

2. INSTRUMENTATION AND SAMPLE PREPARATION

2.5.3. Instrumentation

2.5.3.1. X-ray source and optics

2.5.3.1.1. Beam path in a diffractometer equipped with a 2D detector

The Bragg–Brentano (B-B) parafocusing geometry is most commonly used in conventional X-ray diffractometers with a point detector (Cullity, 1978; Jenkins & Snyder, 1996). In the Bragg–Brentano geometry, the sample surface normal is always a bisector between the incident beam and the diffracted beam. A divergent incident beam hits the sample surface with an incident angle θ . The area of the irradiated region depends on the incident angle θ and the size of the divergence slit. The diffracted rays leave the sample at an angle 2θ , pass through the anti-scatter slit and receiving slit, and reach the point detector. Soller slits are used on both the primary side and secondary side to minimize the effects of axial divergence due to the line-focus beam. The primary line-focus beam sliced by the Soller slits can also be considered as an array of point beams parallel to the diffractometer planes. Each of these point beams will produce a diffraction cone from the sample. The overlap of all the diffraction cones will create a smeared diffraction peak. The Soller slits on the receiving side allow only those diffracted beams nearly parallel to the diffractometer plane to pass through, so the smearing effect is minimized. In another words, the so-called ‘line-focus geometry’ in conventional diffractometry is actually a superposition of many layers of ‘spot-focus geometry’.

The beam path in a diffractometer equipped with a 2D detector is different from that in a conventional diffractometer in many respects (He & Preckwinkel, 2002). In a 2D-XRD system the whole or a large portion of the diffraction rings are measured simultaneously, and neither slits nor monochromator can be used between the sample and detector. Therefore, the X-ray source and optics for 2D-XRD systems have different requirements in terms of the beam spectral purity, divergence and beam cross-section profile. Fig. 2.5.9 shows the beam path in a 2D-XRD system with the θ – θ configuration. The geometry for the θ – 2θ configuration is equivalent. The X-ray tube, monochromator and collimator assembly are all mounted on the primary side. The incident-beam assembly rotates about the instrument centre and makes an incident angle θ_1 to the sample surface. The first main axis is also called the θ_1 axis. The diffracted beams travel in all directions and some are intercepted by a 2D detector. The detector is mounted on the other main axis, θ_2 . The detector position is determined by the sample-to-detector distance D and the detector swing angle α ($= \theta_1 + \theta_2$).

All the components and space between the focal spot of the X-ray tube and sample are collectively referred to as the primary beam path. The primary beam path in a 2D-XRD system is typically sheltered by optical components except between the exit of the collimator and the sample. The X-rays travelling through this open incident-beam path are scattered by the air with two adverse effects. One is the attenuation of the primary beam intensity. The more harmful effect is that the scattered X-rays travel in all directions and some reach the detector, as is shown by the dashed lines with arrows in Fig. 2.5.9. This air scatter introduces a background over the diffraction pattern. Weak diffraction patterns may be buried under the background. Obviously, the air scatter from the incident beam is significantly stronger than that from diffracted X-rays. The intensity of the air scatter from the incident beam is proportional to the length of the open incident-beam path. The effect of air scatter also depends on the wavelength of the X-rays. The longer the wavelength is,

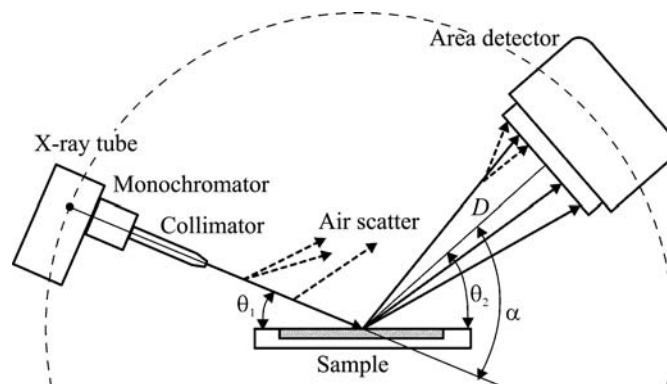


Figure 2.5.9
X-ray beam path in a two-dimensional X-ray diffraction system.

the more severe is the air scatter. The secondary beam path is the space between the sample and the 2D detector. The diffracted X-rays are also scattered by air and the diffraction pattern is both attenuated and blurred by the air scattering. In a conventional diffractometer, one can use an anti-scatter slit, diffracted-beam monochromator or detector Soller slits to remove most of the air scatter that is not travelling in the diffracted-beam direction. These measures cannot be used for a 2D-XRD system, which requires an open space between the sample and the 2D detector. Therefore, the open incident-beam path should be kept as small as possible. In order to reduce the air attenuation and air scatter of the incident beam, a helium-purged beam path or a vacuum beam path are sometimes used in a diffractometer. The air scatter from the diffracted X-rays is relatively weak and the effect depends on sample-to-detector distance. It is typically not necessary to take measures to remove air scatter from the diffracted X-rays between the sample and 2D detector if the sample-to-detector distance is 30 cm or less with Cu $K\alpha$ radiation. However, if the sample-to-detector distance is larger than 30 cm or longer-wavelength radiation, such as Co $K\alpha$ or Cr $K\alpha$, is used, it is then necessary to use an He beam path or vacuum beam path to reduce the air scatter.

2.5.3.1.2. Liouville's theorem

Liouville's theorem can be used to describe the nature of the X-ray source, the X-ray optics and the coupling of the source and optics (Arndt, 1990). Liouville's theorem can be stated in a variety of ways, but for X-ray optics the best known form is

$$S_1\alpha = S_2\beta, \quad (2.5.14)$$

where S_1 is the effective size of the X-ray source and α is the capture angle determined by the effective size of the X-ray optics and the distance between the source and optics. S_2 is the size of the image focus. β is the convergence angle of the X-ray beam from the optics, which is also determined by the effective size of the X-ray optics and the distance between the optics and the image focus. The β angle is also called the crossfire of the X-ray beam. S_2 and β are typically determined by experimental requirements such as beam size and divergence. Therefore, the product $S_1\alpha$ is also determined by experimental conditions. In another expression of Liouville's theorem, the space volume containing the X-ray photons cannot be reduced with time along the trajectories of the system. Therefore, the brilliance of an X-ray source cannot be increased by optics, but may be reduced because of the loss of X-ray photons passing through the optics. In practice, no optics can have 100% reflectivity or transmission.