

3.1. OPTICS AND ALIGNMENT OF THE LABORATORY DIFFRACTOMETER

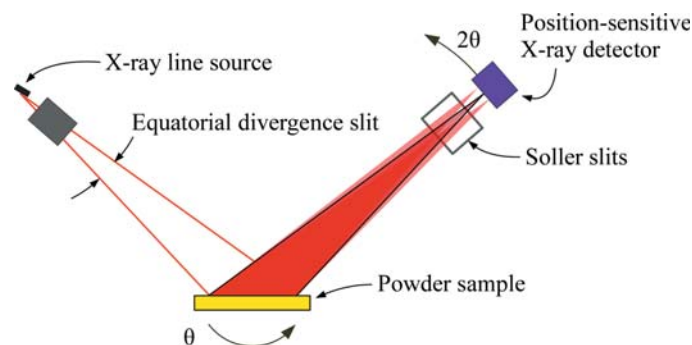


Figure 3.1.2

A schematic diagram illustrating the operation and optical components of a Bragg-Brentano X-ray diffractometer equipped with a position-sensitive detector. Only the rays striking the centre line of the PSD, outlined in black, are in accordance with Bragg-Brentano focusing.

specimen position are critical for the correct interpretation of the data. The goniometer radius is the distance between the rotation axes and the X-ray source (R_1), or the distance between the rotation axes and receiving slit (R_2), as shown in Fig. 3.1.1; these two distances must be equal. The specimen surface is presumed to be on the rotation axes; however, this condition is rarely realized and it is common to have to consider a specimen-displacement error.

Goniometer assemblies themselves can be set up in several configurations. Invariably, two rotation stages are utilized. Fig. 3.1.1 illustrates a machine of $\theta/2\theta$ geometry: the tube is stationary while one stage rotates the specimen through angle θ , sometimes referred to as the angle Ω , while a second stage rotates the detector through angle 2θ . Another popular configuration is θ/θ geometry, where the specimen remains stationary and both the tube and detector rotate through angle θ . However, the diffraction optics themselves do not vary with regard to how the goniometer is set up.

The detector illustrated in Fig. 3.1.1 simply reads any photons arriving at its entrance window as the diffracted signal is analysed by the receiving slit. Such detectors, which often use a scintillation crystal, are typically referred to as point detectors. A diffracted-beam post-sample monochromator is often added to the beam path after the receiving slit to filter out any fluorescence from the sample. The crystal optic of these monochromators typically consists of pyrolytic graphite with a high level of mosaicity that is bent to a radius in rough correspondence to that of the goniometer. This imposes a relatively broad energy bandpass of approximately 200 eV (with 8 keV Cu $K\alpha$ radiation) in width on the diffracted beam. This window is centred so as to

straddle that of the energy of the source radiation being used, thereby filtering fluorescent and other spurious radiation from the detector while transmitting the primary features of the emission spectrum, presumably without distortion.

Within the last decade, however, the popularity of this geometry has fallen markedly, as the use of the post-sample monochromator/point-detector assembly has been largely displaced by the use of a position-sensitive detector (PSD). This geometry is illustrated in Fig. 3.1.2. A line detector replaces the point detector, and offers the ability to discriminate with respect to the position of arriving X-rays within the entrance window of the PSD. A multichannel analyser is typically used to map the arriving photons from the PSD window into 2θ space. Depending on the size of the PSD entrance window, increases in the counting rate by two orders of magnitude relative to a point detector can be easily achieved. Furthermore, this is accomplished by including the signal from additional crystallites, mitigating any problems with particle-counting statistics (Fig. 3.1.2). A drawback to the PSD is that the increased intensity is achieved with the inclusion of signals that are not within the Bragg-Brentano focusing regimen (compare Figs. 3.1.1 and 3.1.2), leading to a broadening of the line profiles. The level of broadening is proportional to the size of the PSD entrance window and inversely proportional to 2θ angle. The move to PSDs has been further augmented by the development of solid-state, silicon strip detectors that offer the advantages of a PSD without the maintenance issues of the early gas-flow proportional PSDs. Fluorescence can be problematic with a PSD; however, the problem can be countered with the use of filters. More recent developments in electronics have improved the ability of these PSDs to discriminate with respect to energy. We discuss only this newer class of solid-state linear PSDs in this chapter.

A monochromator can also be used to condition the incident beam so that it will consist exclusively of $K\alpha_1$ radiation. Monochromators of this nature are inserted into the beam path prior to the beam's arrival at the incident-beam slit shown in Fig. 3.1.1. These devices typically use a Ge(111) crystal as the optic; Ge monochromators have a much smaller energy bandpass than graphite monochromators. They are, therefore, much more complex and difficult to align. Here we discuss an incident-beam monochromator (IBM) using a Johansson focusing optic (Johansson, 1933), as shown in Fig. 3.1.3. When incorporating an IBM assembly into a powder diffractometer using reflection geometry, the focal line of the optic must be positioned on the goniometer radius as per the line source of the tube anode in a conventional setup, shown in the right-hand

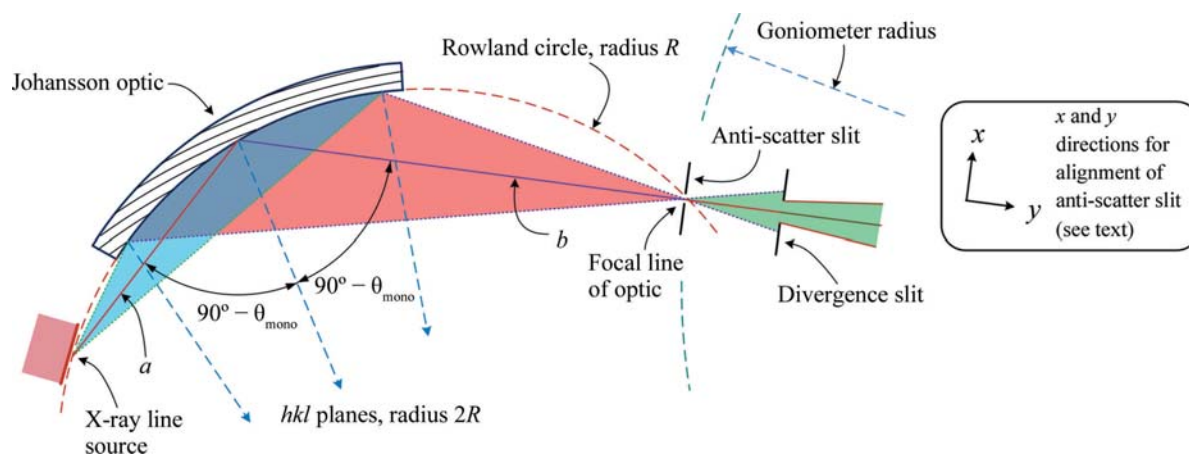


Figure 3.1.3

A schematic diagram illustrating the geometry of a Johansson incident-beam monochromator.