5.4. Electron-diffraction methods

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5.4.1. Determination of cell parameters from single-crystal patterns (By A. W. S. Johnson)

5.4.1.1. *Introduction*

This article treats the recovery of cell axes and angles from (*a*) a single pattern with suitable Laue zones and (*b*) two patterns with different zone axes. It is assumed that instrument distortions, if significant, are corrected and that the patterns are free of artefacts such as twinning, double diffraction *etc.* (Edington, 1975). The treatment is valid for convergent-beam, micro and selected-area electron-diffraction patterns and accelerating voltages above approximately 30 kV. Relevant papers are by LePage (1992) and Zuo (1993), and background reading is contained in Edington (1975), Gard (1976), and Hirsch, Howie, Nicholson, Pashley & Whelan (1965).

The basic requirement in the determination of the unit cell of a crystal is to find, from one or more diffraction patterns, the basis vector set, $\mathbf{a}^*$, $\mathbf{b}^*$, $\mathbf{c}^*$, of a primitive reciprocal unit cell. The Cartesian components of these vectors form an orientation matrix

$$\boldsymbol{UB} = (\mathbf{a}^*, \mathbf{b}^*, \mathbf{c}^*),$$

which, when inverted, gives the vector components of the corresponding real-space cell. The elements of $\boldsymbol{UB}$ can be measured directly from the diffraction pattern in millimetres. Define axes x and y to be in the recording plane and z in the beam direction. A point in the diffraction pattern x, y, z is then related to the indices h, k, l by

$$\begin{pmatrix} x \\ y \\ z \end{pmatrix} = \boldsymbol{UB} \begin{pmatrix} h \\ k \\ l \end{pmatrix}.$$

Note that points with non-zero z are observed on the plane $z = 0$, see Fig. 5.4.1.1.

The metric $\boldsymbol{M}$ of $\boldsymbol{UB}^{-1}$ is used to find the unit-cell edges and angles as

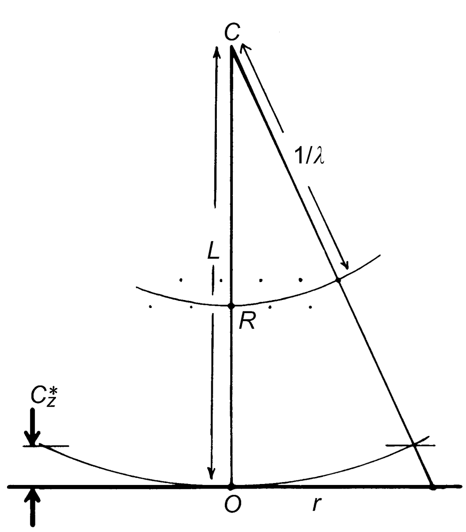

Fig. 5.4.1.1. Diffraction geometry. Crystal at C with the direct transmitted beam, CRO, intersecting the reciprocal-lattice origin at R and the recording plane at normal incidence at O. The camera length L is CO and the reciprocal of the wavelength λ is CR.

Table 5.4.1.1. *Unit-cell information available for photographic recording*

	Pattern type	Constants known	Information available
(1)	Zero zone	None or λ or L	d ratios and interplane angles
(2)		$L\lambda$ or L and λ	d values and interplane angles
(3)	Multiple zone	None or L	As for (1)
(4)		$L\lambda$	As for (2)
(5)		λ	Unit-cell axial ratios and angles
(6)		L and λ	Unit-cell axes and angles
(7)	Two or more zero-zone patterns*	None or L	As for (5)
(8)		$L\lambda$	As for (6)

* See text, Subsection 5.4.1.2.

$$\boldsymbol{M} = \boldsymbol{UB}^{-1} \cdot (\boldsymbol{UB}^{-1})^T,$$

where T means the transpose. Then,

$$\boldsymbol{M} = \begin{pmatrix} \mathbf{a}\cdot\mathbf{a} & \mathbf{a}\cdot\mathbf{b} & \mathbf{a}\cdot\mathbf{c} \\ \mathbf{a}\cdot\mathbf{b} & \mathbf{b}\cdot\mathbf{b} & \mathbf{b}\cdot\mathbf{c} \\ \mathbf{a}\cdot\mathbf{c} & \mathbf{b}\cdot\mathbf{c} & \mathbf{c}\cdot\mathbf{c} \end{pmatrix}$$

gives

$$a = L\lambda(\mathbf{a}\cdot\mathbf{a})^{1/2},$$
$$b = L\lambda(\mathbf{b}\cdot\mathbf{b})^{1/2},$$
$$c = L\lambda(\mathbf{c}\cdot\mathbf{c})^{1/2},$$
$$\cos\gamma = \mathbf{a}\cdot\mathbf{b}/(\mathbf{a}\cdot\mathbf{a}\ \mathbf{b}\cdot\mathbf{b})^{1/2},$$
$$\cos\beta = \mathbf{a}\cdot\mathbf{c}/(\mathbf{a}\cdot\mathbf{a}\ \mathbf{c}\cdot\mathbf{c})^{1/2},$$

and

$$\cos\alpha = \mathbf{b}\cdot\mathbf{c}/(\mathbf{b}\cdot\mathbf{b}\ \mathbf{c}\cdot\mathbf{c})^{1/2},$$

where L is the effective distance between the diffracting crystal and the recording plane and λ is the wavelength. These quantities are defined in Fig. 5.4.1.1 together with the nomenclature and geometrical relationships required in this article.

If necessary, the cell is reduced to the Bravais cell according to the procedures given in *IT* A (1983, Chapter 9.3), before calculating the metric.

In practice, there may be a difficulty in choosing a vector set that describes a *primitive* reciprocal cell. Although a record of any reasonably dense plane of reciprocal space immediately exposes two basis vectors of a cell, the third vector lies out of the plane of the diffraction pattern containing the first two vectors and may not be directly measurable. Hence, some care must be taken to ensure that the third vector chosen makes the cell

references