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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.

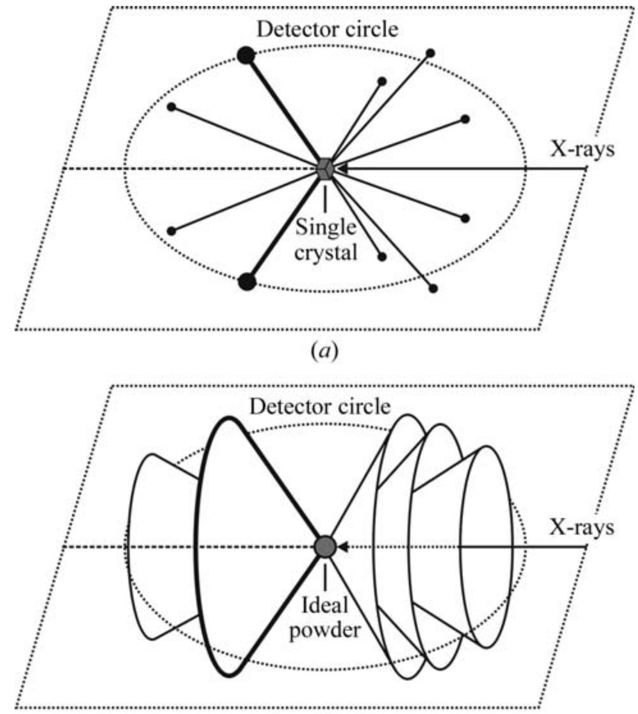

Figure 2.1.1
Diffraction of X-rays by (*a*) a rotating single crystal and (*b*) an ideal powder. The scattered intensity may be measured by a detector placed on the detector circle.

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*

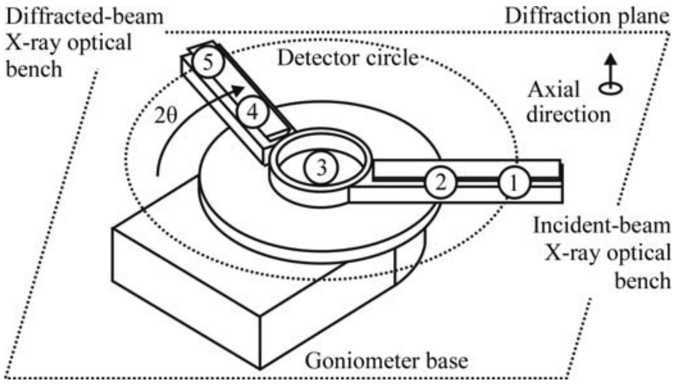

Figure 2.1.2
The basic design principle of modern diffractometers. Currently available instruments are built around a centrally mounted specimen and represent an X-ray optical bench with mounting positions for any (1) X-ray sources, (2) incident-beam optics, (3) specimen stages, (4) diffracted-beam optics 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).

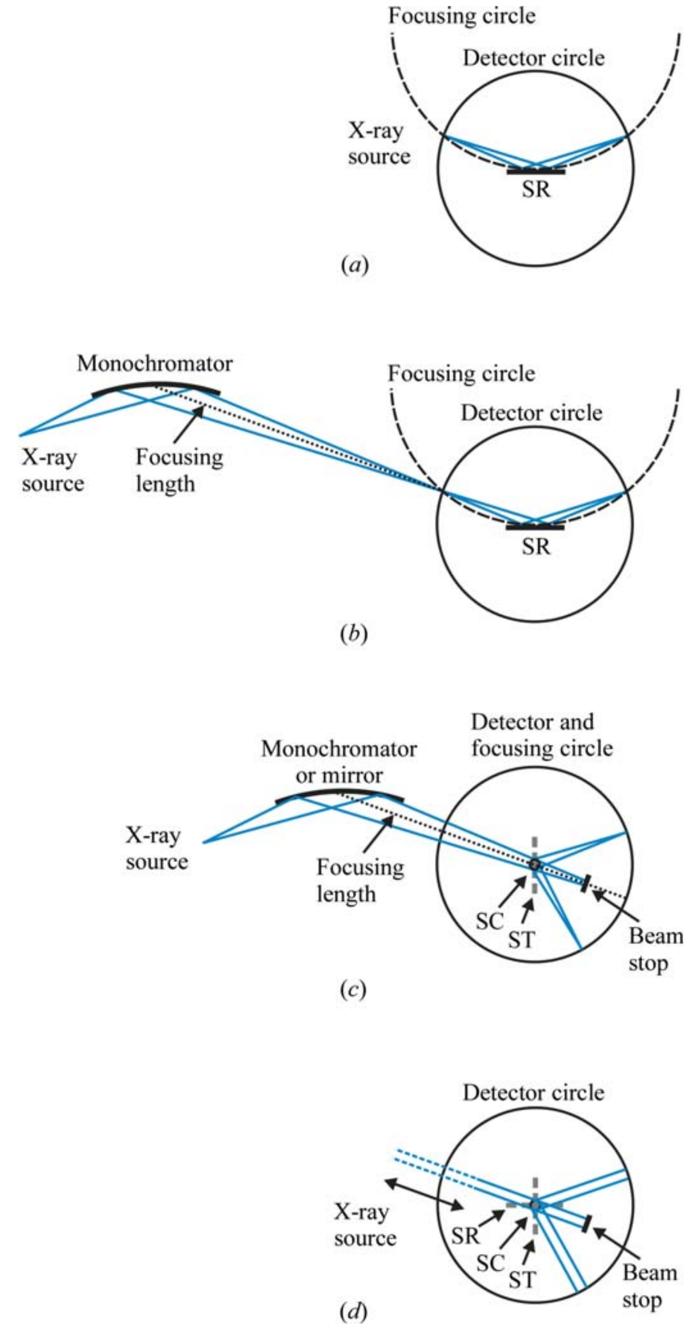

Figure 2.1.3
Transformation between the Bragg–Brentano and Debye–Scherrer geometries using a incident-beam X-ray optical bench. SR: flat specimen, reflection mode; SC: capillary specimen, transmission mode; ST: flat specimen, transmission mode. The actual instrument geometry is a function of the actual 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 predefined 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.

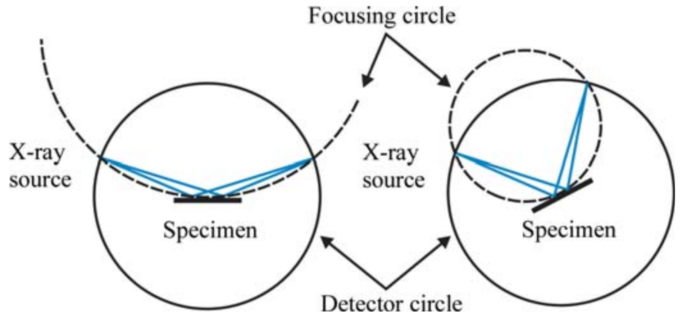

Figure 2.1.4
Bragg–Brentano geometry. The focusing circle, given for two different angles 2θ, is tangential to the specimen surface. Its diameter is given by the intersections between the detector and the focusing circles and is thus 2θ dependent.

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.

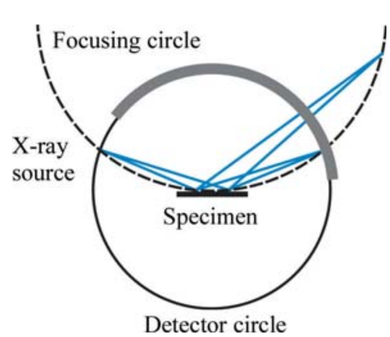

Figure 2.1.5
Bragg–Brentano geometry. While all diffracted beams focus on the (variable-diameter) focusing circle (here shown for two beams), focusing on the detector circle is only achieved at the X-ray source and detector positions (located at the intersections between the detector and the focusing 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-

Table 2.1.1
Types of beam-path components available in laboratory X-ray powder diffraction

The column numbering corresponds to the positions indicated in Fig. 2.1.2 at which individual components can be mounted.

Position 1	Positions 2 and 4	Position 3		Position 5
X-ray sources	X-ray optics	Goniometer base	Specimen stages	Detectors
Fixed target Moving target (rotating anodes, liquid-metal jets)	Absorptive (apertures, metal filters) Diffractive (monochromators, analysers) Reflective (multilayer mirrors, capillary optics)	Vertical [ω–θ (θ–θ), ω–2θ (θ–2θ)] Horizontal [ω–θ (θ–θ), ω–2θ (θ–2θ)]	Fixed, rotating Specimen changer Eulerian cradles Kappa stages Tilt/fixed χ stages *XYZ* stages Flow-through cells Non-ambient (low temperature, high temperature, humidity, high pressure)	Scintillation Gas ionization (metal wire, micro-gap) Semiconductor (SiLi, strip/pixel, CCD/CMOS)

sible angular range may be limited for large components owing to collision issues, while heavy loads on vertical goniometers may impede alignment and lead to early wear and tear. Restrictions will be discussed in Sections 2.1.5 to 2.1.7 for the individual components.

These days, the exchange of lighter components, such as most X-ray optics, specimen stages and detectors, does not require any tools at all (such as when a snap-lock mechanism is employed) or more than a few screws for fixing. Alignment is normally not required when components are factory pre-aligned and handled with care, and when mounts are manufactured with good quality. Intrinsic changes of the beam direction (*e.g.* focusing crystal monochromators or X-ray mirrors) or beam offsets (*e.g.* two-bounce channel-cut monochromators) need compensating translation and/or rotation of the components involved.

The exchange of large, heavy components, or complicated rebuildings such as the conversion of a goniometer (vertical ↔ horizontal, θ–θ ↔ θ–2θ *etc.*), may be still possible for technically skilled users. However, special tools may be necessary, requiring shipment of the component(s), or even the instrument, back into the factory. In addition, X-ray, machine and electrical safety directives by the local authorities have to be obeyed, and conversions may require updating approval to use the instrument. In such cases it may be more economic to operate two dedicated instruments instead.

The instrument control software plays a particularly important role in the context of instrument configuration and automated instrument conversion. In modern instruments, each beam-path component is equipped with an identification chip or hole masks read out by light barriers, which uniquely identify the respective component and link it with all its individual stored or coded properties. This information may range from part numbers, usage history or alignment information such as beam offsets, through to a virtually unlimited wealth of any physical data required to configure and operate that particular component. This 'component recognition' feature provides for completely new and important capabilities of laboratory powder diffractometers, the most important of which are:

(*a*) Any beam-path components, and each change of status, can be automatically detected, validated and configured, allowing true 'plug & play' operation.

(*b*) Real-time conflict detection: detection of incompatible, incorrectly mounted or missing instrument components. This feature can also help the user in choosing compatible instrument components, as already discussed above.

(*c*) Automatic, motorized adjustments of beam direction or beam-offset changes, based on the information stored in the related components' ID chips, as individually determined at the factory *via* pre-alignment.

(*d*) Every instrument detail can be saved together with the measurement data, providing for a complete and accurate documentation of the experiment. In principle, every measurement can be exactly reproduced even years later.

(*e*) Measurement instructions can include instrument information. For example, manufacturers or users can configure the measurement software to propose instrument configurations deemed best for particular applications. A user with appropriate rights can choose to enforce a certain instrument configuration so that measurements will not start unless the instrument has detected the required configuration.

Both the platform concept and the huge advances in instrumentation and instrument control software have dramatically changed the laboratory X-ray instrumentation landscape in the past few years. The ease with which an instrument configuration can be changed is not only useful for less-skilled users. Probably even more importantly, it allows the use of the same instrument, in different configurations, for different X-ray application areas. It can generally be said that laboratory X-ray instrumentation has overcome the (mostly historical) dividing lines between different applications, which were mostly between single-crystal diffraction, powder diffraction and thin-film analysis. As far as differences still remain, these are usually solely the consequence of dedicated instrument components for meeting specific application requirements, resulting in specialized measurement and data-evaluation software, which is rarely included with each instrument.

2.1.4.3. *Range of applications*

It is the flexibility of today's X-ray diffractometers that leads to their usefulness for a wide range of X-ray scattering techniques beyond traditional X-ray powder 'Bragg diffraction'. Table 2.1.2 provides an overview.

X-ray scattering techniques represent the vast majority of techniques that X-ray diffractometers are used for. Properly configured, however, the same instrument can also be used to collect X-ray absorption (X-ray radiography) or X-ray emission (X-ray fluorescence) data, even if the achievable data quality cannot compete with dedicated instruments.

For X-ray radiography, an instrument will be configured in transmission geometry with the X-rays projected towards a

Table 2.1.2
X-ray applications for with modern X-ray diffractometers

X-ray scattering
Powder diffraction
Qualitative (phase identification) and quantitative phase analysis
Indexing, structure determination and structure refinement from powder data
Microstructure analysis (texture, size, strain, microstrain, disorder and other defects)
Pair distribution function analysis ('total scattering')
Thin-film analysis
Grazing incidence X-ray diffraction (GIXRD)
X-ray reflectometry
Stress and texture
High-resolution X-ray diffraction
Reciprocal-space mapping
In-plane GIXRD
Single-crystal diffraction
Chemical crystallography
Protein crystallography
Small-angle X-ray scattering
X-ray topography
X-ray absorption
X-ray radiography (X-ray-absorption-based imaging)
X-ray emission
X-ray fluorescence

specimen. X-rays that pass through the specimen can be detected to give a two-dimensional representation of the absorption contrast within the specimen. For tomography, the X-ray source and detector will be moved to blur out structures not in the focal plane. Multiple images can be used to generate a three-dimensional representation of the specimen by means of computed tomography. Obvious disadvantages are the large effective focal spot size of the X-ray sources and the relatively low resolution of the detectors that are typically used for powder diffraction, which, in combination with a limited adjustability of both the X-ray-source-to-specimen and specimen-to-detector distances, lead to substantial unsharpness issues and poor resolution. High-quality images can be achieved when using microfocus X-ray sources and charge-coupled device (CCD) detectors with focus and pixel sizes smaller than 10 µm, respectively, but such an instrument configuration is not suitable for applications requiring ideal powders (see also Sections 2.1.6 and 2.1.7).

Collecting X-ray fluorescence data is comparatively straightforward. Data can be collected simultaneously to X-ray scattering data when employing a suitable detector, such as an energy-dispersive detector (Section 2.1.7.2.3). There are a couple of disadvantages to be considered, such as absorption issues (the specimen will be normally measured in air rather than in vacuum, hampering the analysis of light elements) and the inefficiency of excitation by the characteristic line energies of the X-ray source anode materials typically used for diffraction (hampering the analysis of elements with higher atomic numbers than that of the anode material).

2.1.5. Goniometer designs

A goniometer, by definition, is an instrument that either measures an angle or allows an object to be rotated to a precise angular position. In an X-ray diffractometer the purpose of the goniometer is to move the X-ray source, specimen and detector in relation to each other. Goniometers are usually categorized by the number of axes available for X-ray source, specimen and detector rotation, and are thus called one-, two-, three-, . . . , *n*-axis (or -circle) goniometers.

Because of practical reasons, most goniometers consist of two distinct components, a goniometer base and a specimen stage, with the specimen stage mounted on the goniometer base.

The goniometer base typically offers two axes, one axis to rotate the X-ray source or the specimen stage, the other axis to rotate the detector. In some designs goniometer bases are omitted, specifically if there is no need to move the X-ray source and the detector, such as in Debye–Scherrer-type diffractometers with large detectors. Such machines are usually dedicated to a particular application without the need for high flexibility.

Depending on the requirements of the application, additional rotational and translational degrees of freedom may be needed to rotate and translate a specimen in space; these are usually implemented in the specimen stage. More rotational degrees of freedom may include the rotation of the X-ray source line focus or a rotation of the detector out of the diffraction plane to measure diffraction by lattice planes (nearly) perpendicular to the specimen surface, so-called non-coplanar diffraction.

2.1.5.1. *Geometrical conventions and scan modes*

In the literature there is some inconsistency related to the naming of axes and the choice of signs for angles (left- *versus* right-handed). A comprehensive treatment of geometrical conventions has recently been given by He (2009); in the following these conventions will be adhered to.

In many texts the notations θ–2θ and θ–θ rather than ω–2θ and ω–θ are used, mostly because of historical reasons. The first diffractometers operated in Bragg–Brentano geometry (see Section 2.1.3.1.2) and were equipped with single-axis goniometers. In such a goniometer the single axis drives two shafts which are mechanically coupled 1:2 or 1:1; thus the notations θ–2θ and θ–θ were coined. Today, the majority of all goniometer bases allow coupled as well as uncoupled rotation of the ω and θ axes. Therefore the ω–2θ and ω–θ notations should be generally preferred, as they represent the more general notations.

2.1.5.1.1. *Goniometer base*

A typical goniometer base provides two coaxial and independently driven axes, ω and 2θ, mounted perpendicular to the diffraction plane. These two axes are the main axes of a goniometer, since they have the most effect on the accuracy and precision of measured Bragg angles. The diffraction plane and the axes are generally described by a right-handed Cartesian coordinate system, as illustrated in Fig. 2.1.6, where the direct X-ray beam propagates along the X_L axis. Z_L is up and coincident with the ω and 2θ axes, and X_L–Y_L define the diffraction plane with the detector circle coplanar to it. Since X_L is coincident with the incident X-ray beam, it is also the axis of the Debye cones. The semi-apex angles of the cones are determined by the 2θ values given by the Bragg equation. The angles 2θ and γ describe the direction of scattering vectors in space (compare Fig. 2.1.1), where γ is defined as the azimuthal angle from the origin at $-Z_L$ with a right-hand rotation axis along the opposite direction of the incident beam ($-X_L$ direction).

The ω and 2θ axes are mechanically arranged as the inner circle and outer circle, respectively. The inner circle usually carries either the specimen stage or the X-ray source, while the detector is mounted on the outer circle. As a consequence, there are two common base goniometer configurations in use: In the ω–2θ (or θ–2θ with $\omega = \theta$) configuration, the incident-beam direction is

references