

4.3. ELECTRON DIFFRACTION

Mamy & Gaultier (1976). The hk lines are no longer straight, but have the shapes described by Bernal (1926) for rotation photographs. It is difficult, however, to prepare good specimens. Other arrangements have been developed recently with advantages for precise intensity measurements. The reflections are recorded consecutively by means of a powder diffractometer fitted with a goniometer head. The relation between the angle of tilt φ and the angle of diffraction (twice the Bragg angle) 2θ depends on the reciprocal-lattice point to be recorded. If the latter is defined by a vector of length $H = (2 \sin \theta)/\lambda$ and by the angle ω between the vector and the plane of orientation (texture basis), the relation $\varphi = \theta - \omega$ permits scanning of reciprocal space along any trajectory by proper choice of consecutive values of ω or θ . In particular, if ω is constant, the trajectory is a straight line passing through the origin at an angle ω to the plane of orientation (Krinary, 1975). Using additional conditions [$\omega = \arctan(D/B)$, $H = (B^2 + D^2)^{1/2}$], Plançon *et al.* (1982) realized the recording and the measurement of intensities along the cylinder-generating hk rods for different shapes of the misorientation function $N(\alpha)$.

In the course of development of electron diffractometry, a deflecting system has been developed that permits scanning the electron diffraction pattern across the fixed detector along any direction over any interval (Fig. 4.3.5.2). The intensities are measured point by point in steps of variable length. This system

is applicable to any kind of two-dimensional intensity pattern, and in particular to texture patterns (Zvyagin, Zhukhlistov & Plotnikov, 1996). Electron diffractometry provides very precise intensity measurements and very reliable structural data (Zhukhlistov *et al.*, 1997).

If the effective thickness of the lamellae is very small, of the order of the lattice parameter c , the diffraction pattern generates into a combination of broad but recognizably distinct $00l$ reflections and broad asymmetrical hk bands (Warren, 1941). The classical treatments of the shape of the bands were given by Méring (1949) and Wilson (1949) [for an elementary introduction see Wilson (1962)].

4.3.5.3. Lattice direction oriented parallel to a direction (fibre texture)

A fibre texture occurs when the crystals forming the specimen have a single direction in common. Each point of the reciprocal lattice describes a circle lying in a plane normal to the texture axis. The pattern, considered as plane sections of the reciprocal-lattice representation, resembles rotation diagrams of single crystals and approximates to the patterns given by cylindrical lattices (characteristic, for example, of tubular crystals).

If the a axis is the texture axis, the hk rods are at distances

$$B_{hk} = (-h \cos \gamma/a + k/b)/\sin \gamma \quad (4.3.5.8)$$

from the texture axis and

$$D_{hk} = h/a \quad (4.3.5.9)$$

from the plane normal to the texture axis (the zero plane b^*c^*). On rotation, they intersect the plane normal to the incident beam and pass through the texture axis in layer lines at distances D_{hk} from the zero line, while the reflection positions along these lines are defined by their distances from the textures axis (see Fig. 4.3.5.3):

$$B_{hkl} = [B_{hk}^2 + (-hx_n - ky_n + l)^2/d_{001}^2]^{1/2}. \quad (4.3.5.10)$$

If the texture axis forms an angle ε with the a axis and $\delta = \varepsilon - \gamma + \pi/2$ with the projection of a^* on the plane ab , then

$$B_{hk} = \{-h(\sin \delta)/a + k[\sin(\gamma' - \delta)]/b\}/\sin \gamma \quad (4.3.5.11)$$

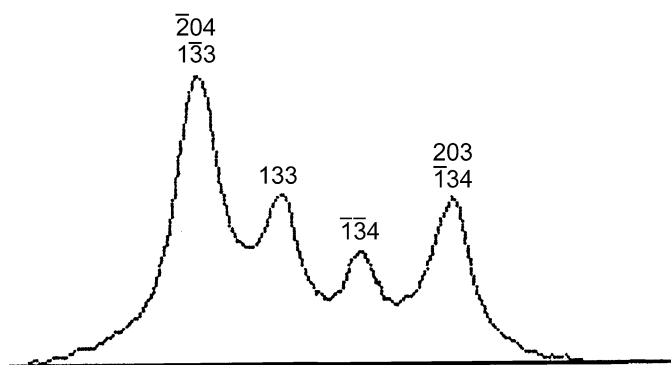
$$= \{-h[\cos(\gamma - \varepsilon)]/a + k \cos \varepsilon/b\}/\sin \gamma \quad (4.3.5.12)$$

$$D_{hk} = \{h(\cos \delta)/a + k[\cos(\gamma' - \delta)]/b\}/\sin \gamma \quad (4.3.5.13)$$

$$= \{h[\sin(\gamma - \varepsilon)]/a + k \sin \varepsilon/b\}/\sin \gamma. \quad (4.3.5.14)$$



(a)



(b)

Fig. 4.3.5.2. (a) Part of the OTED pattern of the clay mineral kaolinite and (b) the intensity profile of a characteristic quadruplet of reflections recorded with the electron diffractometry system. The scanning direction is indicated in (a).

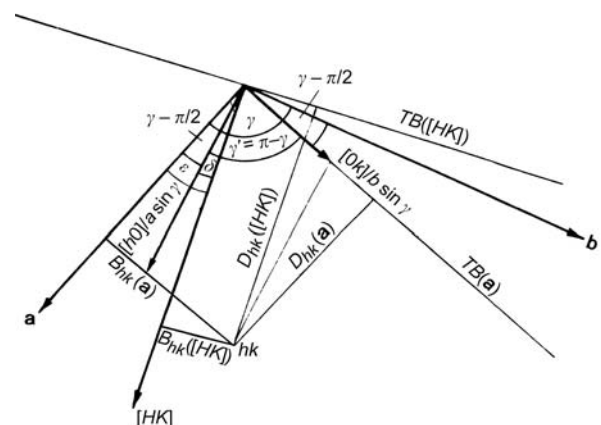


Fig. 4.3.5.3. The projections of the reciprocal axes on the plane ab of the direct lattice, with indications of the distances B and D of the hk rows from the fibre-texture axes a or $[hk]$.