Substitution of (5.4.2.10) into (5.4.2.9) gives

$$2\mathbf{g}_3\mathbf{K} = -|\mathbf{g}_3|^2(1 + \Delta R_3/R_3). \quad (5.4.2.11)$$

From n measurements of intersections, the following equations are obtained:

$$h_{ji}u_j + k_{ji}v_j + l_{ji}w_j = -(1/2)(1/d_{ji})^2(1 + \delta_{i3}\Delta R_{j3}/R_{j3}),$$
$$i = 1, 2, 3, \quad j = 1, 2, \ldots, n, \quad (5.4.2.12)$$

where h_{ji}, k_{ji}, l_{ji} are the indices (given in reciprocal space) of the Kikuchi lines, δ_{i3} is the Kronecker delta, u_j, v_j, w_j are the indices (given in real space) of the direction $\mathbf{K}_j$ to the intersection number $\mathbf{j}$, and d_{ji} is the interplanar spacing corresponding to the reflection h_{ji}, k_{ji}, l_{ji} and can be expressed in terms of the cell dimensions in real space. The length of the $\mathbf{K}_j$ vectors can also be expressed in terms of the lattice parameters in real space a, b, c, α, β, and γ as

$$|\mathbf{K}_j| = (u_j^2a^2 + v_j^2b^2 + w_j^2c^2 + 2u_jv_j\,ab\cos\alpha + 2u_jw_j\,ac\cos\beta + 2v_jw_j\,bc\cos\gamma)^{1/2}. \quad (5.4.2.13)$$

Equations (5.4.2.12) can be solved with respect to u_j, v_j, w_j, and $\mathbf{K}_j$ can then be expressed in terms of the lattice parameters by substituting the expressions for u_j, v_j, w_j from (5.4.2.12) into (5.4.2.13).

If the number of intersections is greater than the number of unknown lattice parameters, a least-squares-refinement procedure can be used. This involves minimizing the function

$$Q = \sum_j (|\mathbf{K}_j| - 1/\lambda)^2. \quad (5.4.2.14)$$

Because the expression for Q is non-linear in the lattice parameters, a refinement procedure can be used to solve the equations derived from (5.4.2.14) using the least-squares procedure. The accuracy in the lattice parameters or wavelength can be found by statistical methods. A computer program for refinement of the lattice parameters (or electron wavelength) is available (Olsen, 1976*b*). In this program, some of the lattice parameters can be held constant or kept equal during the refinement.

Methods based on measurements of distances between zone axes (poles) in the Kikuchi patterns (Thomas, 1970) are not very accurate because optical distortions make long-distance measurements inaccurate on the photographic plate or a magnified print.

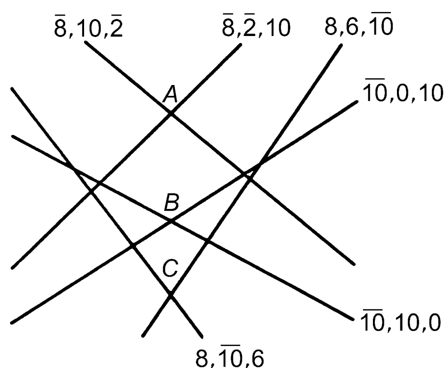

Fig. 5.4.2.3. Schematic diagram showing the indexing of the most prominent lines in the selected-area channelling pattern near the [111] zone of Si. Accelerating voltage: 25 kV.

The methods by Høier (1969) and Olsen (1976*a*) are based on 'near intersections'. Better accuracy may be obtained if the high voltage can be varied in order to obtain exact intersections. A simple method for determination of lattice parameters can be applied for crystals with symmetry as low as orthorhombic (Gjønnes & Olsen, 1984). The method is a simplified version of the Uyeda *et al.* (1965) method. If two parallel Kikuchi lines belonging to different zones overlap, then:

$$2\mathbf{g}_i\mathbf{K} = -|\mathbf{g}_i|^2, \quad i = 1, 2;$$
$$(\mathbf{g}_1 \times \mathbf{g}_2)\mathbf{K} = 0, \quad |\mathbf{K}| = 1/\lambda. \quad (5.4.2.15)$$

These equations can be solved to give the ratio between lattice parameter and electron wavelength.

A simple, rapid procedure for accelerating-voltage (or electron-wavelength) determination of a transmission electron microscope has been described by FitzGerald & Johnson (1984). In their method, it is necessary to measure the ratio of two easily found distances between points defined by the intersections of Kikuchi lines near the $\langle 111 \rangle$ zone of a silicon crystal. It is not necessary to index the Kikuchi lines, because the method is based on (*a*) a particular crystal and (*b*) an easily recognizable crystal orientation, and because the points between which distances need to be measured are specified in their paper and can be easily found. Polynominals for converting the distance measurements both to electron wavelength and to accelerating voltage are given for the range from 100 to 200 kV. A 300 K temperature change must occur (temperature coefficient of 3×10^{-6} K) before the error in the lattice parameter of silicon due to thermal expansion becomes significant.

A method for measuring small local changes in the lattice parameter of bulk specimens based on selected-area channelling patterns has been described by Walker & Booker (1982). The method utilizes the small changes in the position of high-index channelling lines due to small changes in the lattice parameters, and is based on scanning electron microscopy (SEM). The method is rapid and convenient, is suitable for bulk specimens, and can be applied to areas only a few micrometres across. The method has been used for Si and GaP and is in this case based on the intersection of pairs of $\bar{8},10,\bar{2}$, $\overline{10},10,0$, $8,\overline{10},6$ type lines (the points A, B, and C in Fig. 5.4.2.3). Selected-area channelling patterns from Si and GaP obtained under precisely the same conditions have the same characteristic array of lines, but with slightly different distances AB and BC. The detection limit of this method is at present 3 parts in 10^4.

When a small electron probe is used to illuminate a crystal, as in convergent-beam electron diffraction (CBED), very fine lines are often found in the diffraction discs (Steeds, 1979). These lines are called HOLZ lines. They are due to upper-layer diffraction effects and can be used in lattice-parameter determination (Rackham, Jones & Steeds, 1974; Jones, Rackham & Steeds, 1977). For highest accuracy, relatively thick crystals are required. The limitation to the accuracy is set either by the weakness of the lines or by energy losses in the specimen. Relative changes in the lattice parameters can be measured to an accuracy of 1 part in 10^4, whereas the accuracy in the absolute determination of lattice parameters is typically of an order of magnitude worse. The lattice parameters can be measured from crystal regions as small as 20 nm in diameter with an accuracy better than 1 part in 10^3. If more accurate results are desired, it is necessary to make measurements on lines that are not affected by interactions between HOLZ reflections.

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