

2.3. POWDER AND RELATED TECHNIQUES: X-RAY TECHNIQUES

2.3.5.1.3. Source intensity distribution and size

The intensity distribution of the focal line is usually not uniform. This has no apparent effect on the shapes of powder reflections but may cause difficulties with single crystals (Parrish, Mack & Taylor, 1966). The distribution can be measured with a small pinhole placed between the X-ray tube focal line and a dental or Polaroid film. The ratio of the distances between line-to-pinhole and pinhole-to-film determines the magnification of the image. The pinhole diameter should be small for good resolution. About 0.1 mm diameter is satisfactory and can be made with a special microdrill, spark erosion or other methods. The thickness of the metal must be minimal to avoid having the aperture formed by the length and diameter of the pinhole limit the length of focus photographed. Avoid over-exposure which broadens the image. Also, the Polaroid film should be exposed outside the cassette to avoid broadening caused by the intensifying screen.

A more accurate method is to scan a slit and detector (mounted on the same arm) normal to the central ray from the focus as shown in Fig. 2.3.5.2(b) (Parrish, 1967). The slits are a pair of molybdenum rods (or other high-absorbing metal) with opening normal to the scan direction, and the slit width determines the

resolution. This method gives a direct measurement of the intensity distribution from which the projected size can be determined.

The actual size of the focus F'_w is foreshortened to F_w by the small take-off angle ψ , $F_w = F'_w \sin \psi$. A typical 0.5×10 mm focus viewed at 6° appears to be a line 0.05×10 mm or a spot 0.05×1 mm [Fig. 2.3.1.9(a)]. The line focus is generally used for powder diffractometry and focusing cameras and the spot focus for powder cameras and single-crystal diffractometry.

X-rays emerge from three or four Be windows spaced 90° apart around the circumference. Their diameter and position with respect to the plane of the target determine the usable ψ -angle range. The length of line focus that can pass through the window can be seen with a flat fluorescent screen in the specimen holder using the largest entrance slit. The Be window thickness often used is $300 \mu\text{m}$ and the transmission as a function of wavelength is shown in Fig. 2.3.5.2(a).

2.3.5.1.4. Air and window transmission

The absorption of X-rays in air is also wavelength-dependent and increases rapidly with increasing wavelength, Fig. 2.3.5.2(a). The air absorption was calculated using a density

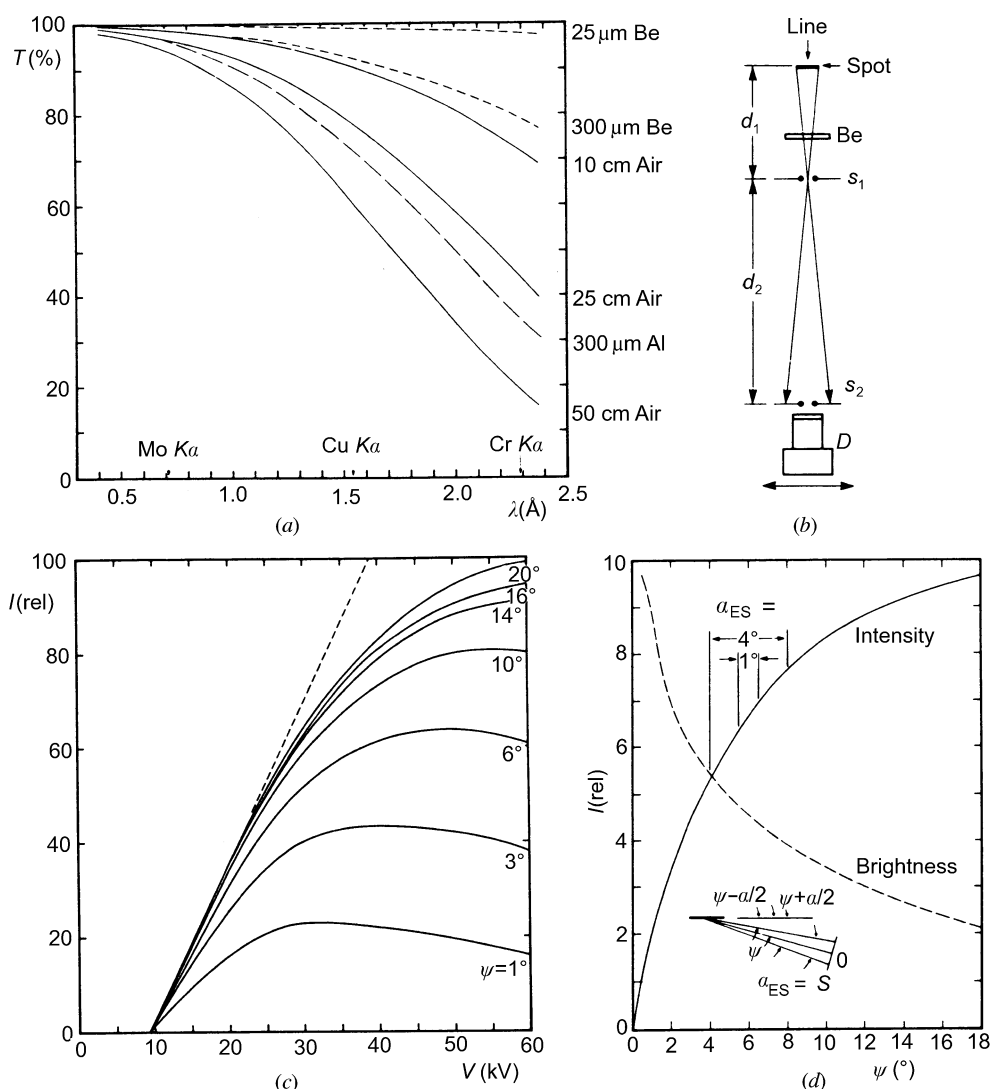


Fig. 2.3.5.2. (a) Transmission of Be, Al and air as a function of wavelength. (b) Method for measuring X-ray tube focus by scanning slit S_2 and detector D . Slit S_1 is fixed and the ratio of the distances d_2/d_1 gives the magnification. (c) Intensity of a copper target tube as a function of kV for various take-off angles. (d) Intensity and brightness as a function of take-off angle of a copper target tube operated at 50 kV. The intensity distributions for 1 and 4° entrance-slit apertures are shown at the top, and terms used to define ψ and α_{ES} are shown in the lower insert.