

A Real-Time Back-Reflection Laue Camera

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(Received 23 February 1978; accepted 14 September 1978)

Abstract

Real-time back-reflection Laue patterns have been made possible through the construction of a specially designed multiwire proportional chamber. This ability to view Laue patterns in live time on an x - y display scope has greatly reduced the magnitude of effort involved in orienting single crystals and of characterizing the nature of less than perfect crystals.

1. Introduction

Various systems have been developed to display and record X-ray diffraction patterns much faster than possible with X-ray film. Review articles by Green (1971, 1977) and Hendricks (1976) appraise the capabilities of detectors based either upon image intensifiers coupled to TV systems or upon gas-filled multiwire proportional chambers. Most of these two-dimensional detectors allow for a rapid crystal alignment. For example, Cork, Fehr, Hamlin, Vernon, Xuong & Perez-Mendez (1974) and Cork, Hamlin, Vernon, Xuong & Perez-Mendez (1975) have rapidly recorded diffraction patterns from protein crystals with a multiwire proportional chamber. Using image intensifiers based on TV systems, Arndt, Gilmore & Boutle (1972) and Minor, Milch & Reynolds (1974) have quickly recorded diffraction patterns from biological structures. Custers & van der Wagt (1960) were the first to utilize transmission Laue patterns made directly viewable with an image intensifier to orient diamond single crystals rapidly. Goetze & Taylor (1962), Reifsnider & Green (1968), Green (1971), Borkowski & Kopp (1970), Picker Corporation (1970) and others have also reported viewing rapid transmission Laue patterns suitable for quick crystal alignment.

None of these systems, however, is particularly suited for the routine orientation of large single crystals. The transmission Laue arrangement is useful only for thin crystals. The back-reflection Laue arrangement, on the other hand, is suitable for orienting large thick crystals as well as thin ones. A partially successful attempt was made to directly image back-reflection patterns by Carlson, Furnas & Beard (1963) and Euler (1966). With a miniature 30 mm diameter X-ray tube mounted between a 220 mm diameter image intensifier and a specimen, a few back-reflection Laue spots were directly viewable in a darkened room if favorable

crystals were used. Photographic exposures of several minutes were necessary, however, to reveal the full Laue pattern. The shadow of the X-ray tube was visible on the viewing screen, reducing the number of Laue spots that could be observed.

2. Back-reflection Laue camera design and operation

A system has been devised that can display a back-reflection Laue pattern in real time on an x - y display scope. If desired Laue patterns may also be integrated over time on the face of the display scope for several seconds if it proves desirable to improve the picture quality. This can be contrasted with the 10 to 15 min exposures typically required for most materials with a standard Polaroid XR-7 cassette. Real-time Laue patterns are particularly advantageous whenever a series of Laue photographs is required, such as for orienting non-cubic crystals.

The essential idea in making a real-time Laue camera is to replace the sheet of film in a normal back-reflection camera with a detector that can display the results immediately, that is more efficient than film in detecting X-rays, and that can allow an intense primary beam of X-rays to pass through its center. A position-sensitive multiwire proportional chamber was built to satisfy these requirements. The development of multiwire proportional chambers by Borkowski & Kopp (1970), Charpak (1970), and others has made possible the construction of a multiwire Laue camera. The basic chamber design was heavily guided by the works of Kaplan, Kaufman, Perez-Mendez & Valentine (1973), Hamlin (1975), Perez-Mendez (1976), and Borkowski & Kopp (1975). Schematic drawings of the chamber are shown in Fig. 1. Three parallel wire planes, each 280×280 mm, are contained in a gas mixture of 90% argon and 10% CO_2 . The center anode plane consists of a parallel grid of $20\mu\text{m}$ gold plated tungsten wires on a 2 mm spacing all electrically tied together and separated by 4 mm from the two outer wire planes. This grid functions in a similar manner to the center wire of an ordinary single-wire proportional counter. The two outer wire planes consisting of $50\mu\text{m}$ gold-plated tungsten wire on a 1 mm spacing, are capacitively coupled to delay lines and are orthogonal to each other. Delay-line construction follows that of Grove, Perez-Mendez & Sperinde (1973). The three wire planes are created by soldering pretensioned fine

wires to precisely spaced copper lands on either end of three large glass epoxy printed-circuit boards (420 × 480 mm). These large boards greatly simplify the chamber construction. The G-10 glass epoxy* frames as well as the 25 μm thick Kapton† windows are assembled together with epoxy glue (Hysol resin R9-2039, hardener H2-2561‡).

The multiwire chamber operates in the following manner. An incoming X-ray ionizes several hundred gas atoms. The free electrons thus created drift to and finally avalanche upon a wire of the center anode plane. As the pulse forms on the center plane, signals are induced on the neighboring wires of the two outer wire planes. These signals are coupled into x and y delay lines which form the basis of the coordinate read-out. The START signal (see Fig. 1) from the center plane is used to start two time-to-amplitude converters (TACs). Signals arriving from the x and y delay lines at a later time (STOP X, STOP Y) are used to stop the TACs. The difference in time (0 to 2 μs) be-

* Great Lakes Plastic Co., Buffalo, New York.

† E. I. DuPont de Nemours & Co., Wilmington, Delaware.

‡ Hysol Division of the Dexter Corp., Olean, New York.

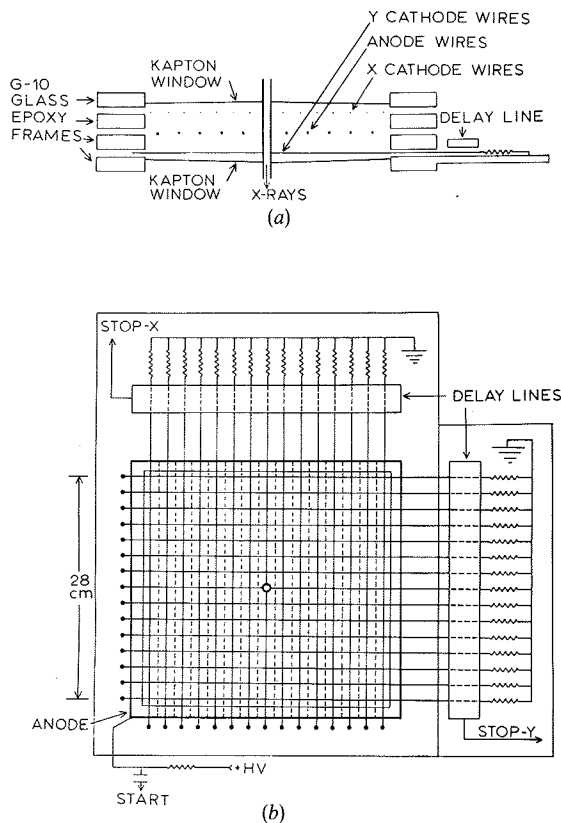


Fig. 1. Schematic diagram of multiwire proportional chamber. (a) Top view showing collimator passing through the detector; (b) front view showing the wire planes and delay lines. The anode wires are all connected together on a common buss.

tween a start and stop pulse gives a coordinate (0 to 280 mm), one in x and the other in y . The resultant x and y voltages are applied to the deflection plates of a Tektronix 607 storage display scope. Subsequently, a pulser briefly intensifies the writing beam of the scope and plots a point. The resultant pattern of dots is viewed as a Laue image. Similar analog display methods have been demonstrated by Borkowski & Kopp (1970), Kaufman, Perez-Mendez, Sperinde & Stoker (1971), Borkowski & Kopp (1972), and Swinth & Crowe (1972).

A pile-up rejector circuit is included to eliminate multiple events in the chamber that occur within the delay interval of 2 μs and the 2 μs dead time of the display scope interface. The total system dead time is 4 μs . The 4 mm separation between the wire planes results in a 14% quantum detection efficiency for 8 kV X-rays with the argon gas mixture flowing at atmospheric pressure through the chamber. For X-rays entering perpendicular to the wire planes, the observed spatial resolution is 1 mm × 2 mm.

One special feature of this detector is the way in which the $\frac{1}{4}$ " (6.3 mm) collimator tube is passed through the center of each of the wire planes. A 0.8 mm thick printed-circuit board (Fig. 2) of G-10 glass epoxy detours the center wires of each frame around the collimator. The curved conductors on the anode plane patch are coated with epoxy glue so that electrons will not avalanche onto the patch. The detoured wires remain active except for the dead area on the printed-circuit patch. The brass collimator is kept at ground potential.

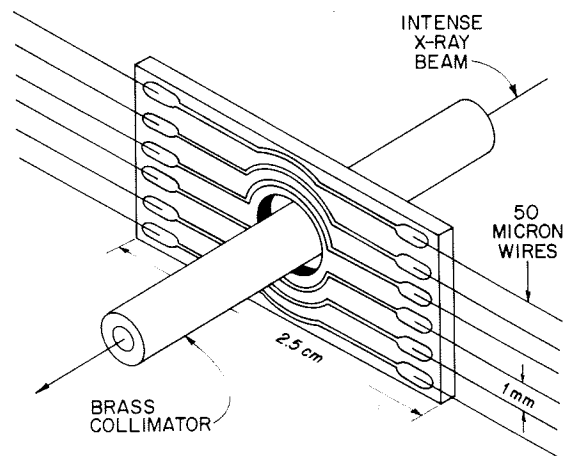


Fig. 2. Wires are detoured around a brass collimator by soldering them to a light weight G-10 glass epoxy printed-circuit board 25 × 12 × 0.8 mm. This allows the primary beam of X-rays to pass safely through the center of the detector. Three of these patches are necessary, one for each of the three wire planes.

3. Performance and applications

Several Laue patterns photographed from the display scope are presented to illustrate the capabilities of the detector. Fig. 3(a) is a one second exposure of the storage scope taken with a Polaroid camera of a Laue pattern from a (110) silicon crystal. This picture most nearly approximates what the eye sees if the display scope is viewed in real time. This is the usual operating mode for rapidly adjusting a crystal to a particular orientation. If better picture quality is required, then the image can be integrated directly on the screen of the display scope. The improvement due to signal averaging is clearly seen in Fig. 3(b) (8 s averaging) and Fig. 3(c) (30 s averaging). Fig. 3(c) also shows the outline of the grid of wires on a 2 mm spacing from the anode plane. The four black dots surrounding the center of the photograph are the shadows of four lead dots taped to the detector to mark the center of the dead area. The elongation of the Laue spots is due to the finite

detector thickness of 8 mm. The radial streaking is most evident for areas around the perimeter of the photograph. Fig. 3(c) is illustrative of the image quality that the system can achieve. The diffraction spots can be directly compared to those of a standard Laue photograph (see Fig. 3d). Of course, for crystal orientation purposes, the real-time display of Fig. 3(a) is the most useful one. With moderate detector-to-specimen distances of 100 to 200 mm, orientations accurate to $\frac{1}{2}$ or $\frac{1}{4}^\circ$ can be realized very quickly.

Several other photographs were taken to demonstrate the capability of the detector. Because of the detector's upper count-rate limit (50000 events/s to the display scope), crystals that produce large amounts of fluorescence in the 5 to 15 kV region become troublesome. Fig. 4(a) shows an 8 s Laue pattern of Zr-20% Nb (111) taken at 15 kV to avoid fluorescence from Zr and Nb and at 1 mA to demonstrate the sensitivity of the detector. Fig. 4(b) is of a Ni (111) crystal at 9 kV. Although the detector has 30% energy resolution as measured with an ^{55}Fe source at 5.9 kV over the whole area of the detector, it has been generally found more useful to reduce the excitation voltage of the X-ray

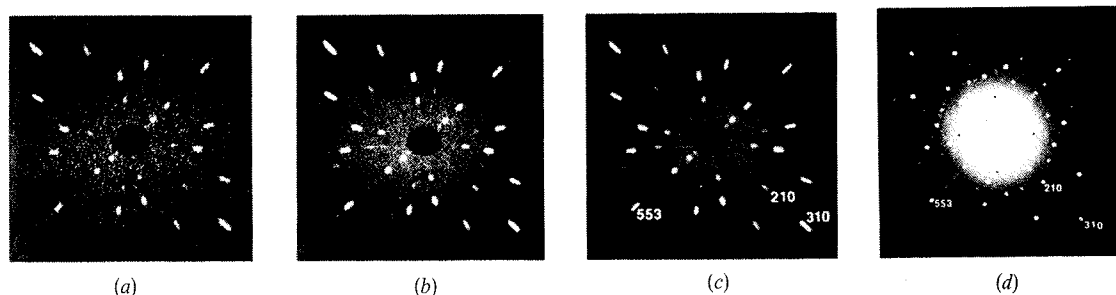


Fig. 3. Back-reflection Laue patterns of (110) Si. (a) Real-time picture, the one second exposure most nearly approximates what the eye sees in live time; (b) 8 s exposure; (c) 30 s exposure, several diffraction spots are labeled with their appropriate Miller indices. Tungsten target at 35 kV, 15 mA, 100mm detector-to-specimen distance, 37 000 events/s to display scope. (d) Standard 30 mm back-reflection Laue photograph using a Polaroid XR-7 cassette, tungsten target at 35 kV, 15mA 10 min exposure. The diffraction spots labeled with their appropriate Miller indices can be compared directly with those in (c).

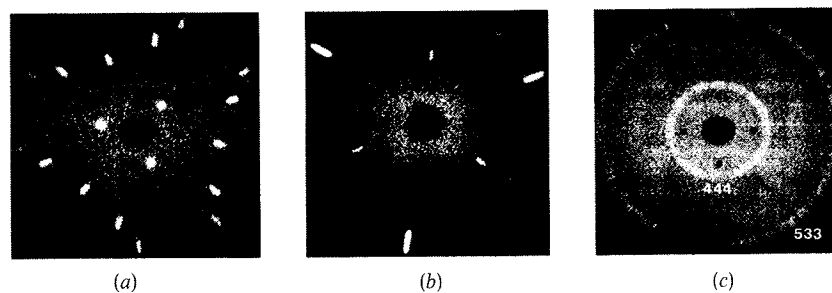


Fig. 4. (a) Zr-20% Nb (111), 15 kV, 1 mA, 4300 events/s to display scope, 100 mm specimen to detector, 8 s exposure, (b) Ni (111), 9 kV, 15 mA 11000 events/s to display scope, 70 mm specimen to detector, 8 s exposure, (c) Debye-Scherrer 533 and 444 rings. The partial arcs are 620. Si powder, 130 mm from detector, filtered Cu radiation, 35 kV, 15 mA, 30 s, 33 000 events/s to the display scope. The back-reflection region is shown, $2\theta > 135^\circ$.

generator and not produce fluorescence at all than to produce it and reject it with a pulse-height discriminator acting on the START pulse. The final picture, Fig. 4(c), is of a Si powder rather than of a single crystal, taken with filtered Cu radiation, 35 kV, 15 mA. Several high-order Debye-Scherrer rings are clearly visible in just 30 s.

The multiwire Laue camera is useful for more than just crystal orienting, however. With dynamic Laue patterns to view, it becomes trivial to determine whether or not an ingot of crystalline material is composed of one single crystal. By translating the ingot perpendicular to the incident beam, any boundary between single-crystal regions will become evident since the Laue patterns are different on each side of the boundary. With this technique, several single-crystal boundaries of a Ru crystal 60 mm in length were marked out in under 30 s. With standard methods, 5 or 6 ten-minute photographs would have allowed a crude determination of the boundary lines of the various single-crystal portions. Twinned crystals are easily noticed by the doubling or tripling of each of the Laue spots. Translating the crystal perpendicular to the incident beam allows the spatial extent of these twinned regions to be quickly ascertained. If the crystal surface has been damaged from cutting or polishing, etc., then the absence of Laue spots is immediately noticed. Of course, the ability to do these kinds of things has been available for quite some time, but the time and effort involved in obtaining a series of Laue photographs was considerable. With dynamic Laue images available, a more complete characterization of a crystal and its orientation can be made on a time scale of a few minutes rather than hours of work.

We acknowledge the assistance of Dan Resler and John Budai in fabricating the chamber, and the facilities made available by the Laboratory for Nuclear Studies and the X-ray Facility of the Materials Science Center, both of Cornell University. Dr V. Perez-Mendez and G. Stoker of the Lawrence Berkeley Laboratory kindly supplied design information about their chambers. This work was supported by the NSF through the Materials Science Center at Cornell University.

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