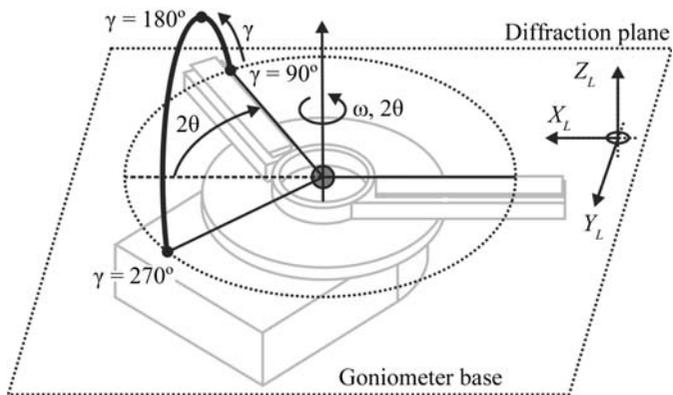


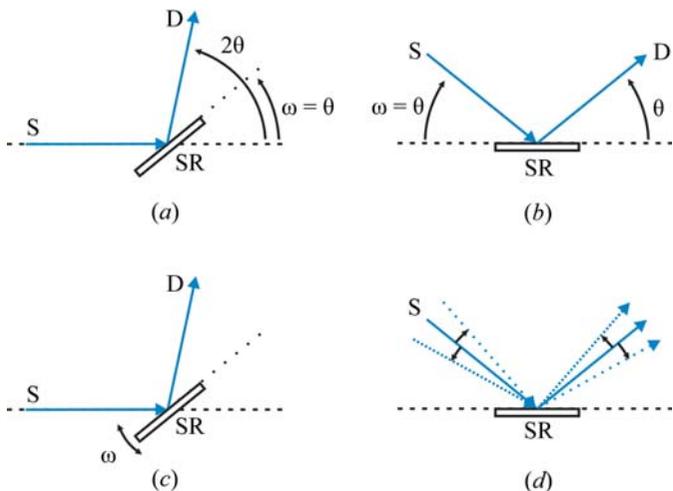
2.1. LABORATORY X-RAY SCATTERING



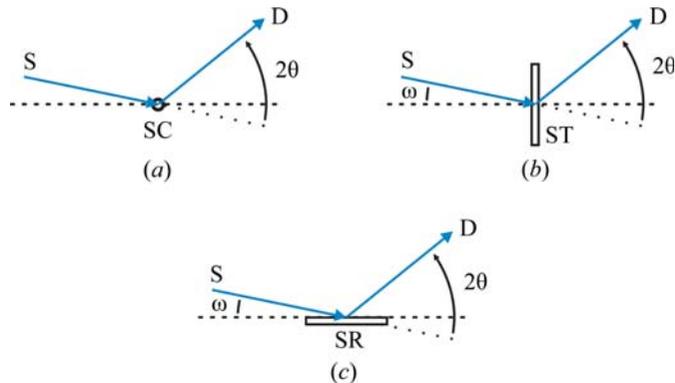
**Figure 2.1.6** Laboratory coordinates and geometric definition of the coaxial goniometer axes  $\omega$  and  $2\theta$ . If the azimuthal angle  $\gamma$  takes all values from 0 to  $360^\circ$  at a given Bragg angle  $2\theta$ , the trace of the diffracted beams forms a Debye cone (compare with Fig. 2.1.1).

mechanically fixed. The  $\omega$  axis rotates the specimen stage, while the  $2\theta$  axis rotates the detector. In the  $\omega$ - $\theta$  (or  $\theta$ - $\theta$  with  $\omega = \theta$ ) configuration, the  $\omega$  axis defines the incident-beam angle by rotating the X-ray source, while the other axis scans the detector. In this configuration the specimen stage is mechanically fixed. Both configurations allow identical positioning of the X-ray source, specimen and detector relative to each other.

The goniometer base orientation is defined by the diffraction plane, which can be either horizontal or vertical. Vertical-base goniometers in  $\omega$ - $\theta$  configuration are particularly popular, as the specimen is always kept horizontal, effectively preventing it from falling off. However, heavy specimens and beam-path components require particular attention in both the goniometer base design and choice of orientation, as they have a strong impact on goniometer accuracy, precision and early wear and tear (see also Section 2.1.5.2). Where loads exceed the maximum specifications for a vertically mounted goniometer base, and whenever horizontal specimen positioning is not imperative, either a vertical goniometer base in  $\omega$ - $2\theta$  configuration or a horizontal goniometer should be chosen.



**Figure 2.1.7** Goniometer base configurations and scan modes suitable for both Bragg-Brentano or Debye-Scherrer geometry. Symmetric beam path setup in (a)  $\omega$ - $2\theta$  and (b)  $\omega$ - $\theta$  configuration. Rocking curve setup in (c)  $\omega$ - $2\theta$  and (d)  $\omega$ - $\theta$  configuration. Only the central beams are shown for clarity, rotations are indicated by arrows. S: X-ray source, D: detector, SR: flat specimen, reflection mode.



**Figure 2.1.8** Goniometer base configurations and scan modes suitable for the Debye-Scherrer geometry only. Only the central beams are shown for clarity. (a) Capillary specimen in transmission mode, (b) flat specimen in transmission mode, (c) flat specimen in reflection mode, grazing incidence with fixed  $\omega$ . S: X-ray source, D: detector, SR: flat specimen, reflection mode; SC: capillary specimen, transmission mode; ST: flat specimen, transmission mode.

In Figs. 2.1.7 and 2.1.8 a range of typical goniometer base configurations and scan modes are illustrated.

A symmetric beam-path setup in reflection mode, where both the incident and diffracted beam form an angle of  $\theta$  with respect to the specimen surface, is mandatory for instruments operating in Bragg-Brentano geometry to maintain the focusing condition (see Section 2.1.4.1), but is also commonly used in Debye-Scherrer geometry. Scanning involves coupling of the  $\omega$  (with  $\omega = \theta$ ) and  $2\theta$  axes in a ratio of 1:2 for the  $\omega$ - $2\theta$  configuration (Fig. 2.1.7a) and 1:1 for the  $\omega$ - $\theta$  configuration (Fig. 2.1.7b), but only allows probing of lattice planes essentially parallel to the specimen surface. Where determination of a texture parameter is sought, a so-called ‘rocking-curve’ measurement can be performed by rocking either the specimen or the X-ray source and detector around the position of a Bragg peak. Two scenarios need to be considered and are illustrated in Fig. 2.1.7(c) and Fig. 2.1.7(d). In the  $\omega$ - $2\theta$  configuration with fixed X-ray source (Fig. 2.1.7c), the detector will be fixed at the  $2\theta$  position of a selected Bragg peak, while the specimen is rotated (‘rocked’) independently, to perform a so-called ‘ $\omega$ -scan’. To achieve the same in an  $\omega$ - $\theta$  configuration with fixed specimen, the X-ray source and the detector will be coupled 1:–1 or –1:1 to perform a clockwise or anticlockwise scan while maintaining the selected  $2\theta$  position, respectively, as illustrated in Fig. 2.1.7(d).

In Debye-Scherrer geometry there is no geometrical restraint requiring coupled scans to maintain  $2\theta$ -dependent focusing conditions, as is the case in the Bragg-Brentano geometry, providing high flexibility. Specimens can be measured in both reflection as well as transmission mode. In principle, the incident-beam direction may be any relative to the specimen surface, and can be fixed or variable, while the detector performs a ‘detector scan’. The  $\omega$  and  $2\theta$  axes may be coupled or not. Choices solely depend on the specimen properties and the requirements of the application. This is demonstrated in Fig. 2.1.8 for a few representative examples. The classic Debye-Scherrer geometry using a capillary specimen is shown in Fig. 2.1.8(a). The capillary specimen can be readily exchanged for a flat-plate specimen as shown in Fig. 2.1.8(b) and Fig. 2.1.8(c) for flat-plate transmission and reflection, respectively.  $\omega$  can be set to different angles or perform a coupled scan to allow access to higher  $2\theta$  angles (Fig. 2.1.8b) or can be set to a different angle for grazing-incidence measurements (Fig. 2.1.8c). In some applications it may be beneficial to perform a (usually coupled) scan of both  $\omega$  and  $2\theta$  to