

## 2. DIFFRACTION GEOMETRY AND ITS PRACTICAL REALIZATION

can receive X-rays only from the specimen area. They must be carefully aligned to avoid touching the beam.

The use of the long X-ray source makes it necessary to reduce the axial divergence, which would cause very large asymmetry. This is done with two sets of thin (25 to 50  $\mu\text{m}$ ) parallel metallic foils PS ('Soller slits'; Soller, 1924) placed before and after the specimen. If a monochromator is used, the set on the side of the monochromator is not essential because the crystal reduces the divergence. The angular aperture of a set of slits is

$$\delta = 2 \arctan(\text{spacing/length}). \quad (2.3.1.7)$$

The overall width of the set and  $\delta$  determine the width of the specimen irradiated in the axial direction, which remains constant at all  $2\theta$ 's. The construction is illustrated in Fig. 2.3.1.5(d). The aperture  $\delta$  is usually 2 to 5°. Each set of parallel slits reduces the intensity; for example, with 12.5 mm long foils with 1 mm spacings, the intensity is about one-half of that without the parallel slits. The aperture can be selected with any combination of spacings and lengths but the greater the length, the fewer foils are needed, and the less is the intensity loss due to thickness of the metal foils (usually 0.025 mm). These slits can be made as interchangeable units of different apertures.

## 2.3.1.1.2. Use of monochromators

Many diffractometers are equipped with a curved highly-oriented pyrolytic graphite monochromator placed after the receiving slit as shown in Fig. 2.3.1.3. Although graphite has a large mosaic spread ( $\sim 0.35$  to  $0.6^\circ$ ), the diffracted beam from the specimen is defined by the receiving slit, which determines the profile shape and width rather than the monochromator. The same results are obtained whether the monochromator is set in the parallel or antiparallel position with respect to the specimen. The most important advantage of graphite is its high reflectivity, which is about 25–50% for Cu  $K\alpha$ . This is much higher than LiF, Si or quartz monochromators that have been used for powder diffraction. The  $K\beta$  filter and the parallel slits in the diffracted beam can be eliminated and, since each reduces the  $K\alpha$  intensity by about a factor of two, the use of a graphite monochromator actually increases the available intensity. The diffracted-beam monochromator eliminates specimen fluorescence and the scattered background whose wavelengths are different from that of the monochromator setting. For example, a Cu tube can be used for specimens containing Co, Fe, or other elements with absorption edges at longer wavelengths than Cu  $K\alpha$  to produce patterns with low background. Several monochromator geometries are described by Lang (1956).

A specimen in the reflection mode may be used with an incident-beam monochromator and  $\theta$ - $2\theta$  scanning as shown in Fig. 2.3.1.1(c). One of the principal advantages is that it is possible to adjust the monochromator and slits to remove the  $K\alpha_2$  component and produce patterns with only  $K\alpha_1$  peaks. The profile symmetry, resolution and instrument function are thus greatly improved; see, for example, Warren (1969), Wölfel (1981), Göbel (1982) and Louër & Langford (1988). The high-quality crystal required causes a large loss of intensity and reduces specimen fluorescence but does not eliminate it. However, Soller slits in the incident beam and a  $\beta$  filter are no longer required and the net loss of intensity can be as low as 20%. Such monochromators can now be provided as standard by diffractometer manufacturers and their use is increasing, but they are not as widely used as the diffracted-beam monochromator.

## 2.3.1.1.3. Alignment and angular calibration

It is essential to align and calibrate the diffractometer properly. Failure to do so degrades the performance of the instrument, leading to a loss of intensity and resolution, increased background, incorrect profile shapes, and errors that cannot be readily diagnosed. Procedures and devices for this purpose are often provided by the manufacturer. The principles and mechanical devices to aid in making a proper alignment have been described by Parrish & Lowitzsch (1959) and the general procedure by Klug & Alexander (1974, p. 280).

The alignment requires setting the diffractometer axis of rotation to the selected X-ray tube take-off angle at a distance equal to the radius of the diffractometer. The long axes of the X-ray tube focal line, entrance, receiving, and antiscatter slits must be centred, be parallel to the axis of rotation, and lie in the same plane when the instrument is at  $0^\circ$ . The slits are made parallel to the axis of rotation in the manufacture of the diffractometer, and these steps require positioning of the instrument with respect to the line focus. The parallel-slit foils must also be normal to the rotation axis. A flat fluorescent screen made as a specimen to fit into the diffractometer specimen post is used to centre the primary beam by small movements of the ES and/or diffractometer. The diffracted beam can be centred on the curved monochromator with a narrow slit placed at the centre of the monochromator position (with the monochromator removed). The detector arm is then moved to the highest intensity. The procedure is repeated with the receiving slit in position. This is very close to the  $0^\circ$  position described below.

The angular calibration of the diffractometer is usually made by accurately measuring the  $0^\circ$  position to establish a fiducial point. It assumes that the gear system is accurate and that the receiving-slit arm moves exactly to the angle indicated on the scale at all  $2\theta$  positions. The determination of the angular precision of the gear train requires special equipment and methods; see, for example, Jenkins & Schreiner (1986). It is

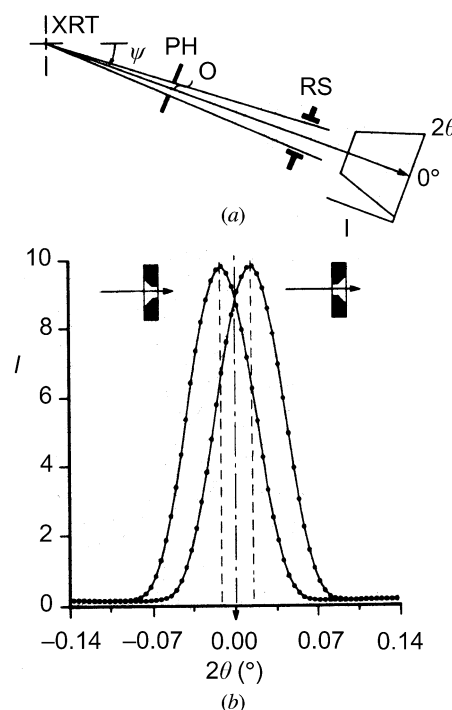


Fig. 2.3.1.6. Zero-angle calibration. (a) XRT X-ray tube anode,  $\psi$  take-off angle,  $O$  axis of rotation, PH pinhole, RS receiving slit. Intensity distribution at right. (b)  $0^\circ$  position is median of two curves recorded with  $180^\circ$  rotation of PH.