

## 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.

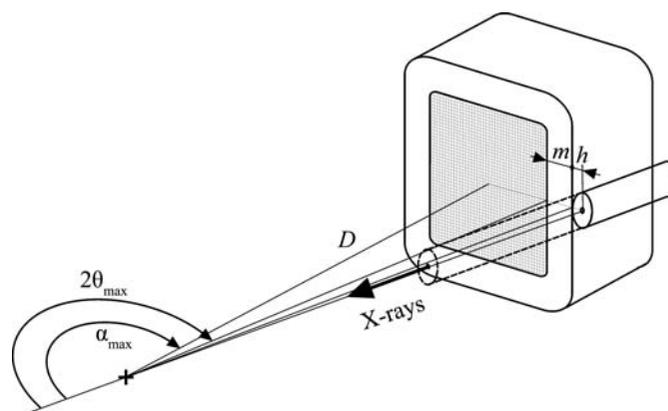
## 2.5.3.2.1. Active area and pixel size

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  $2\theta$  angle and the detector blank margin. For high  $2\theta$  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  $2\theta$ .

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  $\Delta x$  and  $\Delta y$  (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

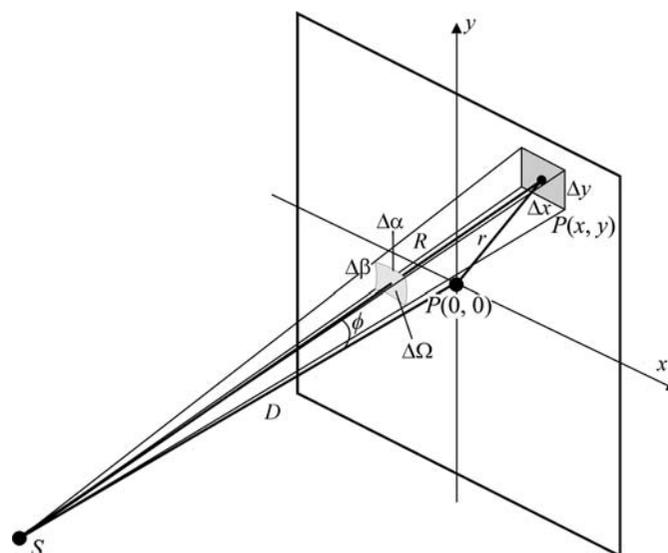
$$\Delta\Omega = \Delta\alpha\Delta\beta = \frac{D}{R^3}\Delta y\Delta x = \frac{D}{R^3}\Delta A, \quad (2.5.17)$$

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.