

5.4. ELECTRON-DIFFRACTION METHODS

have been developed based on Kikuchi or HOLZ (high-order Laue zone) lines (Uyeda, Nonoyama & Kogiso, 1965; Høier, 1969; Olsen, 1976a; Jones, Rackham & Steeds, 1977). In these methods, the lattice parameters can be determined, provided the electron wavelength is known (or *vice versa*) without knowing the value of K .

In the method of Uyeda *et al.* (1965), the electron wavelength can be determined from a single Kikuchi pattern provided that the lattice parameters of the crystal are known. Fig. 5.4.2.1 illustrates the geometry involved in the method. **A** and **B** are two zone axes of the specimen. The pairs of Kikuchi lines g , $-g$ and h , $-h$ belong to the zones **A** and **B**, respectively. The points P and Q are the intersections of the Kikuchi lines g and h with the line AB .

As a first approximation, the wavelength of the electrons is given by

$$\lambda = \theta_{AB} / [(1/2d_P) + (1/2d_Q) + (\Delta/D)(1/d)], \quad (5.4.2.1)$$

where D is the measured distance between the two Kikuchi lines in either of the pairs and d is the corresponding interplanar spacing. θ_{AB} is the angle between the zone axes **A** and **B**. When the Kikuchi pattern is indexed, θ_{AB} and d can be calculated from the known lattice parameters of the specimen. d_P and d_Q are effective interplanar spacings and are given by

$$\begin{aligned} d_P &= d_g \sin \varphi_g, \\ d_Q &= d_h \sin \varphi_h; \end{aligned} \quad (5.4.2.2)$$

d_g and d_h are the interplanar spacings corresponding to g and h , respectively. The angle φ_g (or φ_h) is the angle between the lattice plane g (or h) and the plane defined by **A** and **B**. These angles are given by

$$\begin{aligned} \sin \varphi_g &= \mathbf{g} \times (\mathbf{A} \times \mathbf{B}) / (|\mathbf{g}| \cdot |\mathbf{A} \times \mathbf{B}|), \\ \sin \varphi_h &= \mathbf{h} \times (\mathbf{A} \times \mathbf{B}) / (|\mathbf{h}| \cdot |\mathbf{A} \times \mathbf{B}|). \end{aligned} \quad (5.4.2.3)$$

The values of d_P and d_Q to be used in (5.4.2.1) can be calculated from (5.4.2.2) and (5.4.2.3).

If Δ and D are measured on the photographic plate or a print of any magnification, the electron wavelength can be calculated by using (5.4.2.1). Only the ratio (Δ/D) is required for the determination of λ . The distance AB is not required. The points A and B are used only to fix the points P and Q . A and B do not need to fall inside the photographic plate when a Kikuchi pattern is symmetrical across the line AB , because in such cases P and Q can be determined from intersections of Kikuchi lines that are symmetrical with each other. The expression for λ in (5.4.2.1) is only a first approximation. More accurate expressions can be found in the paper by Uyeda *et al.* (1965).

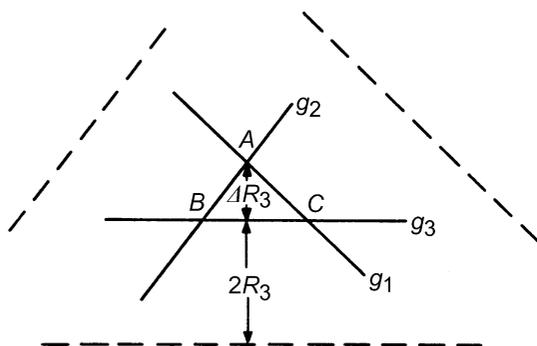


Fig. 5.4.2.2. Schematic diagram of three Kikuchi lines that nearly intersect at the same point.

A simpler and more accurate method has been developed by Høier (1969). He showed that in the cubic case it is possible to determine the ratio between the lattice parameter a and the electron wavelength λ with a relative accuracy of 0.1%. Only two quantities have to be measured on the photographic plate or a print at any magnification: the height of a triangle formed by three Kikuchi lines and one separation between a defect-excess line pair (Fig. 5.4.2.2). If three indexed Kikuchi lines g_i intersect at the same point on a photographic plate, the following equations can be derived from Bragg's law:

$$2\mathbf{g}_i \mathbf{K} = -|\mathbf{g}_i|^2, \quad i = 1, 2, 3. \quad (5.4.2.4)$$

In addition, the length of \mathbf{K} is equal to the electron wavelength

$$|\mathbf{K}| = 1/\lambda_i, \quad (5.4.2.5)$$

where the wavelength is written λ_i for generality (for electrons, $\lambda_i = \lambda$). If the Kikuchi lines g_i do not belong to the same zone, (5.4.2.4) and (5.4.2.5) can be solved and a/λ determined.

Exact triple intersections are rare. A practical method as proposed by Høier (1969) is therefore based on three lines that nearly intersect at the same point (Fig. 5.4.2.2). The dimensions of the triangle ABC in Fig. 5.4.2.2 change with λ . Let us assume that only the wavelength in the beam giving the g_3 reflection varies. The increment $\Delta\lambda_3$ necessary to shift the line g_3 to A is then determined by

$$\lambda_3 = \lambda + \Delta\lambda_3 \sim \lambda(1 + \Delta R_3/R_3). \quad (5.4.2.6)$$

By measuring the height in the triangle ABC and the line separation $2R_3$, the wavelength to be used in (5.4.2.5) can be calculated and the ratio a/λ determined. Care must be taken to avoid areas where the lines are displaced from kinematical positions owing to dynamical interactions.

The method of Høier (1969) for cubic crystals was later extended to lower-symmetry cases (triclinic) by Olsen (1976a), who also developed computer programs for least-squares refinement of the lattice parameters. The derivation of the equations for the procedure is carried out in the following in a way slightly different from that described by Olsen (1976a).

If three Kikuchi lines $\mathbf{g}_i(h_i, k_i, l_i)$ not belonging to the same zone intersect at the same point on the photographic plate, the direction \mathbf{K} from the origin of the Ewald sphere to the intersection point of the Kikuchi lines is given by Bragg's law:

$$2\mathbf{g}_i \mathbf{K} = -|\mathbf{g}_i|^2, \quad i = 1, 2, 3. \quad (5.4.2.7)$$

The length of the vector \mathbf{K} is given by

$$|\mathbf{K}| = 1/\lambda, \quad (5.4.2.8)$$

where λ is the electron wavelength.

Because triple intersections are rare, a practical method is therefore based on three lines that nearly intersect at the same point, as proposed by Høier (1969). In order to obtain an exact intersection, one of the lines can be changed from \mathbf{g}_i to $\mathbf{g}_i + \Delta\mathbf{g}_i$, where $\Delta\mathbf{g}_i$ is a vector approximately parallel to \mathbf{g}_i . For this Kikuchi line (in the following assumed to be line no. 3), the Bragg condition (5.4.2.7) gives

$$2(\mathbf{g}_3 + \Delta\mathbf{g}_3) \mathbf{K} = -(\mathbf{g}_3 + \Delta\mathbf{g}_3)^2. \quad (5.4.2.9)$$

For a pair of Kikuchi lines, the simple relation

$$2\Delta\mathbf{g}_3 = (\Delta R_3/R_3)\mathbf{g}_3 \quad (5.4.2.10)$$

holds, where $2R_3$ is the line separation measured on the photographic plate of the centres of the two Kikuchi lines. ΔR_3 is the shift (measured on the plate) in the position of one of the lines in order to obtain an exact triple intersection.