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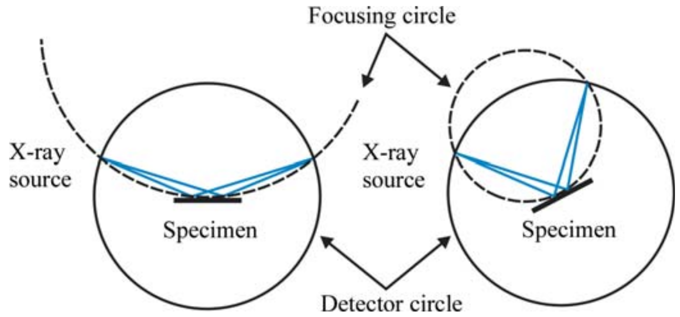

Figure 2.1.4
Bragg–Brentano geometry. The focusing circle, given for two different angles 2θ, is tangential to the specimen surface. Its diameter is given by the intersections between the detector and the focusing circles and is thus 2θ dependent.

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.

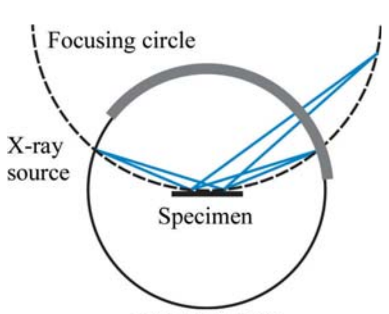

Figure 2.1.5
Bragg–Brentano geometry. While all diffracted beams focus on the (variable-diameter) focusing circle (here shown for two beams), focusing on the detector circle is only achieved at the X-ray source and detector positions (located at the intersections between the detector and the focusing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

references