

2. INSTRUMENTATION AND SAMPLE PREPARATION

energy range of a detector. The DQE drops significantly if a detector is used out of its energy range. For instance, the energy range of MWPC and microgap detectors is about 3 to 15 keV. The DQE with Cu $K\alpha$ radiation (8.06 keV) is about 80%, but drops gradually when approaching the lower or higher energy limits. The energy range of imaging plates is much wider (4–48 keV). The energy range of a CCD, depending on the phosphor, covers from 5 keV up to the hard X-ray region.

2.5.3.2.4. Detection limit and dynamic range

The detection limit is the lowest number of counts that can be distinguished from the absence of true counts within a specified confidence level. The detection limit is estimated from the mean of the noise, the standard deviation of the noise and some confidence factor. In order to have the incoming X-ray photons counted with a reasonable statistical certainty, the counts produced by the X-ray photons should be above the detector background-noise counts.

The dynamic range is defined as the range extending from the detection limit to the maximum count measured in the same length of counting time. The linear dynamic range is the dynamic range within which the maximum counts are collected within the specified linearity. For X-ray detectors, the dynamic range most often refers to linear dynamic range, since only a diffraction pattern collected within the linear dynamic range can be correctly interpreted and analysed. When the detection limit in count rate approaches the noise rate at extended counting time, the dynamic range can be approximated by the ratio of the maximum count rate to the noise rate.

Dynamic range is very often confused with the maximum count rate, but must be distinguished. With a low noise rate, a detector can achieve a dynamic range much higher than its count rate. For example, if a detector has a maximum linear count rate of 10^5 s^{-1} with a noise rate of 10^{-3} s^{-1} , the dynamic range can approach 10^8 for an extended measurement time. The dynamic range for a 2D detector has the same definition as for a point detector, except that with a 2D detector the whole dynamic range extending from the detection limit to the maximum count can be observed from different pixels simultaneously. In order to record the entire two-dimensional diffraction pattern, it is necessary for the dynamic range of the detector to be at least the dynamic range of the diffraction pattern, which is typically in the range 10^2 to 10^6 for most applications. If the range of reflection intensities exceeds the dynamic range of the detector, then the detector will either saturate or have low-intensity patterns truncated. Therefore, it is desirable that the detector has as large a dynamic range as possible.

2.5.3.2.5. Types of 2D detectors

2D detectors can be classified into two broad categories: photon-counting detectors and integrating detectors (Lewis, 1994). Photon-counting area detectors can detect a single X-ray photon entering the active area. In a photon-counting detector, each X-ray photon is absorbed and converted to an electrical pulse. The number of pulses counted per unit time is proportional to the incident X-ray flux. Photon-counting detectors typically have high counting efficiency, approaching 100% at low count rate. The most commonly used photon-counting 2D detectors include MWPCs, Si-pixel arrays and microgap detectors. Integrating area detectors, also referred to as analogue X-ray imagers, record the X-ray intensity by measuring the analogue electrical signals converted from the incoming X-ray flux. The

signal size of each pixel is proportional to the fluence of incident X-rays. The most commonly used integrating 2D detectors include image plates (IPs) and charge-coupled devices (CCDs).

The selection of an appropriate 2D detector depends on the X-ray diffraction application, the sample condition and the X-ray beam intensity. In addition to geometry features, such as the active area and pixel format, the most important performance characteristics of a detector are its sensitivity, dynamic range, spatial resolution and background noise. The detector type, either photon-counting or integrating, also leads to important differences in performance. Photon-counting 2D detectors typically have high counting efficiency at low count rate, while integrating 2D detectors are not so efficient at low count rate because of the relatively high noise background. An MWPC has a high DQE of about 0.8 when exposed to incoming local fluence from single photons up to about $10^3 \text{ photons s}^{-1} \text{ mm}^{-2}$. The diffracted X-ray intensities from a polycrystalline or powder sample with a typical laboratory X-ray source fall into this fluence range. This is especially true with microdiffraction, where high sensitivity and low noise are crucial to reveal the weak diffraction pattern. Owing to the counting losses at a high count rate, the DQE of an MWPC decreases with increasing count rate and quickly saturates above $10^3 \text{ photons s}^{-1} \text{ mm}^{-2}$. Therefore, an MWPC is not suitable for collecting strong diffraction patterns or for use with high intensity sources, such as synchrotron X-ray sources. An IP can be used in a large fluence range from $10 \text{ photons s}^{-1} \text{ mm}^{-2}$ and up with a DQE of 0.2 or lower. An IP is suitable for strong diffraction from single crystals with high-intensity X-ray sources, such as a rotating-anode generator or synchrotron X-ray source. With weak diffraction signals, the image plate cannot resolve the diffraction data near the noise floor. A CCD detector can also be used over a large X-ray fluence range from $10 \text{ photons s}^{-1} \text{ mm}^{-2}$ to very high fluence with a much higher DQE of 0.7 or higher. It is suitable for collecting diffraction of medium to strong intensity from single-crystal or polycrystalline samples. Owing to the relatively high sensitivity and high local count rate, CCDs can be used in systems with either sealed-tube X-ray sources, rotating-anode generators or synchrotron X-ray sources. With a low DQE at low fluence and the presence of dark-current noise, a CCD is not a good choice for applications with weak diffraction signals. A microgap detector has the best combination of high DQE, low noise and high count rate. It has a DQE of about 0.8 at an X-ray fluence from single photons up to about $10^5 \text{ photons s}^{-1} \text{ mm}^2$. It is suitable for microdiffraction when high sensitivity and low noise are crucial to reveal weak diffraction patterns. It can also handle high X-ray fluence from strong diffraction patterns or be used with high-intensity sources, such as rotating-anode generators or synchrotron X-ray sources.

2.5.3.3. Data corrections and integration

2D diffraction patterns contain abundant information. In order to interpret and analyse 2D patterns accurately it is necessary to apply some data-treatment processes (Sulyanov *et al.*, 1994; Scheidegger *et al.*, 2000; Cervellino *et al.*, 2006; Boesecke, 2007; Rowe, 2009). Most data-treatment processes can be categorized as having one of the following four purposes: to eliminate or reduce errors caused by detector defects; to remove undesirable effects of instrument and sample geometry; to transfer a 2D frame into a format such that the data can be presented or further analysed by conventional means and software; and cosmetic treatment, such as smoothing a frame for reports and publications.