORNL beamline at the NSLS essential to providing an intense and focused beam of monochromatic radiation for both X-ray scattering and spectroscopy were shown to be the sagittal focusing crystal and meridional focusing mirror. The measurements show that photon flux near to their theoretical values are obtained and that the beam is well focused in the horizontal by the crystal. Performance limitations are primarily attributed to mirror surface roughness of about 20Å RMS.

Acknowledgement

We express our appreciation to all those who struggled with us to assemble this facility.

References

[1] C. J. Sparks, G. E. Ice, J. Wong, B. W. Batterman, *Nucl. Instrum. Methods* 194, 73 (1982).

[2] G. E. Ice and C. J. Sparks, *J. Opt. Soc. Am.* to be published.

[3] G. E. Ice and C. J. Sparks, *Nucl. Instrum. Methods*, Synchrotron Radiation Instrumentation 5th National Conference, Madison, Wisconsin, June 21–25, 1987; and R. A. Neiser, Master's Thesis, Virginia Polytechnic Institute and State University, Blacksburg, Virginia, 1985.

[4] M. S. Troitsky, *Stiffened Plates, Bending, Stability, and Vibration* (Elsevier, New York, 1976).

[5] B. W. Batterman and L. Berman, *Nucl. Instrum. Methods* 208, 327 (1983).

[6] C. J. Sparks, B. Borie, J. Hastings, *Nucl. Instrum. Methods* 172, 237 (1980).

[7] B. E. Warren, p. 329 in *X-Ray Diffraction*, (Addison-Wesley, Reading, Mass., 1969).

[8] P. Z. Takacs and J. Colbert, *Nucl. Instrum. Methods*, 246, 223 (1986).

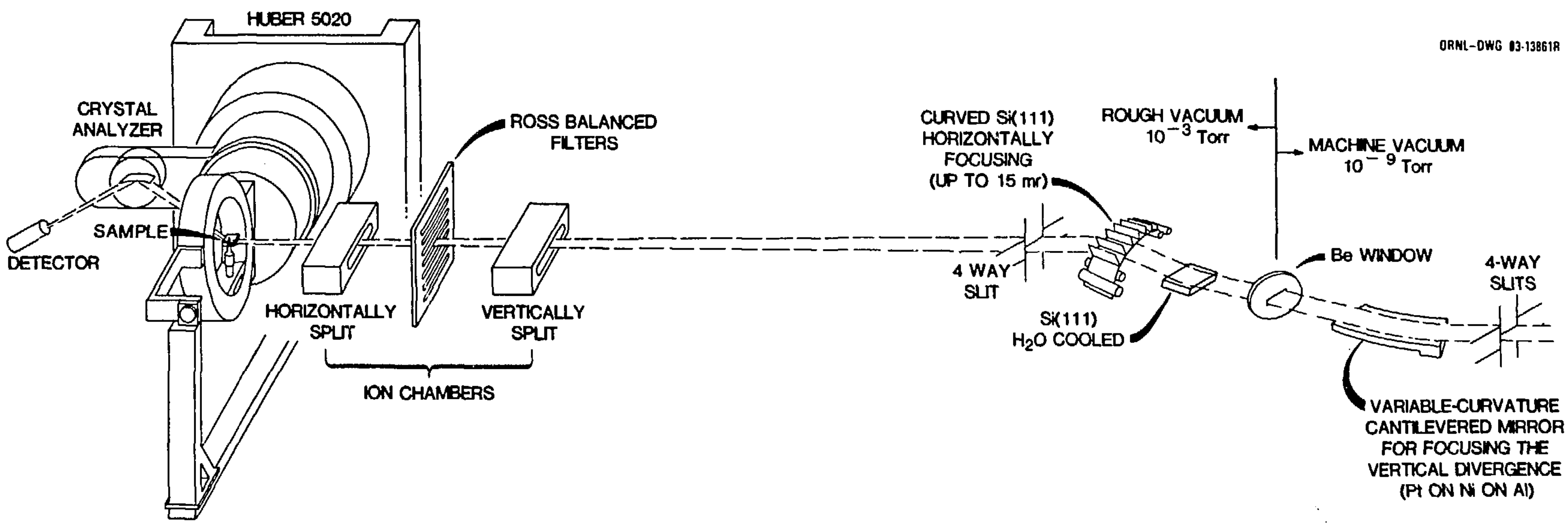
FIGURE CAPTIONS

Fig. 1 Principle optics of the Oak Ridge National Laboratory beamline at the NSLS. The optics provide independent control of the extent of horizontal and vertical focusing. The six circle Huber diffractometer is located inside a shielded hutch. The split ion chambers monitor the incident beam's intensity and position.

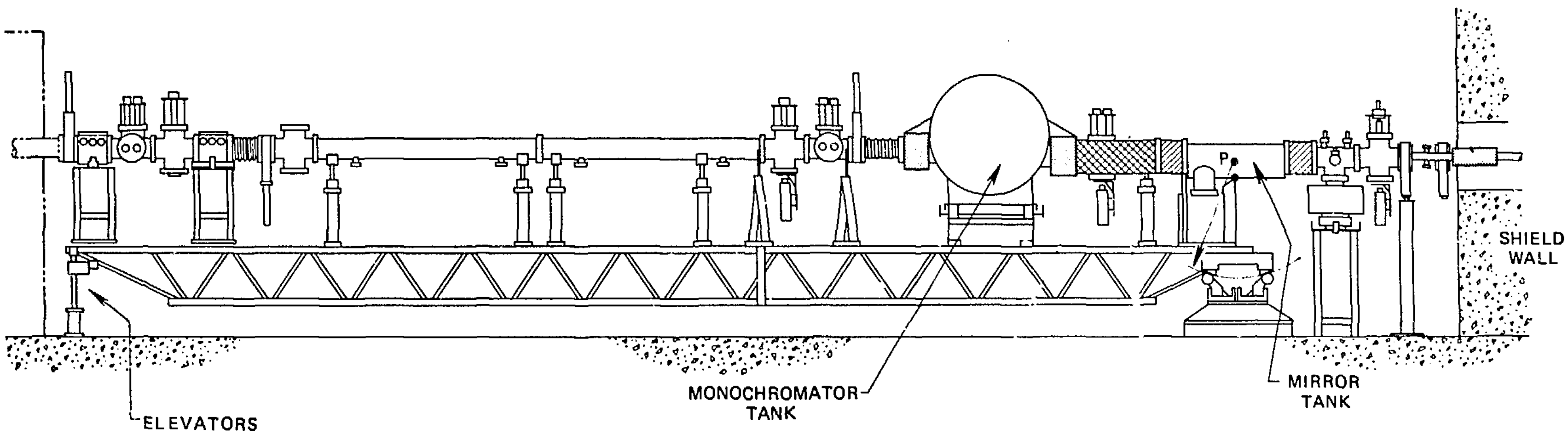
Fig. 2 Diagram of the ORNL beamline's pivoting mechanism. The beamline is rotated about the center of the mirror's surface at P. The elevators raise the end of the beamline, which rotates on the roller bearings beneath the mirror.

Fig. 3 The horizontally focusing two crystal monochromator. The second crystal can be dynamically bent into a conical shape to provide sagittal focusing of the radiation. The stiffening ribs are provided to prevent anticlastic bending.

Fig. 4 The intensity profile of the focused radiation at 8.33 keV. (a) Measured horizontal intensity distribution, (b) measured vertical intensity distribution.

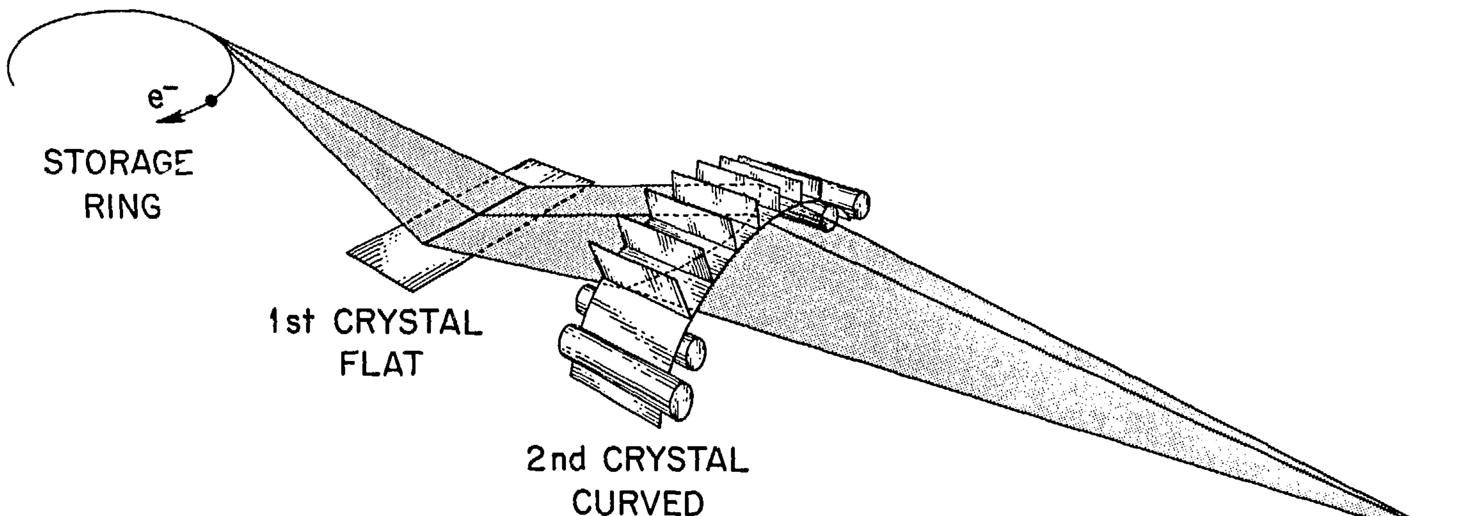


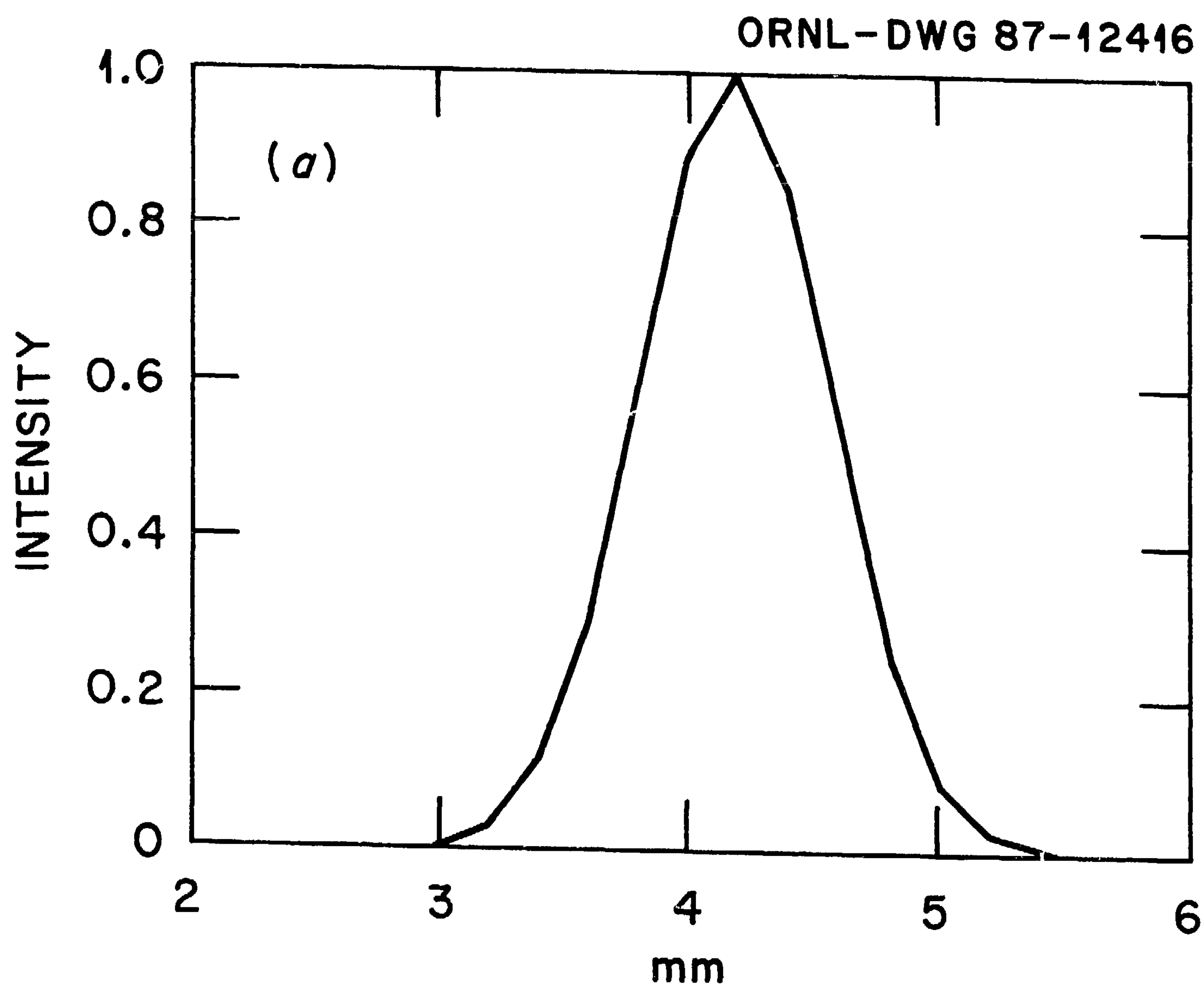
HUBER 5020
ORNL-DWG 83-13861R
CRYSTAL ANALYZER
ROSS BALANCED FILTERS
CURVED Si(111) HORIZONTALLY FOCUSING (UP TO 15 mr)
ROUGH VACUUM 10^{-3} Torr
MACHINE VACUUM 10^{-9} Torr
SAMPLE
DETECTOR
4 WAY SLIT
Be WINDOW
4-WAY SLITS
HORIZONTALLY SPLIT
VERTICALLY SPLIT
ION CHAMBERS
Si(111) H_2O COOLED
VARIABLE-CURVATURE CANTILEVERED MIRROR FOR FOCUSING THE VERTICAL DIVERGENCE (Pt ON Ni ON Al)

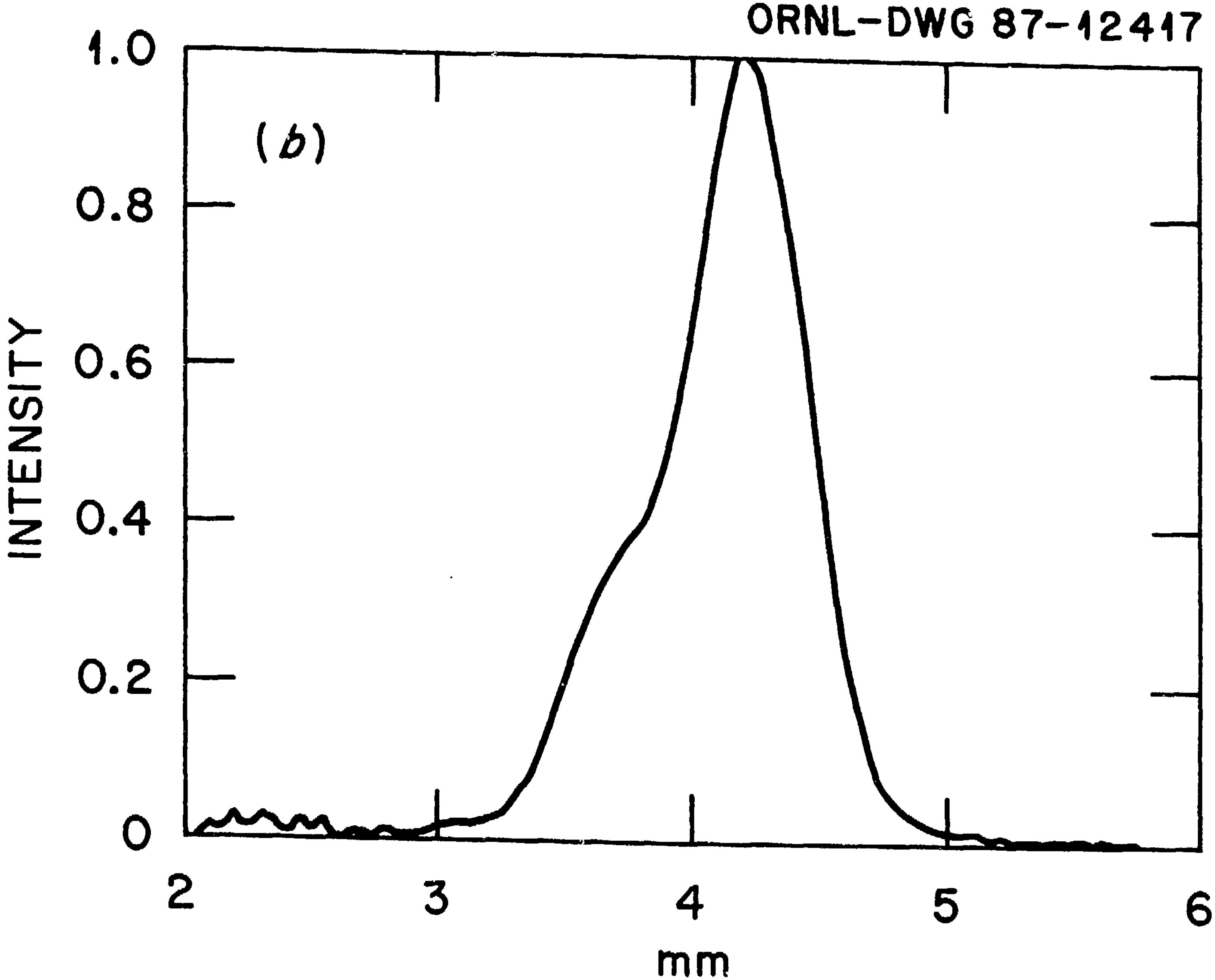

SHIELD WALL
P
MONOCHROMATOR TANK
MIRROR TANK
ELEVATORS

ORNL-DWG 81-13501

HORIZONTALLY FOCUSING
TWO CRYSTAL MONOCHROMATOR




ORNL-DWG 87-12416
(a)
INTENSITY
1.0
0.8
0.6
0.4
0.2
0
2
3
4
5
6
mm


ORNL–DWG 87–12417
(b)
INTENSITY
1.0
0.8
0.6
0.4
0.2
0
2
3
4
5
6
mm