International Tables for Crystallography (2018). Vol. H, Section 2.1.4.1, pp. 28–30.

Brentano geometry, and for thin-film analysis, usually based on the Debye–Scherrer geometry.

2.1.3.2. *Recent years*

In the 1990s, more and more laboratories started to deal with a full range of materials and related applications - from powders through polycrystalline thin films to epitaxial thin films. Dedicated and inflexible instruments were no longer economic for serving the increasing range of applications and also their increasing data-quality requirements.

The growing need for multipurpose instrumentation led to a new generation of X-ray diffractometers in the late 1990s, from all of the major manufacturers, based on a platform concept covering all relevant beam-path components including X-ray sources, optics, specimen stages and detectors. This concept, described in Section 2.1.4, allowed for a faster development of more and more differentiated instrumentation to optimally meet the requirements of all possible applications and sample types. Particularly successful were design improvements that allow the user to transform an instrument on-site by changing beam-path components, often without any need for alignment or even tools, to cover a larger range of applications and sample types using a single instrument.

A major contribution to the platform concept came from the continued development of beam conditioners based on multilayers, resulting in a wealth of X-ray beam optics for different applications. Advanced sputtering techniques allow the fabrication of multilayer optics with virtually arbitrary beam divergence, which can be used to generate focusing, parallel and divergent beams for both point- and line-focus applications.

The introduction of a series of new detector technologies in the early 2000s represented another technological quantum leap, which completely changed the X-ray detection landscape for laboratory diffraction. Within only a few years, detectors based on silicon micro-strip, silicon pixel and micro-gap technologies reached a market share of more than 90% in newly sold systems. Proportional and scintillation point detectors will probably become obsolete in only a few years from now, but can still be found, usually in lower-budget systems.

Today's instruments, with their different possible configurations of beam-path components, are now capable of performing a wider range of X-ray scattering applications than ever (see Section 2.1.4.3). Not surprisingly, the platform concept has become so successful that all modern X-ray diffractometers are now, at least to some extent, equipped with interchange capabilities for beam-path components. However, the fundamental principles remain the same and date back to the first film cameras and diffractometers, no matter how advanced today's instrumentation is.

2.1.4. The platform concept – fitting the instrument to the need

Modern X-ray diffractometers are highly modular assembly systems based on a platform concept, with a shared set of major components over a number of distinct diffractometer models, serving different X-ray scattering application areas. Such a platform concept has two important advantages. Firstly, a common design allows differentiated instruments to be developed faster, and eases the integration of new or improved beam-path components, potentially over the whole model range. Secondly, it enables the design of an X-ray optical bench with on-site interchange capabilities, allowing the mounting of selected beam-path components to meet specific application and specimen-property requirements.

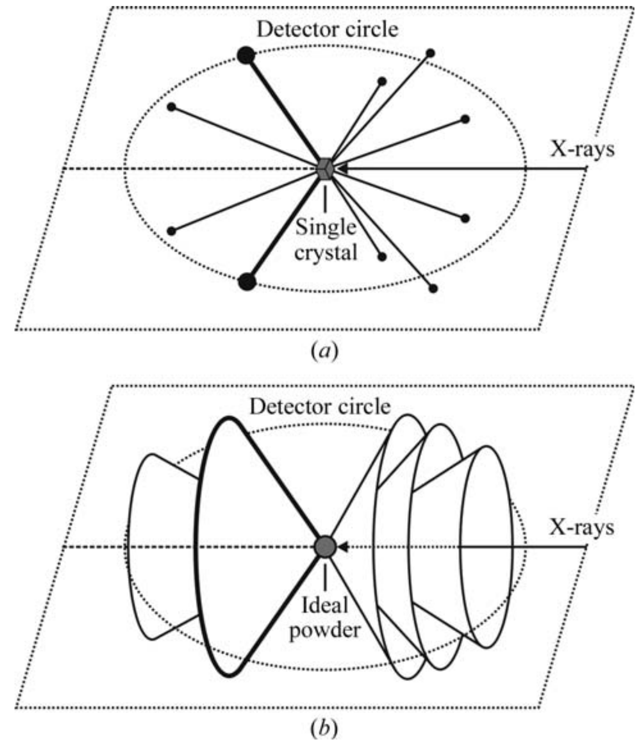

Figure 2.1.1
Diffraction of X-rays by (*a*) a rotating single crystal and (*b*) an ideal powder. The scattered intensity may be measured by a detector placed on the detector circle.

2.1.4.1. *Basic design principles and instrument geometry considerations*

X-ray scattering data are generally recorded in what is virtually the simplest possible manner, where the scattered intensity is measured by a detector mounted at some distance from the specimen. This is illustrated in Fig. 2.1.1, where a narrow, essentially monochromatic beam illuminates a small spherical specimen. For a rotating single crystal, the diffracted beams point in discrete directions in space as given by Bragg's law for each lattice vector d_{hkl} (Fig. 2.1.1*a*). For an ideal powder consisting of a virtually unlimited number of randomly oriented crystallites, the diffracted beams will form concentric cones ('Debye cones') with a semi-apex angle of 2θ, representing all randomly oriented identical lattice vectors d_{hkl} (Fig. 2.1.1*b*). Note that in contrast to a single crystal, an ideal powder does not need to be rotated to obtain a complete powder diffraction pattern.

Most instruments are built around a central specimen and consist of the following beam-path components, the numbering of which is consistent with the mounting positions shown in Fig. 2.1.2:

(1) X-ray source;
(2) incident-beam optics;
(3) goniometer base or specimen stage;
(4) diffracted-beam optics;
(5) detector.

The directions of the *incident* and *diffracted beams* (also called 'primary' and 'secondary' beams) form the *diffraction plane* (also called the 'equatorial plane' or 'scattering plane'). The goniometer base can be mounted horizontally (horizontal diffraction plane) or vertically (vertical diffraction plane). The direction perpendicular to the equatorial plane is known as the *axial*

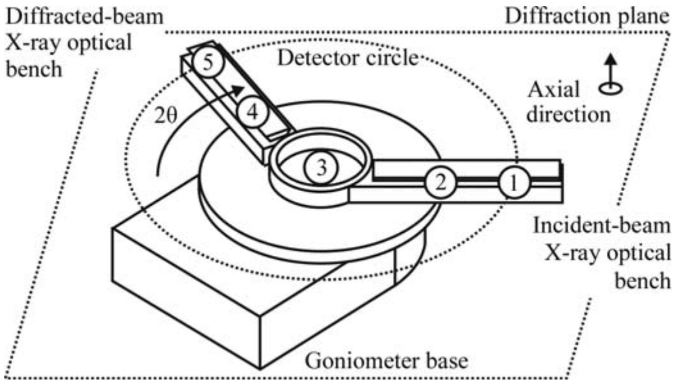

Figure 2.1.2
The basic design principle of modern diffractometers. Currently available instruments are built around a centrally mounted specimen and represent an X-ray optical bench with mounting positions for any (1) X-ray sources, (2) incident-beam optics, (3) specimen stages, (4) diffracted-beam optics and (5) detectors. The 2θ position of the scattered-X-ray optical bench refers to the 2θ angle of the Debye cone shown in bold in Fig. 2.1.1(*b*).

direction. The *detector circle* (also called the 'goniometer circle' or 'diffractometer circle') is defined either by the centre of the active window of a stationary detector, or, in most cases, by a detector moving around the specimen, and is coplanar to the diffraction plane. The 2θ angle of both the diffracted beam in Fig. 2.1.1(*a*) and the Debye cone in Fig. 2.1.1(*b*) (shown in bold) refers to the 2θ position of the diffracted-beam X-ray optical bench in Fig. 2.1.2. It is obvious from Figs. 2.1.1 and 2.1.2 that, in principle, diffraction from single crystals and (ideal) powders can be measured using the same instrument.

An instrument design with a centrally mounted specimen has the important advantage that it implicitly allows the operation of one and the same instrument in both Bragg–Brentano and Debye–Scherrer geometry, depending on the beam divergence chosen. The actual instrument geometry is thus a function of the actual beam propagation angle (divergent, parallel or convergent), making the X-ray optics the most important part of any instrument-geometry conversion. The relationship between the two geometries and their implementation in a single instrument using an incident-beam X-ray optical bench is illustrated in Fig. 2.1.3.

As laboratory X-ray sources invariably produce divergent beams, the 'natural' instrument geometry is self-focusing, 'automatically' leading to the Bragg–Brentano geometry as shown in Fig. 2.1.3(*a*). In this geometry the angle of both the incident and the diffracted beam is θ with respect to the specimen surface. The X-ray-source-to-specimen and the specimen-to-detector distances are equal. The diffraction pattern is collected by varying the incidence angle of the incident beam by θ and the diffracted-beam angle by 2θ. The focusing circle is defined as positioned tangentially to the specimen surface. The focusing condition is fulfilled at the points where the goniometer circle intersects the focusing circle, and thus requires measurements in reflection mode.

The Bragg–Brentano geometry may be extended by an incident- or a diffracted-beam monochromator. In the case of an incident-beam monochromator as shown in Fig. 2.1.3(*b*), the focus of the X-ray source is replaced by the focus of the monochromator crystal. This involves mounting the monochromator crystal (and the X-ray source) a certain distance away along the incident-beam X-ray optical bench, as given by the focusing length of the monochromator crystal (the dotted line in Fig. 2.1.3*b*). For a diffracted-beam monochromator or mirror, the geometry shown in Fig. 2.1.3(*b*) can be thought of as reversed (simply consider the X-ray source and detector switching their positions).

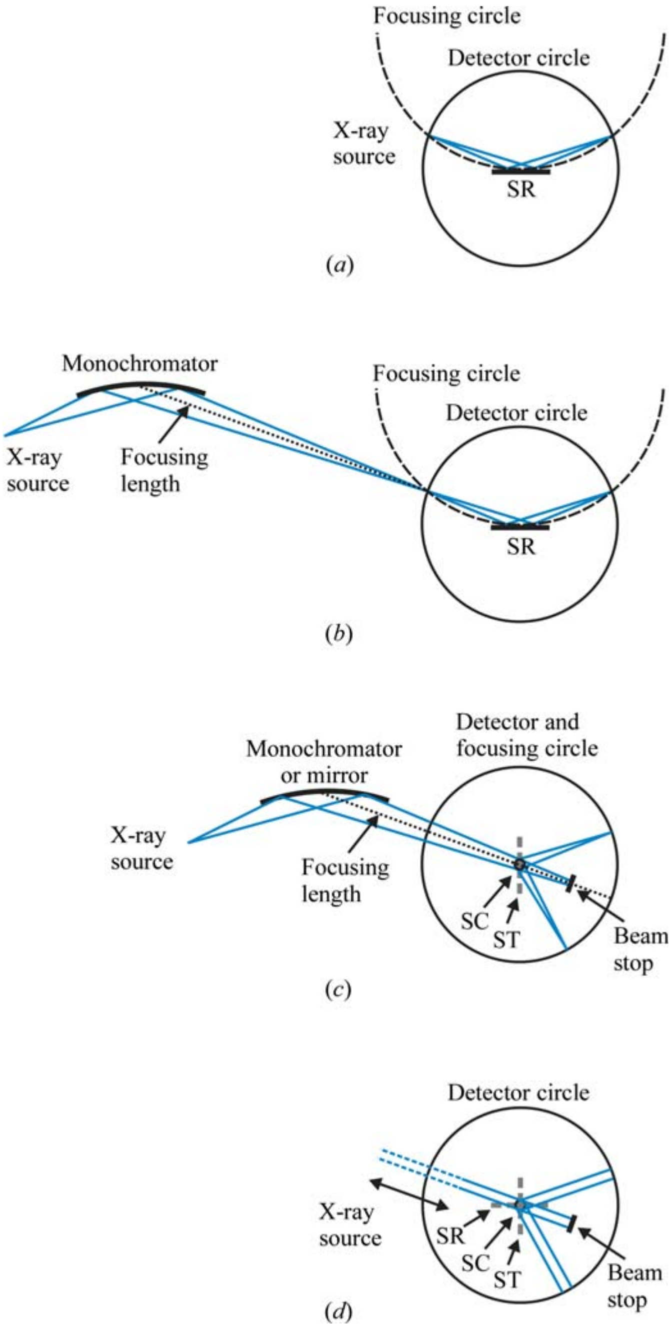

Figure 2.1.3
Transformation between the Bragg–Brentano and Debye–Scherrer geometries using a incident-beam X-ray optical bench. SR: flat specimen, reflection mode; SC: capillary specimen, transmission mode; ST: flat specimen, transmission mode. The actual instrument geometry is a function of the actual beam-propagation angle, making the X-ray optics the most important part of any instrument-geometry conversion. (*a*) Divergent beam: Bragg–Brentano geometry, (*b*) divergent beam: Bragg–Brentano geometry extended by an incident-beam monochromator. (*c*) Convergent beam: focusing Debye–Scherrer geometry, (*d*) parallel beam: Debye–Scherrer geometry. Transformation is achieved by mounting the X-ray tube and pre-aligned optical components at pre-defined positions of the optical bench. None of the figures are to scale.

The conversion from Bragg–Brentano to Debye–Scherrer geometry involves the mounting of some kind of optics designed to convert the divergent beam coming from the X-ray source into a focusing or parallel beam; this is shown in Figs. 2.1.3(*c*) and (*d*), respectively.

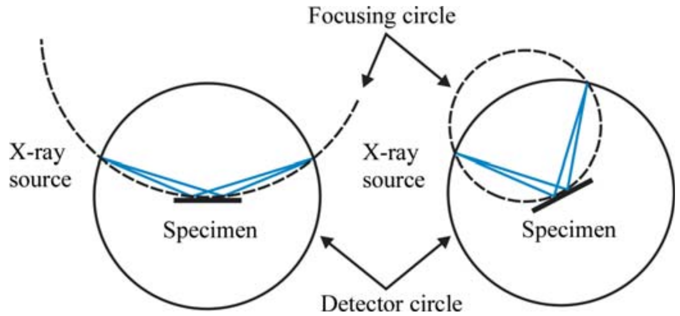

Figure 2.1.4
Bragg–Brentano geometry. The focusing circle, given for two different angles 2θ, is tangential to the specimen surface. Its diameter is given by the intersections between the detector and the focusing circles and is thus 2θ dependent.

In a focusing Debye–Scherrer geometry setup, as shown in Fig. 2.1.3(*c*), the divergent beam coming from the X-ray source is normally focused on the detector circle (for highest resolution) by means of an incident-beam monochromator or a focusing mirror. The focusing circle is identical to the detector circle and the focusing condition requires measurements in transmission mode. When employing an incident-beam monochromator with sufficient focusing length, then conversion between the Bragg–Brentano geometry and the focusing Debye–Scherrer geometry involves a shift of the monochromator crystal (and the X-ray source) along the incident-beam X-ray optical bench (note the identical focusing length of the monochromator shown in Figs. 2.1.3*b* and *c*).

For a parallel-beam setup, as shown in Fig. 2.1.3(*d*), parallelization of the divergent beam coming from the X-ray source may be achieved by different means, such as collimators (classic Debye–Scherrer geometry) or reflective optics such as mirrors or capillaries. In principle, the X-ray source and the detector may be placed at any distance from the specimen, as there are no focusing requirements. As a consequence, measurements can be performed in both reflection and transmission mode.

In a simplified scheme, conversion between the geometries discussed above involves repositioning of the X-ray source, together with mounting of X-ray optics with suitable beam divergence. To make this possible, the incident-beam optical X-ray bench offers the necessary predefined mounting positions including relevant translationary and rotationary degrees of freedom.

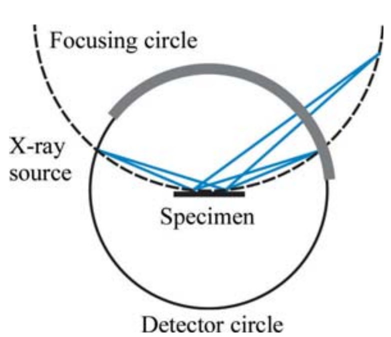

Figure 2.1.5
Bragg–Brentano geometry. While all diffracted beams focus on the (variable-diameter) focusing circle (here shown for two beams), focusing on the detector circle is only achieved at the X-ray source and detector positions (located at the intersections between the detector and the focusing circles). This prevents the use of larger position-sensitive detectors because of defocusing, as indicated by the hypothetical large position-sensitive detector represented by the bold grey line.

An important aspect directly related to the choice of the instrument geometry is the geometric compatibility with position-sensitive detectors. In contrast to Debye–Scherrer geometry, large line and area detectors may not be used in Bragg–Brentano geometry. This is an important limitation of the latter, as the focusing circle does not coincide with the detector circle and has a 2θ-dependent diameter, as illustrated in Fig. 2.1.4. As a consequence, the diffracted beam is only focused on a single point of the goniometer circle, as shown in Fig. 2.1.5. However, small position-sensitive detectors with an angular coverage of not more than about 10° 2θ are used with great success, as defocusing can be ignored at diffraction angles larger than about 20° 2θ if high angular accuracy and resolution are not required. For measurements at smaller 2θ angles, or for highest angular accuracy and resolution, the active window size of a position-sensitive detector may be reduced by means of slits and/or electronically down to a point, allowing the use of this detector as a point detector.

2.1.4.2. *Range of hardware*

An X-ray diffractometer is generally characterized by the relationship between a conditioned beam, the specimen orientation and the subsequent interception of the diffracted beams by a detector of given geometry and imaging properties. There are only a very few instrument configurations that will be ideal for any two application areas, or every conceivable sample within a single application area. It is the user's responsibility to match the instrument to the specimen properties, which can be challenging, particularly in multi-user environments with a large variety of sample types. The platform and the X-ray optical-bench concepts allow the user to choose and mount the most appropriate beam-path components in order to optimize an instrument with respect to a specific application and specimen-property requirements. Table 2.1.1 provides an overview of the currently available types of beam-path components from the X-ray source through to the detector.

The length of available X-ray optical benches varies, and is typically in the range of about 15 cm up to 100 cm. Larger benches allow mounting of bulky components (*e.g.* moving-target X-ray sources or large detectors) as well as mounting of several X-ray optics in a row (*e.g.* combinations of mirrors and channel-cut monochromators). Some diffractometer models allow mounting of two incident- and/or diffracted-beam X-ray optical benches to mount different beam-path components in parallel, *e.g.* X-ray sources with different wavelengths or beam shapes (very popular in single-crystal diffraction), X-ray optics with different beam divergence (*e.g.* to switch between Bragg–Brentano and Debye–Scherrer geometry), and different detector types.

While Table 2.1.1 and the above may imply an enormous combinatorial diversity, in practice this is not entirely the case. In general, beam-path components have to be compatible with the selected instrument geometry, which is dictated by the choice of the X-ray source (point or line), the beam characteristics (wavelength distribution, divergence) and the detector (point, linear or area). This automatically narrows down the range of combinations. As an obvious example, many crystal monochromators and X-ray mirrors are only compatible with a particular wavelength. Also, the size and weight of bulky components, such as moving-target X-ray sources, large specimen stages and large two-dimensional detectors, may impose practical constraints that require consideration. For example, the acces-

references