A Focusing X-ray Camera for Recording Low-Angle Diffraction from Small Specimens

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Abstract
An X-ray camera is required in many low-angle diffraction experiments to focus the X-ray beam incident on the specimen. This paper describes an X-ray camera designed to record two-dimensional diffraction data from small specimens with unit cells as large as 50 nm. It matches the 200 μm focal spot of a rotating-anode X-ray generator to an electro-optical detector with a spatial resolution of 300 μm. The camera is very flexible and can accommodate a total source-to-detector distance of 2.5 m. The X-ray beam is focused by two 20 cm mirrors bent by simple two-point benders. The theory of such benders and the design of X-ray cameras incorporating them is discussed briefly. The paper also describes a fast (1 ms) X-ray shutter and simple ion chambers useful for alignment and operation.

Introduction
An essential element of a low-angle X-ray diffraction experiment is the apparatus used to focus and define the X-ray beam incident on the specimen. Since the optimum design of this 'camera' depends on the X-ray source, X-ray detector and specimen, many different cameras have been constructed (Hendrix, Koch & Bordas, 1979; Graham, 1978; Guinier, 1952; Hendricks, 1978; Franks, 1958; Schelten & Hendricks, 1978). I describe in this paper an X-ray camera optimized for the recording of low-angle diffraction using an area X-ray detector with relatively coarse spatial resolution (300 μm) and a rotating-anode X-ray generator with a 200 μm focal spot. It is especially suited for examining the two-dimensional diffraction from small specimens with unit cells as large as 50 nm.

The small specimen size dictates the use of a double-mirror focusing system; the exit beam of a monochromator would be much larger than the specimen and so would be poorly utilized. The detector resolution dictates a long specimen-to-detector distance and therefore, from geometrical considerations, a long source-to-specimen distance. One of the disadvantages of a long beam line is that the solid angle collected by the mirrors is decreased as they are moved away from the source. This loss is compensated for by making the mirrors very long (20 cm each). Since conventional benders would be inconvenient for such large mirrors, these mirrors are triangular plates bent into segments of circles by simple benders. To avoid another potential problem, the loss of intensity due to air absorption, the beam line is filled with helium gas at atmospheric pressure.

Alignment and focusing of the camera is simplified by two ion chambers installed permanently in the beam line and by the use of an X-ray sensitive three-stage image intensifier.

Theory
The geometry and notation used to describe mirror performance is shown in Fig. 1. If the mirror were perfect, that is, if every X-ray incident on the mirror were reflected into the image, the size and intensity of the image would be determined by geometrical optics. The size would be given by

$$y_d = y_s(x_d - x_1 - l/2)/(x_1 - l/2),$$

where $y_s$ is the size of the X-ray source, and the intensity would be proportional to the angle subtended by the mirror from the source:

$$A = \theta_i l/x_1.$$

No mirror shape can reach this ideal. The source is extended rather than a point, and X-rays will reflect from the mirror only if the angle of incidence is less than a critical value, $\theta_c$. For 0.15 nm X-rays reflecting from gold, $\theta_c$ is 8 mrad. An ellipse focuses a point source to a point image, but the angle of incidence varies along the mirror and, if the mirror is set so that

Fig. 1. Geometry of a focusing X-ray camera. The mirror is centered at a distance $x_1$ from the source. A source of size $y_s$ is focused onto a region of size $y_d$. 

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the maximum angle is \(\theta_n\), the average angle is somewhat less than \(\theta_n\), decreasing the intensity in the image. A logarithmic spiral \([r = a \exp(b0)]\) has a constant angle of incidence, but it focuses a point source to an extended image. A circle neither focuses to a point nor has constant angle, but it is the easiest shape to form. These simple facts raise three questions:

1. For an extended source, is an ellipse or log spiral the preferred shape?
2. How badly does a circular arc perform compared to the ideal?
3. How long could one make a mirror and still get a good focus from an extended source?

These questions are not entirely academic. A concave mirror of any shape can be formed by choosing a glass plate with the proper profile and bending the plate with the simple two-point bender described below.

To answer these questions I used a computer to model various mirror shapes and to trace the paths of X-rays through the system. Mirror shapes were generated in two ways: from the analytical equations for ellipses, log spirals and circles; and from the equations describing the shape of a plate of arbitrary cross section loaded at a point as described below. Numerical results are shown in Fig. 2. In summary:

1. An ellipse is much better than a log spiral.
2. A circle is an excellent approximation to an ellipse for short to medium mirror lengths.
3. An elliptical mirror can be made extremely long and still focus an extended source nearly perfectly.

Simply bent mirrors

The conventional Franks' bender applies a force couple to each end of a rectangular glass plate, bending it into a circular arc (Franks, 1958). A more general, and simpler to implement, approach is shown in Fig. 3. [This technique is now used most often for bending monochromators; see, for example, Hendrix, Koch & Bordas (1979).] A glass plate is held rigidly at one end and loaded by a force \(F\) at the other. The plate can have an arbitrary cross-sectional shape (a triangle is shown in Fig. 3) and need not have a constant thickness. The shape of the plate under loading is described by the deflection \(y(x)\) as a function of position along the beam (Huddleston, 1961). This deflection is determined by the curvature

\[
\frac{d^2 y}{dx^2} = \frac{M(x)}{EI(x)},
\]

where \(E\) is the Young's modulus of the material \((\sim 10^9 \text{ g/cm}^2\)), \(l(x)\) is the moment of the beam \((wt^3/12\) for a homogeneous beam of thickness \(t\) and width \(w\)), and \(M(x)\) is the bending moment in the plate which, for the case shown in Fig. 3, is given by

\[
M(x) = F(L - x).
\]

For a triangular plate with the force applied at the apex,

\[
l(x) = \frac{t^3 w_0}{12L} (L - x).
\]

For such a plate, substitution of (4) and (5) into (3) gives a constant curvature, so the surface follows a circle. It is quite clear, however, that any smooth surface can be produced by suitably varying the width or thickness of the beam and thus \(l(x)\). (The preceding discussion is strictly valid only for small curvatures.)

The ray-tracing program was used to investigate the effects of imperfections in shape, sagging due to gravity, and loading the beam a small distance from the apex. The effects on the focal spot were small.

Construction

The X-ray camera was designed from readily available inexpensive components. Only the simpler parts of the system were built in Princeton University shops; all precision components were purchased. Fig. 4 shows
an overview of the camera and identifies the individual components.

The X-ray source (A) is a Rigaku RU200 rotating-anode generator capable of producing a 0.2 × 0.2 mm focal spot with a loading of 3000 W. The camera components were assembled on a hollow aluminum block (C) manufactured by Ardel Kinamatic Corp. These blocks bolt together to form a very stable table and have pre-tapped holes on 2" (5.08 cm) centers covering the upper surface. They are very convenient for rapid setup and easy modification of the camera configuration. The table is supported by 3" (7.62 cm) diameter (D) and 5" (12.70 cm) diameter (E) thick-walled aluminum tubes bolted to the floor.

The major components of the camera are the mirrors (J and L), the fast shutter (Q), the slit assembly (R), and the ion chambers (S and U). Each of these will be described in detail below. Careful attention was paid to the remainder of the camera to ensure that it was easy to use and provided proper radiation protection. The major components are connected by 1" (2.54 cm) diameter, 0.06" (0.15 cm) wall brass tubing, cut to various lengths and jointed by pieces of bicycle inner tube (H). All joints from the X-ray port (B) to the first mirror (J) must either overlap or be wrapped with lead foil (H) to absorb hard X-rays. The tubes are supported on simple telescoping stands (N). The mirrors are supported by optical positioners from Ardel Kinamatic Corp. The support (K) for the vertical focusing mirror (J) allows adjustment of height and rotation about a horizontal axis (Ardel TRx105). The support (M) for the horizontal focusing mirror (L) allows motion across the table and rotation about a vertical axis (Ardel Ry and TT-100). The specimen is mounted on a combination (T) of Ardel components (RT-31, T-105, TT-105), which can be assembled in many different ways to allow various degrees of freedom. The slit assembly is mounted on a heavy aluminum fixed arm.

The front portion of the beam line is filled with helium gas through tube (P). Valve (G) is opened to purge the line for a short period during setup; thereafter it is closed and a flow into the tube (P) of 0.5 SCFH keeps the system free of air. The assembly marked (F) is a short, vented tube with thin Mylar windows at each end. The Be window of the X-ray machine is quite permeable to helium and this arrangement avoids contamination of the X-ray vacuum system:

The region beyond the specimen is evacuated to remove the background due to air-scattered X-rays. A large thick-walled enclosure is required and it would be very heavy if fabricated from aluminum, so 4" (10.16 cm) diameter, 0.25" (0.64 cm) wall PVC pipe (V) (a standard plumbing item) was used. Standard PVC end caps machined to form a vacuum seal to the pipe hold Mylar windows to complete a vacuum enclosure. This assembly is light, modular and easily modified. A small piece of self-adhesive lead tape (William Farnell Co., Philadelphia, PA) stuck to the inner surface of one Mylar window (on W) serves as a beam stop. The endcap (W) is supported by an x-y translator (X) manufactured by VelMex, Inc. (Unislide 4000 series).

The X-ray detector (Y) is the Princeton SIT X-ray detection system (Milch, Gruner & Reynolds, 1982; Gruner, Milch & Reynolds, 1982). This is an integrating area detector with high detection efficiency. It sits on a rolling table (Z) which was designed to slide over the bench assembly (C–D–E). That is, if the beam line is shorter than the bench, the detector will roll up the bench as far as required. The table can be lifted from its wheels by jackscrews for stability.

Mirrors

A key component of the camera is the X-ray mirror. An ideal X-ray mirror has a surface which is both flat (globally) to give good focus and smooth (on a scale of 1 nm) to reduce diffuse scatter. Excellent mirrors are available from several commercial vendors at very high prices. The mirrors for this camera are made from industrial-grade float glass from the standard stock of a local glass supplier. The original intention was to test the design concept using these inexpensive mirrors.

Fig. 4. A schematic of the X-ray camera. See text for explanation of individual components. All units can be positioned freely on the table surface and later can be clamped securely.

Fig. 5. Two views of the mirror box and mirror bender. Individual parts are identified in the text.
and then to purchase quality mirrors. However, the float glass mirrors worked so well that they are still in use.

The mirrors were cut by hand from a selected piece of 0.25" (0.64 cm) thick window glass. With practice fairly smooth triangular pieces can be cut with a base of 5 cm and a length of 28 cm. These pieces were coated with 10 nm of gold by a local optical jobber. The glass is in fact quite smooth but not all pieces are flat. One mirror (of three tried) was found in practice to give a poor focus and was discarded.

The mirror is mounted in the mirror box shown in Fig. 5. The base of triangle (B) is glued with Eastman 910 adhesive to a polished steel block (C). A polished steel tongue (D) is glued to the apex. A differential screw (F, Klinger Scientific No. 385034) pushes on the tongue to bend the mirror; a beryllium-copper spring (E) presses back against the tongue to ensure positive control. Tantalum slits (G) are mounted on non-rotating micrometer heads (H, Starrett No. 261L) to define the active length of the mirror and to block that portion of the incident beam which would not reflect from the mirrors. The entire assembly is mounted in a 2 by 4" (5.08 by 10.16 cm) extruded aluminum box beam. This method of mounting the mirror and slits utilizes standard components and has no critical machining requirements.

The fast shutter

The X-ray camera is often used for experiments in which the diffraction is recorded only during certain time intervals. The Rigaku X-ray generator has an electrically operated shutter, suitable for defining exposure times of more than a few seconds, but for shorter times a fast electrically operated shutter is needed. A stepper motor (Warner SM-0360030AB) is used in an unusual way to accomplish this (Collett, 1982). This motor has a step size of 10°. The motor is driven between two stable states (i.e. one step back and forth) to move a tantalum flag at the end of a 100 mm long arm in and out of the beam path. To define a short (e.g. 50 ms) exposure time precisely, the motor must complete each step rapidly, so the motor windings are driven by a two-level driver. Between steps, the rotor is held in position by a small winding current (0.5 A), but to execute a step, a 1000 μF capacitor charged to 40 V is discharged through the winding. This pulse drives the winding current to 6 A within 0.5 ms and the shutter motion is complete 1 ms after it is commanded. Two carefully positioned rubber stops limit the motion of the flag to 8 mm and damp the motion of the arm after each step.

Slits

Guard slits are needed just in front of the specimen to eliminate scatter around the direct beam. The guard-slit assembly was fabricated from four non-rotating micrometer heads and four tantalum strips. In this critical application, the small residual rotation of these ‘non-rotating’ micrometers proved bothersome, so the slit holders were made to slide along teflon pads. The slit positions could easily be set to an accuracy of 15 μm and were very stable.

Ion chambers

In the course of setting up the camera, I built a small ion chamber to monitor the X-ray flux in the direct beam. It proved so useful that two ion chambers (S and U) were built permanently into the line. Many uses have been found for them, such as verifying that helium is in the line, peaking of mirror angles and centering specimens in the direct beam.

Fig. 6 shows the chamber in detail. Ions are produced in the air within the ring and are collected by the gold-plated wire loop. The current is amplified by an integrated circuit electrometer built into the cham-
ber mount. This local amplifier eliminates the instability common to most ion chambers. The output voltage is linear with X-ray flux and 1 mV corresponds to roughly $4 \times 10^6$ X-rays s$^{-1}$.

The chambers are periodically calibrated by measuring the direct-beam intensity with a plastic scintillator and photomultiplier tube. Our laboratory has used the latter device, in a current-measuring (rather than X-ray-counting) mode, as a secondary standard for many years. It has been compared several times (at low count rates) to a NaI scintillator counter and has proved to be stable and reliable.

Originally the ion chambers had thin Mylar windows to define more precisely the active volume, but these devices proved to be excellent microphones and therefore the Mylar was removed. A collection voltage of several hundred volts was essential for stable response. The sensitivity of the chamber varies by about 30% over its active area and the output is not stable enough to provide a precise measure of X-ray beam intensity, but the chambers have nevertheless been invaluable in operation of the X-ray camera.

**Alignment**

The most difficult aspect of beam-line operation is the initial alignment. Once the beam is coming through the entire system it can be peaked and adjusted easily. An X-ray image intensifier is used to simplify the initial alignment. A three-stage electrostatic image intensifier with fiber-optics input (similar to Varo Model 1122) is made sensitive to X-rays by bonding a 75 µm thick layer of ZnS(Ag) phosphor to the input window. Room light is excluded by a layer of black photographic paper. The output of the intensifier produces a bright, sharp image of the incident X-ray beam.

The design of the camera also simplifies alignment. All components are demountable. Filling the beam line with helium, rather than evacuating it, avoids two problems: all adjustments are accessible, and critical components are not disturbed by motions caused by evacuation of the system.

Alignment of a new camera layout begins by clearing the table. Starting at the X-ray port, each component is placed on the table and adjusted to direct the beam down the center of the table, parallel to it. At this stage, the mirrors are flat and their slits are wide open. The image intensifier is used to locate the beam and set the angle of incidence appropriately. Next the mirrors are bent to focus the doubly reflected beam at the correct distance. The image intensifier is used to monitor the shape of the beam and the ion chamber to monitor the intensity. Finally the slits are moved in to cut wings off the beam without compromising the intensity too badly. Since the length of the beam line exceeds the length of the human arm, a mirror (behind the intensifier) and a small telescope are often required.

**Performance**

The X-ray camera has been used recently for low-angle diffraction studies of muscle, which has a 40 nm unit cell (Huxley & Brown, 1967). The layout for this experiment is shown in Fig. 7. The camera is adjusted to focus the incident beam at the detector. With an X-ray source size of 0.2 × 0.2 mm (0.2 × 2.0 mm fore-shortened) and a loading of 3 kW, the focus is typically 0.4 × 1.0 mm and has a flux of $8 \times 10^7$ X-rays s$^{-1}$. Equation (1) predicts a focus 0.3 × 0.7 mm. A nickel filter is not used in the system; careful mirror settings nearly eliminate the Cu $K\beta$ component. The size of the beam at the specimen is 0.3 × 1 mm.

The beam size stated above does not completely describe the beam quality for low-angle diffraction. More important than this nominal size is the actual profile of the beam, including the very weak wings. This profile was measured by scanning a 35 µm diameter pinhole across the focal plane, and measuring the transmitted intensity with a NaI scintillator counter. The results are shown in Fig. 8. At a full width of 0.4 mm (as above) the intensity falls to 0.25 of its peak, but a full width of 2 mm is reached before the intensity falls to $10^{-4}$ of peak, and 2.6 mm for $10^{-6}$ of the beam intensity.
peak intensity. This rapid falloff is absolutely necessary to allow observation of weak diffraction maxima at low angles. Note that the specimen–detector distance is 600 mm, so that with 0.15 nm radiation a full width of 2.6 mm corresponds to a 70 nm diffraction order.

A substantial portion of this weak wing on the beam is contributed by wings on the X-ray generator focal spot itself; the remainder is due to roughness of the mirrors.

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References


