International Tables for Crystallography (2006). Vol. C, Section 7.1.6, pp. 623–633.

additional circuit to reject pileup pulses that can distort the spectrum at high count rates.

The intrinsic efficiency, defined as the ratio of the number of pulses produced to the number of photons striking the detector, is close to 100% in a large energy range. Because of the penetrating power of high-energy X-rays, the efficiency declines at high energies. The low-energy limit is set mainly by the absorption in the beryllium entrance window of the detector. Fig. 7.1.5.1 shows the intrinsic efficiency for an Si(Li) detector and HPGe detector with typical crystal size and window thickness. It is seen that the useful photo-energy range is about 1–40 keV for the Si(Li) detector and 2–150 keV for the HPGe detector. Some minor complications of the HPGe detector are a dip in efficiency around the germanium K absorption edge at 11 keV and the presence of Ge $K\alpha$ and $K\beta$ escape peaks in the measured spectrum.

The energy resolution is commonly expressed as the full width at half-maximum (FWHM) of a peak in an energy spectrum. For a spectral peak with Gaussian shape, ΔE(FWHM) corresponds to 2.355 times the root mean square of the energy spread. The energy resolution, including both the detector and the associated electronics, is given by

$$\Delta E_{\rm FWHM} = \{e_n^2 + [2.355(F\varepsilon E)^{1/2}]^2\}^{1/2}, \qquad (7.1.5.1)$$

where e_n is the electronic noise contribution, F the Fano factor (about 0.1 for both silicon and germanium), and ε the energy required for creating an electron–hole pair.

The energy resolution is generally specified at 5.9 keV (Mn $K\alpha$) as a reference energy. Typical best values for a detector with 25 mm^2 area are 145 eV (2.5%) for an HPGe detector and 165 eV (2.7%) for an Si(Li) detector. The resolution is degraded for larger detector areas.

Count-rate limitations are particularly obvious in synchrotron-radiation applications, where high photon fluxes are encountered (Worgen, 1982). The count rate is limited to below 10^5 counts s^{-1}, mainly by the pulse processing system.

Cadmium telluride, mercury iodide and other wide-band-gap semiconductors could be good candidates for energy-dispersive room-temperature X-ray detectors. Until now, the best energy resolution of the Hg_2I spectrometer with both the detector and the preamplifier operating at room temperature is 295 eV (FWHM) for the 5.9 keV Mn $K\alpha$ line, corresponding to a relative resolution of 5.0%. By lowering the noise level of the preamplifier FET with cryogenic techniques, a resolution of about 200 eV (3.4%) has been achieved (Warburton, Iwanczyk, Dabrowski, Hedman, Penner-Hahn, Roe & Hodgson, 1986).

7.1.6. Position-sensitive detectors (By U. W. Arndt)

Most X-ray diffraction or scattering problems require the quantitative evaluation of a linear or of a two-dimensional pattern. Recent years have seen the development of many different types of linear and area detectors for X-diffraction purposes, that is, of position-sensitive detectors (PSD's) that allow the recording of the positions of the arrival of X-ray photons (Hendricks, 1976; Hendrix, 1982; Arndt, 1986). In addition, imaging detectors have found increasing use in related fields, such as in X-ray astronomy (Allington-Smith & Schwarz, 1984), in X-ray microscopy, in X-ray absorption spectroscopy, and in topography. Here, the emphasis is on the production of an image for direct viewing rather than on the making of quantitative intensity measurements; these applications, in general, require an ultra-high spatial resolution over a relatively small field of view and the ability to cope with very low contrast images: Imaging detectors for topography are discussed in Section 7.1.7. Lessons can also be learnt, and component parts utilized, from quantitative imaging devices developed for visible light.

Progress in these fields has been covered in the *Symposia on Photoelectronic Image Devices* held every 3 years at Imperial College London (since 1960) and in the *Wire Chamber Conferences* (since 1978) and the *London Position-Sensitive Detector Conferences* (since 1987), both reported in full in *Nuclear Instruments and Methods*. Detectors are always one of the principal subjects considered at synchrotron-radiation conferences and workshops, the highlights usually being reported in *Synchrotron Radiation News*. Detectors feature prominently in the proceedings of the *IEEE Symposia on Nuclear Science*, which appear in the *IEEE Transactions*.

Other recent reviews of X-ray detectors are by Fraser (1989), Stanton (1993), Stanton, Phillips, O'Mara, Naday & Westbrook (1993) and Sareen (1994).

The detection of X-ray photons in the energy range of interest for diffraction studies (3 to 20 keV) always involves the interaction of the photon with an inner-shell electron and its complete absorption. The processes that are of interest for the construction of PSD's are of three kinds:

(1) Photography. The characteristics of X-ray film are discussed in Section 7.1.1.

(2) The use of storage phosphors, such as europium-activated barium halide (BaFX:Eu^{2+}, $X = $ Cl or Br) (Sonada, Takano, Miyahara & Kato, 1983; Miyahara, Takahashi, Amemiya, Kamiya & Satow, 1986), which are exposed like photographic film and then scanned with a laser beam causing photon-stimulated light emission of an intensity proportional to the original exciting X-ray intensity; this is measured with a photomultiplier. The plate is re-useable when the X-ray image has been erased. These X-ray detectors have a low background, a large dynamic range, and an adequate spatial resolution. See Section 7.1.8.

(3) Processes that involve the production of electrons. These may be the result of the ionization of a gas; they may be due to the production of electron–hole pairs in a semiconductor; they may be produced in an X-ray photocathode; finally, a phosphor may be used to convert the X-rays into visible light that then produces photoelectrons from a conventional photocathode.

At the present time, X-ray-sensitive photographic emulsions are mainly of historical interest. Storage-phosphor image plates have not only largely replaced photographic film; they have also taken over many of the applications of electronic detectors.

In the following, we are concerned only with detectors that depend on the production of electrons by incident X-rays.

In all detectors, except in semiconductor detectors, the number of primary electrons is multiplied by gas amplification, or in some device such as a microchannel plate or by some other intermediate process. In a PSD, the electron multiplication must take place with a minimum of lateral spread.

Many methods are available for deriving the position of the amplified electron stream or of the cloud of electron–ion pairs (avalanche) in an ionized gas and some of these are discussed below. However, almost any combination of photon detection, electron multiplication, and localization procedure can be used in the construction of PSD's (Fig. 7.1.6.1).

7.1.6.1. *Choice of detector*

Detectors may either be true counters in which individual detected photons are counted or they may be integrating devices that generate a signal that is a function of the rate of arrival of photons; this signal may then be digitized for recording purposes.

The choice of a linear or of a two-dimensional, or area, PSD depends on its detection efficiency, linearity of response to incident X-ray flux, dynamic range, and spatial resolution, its uniformity of response and spatial distortion, its energy discrimination, suitability for dynamic measurements, its stability, including resistance to radiation damage, and its size and weight.

Before discussing a few of the many types of PSD individually, we shall examine these criteria in a general way. Some of them have been discussed by Gruner & Milch (1982). Table 7.1.6.1 shows the importance, on a scale of 0 to 3, of some of the factors in different fields of study. Other properties, such as stability and sensitivity, are equally important for *all* PSD's.

7.1.6.1.1. *Detection efficiency*

The detection efficiency of a detector is determined in the first instance by the fraction of the number of incident photons transmitted by any necessary window or inactive layer, multiplied by the fraction usefully absorbed in the active region of the detector. This product, which is often called the absorption efficiency or the quantum efficiency, should be somewhere between 0.5 and 1.0 since the information loss due to incident photons not absorbed in the active region cannot be retrieved by subsequent signal amplification. The *useful* efficiency is best described by the so-called detective quantum efficiency (DQE), ε (Rose, 1946; Jones, 1958). For our purposes, this can be defined as

$$\varepsilon = S^2/\sigma^2 N, \qquad (7.1.6.1)$$

where N is the number of quanta incident upon the detector and σ is the standard deviation of the analogue output signal of amplitude S. For a photon counter with an absorption efficiency q, $S = qN$, $\sigma = (qN)^{1/2}$, and $\varepsilon = q$. An analogue detector with a DQE ε thus behaves like a perfect counter that only detects a fraction ε of the incident photons.

Table 7.1.6.1. *The importance of some detector properties for different X-ray patterns*

Detector property	Type of pattern: Solution	Fibre	Powder	Single crystal	Topographic	Orientation Laue
Spatial resolution	1	2	3	3	3	2
Lack of parallax	0	1	3	3	0	1
Accuracy of intensity measurement	3	3	3	3	1	1
High count rate capability	2	2	2	3	1	1
Suitability for short time slices	3	3	3	2	3	0
Suitability for short wavelengths	0	0	2	2	1	3
Energy discrimination	1	1	2	2	0	0

0 = unimportant; 3 = very important.

Under favourable conditions, the DQE of analogue detectors for X-rays is in excess of 0.5, but ε varies with counting rate and is lower for detectors with a very large dynamic range, as shown below.

The DQE of CCD- and vidicon-based X-ray detectors has been discussed by Stanton, Phillips, Li & Kalata (1992*a*).

7.1.6.1.2. *Linearity of response*

The linearity of a counter depends on the counting losses, which are due to the finite dead-time of the counter and its

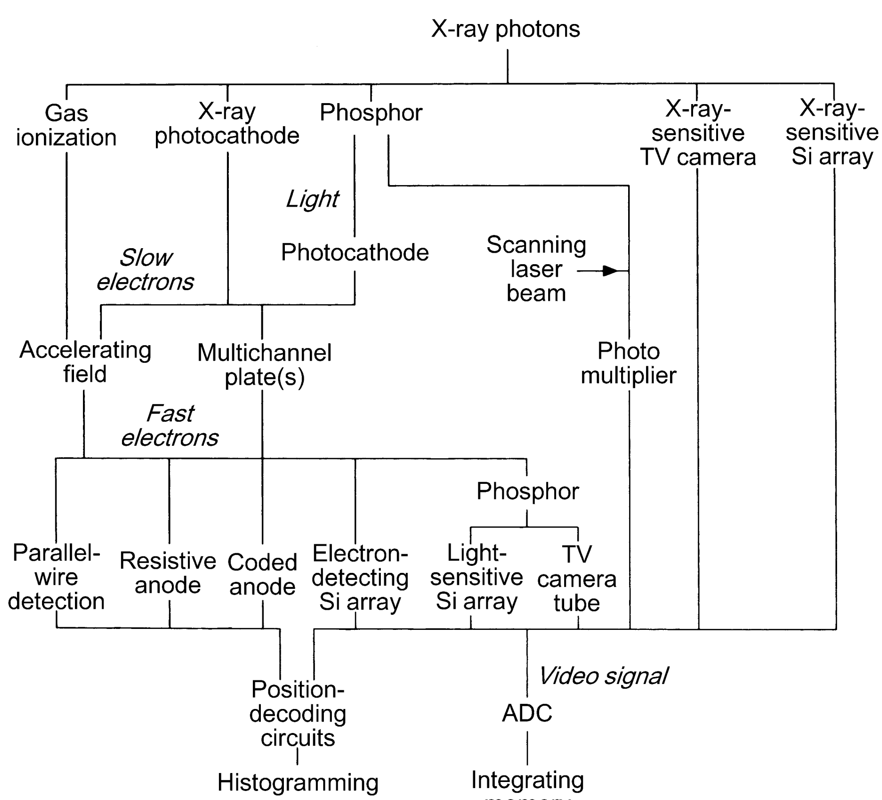

Fig. 7.1.6.1. Possible combinations of detection processes, localization methods and read-out procedures in PSD's.

processing circuits. The counting losses are affected by the time modulation, if any, of the source, as, for example, with storage rings (Arndt, 1978).

Counting losses can affect the behaviour of detectors in two different ways. In most analogue detectors and in counters with parallel read-out, each pixel behaves as an independent detector and the counting loss at any point depends only on the *local* counting rate. In other devices, such as multiwire proportional chambers with delay-line read-out (see Subsection 7.1.6.2), the whole detector becomes dead after an event anywhere in the detector and what matters is the *global* counting rate.

Fortunately, the fractional counting loss is the same for all parts of the pattern so that the *relative* intensities in a stationary pattern are not affected.

7.1.6.1.3. *Dynamic range*

The lowest practically measurable intensity is determined by the inherent background or noise of the detector. Some form of discrimination against noise pulses is usually possible with a detector that counts individual photons, but not, of course, with integrating detectors.

The maximum intensity at which a *counter* can operate is determined by the dead-time. In the case of an *integrating or analogue detector* with a variable gain, there is a trade-off between maximum intensity and DQE. Such a device can often be regarded as having an output signal with an amplitude $S = NV/M$ that is a noise-free representation of N, the number of photons detected in the integrating period of the device, where V, the maximum signal amplitude, is produced by M photons in this period. M can be varied by altering the gain of the detector. The noise can be regarded as a fixed fraction $1/r$ of the maximum amplitude V that is added to the signal. Then the DQE will be

$$\begin{aligned}\varepsilon &= S^2/\sigma^2 N\\ &= (1 + M^2/r^2 N)^{-1}.\end{aligned} \qquad (7.1.6.2)$$

This equation shows the importance of having as small a value of $1/r$ as possible; it also demonstrates that, for a given value of r, M can be increased only at the expense of a reduced DQE. This is valid for X-ray film (Arndt, Gilmore & Wonacott, 1977), for television detectors (Arndt, 1984), for the integrating gas detectors discussed in Subsection 7.1.6.2, and for many semiconductor X-ray detectors.

7.1.6.1.4. *Spatial resolution*

The spatial resolution of a PSD is determined by the number and size of resolution or picture elements (pixels) along the length or parallel to the edge of the detector. In most diffraction experiments, the size of the pattern can be scaled by altering the distance of the detector from the sample and what is important is the angular resolution of the detector when placed at a distance where it can 'see' the entire pattern. We shall see below that linear PSD's can be made with up to 2000 pixels and that area detectors are mostly limited to fewer than 512 × 512 pixels. The sizes of pixels range from about 10 µm for semiconductor PSD's to about 1 mm for most gas-filled detectors.

The *useful* number of pixels of a detector is determined by its point-spread function (PSF). This is the relative response as a function of distance from the centre of a point image on the detector. PSF's are not necessarily radially symmetrical and may have to be specified in at least two directions at right angles, for example along and perpendicular to the lines of a television raster scan. The width of the PSF at the 50% level determines the amount of detail visible in a directly viewed image. The accuracy of intensity measurements may depend more critically upon the width of the PSF at a lower level, since a weak spot may be immeasurable when sitting on the 'tail' of a very intense one. For various physical reasons, the PSF's of *all* PSD's, including X-ray film, have appreciable tails.

The spatial resolution of a detector is affected by parallax: when an X-ray beam is absorbed in a thick planar detector at an angle φ to the normal, the width of the resultant image is smeared out exponentially and its centroid is shifted by an amount $\sin\varphi/\mu$. For 8 keV X-rays incident at 45° on a xenon-filled counter, for example, this shift is about 4 mm for a filling pressure of 1 atm and 0.4 mm for a filling pressure of 10 atm. These figures illustrate the desirability of high-pressure xenon (Fig. 7.1.6.2) for gas-ionization detectors intended for wide-angle diffraction patterns.

7.1.6.1.5. *Uniformity of response*

All PSD's show long-range and pixel-to-pixel variations of response to larger or smaller extents. These can be corrected, in general by means of a look-up table, during data processing, but the measurements necessary for the calibration are often time-consuming. The output signals of many analogue detectors contain fixed-pattern noise that is synchronous with the read-out clock. This noise is usually removed during data processing, which in any case requires the subtraction of the background pattern.

7.1.6.1.6. *Spatial distortion*

In most detectors, there is some spatial distortion of the image. Again, the necessary calibration procedure may be time-consuming. Distortions cause point-to-point variations in pixel size, which produce response variations additional to those from other causes.

Corrections for spatial distortion and for non-uniformity of response have been discussed by Thomas (1989, 1990) and by Stanton, Phillips, Li & Kalata (1992*b*).

7.1.6.1.7. *Energy discrimination*

The amplitude of the signal due to a single photon is usually a function of the photon energy. The variance in this amplitude, or the full width at half-maximum (FWHM) of the pulse-height spectrum, for a monoenergetic input, depends on the statistics of the detection process. A sharp pulse-height-distribution (PHD) curve may permit simultaneous multi-wavelength measurements with a suitable counter, or at least afford a reduction of the background by pulse-height discrimination. In an analogue

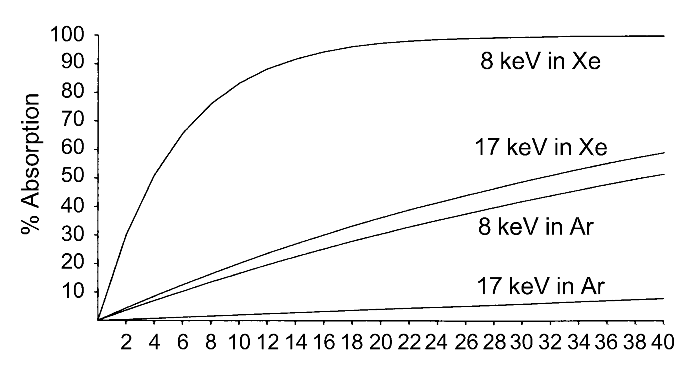

Fig. 7.1.6.2. Absorption of 8 keV and 17 keV photons in argon and xenon as a function of pressure in atm × column length in mm.

detector, the variance in the primary amplitude affects the DQE (Arndt & Gilmore, 1979).

7.1.6.1.8. *Suitability for dynamic measurements*

Many investigations, such as low-angle or fibre diffraction studies on biological materials carried out with synchrotron radiation (Boulin, Dainton, Dorrington, Elsner, Gabriel, Bordas & Koch, 1982; Huxley & Faruqi, 1983), involve the time variation of a diffraction pattern. For very short time slots, only counters can be employed; the incoming pulses must then be gated and stored in an appropriate memory (Faruqi & Bond, 1982).

Stores used for these experiments are described as histogramming memories; the contents of a given storage location are incremented by one whenever the corresponding address, which represents the position and the time of arrival of a photon, appears on the address bus (Hendricks, Seeger, Scheer & Suehiro, 1982; Hughes & Sumner, 1981).

7.1.6.1.9. *Stability*

Stability of the performance of a detector is of paramount importance. Most position-sensitive detectors are used in connection with microcomputers, which make calibration and corrections for spatial distortion, non-uniformity of response, and lack of linearity relatively easy, provided that these distortions remain constant. The long-term stability of many detectors, notably of semiconductor devices, is affected by radiation damage produced by prolonged exposure to intense irradiation.

7.1.6.1.10. *Size and weight*

The size and weight of the detector determine the ease with which the detector can be moved relative to the sample and thus the extent to which the diffractometer can be adapted to varying resolution and collimation conditions. Some detectors cannot be moved easily (Xuong, Freer, Hamlin, Nielsen & Vernon, 1978), or need very heavily engineered rotational and translational displacement devices (Phizackerley, Cork & Merritt, 1986). Others, such as spherical drift chamber multiwire proportional chambers, are designed for use at a fixed distance from the sample and may only be swung about the latter but not translated (Kahn, Fourme, Bosshard, Caudron, Santiard & Charpak, 1982).

In single-crystal diffraction patterns, the Bragg reflections may be visualized as diverging from the X-ray source while the background – fluorescence X-rays, scatter by amorphous material on the specimen crystal and its mount – diverges from the sample. Consequently, the highest reflection-to-background ratio is achieved by using a large detector at a large distance from the specimen. Of all the detectors discussed here, the image plate can most readily and economically be used to cover a large area; its present popularity is chiefly due to this property (Sakabe, 1991).

There is fairly general agreement on the specification of an ideal X-ray area detector. It should be at least 250 mm in diameter, contain at least 1000×1000 pixels, have a large dynamic range and a high detective quantum efficiency for photon energies up to 20 keV and be capable of being read-out rapidly.

Many suggestions have been made for improving the performance of existing detectors. It has become apparent, however, that the development of ideal, or even better, X-ray detectors is extremely expensive and, therefore, that their development and installation can be undertaken only in central national or international laboratories such as storage-ring synchrotron-radiation laboratories.

7.1.6.2. *Gas-filled counters*

In all gas-filled counters, whether one-, two-, or three-dimensional, the initial event is the absorption of the incoming X-ray photon in a gas molecule with the emission of a photo-, or alternatively an Auger, electron. The detection efficiency depends on the fraction of the photons absorbed in the gas and this fraction is shown in Fig. 7.1.6.2 as a function of the product of gas pressure and column length for 8 and 17 keV photons on argon and xenon. The ionization energy of noble gases is about 30 eV so that one 8 keV photon gives rise to about 270 electron–ion pairs. With adequately high collecting fields, the electrons acquire sufficient energy to produce further ionization by collision with neutral filling gas molecules; this process is often referred to as 'avalanche production' or 'gas multiplication'. The factor A by which the number of primary ion pairs is multiplied can be as great as ten to one hundred thousand. Up to a certain value of A, the total amount of ionization remains proportional to the energy of the original X-ray photon. The electrical signal generated at the anode of the counter is due very largely to the movement of the positive ions from the immediate vicinity of that electrode; at the same time, a corresponding pulse is induced on the cathode. The signal can be shaped to produce a pulse with a duration of the order of a microsecond.

In single or multiwire proportional counters, the secondary ionization (avalanche production) takes place in the highest field region, that is, within a distance of a few wire diameters of the anode wire or wires. The electrons are collected on the anode and the positive ions move towards the cathode, with very little spread of the ionization in a direction perpendicular to the field gradient, that is, parallel to the wire direction. It is thus possible to construct position-sensitive devices based on such chambers.

Proportional-counter behaviour is discussed in detail in many standard texts and review articles (Wilkinson, 1950; Price, 1964; Dyson, 1973; Rice-Evans, 1974).

The gas amplification does not have to take place in the same region of the detector as the original absorption. In so-called drift chambers, the primary ionizing event takes place in a low-field region where no avalanching takes place. The electrons drift through a grid or grids into a region where the field is sufficiently high for gas multiplication to occur. The drift field can be made cylindrical in a linear counter (Pernot, Kahn, Fourme, Leboucher, Million, Santiard & Charpak, 1982), or spherical in an area detector (Charpark, 1982; Kahn, Fourme, Bosshard, Caudron, Santiard & Charpak, 1982), centred on the point from which the X-rays diverge, that is on the specimen; the electrons then drift in a radial direction without parallax being introduced (Fig. 7.1.6.3).

In many experiments, use is made of the energy discrimination of the detector. The ratio of the full width at half-maximum to the position of the maximum of the pulse-height distribution is given by

$$w = 2.36[(F + f)/N], \qquad (7.1.6.3)$$

where N is the number of primary ion pairs produced, F is the Fano factor (Fano, 1946, 1947), which takes into account the partially stoichastic character of the gas multiplication process, and f is the avalanche factor. For proportional counters filled with typical gas mixtures (argon + methane), $F = 0.17$ and $f = 0.65$, so that for 8 keV photons $w \sim 13\%$, but, in the so-called Penning gas mixtures (*e.g.* noble gas and ethylene), f can approach zero at a certain field strength. In a wire counter with its rapidly varying field strength, f is small only for a gas amplification of less than 50. The energy resolution for 8 keV photons could then be as low as 6%, but the pulses induced on the cathode wires of a MWPC are then too small to permit a precise localization. This problem has been overcome by using uniform-field avalanching in two regions in tandem, separated by a drift space (Schwarz & Mason, 1984, 1985). The energy information was derived after the first low-gain gas multiplication process ($A \sim 500$): a proportion of the electrons from the first avalanche then drifted into the second avalanche region which boosted the gas gain to more than 10^5, necessary to give a high spatial resolution.

In an alternative method (Charpak, 1982; Siegmund, Culhane, Mason & Sanford, 1982), the additive avalanche factor f is eliminated by deriving the energy information, not from the collected charge, but from the visible light pulse produced by the individual avalanches of each primary electron.

7.1.6.2.1. *Localization of the detected photon*

There are several methods of deriving the position of the detected photon that are applicable to both linear and area detectors.

(1) The charge produced in the avalanche can be collected on a resistive anode. In the case of linear detectors, the central wire can be given a low or a very high resistance. The latter type is most commonly made from a quartz fibre coated with carbon. The emerging pulse is detected at both ends of the wire (Borkowski & Kopp, 1968; Gabriel & Dupont, 1972). Area detectors with a resistive disc anode must have at least three read-out electrodes (Stümpel, Sanford & Goddard, 1973). With low-resistance electrodes, the position of the event can be computed by analogue circuits from the relative pulse amplitudes (Fig. 7.1.6.4*a*); a preferred method with high-resistance anodes is to measure the rise times of the output pulses that are determined by the time constant formed by the input capacity of the pulse amplifier at each output and the resistance of the path from the detection point to the output electrode (Fig. 7.1.6.4*b*).

(2) The anode or cathode can be constructed in the form of two or more interleaved resistive electrodes insulated from each other. Provided that the charge distribution covers at least one

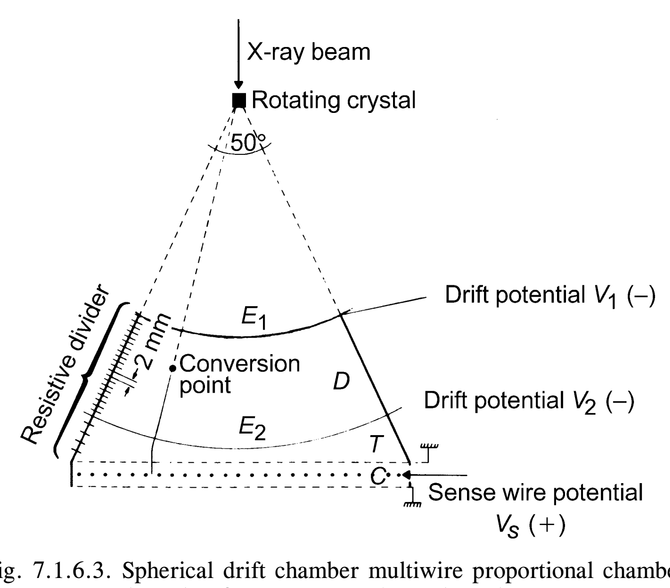

Fig. 7.1.6.3. Spherical drift chamber multiwire proportional chamber (MWPC) (Charpak, 1982; courtesy of G. Charpak).

unit of the pattern, positional information can be derived by relative pulse height or by timing methods. Examples of this type of read-out are the linear backgammon (*jeu de jacquet*) counter together with its two-dimensional variant (Allemand & Thomas, 1976), the wedge-and-strip anode developed by Anger and his collaborators (Anger, 1966; Martin, Jelinsky, Lampton, Malina & Anger, 1981), and its polar coordinate analogue (Knibbeler, Hellings, Maaskamp, Ottewanger & Brongersma, 1987), for two-dimensional read-out. The method seems capable of a higher spatial resolution than any other (Schwarz & Lapington, 1985).

(3) The anode or cathode can be made from a number of sections connected to a tapped delay line (Fig. 7.1.6.4*c*). Positional information is derived from the time delay of the pulse relative to the arrival of an undelayed prompt pulse. Linear PSD's (LPSD's) with delay-line read out are usually made straight, but variants have been produced in the form of circular arcs (Wölfel, 1983; Ballon, Comparat & Pouxe, 1983).

Area detectors of this type require two parallel planes of parallel wires with the wires in the two planes at right angles to one another placed on either side of the anode, which also consists of parallel wires. The prompt pulse in such a detector, the multiwire proportional chamber (MWPC), is usually taken from the anode (Fig. 7.1.6.5). In counters without a drift space, the electron avalanche always ends up on one anode wire, and there is then a pseudo-quantization in the position measurement made at right angles to the direction of the anode wires. In drift-space detectors with a narrow anode-wire spacing, the avalanche lands on more than one wire and some interpolation is possible. In the direction parallel to the anode wires, there is never any quantization and the resolution can be better than the cathode wire spacing: Although pulses are induced on several wires, the centroid of the delayed group of pulses can be measured with precision. Delay-line read-out LPSD's have reached the highest resolution in the hands of Radeka and his group (Smith, 1984). MWPC's of this type have been used for several years (Xuong, Freer, Hamlin, Nielsen & Vernon, 1978; Bordas, Koch, Clout, Dorrington, Boulin & Gabriel, 1980; Baru, Proviz, Savinov Sidorov, Khabakhshev, Shuvalov & Yakovlev, 1978; Anisimov, Zanevskii, Ivanov, Morchan, Peshekhonov, Chan Dyk Tkhan, Chan Khyo Dao, Cheremukhina & Chernenko, 1986). They have a relatively low maximum count rate ($< 10^5\ s^{-1}$) determined by the space charge due to earlier events and by the fact that position digitization takes of the order 1 µs. Limitations in the closeness of practicable wire spacing leads to a pixel size of the order of 1 mm.

(4) A faster read out is possible with MWPC's in which the positional information is derived from the centroid in amplitude of the group of induced cathode pulses (Fig. 7.1.6.4*d*). Individual amplifiers of carefully equalized gain are required for each individual wire or at least for small groups of adjacent wires (Pernot, Kahn, Fourme, Leboucher, Million, Santiard & Charpak, 1982). Counting rates in excess of $10^6\ s^{-1}$ are then possible.

7.1.6.2.2. *Parallel-plate counters*

In the gas-filled detectors that we have considered so far, the electric field is cylindrically symmetrical in the immediate vicinity of the wire or wires near which gas multiplication takes place and the maximum count rate is limited ultimately by the electrostatic shielding effect of the ion sheath owing to previous X-ray photons. In parallel-plate chambers, the electrodes are in the form of very fine electro-formed grids: With this structure, the pulse shape is quite different; the very sharp initial part, due to the rapidly moving electrons, can be separated, at the expense of a loss of signal amplitude, from the slow component due to the positive ions; in addition, the shielding effect is much less pronounced. Accordingly, counting rates up to at least $10^{11}\,\mathrm{s}^{-1}\,\mathrm{m}^{-2}$ are possible with parallel-plate PSD's (Stümpel, Sanford & Goddard, 1973; Peisert, 1982; Hendrix, 1984).

7.1.6.2.3. *Current ionization PSD's*

For the very highest counting rates, it is necessary to abandon all methods in which individual X-ray photons are counted and instead to measure the ionization current produced by the incident X-rays on either cathode or anode. Fig. 7.1.6.6 shows the principles of a cathode read-out linear PSD. The cathode is divided into strips, each of which is connected to a capacitor and to an input terminal of a CMOS analogue multiplexer. The charge accumulated on each capacitor in a given time period is transferred to a charge-sensitive amplifier when the associated channel is selected by an addressing signal. The output voltage of the amplifier is digitized by means of an analogue-to-digital converter. The complete pattern is scanned by incrementing the addresses sequentially: The resolution is that of the strip spacing (~0.5 mm) and the principle can be extended to two dimensions (Hasegawa, Mochiki & Sekiguchi, 1981; Mochiki, Hasagawa, Sekiguchi & Yoshioka, 1981; Mochiki, 1984; Mochiki & Hasegawa, 1985). Global count rates in excess of $10^9\,\mathrm{s}^{-1}$ are possible with this method. Lewis (1994) has published a

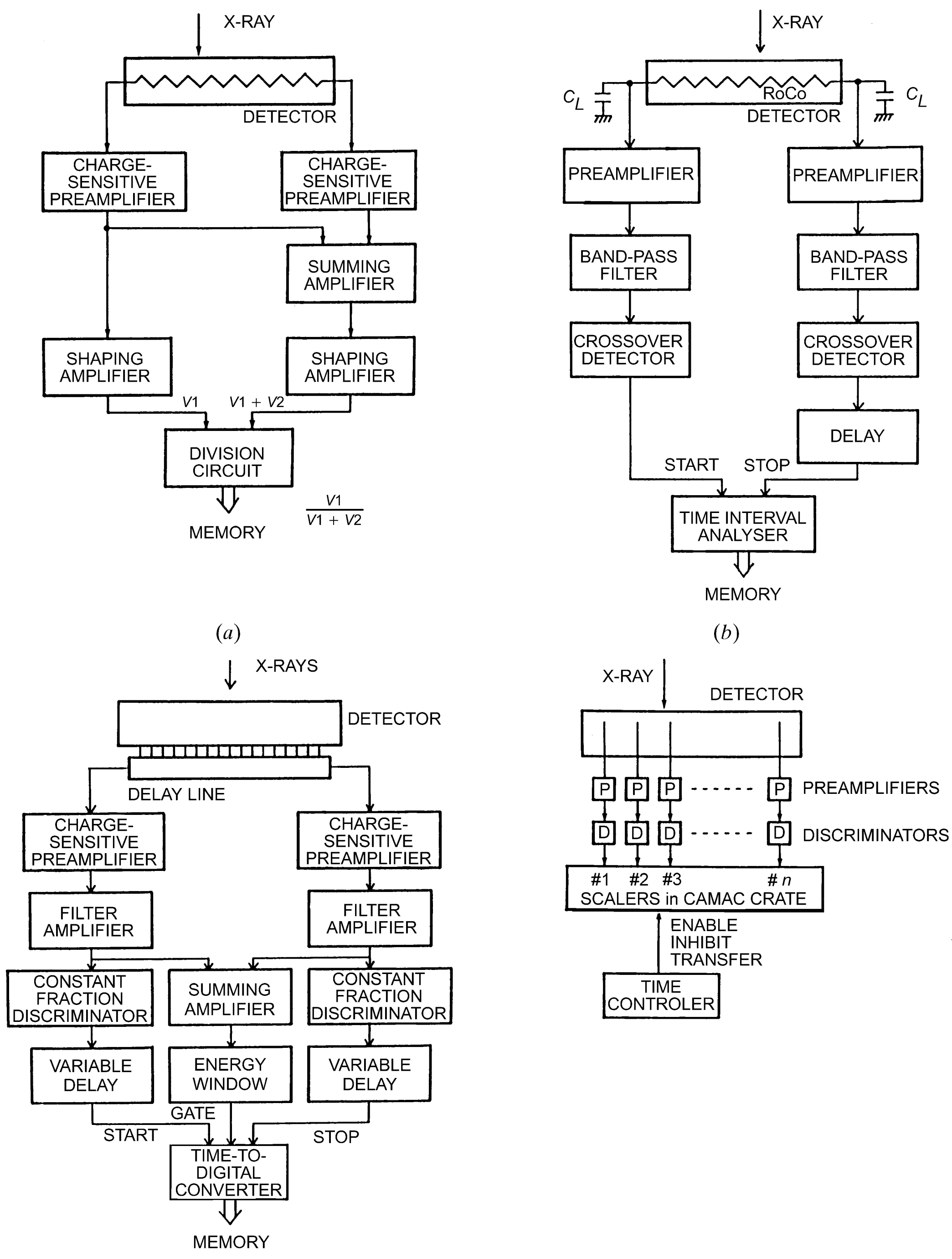

Fig. 7.1.6.4. Read-out methods for gas-filled LPSD's. (*a*) Charge division with low-resistance anode wire. (*b*) Rise-time method with high-resistance anode. (*c*) Delay-line read-out. (*d*) Amplifier-per-wire method. From Mochiki (1984); courtesy of K. Mochiki.

comprehensive survey of the present status and the future potentialities of gas-filled position-sensitive detectors.

7.1.6.3. *Semiconductor detectors*

Semiconductor detectors are essentially solid-state ionization chambers in which the incoming photon generates electron–hole pairs instead of electron–ion pairs. Semiconductor detectors are sensitive to the entire electromagnetic spectrum from visible to X-rays, and they can also detect electrons directly. There are, therefore, three possible methods of constructing semiconductor X-ray detectors, each of which has its advantages and disadvantages.

(1) They can be exposed directly to the incoming X-rays.

(2) The X-ray photons can be made to produce visible light photons that are then detected in a light sensor.

(3) The X-rays can be made to produce electrons that are detected by the semiconductor detector.

In semiconductor one- or two-dimensional PSD's, the detector and the read-out circuitry are usually integrated on the same chip. In these devices, the pixel size and position are fixed once and for all so that they are geometrically completely stable. Integrated read-out circuitry has a low input capacity and thus an extremely low read-out noise.

All semiconductor X-ray PSD's are derived from imagers for visible light, which are of two basic types: photodiode arrays (PDA's) and charge-coupled devices (CCD's) (Lowrance, 1979; Allinson, 1982; Borso, 1982; Third European Symposium on Semiconductor Detectors, 1984).

In PDA's, the detection of a photon-generated charge takes place in a depletion layer that is formed either in a suitably biased *p–n* diode or in a metal-oxide-semiconductor (MOS) capacitor. The electron–hole pairs are separated by the field associated with the depletion layer. The individual diodes store charge during the integration period; this is read out into a common video output line *via* MOS multiplexing switches. This architecture is usually adopted for linear arrays where the switches can be arranged around the periphery of the diodes with a minimum amount of dead space between them and where they can be shielded from the incident X-rays. PDA's tend to suffer from a high fixed-pattern noise due to differences in the performance of individual MOS switches.

In CCD's (Howes & Morgan, 1979), the sensing elements are always MOS capacitors suitably biased to establish charge-storage volumes. During read out, the charge is transferred in a 'bucket-brigade' fashion from one MOS capacitor to the next until it reaches the output. In linear CCD's, this output is at one end of what is essentially an analogue shift register. Most two-dimensional CCD's are frame-transfer devices: at the end of the exposure, the charge is transferred line by line to an identical array of MOS elements; while the next 'frame' is exposed in the image array, the contents of the buffer array are transferred, one line at a time, to a single-line buffer from which they are shifted out element by element. Since the transfer circuitry in CCD's is interlaced with the detector elements, they are more difficult to shield and are more subject to radiation damage.

7.1.6.3.1. *X-ray-sensitive semiconductor PSD's*

For X-ray diffraction applications, the main disadvantage of semiconductor devices is that the universal trend in their manufacture is in the direction of miniaturization, leading to a very small pixel size. Thus, imaging devices with up to 2000×2000 pixels have been produced but the pixel size is typically $\sim 10\,\mu m$ for CCD's and $\sim 20\,\mu m$ for PDA's; in most X-ray diffraction applications, it would be difficult to scale down sample and source sizes to a point where the pattern size is

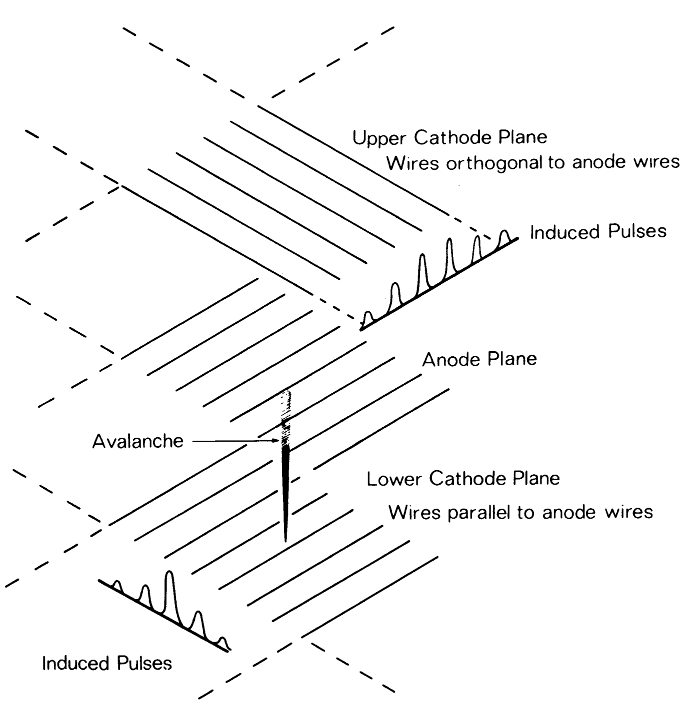

Fig. 7.1.6.5. Three-plane MWPC. Note the pseudo-quantization due to charge collection on one anode wire. The cathode wires may either be connected to a tapped delay line as in Fig. 7.1.6.4(*c*) or to individual amplifiers as in Fig. 7.1.6.4(*d*) (courtesy of A. R. Faruqi).

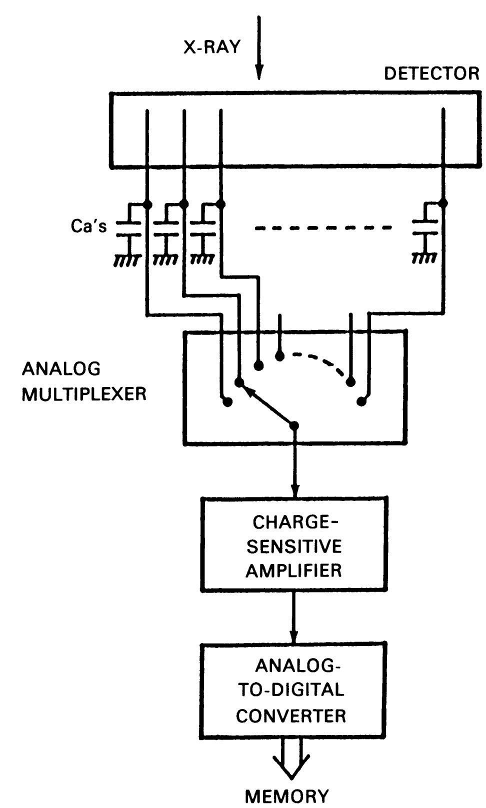

Fig. 7.1.6.6. Integrating LPSD. From Mochiki (1984); courtesy of K. Mochiki.

appropriate to a semiconductor imager, even though a 2000×2000 CCD with $27 \times 27\,\mu m$ pixels is available.

Semiconductor point counters cooled to 80 K are characterized by their very high energy resolution (better than 2% in the 8 keV region). Some counting PSD's potentially have a similar energy resolution. Two-dimensional X-ray-sensitive CCD's for X-ray astronomy research have been used as photon counters (Walton, Stern, Catura & Culhane, 1985; Lumb, Chowanietz & Wells, 1985) but they can only be used at very low counting rates. In integrating devices, the energy discrimination is lost. Silicon detectors for visible-light applications are made with depletion depths of the order of $10\,\mu m$. For the detection of 8 keV photons with more than 90% efficiency, depletion depths of $165\,\mu m$ are necessary and these can be produced only from very high resistivity material (Howes & Morgan, 1979). Moreover, in commercial visible-light imagers, the depletion region is covered by circuitry or by an inactive layer that constitutes an absorbing window for X-ray detection. For the detection of X-rays or electrons, it is, therefore, customary to thin the device and to illuminate it from the back (see, for example, Meyer-Ilse, Wilhelm & Guttmann, 1993).

One-dimensional X-ray detectors utilizing PDA's, such as those made by the Reticon Corporation, have found a number of applications, especially in dispersive X-ray absorption spectroscopy (EXAFS) (Jucha, Bonin, Dartyge, Flank, Fontaine & Raoux, 1984).

A particular problem with silicon detectors is the damage caused by the incidence of X-rays or of energetic electrons. The effects can be minimized by masking all but the active part of their device and by operating it at low temperatures (Jucha *et al.*, 1984).

The use of the room-temperature semiconductor mercuric iodide in place of silicon seems promising (Patt, Delduca, Dolin & Ortale, 1986).

The X-ray diffraction applications of directly sensitive semiconductor PSD's are likely to remain limited. A previous conversion to visible light or to electrons offers the possibility of an optical or electron-optical demagnification onto the imager, as well as of avoiding some of the other problems discussed above (Deckman & Gruner, 1986).

7.1.6.3.2. *Light-sensitive semiconductor PSD's*

Standard light-sensitive semiconductor imaging devices can be used for X-ray detection if the X-rays are first converted to light by means of a phosphor. One 8 keV X-ray photon produces several hundred light photons in a good phosphor (see Table 7.1.6.2). Because of the low noise levels possible with cooled CCD's ($\sim$10 electrons r.m.s.), only 1 to 3% of these photons need to reach the device to produce a perfect X-ray detector. Unfortunately, even this is possible only with optics that do not demagnify to any considerable extent (see Subsection 7.1.6.5) and one is thus restricted to a small detector. However, CCDs are now available that can be butted along two or three edges and these make possible the construction of 'tiled' detectors that contain four or six of such CCD chips (Burke, Mountain, Harrison, Bautz, Doty, Ricker & Daniels, 1991; Fordham, Bellis, Bone & Norton, 1991; Allinson, 1994). Individual channels can be read simultaneously (Hopf & Rodricks, 1994), thus making possible a relatively rapid read out of a large number of pixels.

7.1.6.3.3. *Electron-sensitive PSD's*

In image intensifiers, an output image is produced on a phosphor screen by electrons with an energy of a few keV. A promising device with direct positional read out consists of a demagnifying intensifier in which the phosphor is replaced by a CCD. Various intensifiers have been described in which the electrons reaching the CCD have an energy of several keV so that the electron–hole generation is amplified (EBS process) (Lowrance, Zucchino, Renda & Long, 1979; Lemonnier, Richard, Piaget, Petit & Vittot, 1985). High-energy electrons are liable to cause radiation damage and similar precautions as for X-rays, such as thinning and back-illumination, are necessary. Experiments have been reported with less-damaging low-energy (200 eV) electrons produced in a microchannel plate (MCP) image intensifier (Dereniak, Roehrig, Salcido, Pommerrenig, Simms & Abrahams, 1985). Both approaches look promising but it is questionable whether a device with an X-ray phosphor input and of adequate size and resolution at an affordable price will emerge in the near future.

7.1.6.4. *Devices with an X-ray-sensitive photocathode*

Image converters and television cameras with X-ray-sensitive photocathodes have been reported. These cathodes must be thin, or they must have a low bulk density to allow the photoelectrons to escape (Bateman & Apsimon, 1979; Haubold, 1984).

Television tubes with a beryllium window and a 25 mm diameter lead oxide target are available and experimental 125 mm tubes have been described (Suzuki, Uchiyama & Ito, 1976).

X-ray-sensitive Saticon television camera tubes with an amorphous selenium–arsenic target (Chikawa, Sato & Fujimoto, 1984) have an active diameter of only 10 mm but a limiting resolution (MTF = 5%) of $6\,\mu m$. They have an absorption efficiency of 52% for Mo $K\alpha$ radiation and are used mainly for X-ray topography. Other X-ray-sensitive television camera tubes have been described by Matsushima, Koyama, Tanimoto & Tano (1987), Suzuki, Hayakawa, Usami, Hirano, Endoh & Okamura (1989) and Sato, Maruyama, Goto, Fujimoto, Shidara, Kawamura, Hirai, Sakai & Chikawa (1993). Such tubes are discussed further in Section 7.1.7.

7.1.6.5. *Television area detectors with external phosphor*

Much development has gone into quantitative measurements with area detectors in which the diffraction pattern is formed on an external phosphor fibre-optically coupled to a low-light-level television camera. Mostly the television camera embodies a demagnifying image intensifier coupled *via* demagnifying optics to a sensitive television camera tube (Arndt & Gilmore, 1979; Arndt, 1982; 1985; Arndt & In't Veld, 1988; Kalata, 1982, 1985; Gruner, Milch & Reynolds, 1982) or to a CCD or an array of CCD's (Strauss, Naday, Sherman, Kraimer & Westbrook, 1987; Strauss, Westbrook, Naday, Coleman, Westbrook, Travis, Sweet, Pflugrath & Stanton, 1990; Templer, Gruner & Eikenberry, 1988; Widom & Feng, 1989; Fuchs, Wu & Chu, 1990; Karellas, Liu, Harris & D'Orsi, 1992). Camera tubes were frequently read at commercial television rates (625 lines with a field repetition rate of 25 Hz or 525 lines with a field repetition rate of 30 Hz in Europe and in North America and Japan, respectively), leading to pixel rates of about 10 MHz. Successive images were then digitized and their sums stored in memory (see Fig. 7.1.6.7). Milch, Gruner & Reynolds (1982) developed a slow-scan method for a silicon-intensifier-target (SIT) tube. This latter method has been facilitated by the development of very large scale integration memory circuits, which have made it possible to construct economical image stores into which the camera can write at a slow rate and which can then be read at normal television rates for display purposes.

Table 7.1.6.2. *X-ray phosphors (from Arndt, 1982)*

Phosphor	Type	Bulk density	No. of photons per 8 keV quantum		Max. emission wavelength (nm)	Decay to 1% (s)
			Produced	Collected*		
ZnS (Ag)	Polycrystal	4.1	750	300	450	3×10^{-7} + slow components
Gd_2O_2S	Polycrystal	7.1	500	200	550	10^{-3}
CsI (Tl)	Monocrytal	4.5	240	62	580	10^{-6}

* These figures are for collection on a photocathode on the opposite side of a fibre-optics face plate, in the absence of a reflective coating.

CCD's are usually operated in this fashion, so that the read–out circuits can have a narrow band-width and produce an excellent signal-to-noise ratio.

For very low X-ray intensities in which the probability of the arrival of a photon per pixel per frame period is much less than one, the camera can be operated at normal frame frequencies in a digital mode. Specially designed circuits detect the charge image produced by a single X-ray photon and find the centroid of this image (Kalata, 1982); the events are 'counted' in a histogramming memory. The method is capable of some energy discrimination and has a high spatial resolution because the centroid of the image can be found to a high precision.

7.1.6.5.1. *X-ray phosphors*

The incoming X-rays are converted to light in a phosphor that is coupled to the first photocathode of the system. Both polycrystalline and monocrystalline phosphors are used for X-ray detection. The former give a higher light output but have a limited resolution; the latter tend to have a poorer light-conversion efficiency but have the best resolution. The most useful phosphors are shown in Table 7.1.6.2.

Many attempts have been made to improve the spatial resolution of phosphor screens by constructing them in the form of scintillating fibres that are optically isolated from one another so that the scintillation does not spread. This can be achieved by growing columnar scintillating crystals (Oba, Ito, Yamaguchi & Tanaka, 1988), by intagliating polycrystalline phosphors (Fouassier, Duchenois, Dietz, Guillemet & Lemonnier, 1988) and by using arrays of scintillating fibres (Bigler, Polack & Lowenthal, 1986; Ikhlef & Skowronek, 1993, 1994).

Image intensifiers designed for radiography with relatively hard radiation usually have an X-ray-transparent window – which may be up to 300 mm in diameter – and an *internal* phosphor–photocathode sandwich deposited on an X-ray-transparent substrate. Problems of compatibility of phosphor and photocathode have restricted the phosphor used, but CsI works well with multialkali photocathodes. Moy and his collaborators have constructed a large-diameter television detector in which the image intensifier has been modified by using beryllium for the window and for the sandwich substrate; this intensifier is coupled to a slow-scan CCD camera (Moy, 1994).

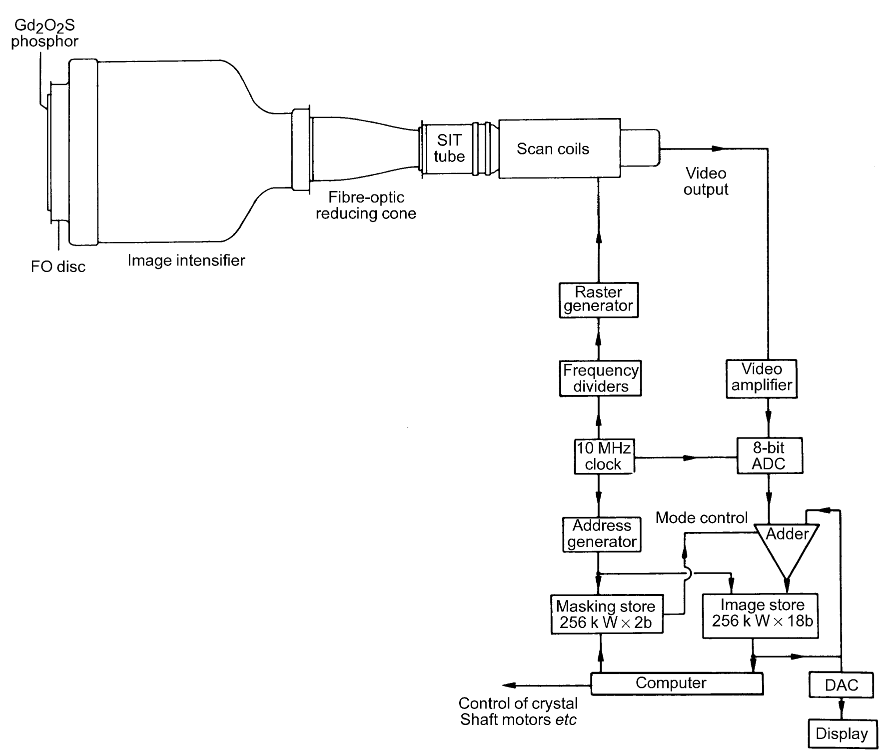

Fig. 7.1.6.7. Fast-scanning television X-ray detector (after Arndt, 1985).

7.1.6.5.2. *Light coupling*

Each incident X-ray photon should give rise to several photoelectrons from the first photocathode in order to achieve a high DQE (Arndt & Gilmore, 1979). The best photocathodes have a yield of about 0.2 electrons per light photon; only fibre-optics coupling between the phosphor and the photo-cathode can give an adequate light-collection efficiency (in excess of 80% for 1:1 imaging). With demagnifying fibre optics, the light loss is considerable for purely geometrical reasons. Fibre-optic cones, in which each individual glass fibre is conical, are available for magnification or demagnification up to about 5:1. It should be noted that image intensifiers and TV tubes with electrostatic focusing are normally made with fibre-optics face-plates and that some CCD's are also available with fibre-optics windows.

Where lens optics must be employed, it is best to use two infinity-corrected objectives of the same diameter, but not necessarily of the same focal length, back to back. In practice, the best light-collection efficiency that can be achieved at a demagnification of M is about $(2M)^{-2}$. It is for this reason that there are limitations on the maximum size of image that can be projected onto a CCD without the use of an image intensifier (but see Naday, Westbrook, Westbrook, Travis, Stanton, Phillips, O'Mara & Xie, 1994; Koch, 1994; Allinson, 1994). The function of this intensifier is to match a relatively large diameter X-ray phosphor to a small-size read-out device. As a result of the high sensitivity of CCD's, especially of slow-scan CCD's, a low photon gain in the intensifier and a low light-coupling efficiency in the coupling between intensifier and read-out device are quite adequate. It is, however, essential to couple the X-ray phosphor as efficiently as possible to the photocathode.

Roehrig *et al.* (1989) have described a design in which an image intensifier with 150 mm diameter input and output face plates is coupled by means of six demagnifying fibre-optic cones to six CCD's. Allinson (1994) has examined the need for image intensification and has shown that it is possible to construct a 150 mm square detector that has a performance approaching that of an ideal detector without using an image intensifier. Different methods of light coupling and format alteration have been discussed by Deckman & Gruner (1986).

7.1.6.5.3. *Image intensifiers*

In an image intensifier, the photoelectrons from a photocathode are made to produce a visible intensified image on an output phosphor. In so-called first-generation tubes, the intensification is produced by subjecting the electrons to accelerating voltages of up to about 15 keV: the number of visible-light photons at the output per keV of electron energy is about 80. The photon gain of the devices is typically about 100; there may be a brightness gain factor of M^2 if the electrostatic electron-optical system produces a demagnification of M. Standard first-generation image intensifiers of this type are made with input field diameters up to 80 mm, they always have fibre-optics input face plates on which X-ray phosphor may be deposited, they are stable and robust, and have a good resolution of better than 100 μm at the input. Their low gain requires the use of a low-light-level TV camera tube in the next stage (Arndt & Gilmore, 1979) or of two or more intensifiers of this type in tandem (Kalata, 1982) or of an intrinsically more sensitive slow-scan read out (Eikenberry, Gruner & Lowrance, 1986).

For military and civilian night-vision applications, first-generation image intensifiers have largely been replaced by devices embodying one or two microchannel plates (MCP's) that produce an electron gain of up to 1000 per stage (see, for example, Emberson & Holmshaw, 1972; Garfield, Wilson, Goodson & Butler, 1976). Commercial second- and third-generation intensifiers (Pollehn, 1985) are less suitable for quantitative scientific purposes than the first-generation devices: their GaAs photocathodes are less well matched to most X-ray phosphors, the gain of MCP's decreases with time, and the tubes have a slightly lower resolution than diode types of comparable diameter. Most third-generation intensifiers have plain rather than fibre-optics face plates and none appear to be available with a diameter greater than 50 mm (Airey & Morgan, 1985). Nevertheless, these high-gain intensifiers do make it possible to construct relatively cheap moderate-performance X-ray detectors using standard-sensitivity TV pick-up devices, including CCD's (Dalglish, James & Tubbenhauer, 1984), instead of the low-light-level camera tubes necessary with a lower pre-amplification.

An intensifier can, in principle, employ a variety of read-out methods, *e.g.* by substituting a resistive disc anode, a coded anode or a CCD for the output phosphor. However, the only way of employing standard modules is to couple them to a TV pick-up device.

7.1.6.5.4. *TV camera tubes*

Vacuum-tube television cameras have been largely replaced by semiconductor devices but of the former the preferred tube for use in an X-ray detector is still the silicon-intensifier-target (SIT) tube (Santilli & Conger, 1972). It has an adequate resolution for images up to 512×512 pixels and a linear transfer function (unity gamma) and its sensitivity is well matched for use with a single-stage image intensifier with a gain of 100. When cooled, this tube can be used for long exposures in an integrated slow read-out mode (Milch, Gruner & Reynolds, 1982).

When better high-gain image intensifiers become available, the preferred choice may be tubes like the Saticon (Goto, Isozaki, Shidara, Maruyama, Hirai & Fujita, 1974) whose diode gun gives them a superior resolution (Isozaki, Kumada, Okude, Oguso & Goto, 1981) and which have superior 'lag' or 'sticking' performance, that is a short 'memory' for previous high-intensity patterns to which they have been exposed (Shidara, Tanioka, Hirai & Nonaka, 1985).

7.1.6.6. *Some applications*

The use of linear and area detectors has increased markedly in recent years (Arndt, 1988). No new principles for the construction of linear devices have emerged, but more examples of each type have become commercially available.

Many more structures have been determined with area-detector diffractometers. The most commonly used gas-filled detectors at present are the delay-line read out MWPC first described by Xuong *et al.* (1978), as developed by Hamlin (1985), and a detector using a modification of the coded-anode read out due to Burns (Durbin, Burns, Moulai, Metcalf, Freymann, Blum, Anderson, Harrison & Wiley, 1986; Howard, Gilliland, Finzel, Poulos, Ohlendorf & Salemne, 1987; Derewenda & Helliwell, 1989). Corresponding instruments in the USSR and their use have been described by Anisimov, Zanevskii, Ivanov, Morchan, Peshekhonov, Chan Dyk Tkhan, Chan Khyo Dao, Cheremukhina & Chernenko (1986) and by Andrianova, Popov, Kheiker, Zanevskii, Ivanov, Peshekhonov & Chernenko (1986).

A two-dimensional photon-counting X-ray detector has been described by Collett & Podolsky (1988).

The widespread use of area-detector methods in single-crystal studies, especially for macromolecular material, has been greatly aided by the development of complete software packages to deal with all aspects of data collection and handling. Earlier program packages (Howard, Nielson & Xuong, 1985; Pflugrath & Messerschmidt, 1987; Thomas, 1987) tended to be specific to one particular detector and its associated diffractometer. However, following the initiative of Bricogne (1987) and with financial assistance from EEC funds, a group of scientists, including most of the originators of the earlier packages, are now collaborating in writing, extending, and maintaining a comprehensive device-independent position-sensitive-detector software package.

Acknowledgements

Many helpful comments on this article by Drs A. R. Faruqi, H. E. Schwarz, and D. J. Thomas are gratefully acknowledged.

7.1.7. X-ray-sensitive TV cameras (By J. Chikawa)

High-resolution X-ray imaging systems are required for the topographic study of spatial change in crystal structures, such as that which takes place in phase transformations. From this point of view, high-resolution TV camera tubes will be described.

7.1.7.1. *Signal-to-noise ratio*

Video displays of X-ray and optical images have different features. Although X-ray photon energies are very large, the intensities available in X-ray diffraction are extremely low compared with optical images. Therefore, the photon noise resulting from the statistical fluctuation of the number of photons incident upon an image system gives a detection limit of the image.

Consider the case of defects in a crystal viewed by an imaging system: ν_p photons s^{-1} mm^{-2} are diffracted from the perfect region of a crystal, and $q\nu_p$ $(q < 1)$ are absorbed by the X-ray-sensing layer of the system. An absorbed photon produces a mean number η_1 of electrons or visible photons, each of which may be rescattered to produce a mean number η_2 of electrons or photons. By repeating s such processes, the mean signal height

$$S_p = q\nu_p\eta_1\eta_2\ldots\eta_s\delta^2 t \qquad (7.1.7.1)$$

is obtained from each square-shaped picture element $\delta \times \delta$ mm for t s. The value of δ may be taken as the limiting resolution of the system. Since $\eta_1 > 100$ owing to the large photon energy and the values of $\eta_2, \eta_3, \ldots, \eta_s$ are considered to be less than 100, the photon noise σ_p as a standard deviation of S_p is given by (Arcese, 1964)

$$\sigma_p = \eta_s\eta_{s-1}\ldots\eta_1(q\nu_p\delta^2 t)^{1/2}. \qquad (7.1.7.2)$$

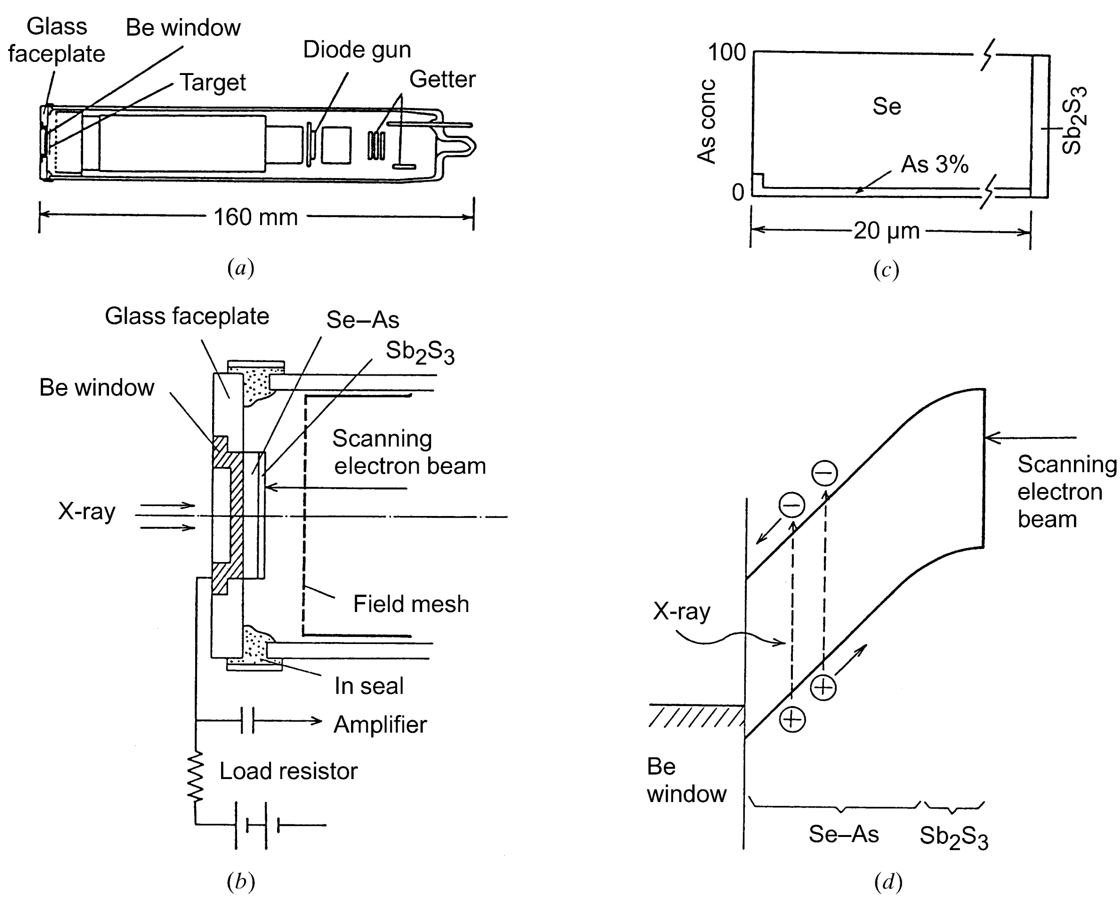

Fig. 7.1.7.1. Schematic illustration of an X-ray sensing Saticon camera tube. (*a*) Schematic representation of the tube. (*b*) Structure of the target. (*c*) Concentration (wt%) of As in the target (Se–As photoconductive layer). Since crystalline Se is metallic, As acts to stabilize the amorphous state. (*d*) Potential in the target. A blocking contact is formed between the X-ray window material and the Se–As alloy layer to prevent holes from flowing into the layer. Incident X-rays form electrons and holes in the layer, and the latter migrate toward the scanning-electron-beam side and contribute to the video signal. On the surface of the Se–As layer, Sb_2S_3 was evaporated to form another blocking contact that improves landing characteristics of the electron beam. By applying a high voltage on the layer, the holes are accelerated to produce multiplication (avalanche amplification), resulting in a great increase of the signal current.

references