

## 2. RECIPROCAL SPACE IN CRYSTAL-STRUCTURE DETERMINATION

spherical surface with the centre at the point 000; the oblique cross section of such bands produces reflections in the form of arcs. The main interference curves for texture patterns are ellipses imaging oblique plane cross sections of the cylinders  $hk$  (Fig. 2.5.4.3).

At the normal electron-beam incidence (tilting angle  $\varphi = 0^\circ$ ) the ED pattern represents a cross section of cylinders perpendicular to the axis  $c^*$ , i.e. a system of rings.

On tilting the specimen to an angle  $\varphi$  with respect to its normal position (usually  $\varphi \simeq 60^\circ$ ) the patterns image an oblique cross section of the cylindrical RL, and are called oblique-texture (OT) ED patterns. The ellipses ( $hk = \text{constant}$ ) and layer lines ( $l = \text{constant}$ ) for orthogonal lattices are the main characteristic lines of ED patterns along which the reflections are arranged. The shortcoming of oblique-texture ED patterns is the absence of reflections lying inside the cone formed by rotation of the straight line coming from the point 000 at an angle  $(90^\circ - \varphi)$  around the axis  $c^*$  and, in particular, of reflections  $00l$ . However, at  $\varphi \lesssim 60\text{--}70^\circ$  the set of reflections is usually sufficient for structural determination.

For unit-cell determination and reflection indexing the values  $d$  (i.e.  $|\mathbf{h}|$ ) are used, and the reflection positions defined by the ellipses  $hk$  to which they belong and the values  $\eta$  are considered. The periods  $a^*$ ,  $b^*$  are obtained directly from  $h_{100}$  and  $h_{010}$  values. The period  $c^*$ , if it is normal to the plane  $a^*b^*$  ( $\gamma^*$  being arbitrary), is calculated as

$$c^* = \eta/l = (h_{hkl}^2 - h_{hk0}^2)^{1/2}/l. \quad (2.5.4.5a)$$

For oblique-angled lattices

$$c^* = [(h_{h+l}^2 + h_{h-l}^2 - 2h_l^2)/2]^{1/2}/l. \quad (2.5.4.5b)$$

In the general case of oblique-angled lattices the coaxial cylinders  $hk$  have radii

$$b_{hk} = (1/\sin \gamma)[(h^2/a^2) + (k^2/b^2) - (2hk \cos \gamma/ab)]^{1/2} \quad (2.5.4.6)$$

and it is always possible to use the measured or calculated values  $b_{hk}$  in (2.5.4.5a) instead of  $h_{hk0}$ , since

$$\eta = (h_{hkl}^2 - b_{hk}^2)^{1/2}. \quad (2.5.4.7)$$

In OT patterns the  $b_{hk}$  and  $\eta$  values are represented by the lengths of the small axes of the ellipses  $B_{hk} = L\lambda b_{hk}$  and the distances of the reflections  $hkl$  from the line of small axes (equatorial line of the pattern)

$$D_{hkl} = L\lambda\eta/\sin \varphi = hp + ks + lq. \quad (2.5.4.8)$$

Analysis of the  $B_{hk}$  values gives  $a$ ,  $b$ ,  $\gamma$ , while  $p$ ,  $s$  and  $q$  are calculated from the  $D_{hkl}$  values. It is essential that the components of the normal projections  $c_n$  of the axis  $c$  on the plane  $ab$  measured in the units of  $a$  and  $b$  are

$$\begin{aligned} x_n &= (c/a)(\cos \beta - \cos \alpha \cos \gamma)/\sin^2 \gamma \\ &= -p/q, \\ y_n &= (c/b)(\cos \alpha - \cos \beta \cos \gamma)/\sin^2 \gamma \\ &= -s/q. \end{aligned} \quad (2.5.4.9)$$

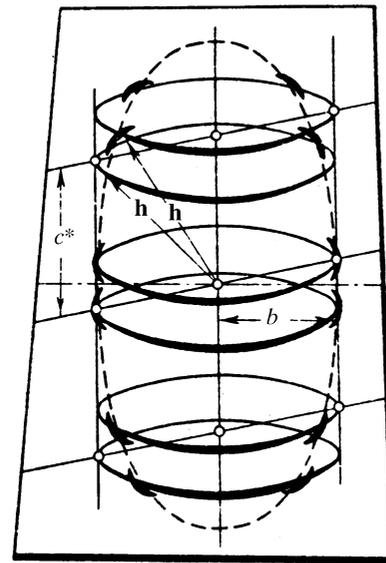


Fig. 2.5.4.3. Formation of ellipses on an electron-diffraction pattern from an oblique texture.

Obtaining  $x_n$ ,  $y_n$  one can calculate

$$c_n = [(x_n a)^2 + (y_n b)^2 + 2x_n y_n ab \cos \gamma]^{1/2}.$$

Since

$$\begin{aligned} d_{001} &= L\lambda/q \sin \varphi, \\ c &= (c_n^2 + d_{001}^2)^{1/2}. \end{aligned} \quad (2.5.4.10)$$

The  $\alpha$ ,  $\beta$  values are then defined by the relations

$$\begin{aligned} \cos \alpha &= (x_n a \cos \gamma + y_n b)/c, \\ \cos \beta &= (x_n a + y_n b \cos \gamma)/c. \end{aligned} \quad (2.5.4.11)$$

Because of the small particle dimensions in textured specimens, the kinematic approximation is more reliable for OT patterns, enabling a more precise calculation of the structure amplitudes from the intensities of reflections.

*Polycrystal ED patterns.* In this case, the RL is a set of concentric spheres with radii  $h_{hkl}$ . The ED pattern, like an X-ray powder pattern, is a set of rings with radii

$$r_{hkl} = h_{hkl}L\lambda. \quad (2.5.4.12)$$

## 2.5.4.3. Intensities of diffraction beams

The intensities of scattering by a crystal are determined by the scattering amplitudes of atoms in the crystal, given by (see also Section 5.2.1)

$$\begin{aligned} f_e^{\text{abs}}(s) &= 4\pi K \int \varphi(r)r^2 \frac{\sin sr}{sr} dr; \\ K &= \frac{2\pi m e}{h^2}; f_e = K^{-1}f_e^{\text{abs}}, \end{aligned} \quad (2.5.4.13)$$

where  $\varphi(r)$  is the potential of an atom and  $s = 4\pi(\sin \theta)/\lambda$ . The absolute values of  $f_e^{\text{abs}}$  have the dimensionality of length  $L$ . In EDSA it is convenient to use  $f_e$  without  $K$ . The dimensionality of