International Tables for Crystallography (2006). Vol. C, Figure 2.3.4.1, p. 70.

2.3.4. Powder cameras

The use of powder cameras has greatly diminished in recent years, having been largely replaced by diffractometers. Detailed descriptions of the many types of camera, their use, film measurement, and interpretation have been published in the books by Peiser, Rooksby & Wilson (1955), Azároff & Buerger (1958), Taylor (1961), Alexander (1969), Lipson & Steeple (1970), Klug & Alexander (1974), Cullity (1978), and Barrett & Massalski (1980). The following is an outline of the more important features.

The most commonly used cameras are:

(*a*) Cylindrical camera with narrow fibre-shaped specimen and Straumanis film mounting.

(*b*) Guinier focusing monochromator camera with flat transmission specimen and cylindrical film.

(*c*) Flat-film camera for Laue patterns and crystal orientation.

The best results are obtained using the X-ray tube spot focus for non-focusing methods as in (*a*) and (*c*), and the line focus for focusing cameras as in (*b*). A filter is used to eliminate the $K\beta$ lines in the methods that do not use a monochromator. Double-coated film is used for cameras in which the reflections are normal to the film. Single-coated film is used for focusing cameras; alternatively, double-coated film can be used if the second image is prevented from developing (Parrish, 1955).

In all film methods, it is necessary to account for film shrinkage in the development processing to obtain correct angle measurements. In the Straumanis film mounting, Fig. 2.3.4.1(*a*), the arcs can be measured around the incident and exit holes to obtain a linear measure of the effective camera diameter, *i.e.* $180^\circ 2\theta$. Other methods include exposing a transparent scale on the film prior to development, installing a pair of knife edges with accurately measured separation just above the film to cast sharp images on both ends of the film, or incorporating a standard material in the specimen. Exposure times vary from a few minutes to an hour or more depending on the specimen and the various camera parameters.

2.3.4.1. *Cylindrical cameras (Debye–Scherrer)*

The design of cylindrical powder cameras with Straumanis film mounting was described by Buerger (1945) and the collimators by Parrish & Cisney (1948). Straumanis developed the method to an art and used it to measure lattice parameters, thermal expansion, and other properties of many materials; see, for example, Straumanis (1959), which contains references to many of his papers. In the USA, the camera diameter was usually made 57.3 or 114.6 mm to simplify measuring the film with a millimetre scale, 1 mm = 1° or $2^\circ 2\theta$. One of the major advantages of the method is that the full reflection range is recorded simultaneously on the film. Other advantages are that the effects of preferred orientation are immediately apparent on a film, lines can have non-uniform intensity ('spottiness') owing to size effects or there can be broadening owing to structural imperfections. These visual effects, which are less evident with diffractometer data, can be valuable aids in identifying a mixture of substances.

The camera is basically a cylindrical light-tight metal body with removable cover, and the film is pressed around the inside circumference. The beam is defined by an entrance collimator and the undiffracted portion is conducted out by an exit tube; both are mounted on the central plane of the camera and extend inside nearly to the specimen. The specimen is centred and rotated continuously during the exposure; translation may be added to bring more particles into the beam. Evacuating the camera or filling it with helium removes the air scattering which darkens the film in the vicinity of the 0° hole.

If the specimen is too thick or has high absorption, the forward reflection lines split because the beam penetrates only the top and bottom of the rod. The diameter of the rod determines the widths of the lines. The line widths are about twice the diameter of the rod at small 2θ's and decrease with increasing 2θ. The absorption causes a systematic error in the positions of the lines, which can be handled with a $\cos^2\theta$ or Nelson–Riley plot (Section 5.2.8). The sample may be small – only about 0.1 mg is required. Axial divergence causes the well known 'umbrella' or 'broom' broadening illustrated in Fig. 2.3.4.1(*b*). It is essential to measure the film along the equator where the lines are narrowest and shifts the smallest. The specimens should be less than 0.5 mm diameter and may be coated on a fine wire or glass fibre (silica or Lindemann glass), or packed into a capillary (commercially available).

Read & Hensler (1972) modified a Debye–Scherrer camera to use flat specimens for thin-film analysis (Tao & Hewett, 1987).

2.3.4.2. *Focusing cameras (Guinier)*

The Guinier camera (Guinier, 1937, 1946; Guinier & Dexter, 1963) uses a high-quality asymmetric focusing monochromator and cylindrical camera with a thin transmission specimen, Fig. 2.3.4.1(*c*). The film must be placed at the focal point of the monochromator, which can be adjusted to reflect only the $K\alpha_1$ line. When the camera is in the position shown, the angular range is larger on one side of the film than the other (asymmetric

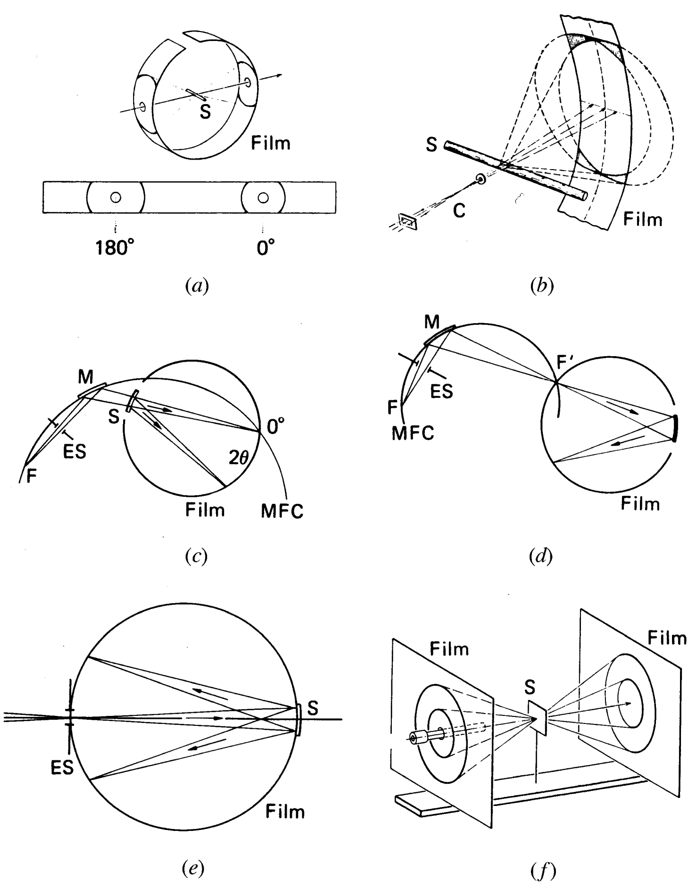

Fig. 2.3.4.1. Powder-camera geometries. (*a*) Straumanis film setting. (*b*) Origin of 'umbrella' effect (axial divergence). (*c*) Guinier camera with specimen in transmission and (*d*) in reflection. (*e*) Symmetrical back-reflection focusing camera. (*f*) Flat-film camera for forward- and back-reflection.

 references