International Tables for Crystallography (2018). Vol. H, Section 2.1.7.3, pp. 48–49.

noise levels result in very high dynamic ranges. Notably, in contrast to wire detectors, micro-gap detectors are not likely to be damaged by accidental exposure to a high-intensity direct beam, as a patterned anode plane is used rather than wires.

2.1.7.2.3. *Semiconductor detectors*

Semiconductor (or solid-state) detectors are solid-state ionization devices in which electron–hole pairs instead of electron–ion pairs are generated by incoming photons, and they are sensitive to the entire electromagnetic spectrum from visible light to X-rays. The energy required for production of an electron–hole pair is very low compared to the energy required for production of paired ions in a noble-gas-filled detector. As a consequence, a larger number of charge pairs with a smaller statistical variation are generated in semiconductor detectors, resulting in intrinsically higher energy-resolution capabilities. The efficiency of semiconductor detectors is very high due to the high absorption of the semiconductor materials, usually reaching 100%, but may decline at higher photon energies if the photons are not fully absorbed in the semiconductor *e.g.* because of insufficient thickness.

2.1.7.2.3.1. The Si(Li) detector

The Si(Li) detector sensor consists of a lithium-drifted silicon crystal which must be cooled to prevent lithium diffusion and to reduce dark noise. An important advantage of this detector is its excellent energy resolution of even better than 200 eV (4%) at 8 keV (Cu radiation), allowing very effective filtering of $K\beta$ and fluorescence radiation and thus operation without a metal filter or a diffracted-beam monochromator. As Peltier cooling is sufficient, the Si(Li) detector type has found wide interest for applications benefitting from high energy resolution, unlike energy-dispersive detectors requiring operation under cryogenic conditions [*e.g.* Ge(Li)]. In particular, the Si(Li) detector significantly extends the application range of today's X-ray diffractometers by allowing energy-dispersive X-ray powder diffraction (EDXRD) as well as – to some extent – XRF (see Section 2.1.4.3).

An important disadvantage of Si(Li) detectors is their large dead time, which prohibits the handling of higher count rates. Moderate noise levels result in low-to-moderate dynamic ranges. An additional important disadvantage is the limitation to 0D detection.

2.1.7.2.3.2. Silicon micro-strip and silicon pixel detectors

Silicon micro-strip and silicon pixel detectors employ silicon sensors, which are one- or two-dimensional arrays of p–n diodes in the form of strips or pixels, respectively, individually connected to an array of readout channels. The development of this type of detector technology has obviously been driven by the idea of massive parallelism: each strip or pixel actually represents an individual detector. Accordingly, the silicon micro-strip and silicon pixel detectors are therefore of the 1D and 2D detection type, respectively.

Count rates recorded by silicon micro-strip and silicon pixel detectors are very high with very low noise levels, resulting in very large dynamic ranges. The energy resolution of most silicon micro-strip and silicon pixel detectors is of the order of 1600 eV (20%) at 8 keV (Cu radiation). Recently, a silicon micro-strip detector with an energy resolution of better than 380 eV at 8 keV has been introduced (Wiacek *et al.*, 2015). At such high energy resolution Cu $K\beta$ is filtered out to below the detection limit while Mn, Fe and Co fluorescence is filtered completely, allowing this detector to be operated without a metal filter or a diffracted-beam monochromator for most applications.

2.1.7.2.3.3. CCD and CMOS detectors

Charge-coupled device (CCD) detectors are represented by one- or two-dimensional arrays of square or rectangular pixels consisting of metal–oxide–semiconductor (MOS) capacitors, and can detect X-ray photons directly or indirectly. The pixel size may be less than 10 µm. The majority of detectors use indirect detection, where the incoming X-ray photons are first converted to visible-light photons by a phosphor layer. CCD detectors employ the 'bucket brigade' readout method, in which charge is shifted one pixel at a time by phasing the bias on the gate electrodes that overlay each pixel until it reaches the output, resulting in relatively large readout times ranging from a few tenths of a second up to several seconds per frame. Cooling (Peltier-type) is required to reduce the dark-current noise representing the dominant noise source for long exposures. In some detector designs fibre-optic demagnification is used to increase the effective active detector area, resulting in an imaging area larger than the active area of the CCD chip at the cost of detector sensitivity and spatial resolution.

CCD detectors are usually operated as integrating detectors. As such, they have no dead time and therefore provide excellent linearity over a moderate dynamic range, but cannot have energy resolution. CCD detectors are the detectors of choice for single-crystal diffraction and imaging, but are not favourable for applications with weak diffraction signals, such as powder X-ray diffraction, owing to the relatively large dark-current noise.

CCD detectors may also function as counting detectors by making the exposure time sufficiently short. In single-event mode the energy of each photon can be determined, providing an energy resolution down to about 300 eV at 8 keV (Cu radiation) and allowing a spectrum at each pixel of the CCD array to be built up by a series of consecutive measurements. Such a detector can record energy-dispersive X-ray powder diffraction (EDXRD) as well as – to some extent – XRF (see Section 2.1.4.3); however, owing to the readout time, count rates are extremely low with high statistical noise.

Unlike the bucket-brigade readout of a CCD, the complementary metal–oxide–semiconductor (CMOS) active-pixel sensor (He *et al.*, 2011) uses a completely different architecture in which each pixel incorporates a readout preamplifier and is then read out through a bus, as in random-access memory (He *et al.*, 2011). Cooling is not required. CMOS detectors are immune to the blooming effect (in which a light source overloads the sensitivity of the sensor, causing the signal to bleed vertically into surrounding pixels forming vertical streaks). Additionally, they offer the very significant advantage of shutter-free operation, that is dead-time-free continuous scans which improve the efficiency of data collection and also improve data quality by eliminating shutter-timing jitter.

As a consequence of these characteristics, CMOS-detector active-pixel sensors are now replacing CCD chips in a number of high-end applications (*e.g.* professional digital photography and high-definition television), and have reached a level of performance where they are also starting to displace CCD chips in the most demanding scientific applications.

2.1.7.3. *Position sensitivity and associated scanning modes*

2.1.7.3.1. *Pixel size, spatial resolution and angular resolution*

Detectors of the line (1D) or area (2D) type have the important property of position sensitivity, which is characterized by the two parameters pixel size and spatial resolution.

The pixel size of a position-sensitive detector (PSD) can be represented either by the intrinsic size of the smallest addressable sensitive component of a detector (*e.g.* the actual size of the diodes), which can be binned to form larger pixels, or is set by the readout electronics (*e.g.* for wire-based detectors such as proportional counters). The spatial resolution is determined by the actual pixel size, the point-spread function (PSF) and parallax. The PSF represents the spread of a signal produced by a single photon over several pixels by mapping the probability density that a photon is recorded by a pixel in the vicinity of the point that the photon hit. Parallax will lead to an additional smearing if the photon travels at an angle to the detector normal. The final angular resolution of a detector system is given by the spatial detector resolution and the specimen-to-detector distance.

Point (0D) detectors do not provide position sensitivity, regardless of the actual size of the active window (representing a single pixel). Simply speaking, in analogy to PSDs, the spatial resolution of a point detector is determined by the goniometer step size representing the actual pixel size, and the size of the detector slit representing the PSF. As for PSDs, the angular resolution is given by the spatial resolution and the specimen-to-detector distance.

Detectors can be operated in fixed as well as in (2θ) scanning mode, where the step size is usually determined by the detector pixel size. Subsampling, that is scanning using an angular step size smaller than the angular pixel resolution, may be used to improve observed line profile shapes if the pixel resolution is too small. As a rule of thumb some 5–8 data points need be collected over the FWHM of a diffraction peak to allow for an appropriate description of the line-profile shape.

2.1.7.3.2. *Dimensionality*

Area detectors can be operated as line or point detectors. Electronic binning of the pixels into columns will form a line detector, while binning all pixels together will form a point detector, each associated with improvements of count rates and thus dynamic ranges. Alternatively, 1D or 0D 'regions of interest' can be defined electronically and/or by mounting suitable diffracted-beam-path X-ray optics. Area detectors – when operated as such – require point-focus operation.

Line detectors can be used as point detectors, which may be formed in several ways. One way is to only use one or more central pixels by either electronically switching off outer pixels and/or by mounting suitable X-ray optics. Another way is to turn the detector by 90° and to bin all pixels, leading to an improved count rate and thus dynamic range.

Obviously, when turning a line detector by 90°, it will function as an area detector if it is scanned over an angular range; the trace of the scan will form a cylindrical surface that is a two-dimensional diffraction image (He, 2009). This scan mode may be associated with a few advantages, in addition to lower costs. For example, the elimination of parallax and the possibility of using diffracted-beam-path optics improve the angular resolution in the 2θ direction and allow air scattering to be reduced.

2.1.7.3.3. *Size and shape*

PSDs are available in different sizes with flat (1D, 2D), curved (1D), cylindrical (2D) and spherical (2D) detection surfaces. Curved, cylindrical and spherical detectors are designed for focusing or parallel-beam geometries with a fixed specimen-to-detector distance, and cannot normally be used with the Bragg–Brentano geometry because of its 2θ-dependent focusing circle (Section 2.1.4.1). Flat detectors can be used at different specimen-to-detector distances, with either high angular resolution at a large distance or large angular coverage at a short distance. For large flat detectors, parallax errors must be addressed. Small flat detectors are perfectly suited for operation in Bragg–Brentano geometry but the angular coverage should not exceed about 10° 2θ (Section 2.1.4.1) to minimize defocusing, particularly at small 2θ angles.

References

Bartels, W. J. (1983). *Characterization of thin layers on perfect crystals with a multipurpose high resolution X-ray diffractometer*. *J. Vac. Sci. Technol. B*, **1**, 338–345.

Bilderback, D. H. (2003). *Review of capillary X-ray optics from the 2nd International Capillary Optics Meeting*. *X-ray Spectrom.* **32**, 195–207.

Bohlin, H. (1920). *Eine neue Anordnung für röntgenkristallographische Untersuchungen von Kristallpulver*. *Ann. Phys.* **366**, 421–439.

Bonse, U. & Hart, M. (1965). *Tailless X-ray single crystal reflection curves obtained by multiple reflection*. *Appl. Phys. Lett.* **7**, 238–240.

Bowen, D. K. & Tanner, B. K. (1998). *High Resolution X-ray Diffractometry and Topography*. London: Taylor & Francis.

Brentano, J. C. M. (1924). *Focussing method of crystal powder analysis by X-rays*. *Proc. Phys. Soc.* **37**, 184–193.

Charpak, G., Bouclier, R., Bressani, T., Favier, J. & Zupančič, Č. (1968). *The use of multiwire proportional counters to select and localize charged particles*. *Nucl. Instrum. Methods*, **62**, 262–268.

Clearfield, A., Reibenspiess, J. & Bhuvanesh, N. (2008). *Principles and Applications of Powder Diffraction*. New York: Wiley.

Debye, P. & Scherrer, P. (1916). *Interference of X-rays, employing amorphous substances*. *Phys. Z.* **17**, 277–283.

Durst, R. D., Diawara, Y., Khazins, D. M., Medved, S., Becker, B. L. & Thorson, T. A. (2003). *Novel, photon counting X-ray detectors*. *Powder Diffr.* **18**, 103–105.

EN 1330–11 (2007). *Non-Destructive Testing*. Part 11. *Terms used in X-ray Diffraction from Polycrystalline and Amorphous Materials*. Brussels: European Committee for Standardization (CEN).

Fankuchen, I. (1937). *A condensing monochromator for X-rays*. *Nature (London)*, **139**, 193–194.

Fewster, P. F. (2003). *X-ray Scattering from Semiconductors*. London: Imperial College Press.

Friedmann, H. (1945). *Geiger counter spectrometer for industrial research*. *Electronics*, **18**, 132–137.

Göbel, H. E. (1980). *The use and accuracy of continuously scanning position-sensitive detector data in X-ray powder diffraction*. *Adv. X-ray Anal.* **24**, 123–138.

Guinier, A. (1937). *Arrangement for obtaining intense diffraction diagrams of crystalline powders with monochromatic radiation*. *C. R. Acad. Sci. Paris*, **204**, 1115–1116.

Hanawalt, J. D., Rinn, H. W. & Frevel, L. K. (1938). *Chemical analysis by X-ray diffraction*. *Ind. Eng. Chem. Anal.* **10**, 457–512.

Hart, M. (1971). *Bragg-reflection X-ray optics*. *Rep. Prog. Phys.* **34**, 435–490.

He, B. B. (2009). *Two-Dimensional X-ray Diffraction*. New York: Wiley.

He, T., Durst, R. D., Becker, B. L., Kaercher, J. & Wachter, G. (2011). *A large area X-ray imager with online linearization and noise suppression*. *Proc. SPIE*, **8142**, 81421Q.

Hemberg, O. E., Otendal, M. & Hertz, H. M. (2003). *Liquid-metal-jet anode electron-impact X-ray source*. *Appl. Phys. Lett.* **83**, 1483–1485.

Hull, A. W. (1917). *A new method of X-ray crystal analysis*. *Phys. Rev.* **10**, 661–696.

Hull, A. W. (1919). *A new method of chemical analysis*. *J. Am. Chem. Soc.* **41**, 1168–1175.

International Tables for Crystallography (2004). Volume C, 3rd ed., edited by E. Prince. Dordrecht: Kluwer Academic Publishers.