

2. INSTRUMENTATION AND SAMPLE PREPARATION

1999; Durst *et al.*, 2002; Blanton, 2003; Khazins *et al.*, 2004). X-ray photographic plates and films were the first generation of two-dimensional X-ray detectors. Now, multiwire proportional counters (MWPCs), image plates (IPs), charge-coupled devices (CCDs) and microgap detectors are the most commonly used large area detectors. Recent developments in area detectors include X-ray pixel array detectors (PADs), silicon drift diodes (SDDs) and complementary metal-oxide semiconductor (CMOS) detectors (Ercan *et al.*, 2006; Lutz, 2006; Yagi & Inoue, 2007; He *et al.*, 2011). Each detector type has its advantages over the other types. In order to make the right choice of area detector for a 2D-XRD system and applications, it is necessary to characterize area detectors with consistent and comparable parameters. Chapter 2.1 has more comprehensive coverage on X-ray detectors, including area detectors. This section will cover the characteristics specifically relevant to area detectors.

2.5.3.2.1. Active area and pixel size

A 2D detector has a limited detection surface and the detection surface can be spherical, cylindrical or flat. The detection-surface shape is also determined by the detector technology. For example, a CCD detector is made from a large semiconductor wafer, so that only a flat CCD is available, while an image plate is flexible so that it is easily bent to a cylindrical shape. The area of the detection surface, also referred to as the active area, is one of the most important parameters of a 2D detector. The larger the active area of a detector, the larger the solid angle that can be covered at the same sample-to-detector distance. This is especially important when the instrumentation or sample size forbid a short sample-to-detector distance. The active area is also limited by the detector technology. For instance, the active area of a CCD detector is limited by the semiconductor wafer size and fabrication facility. A large active area can be achieved by using a large demagnification optical lens or fibre-optical lens. Stacking several CCD chips side-by-side to build a so-called mosaic CCD detector is another way to achieve a large active area.

In addition to the active area, the overall weight and dimensions are also very important factors in the performance of a 2D detector. The weight of the detector has to be supported by the goniometer, so a heavy detector means high demands on the size and power of the goniometer. In a vertical configuration, a heavy detector also requires a heavy counterweight to balance the driving gear. The overall dimensions of a 2D detector include the height, width and depth. These dimensions determine the manoeuvrability of the detector within a diffractometer, especially when a diffractometer is loaded with many accessories, such as a video microscope and sample-loading mechanism. Another important parameter of a 2D detector that tends to be ignored by most users is the blank margin surrounding the active area of the detector. Fig. 2.5.10 shows the relationship between the maximum measurable 2θ angle and the detector blank margin. For high 2θ angle measurements, the detector swing angle is set so that the incident X-ray optics are set as closely as possible to the detector. The unmeasurable blank angle is the sum of the detector margin m and the dimension from the incident X-ray beam to the outer surface of the optic device h . The maximum measurable angle is given by

$$2\theta_{\max} = \pi - \frac{m+h}{D}. \quad (2.5.16)$$

It can be seen that either reducing the detector blank margin or optics blank margin can increase the maximum measurable angle.

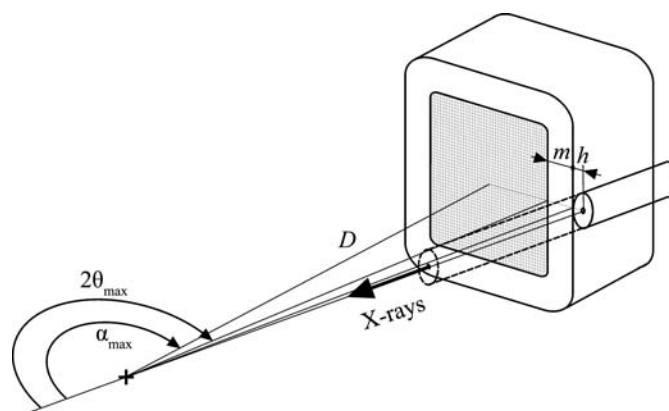


Figure 2.5.10
Detector dimensions and maximum measurable 2θ .

The solid angle covered by a pixel in a flat detector is dependent on the sample-to-detector distance and the location of the pixel in the detector. Fig. 2.5.11 illustrates the relationship between the solid angle covered by a pixel and its location in a flat area detector. The symbol S may represent a sample or a calibration source at the instrument centre. The distance between the sample S and the detector is D . The distance between any arbitrary pixel $P(x, y)$ and the detector centre pixel $P(0, 0)$ is r . The pixel size is Δx and Δy (assuming $\Delta x = \Delta y$). The distance between the sample S and the pixel is R . The angular ranges covered by this pixel are $\Delta\alpha$ and $\Delta\beta$ in the x and y directions, respectively. The solid angle covered by this pixel, $\Delta\Omega$, is then given as

$$\Delta\Omega = \Delta\alpha\Delta\beta = \frac{D}{R^3}\Delta y\Delta x = \frac{D}{R^3}\Delta A, \quad (2.5.17)$$

where $\Delta A = \Delta x\Delta y$ is the area of the pixel and R is given by

$$R = (D^2 + x^2 + y^2)^{1/2} = (D^2 + r^2)^{1/2}. \quad (2.5.18)$$

When a homogeneous calibration source is used, the flux to a pixel at $P(x, y)$ is given as

$$F(x, y) = \Delta\Omega B = \frac{\Delta ADB}{R^3} = \frac{\Delta ADB}{(D^2 + x^2 + y^2)^{3/2}}, \quad (2.5.19)$$

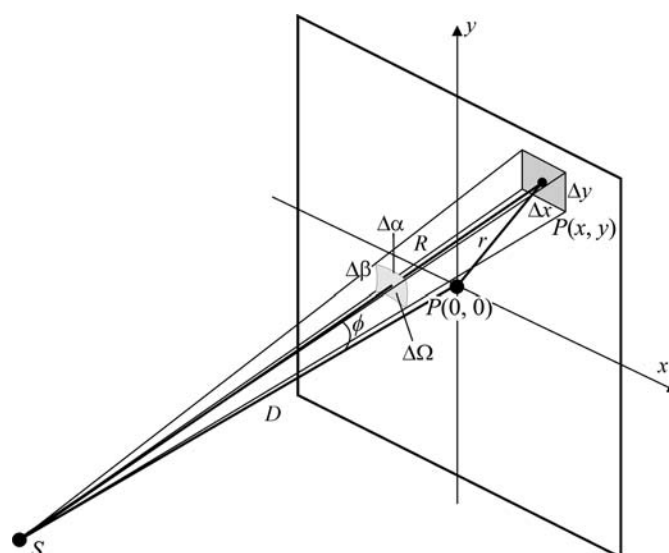


Figure 2.5.11
Solid angle covered by each pixel and its location on the detector.