

2. INSTRUMENTATION AND SAMPLE PREPARATION

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

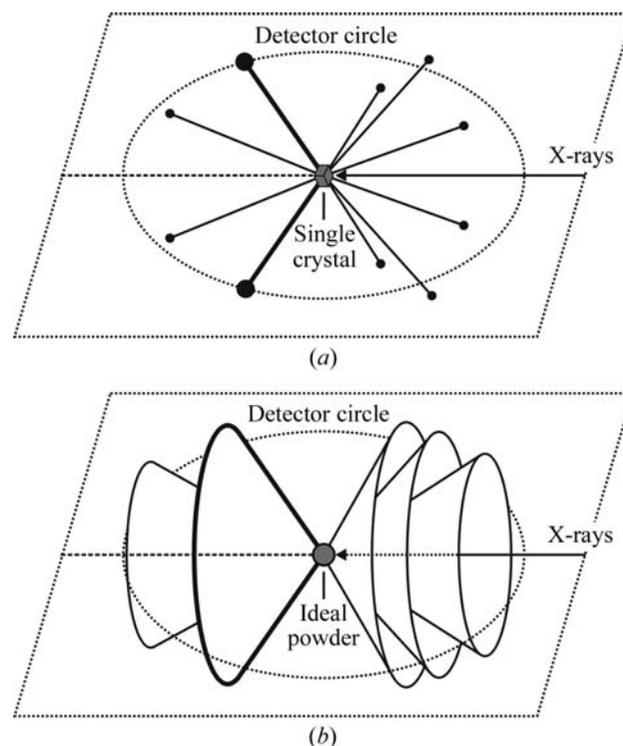


Figure 2.1.1

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.

components to meet specific application and specimen-property requirements.

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. 2.1.1a). 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. 2.1.1b). 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*