International Tables for Crystallography (2006). Vol. F, Section 7.2.3.1, p. 149.

Fibre optics and lenses. Light transmission in image reduction is limited by n.a. $\times M^2$, where n.a. is the numerical aperture of the optical system and M is the linear magnification factor ($M < 1$ corresponds to reduction). Lens systems have low values of n.a. when used in reduction, so there is only 2% light transmission for a 3:1 reduction with an f/1.0 lens. With a much higher n.a., fibre optics can typically transmit 13% with the same 3:1 reduction (Coleman, 1985). Such reducing tapers are produced by locally heating, then pulling, a fused bundle of optical fibres. Each fibre within the bundle becomes tapered in this process, thereby reducing the image scale from front to back of the bundle. The bundle structure introduces a characteristic 'chicken-wire' pattern into the image which must be removed *via* intensity calibration (Section 7.2.3). Tapers up to 165 mm in diameter are available.

To obtain good resolution, extramural absorbing fibres (EMAs) must be placed in the fibre optic to absorb light that propagates between fibres. These EMAs are often a more effective absorber in the blue part of the spectrum, with the result that a red-emitting phosphor yields somewhat poorer resolution. Another concern with fibre optics is radioactivity in the glass used to produce the optic, which manifests itself as random flashes ('zingers') in both the phosphor and the CCD (Section 7.2.3).

Although fibre-optic coupling is preferred in many situations, lenses are appropriate for use with image intensification, or in cases of image magnification, such as for microtomography, using high-resolution screens.

Image intensifiers allow large demagnification ratios without undue DQE loss (Moy, 1994). In these vacuum-tube devices, visible light produces photoelectrons in the photocathode of the intensifier, which in turn are accelerated under high potential onto a secondary phosphor screen. Each photoelectron excites many photons in the secondary phosphor, giving light amplification. Image resolution is retained either by magnetic, electrostatic or proximity focusing of the electrons. In the case of microchannel plate intensifiers, photoelectrons are restricted to cascade down hollow fibres. Intensifiers often have problems with stability and linearity, and they degrade resolution. Phosphor afterglow in the secondary phosphors is also a consideration. Intensifiers typically limit the input format to less than 80 mm in diameter, although one design uses a large (230 mm) radiographic intensifier (Moy, 1994; Hammersley *et al.*, 1997). The greatest drawbacks of image intensifiers are cost, availability, susceptibility to magnetic fields and lack of robustness.

CCDs. The low noise and high sensitivity of scientific CCDs have made CCDs the preferred imaging device in X-ray applications. Visible photons are converted to charge carriers in the silicon of the CCD, with a 30–40% quantum efficiency (q.e.) in the red, dropping to 5% in the blue owing to increased absorption by the controlling gate structure (the q.e. can be considerably higher for back-illuminated devices which, however, are expensive, fragile and difficult to obtain). The 'read noise' (noise with zero charge in the pixel) is routinely less than 10 electrons for scientific grade chips with pixel read rates of less than 1 MHz. When coupled to a good phosphor screen with a modest (*circa* 3:1) fibre-optic taper, signal levels in the CCD are typically 10–30 recorded electrons per 10 keV X-ray. This is above the CCD read-noise level, thereby allowing low-dose quantum-limited X-ray imaging.

A physically large CCD chip is usually desired to maximize the detector area, given the limited image reduction ratio. Devices from 2–4 cm on an edge with 1000×1000 to 2000×2000 pixels are available. These sizes match well to the largest of the fibre-optic tapers and are therefore most often used in detectors. Larger-format CCDs are available, but are harder to obtain and are expensive.

CCDs are usually cooled well below room temperature to minimize thermally generated dark current, an unwanted source of noise. The dark current drops by a factor of 2 for every 5–7 K drop in temperature. At 233 K, there is roughly one electron per pixel per second dark current for normal clocking operation. Since the dark current changes with temperature, the temperature must be well regulated (to within ± 0.1 K) to subtract the dark charge from an image reproducibly.

Most of the dark current comes from surface defects, not from the bulk silicon. Multiphase pinned (MPP) CCDs use charge implants within the pixel structure to move the charge collection region away from the surface, thereby reducing the dark current by several orders of magnitude. This is usually accompanied by a significant reduction in the maximum charge capacity ('full well') of a pixel. Even so, MPP CCDs are becoming the norm in most X-ray detectors.

Directly exposed CCDs. CCDs can directly image X-rays, although typical CCDs are not very efficient, since the charge collection region in the silicon is very thin (< 10 μm). Thicker depletion regions can be fabricated in high-resistivity silicon wafers, improving X-ray collection efficiency to greater than 30% at 8 keV. Direct conversion of X-rays in silicon produces a large signal with excellent energy resolution. It is therefore possible to use the chip as an X-ray counting detector with the ability to discriminate in energy. To retain X-ray-energy measurement capability, however, there must be less than one X-ray per pixel per frame to avoid signal overlap. This requires a fast readout as well as large amounts of disk storage to handle the large number of files. The X-rays damage the CCD's electronic structure, resulting in a higher dark current within the exposed pixels. Care must be taken to shield non-imaging parts of the CCD, such as the output amplifier, which will adversely affect chip performance at even lower radiation dose.

7.2.3. Calibration and correction

Inhomogeneities within the detector components introduce non-uniformities in the output image of several per cent or more, both as geometric distortion and as nonuniformity of response. The response of the system varies not only with position, but also with the angle of incidence and X-ray energy. Optimal calibration of the detector should take into account the parameters of the X-ray experiment, seeking to mimic the experimental conditions as closely as possible: a uniform source of X-rays of the proper energy positioned in place of the diffracting crystal would be ideal. Realizing such a source is somewhat problematic, so the calibration procedure is often broken down into several independent steps. Calibration procedures are detailed in Barna *et al.* (1999) and are summarized below.

7.2.3.1. *Dark-current subtraction*

It is important to remove both the electronic offset and the accumulated dark charge from an X-ray image. Since the integrated dark current varies from pixel to pixel and with time, a set of images needs to be taken (with no X-rays), matched in integration time to the X-ray exposures. With a properly temperature-stabilized detector, the background images may be acquired in advance and used throughout an experiment. Because the background image has noise, it is common to average a number of separate backgrounds to minimize the noise.

7.2.3.2. *Removal of radioactive decay events*

Cosmic rays and radioactive decay of actinides in the fibre-optic glass produce large-amplitude isolated signals ('zingers') within an X-ray image. These accumulate randomly in position and in time. For the short exposures typical at synchrotron sources and for data sets with highly redundant information, the few diffraction spots

references