(2) If, by additional heating, a temperature gradient was introduced, perpendicular to the plane formed by the incoming and reflected beam, the excursion also disappeared.

Recent measurements by Umbayashi, Frazer, Shirane & Daniels (1967) which were done with a small sample and an extremely low cooling rate (1 °/200 min) still showed a small excursion. This leaves open the question whether the spread in d is only due to a temperature gradient or partly to some fluctuation mechanism that is associated with the phase transition.

As an application of our results we suggest that the reflectivity of nearly perfect crystals used as neutron monochromators may be increased by introducing large temperature gradients normal to the reflecting planes.

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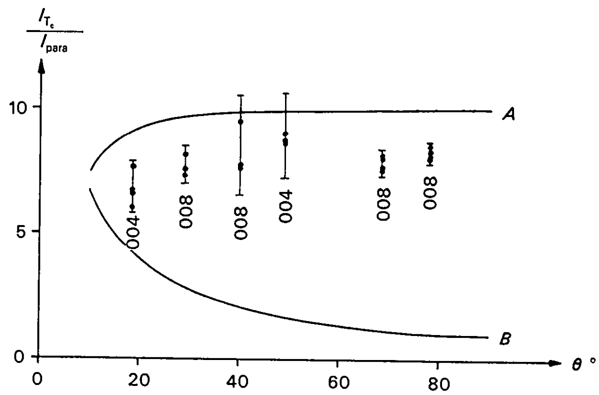

Fig. 2. Ratio J_{Tc}/J_{para} for (004) and (008) reflexions *versus* Bragg angle.

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A Method to Determine the Ratio between Lattice Parameter and Electron Wavelength from Kikuchi Line Intersections

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Three Kikuchi lines not belonging to the same zone, which nearly intersect at the same point, can be utilized for determination of the ratio between lattice parameter and electron wavelength. The method given is analogous to a method based on Kossel line intersections in the X-ray case. One example is given in which the electron wavelength is determined with an uncertainty of *ca.* 0·1%.

Determinations of lattice parameters in selected area electron diffraction necessitate careful calibrations of the camera constant λL, because neither the camera length, L, nor the wavelength, λ, can be determined separately. It is possible, however, to utilize the Kikuchi lines in analogy with the highly efficient use of Kossel lines in the X-ray case. The lattice parameter (a_0) can thus be determined, provided the wavelength is known (or *vice versa*), as shown by Uyeda, Nonoyama & Kogiso (1965).

The purpose of this note is to give an alternative method which enables a determination of the ratio a_0/λ with a relative uncertainty of *ca.* 0·1%. Only two quantities have to be measured on the photographic plate, or a reproduction at any magnification: the height of a triangle formed by three Kikuchi lines and one separation between a deficient-excess line pair.

It is assumed that diffuse scattering from the direct beam involves only negligible energy losses. Only cubic crystals are discussed, although the method can be extended to crystals with lower symmetry, as has been done in the X-ray case by Mackay (1962).

If three indexed Kikuchi lines $\mathbf{H}_i(h_i,k_i,l_i)$ intersect at the same point on the photographic plate, the following equations can be taken over from the Kossel technique (Sharpe, 1965):

$$\mathbf{H}_i \,.\, \mathbf{r}_0 = \tfrac{1}{2} a_0 \lambda_i |\mathbf{H}_i|^2 \,, \quad i=1,2,3 \,, \qquad (1a,b,c)$$

where

$$|\mathbf{r}_0|^2 = x_0^2 + y_0^2 + z_0^2 = 1 \,. \qquad (2)$$

$\mathbf{H}_i$ is a reciprocal lattice vector and $-\mathbf{r}_0$ a unit vector in the direction of the beam which satisfies Bragg's law simultaneously for the three $\mathbf{H}_i$. The wavelength is written λ_i for generality; with electrons we have $\lambda_i = \lambda$.

Provided the $\mathbf{H}_i$'s do not belong to the same zone equations (1) can be solved to give x_0, y_0 and z_0 as functions of a_0 and λ. Substituting in equation (2) the expressions thus obtained we get

$$a_0/\lambda = f(h_i, k_i, l_i) \,. \qquad (3)$$

The analytical form of the right hand side of equation (3) is cumbersome. It is not given here because practical calculations are most readily performed by introducing numerical values directly into equations (1).

Exact triple intersections are rare. A practical method must therefore be based on three lines which nearly intersect at the same point, as shown schematically in Fig. 1. The dimensions of the triangle ABC in the Figure change with λ, and at a certain $\Delta\lambda > 0$ the $\mathbf{H}_i$ lines intersect at D. This $\Delta\lambda$ might be calculated and a_0/λ determined substituting $\lambda_i = \lambda + \Delta\lambda$ in equations (1). However, the expression thus obtained for $\Delta\lambda$ is unwieldy for practical use. In order to arrive at a simpler form, let us start with the non-physical assumption that only the wavelength in the beam giving the $\mathbf{H}_3$ reflexion varies. The increment $\Delta\lambda_3$ necessary to shift the $\mathbf{H}_3$ line to A (Fig. 1) is determined on derivation of Bragg's law, giving:

$$\lambda_3 = \lambda + \Delta\lambda_3 \simeq \lambda(1 + \Delta R_3/R_3) \,. \qquad (4)$$

By measuring the height ΔR_3 in the triangle ABC and the line separation $2R_3$, we can thus calculate the wavelength to be used in equation (1c), and as before, through elimination of x_0, y_0 and z_0, determine the ratio a_0/λ, *viz.*

$$a_0/\lambda = f(h_i, k_i, l_i, \Delta R_3/R_3) \,.$$

(In many cases $2R_3$ cannot be measured directly, but it can be deduced from any measured line separation on the plate.)

To illustrate the calculation procedure the case $\mathbf{H}_1 = (\bar{8}84)$, $\mathbf{H}_2 = (\overline{16},\bar{4},\bar{4})$ and $\mathbf{H}_3 = (16,0,0)$ in Table 1, is chosen. Thus, $\Delta R_3/R_3 = \Delta R_{16,0,0}/R_{16,0,0}$ in equation (4) and equation (1) become:

$$-8x_0 + 8y_0 + 4z_0 = 72\lambda/a_0 \,,$$
$$-16x_0 - 4y_0 - 4z_0 = 144\lambda/a_0 \,,$$
$$16x_0 = 128\lambda\,(1 + \Delta R_{16,0,0}/R_{16,0,0})/a_0 \,.$$

$\Delta R_{16,0,0}$ is the height from the $(\bar{8}84)$, $(\overline{16},\bar{4},\bar{4})$ intersection point to the (16,0,0) line in Fig. 2, and $2R_{16,0,0}$ the $\pm(16,0,0)$ line pair separation. These equations are easily solved to give x_0, y_0 and z_0, and the expressions thus obtained are then substituted in equation (2). Using the measured value $\Delta R_{16,0,0}/R_{16,0,0} = 0{\cdot}136$ and the value of a_0 given below, we get $\lambda = 0{\cdot}03827$ Å.

The error involved in neglecting higher order terms in equation (4) is in normal cases negligible. The major uncertainty arises from the measurement of ΔR_3. To reduce the influence of the error in this measurement it is therefore desirable to have a rapid change in ΔR_3 with variations in the wavelength which occur when all $\mathbf{H}_i$ have high indices, provided $\mathbf{H}_3$ is not too far from being parallel to $-(\mathbf{H}_1 + \mathbf{H}_2)$.

Care must be taken to avoid areas where the lines are displaced owing to dynamical interactions which may introduce displacements from the kinematical positions.

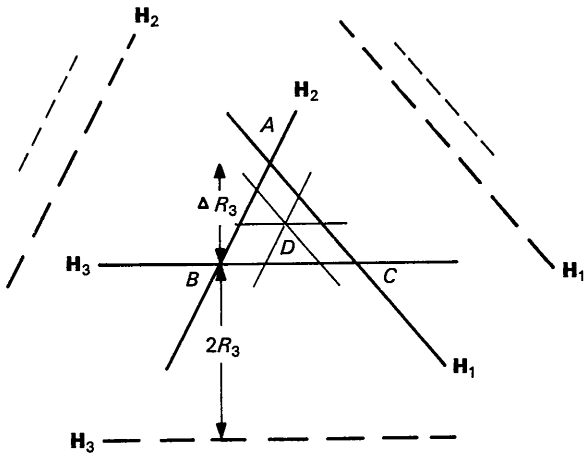

Fig. 1. Schematic drawing of a near triple intersection of Kikuchi lines. Heavy line: position at wavelength λ. Thin line: position at wavelength $\lambda + \Delta\lambda$, $\Delta\lambda > 0$.

Table 1. *Calculated electron wavelength and acceleration voltage from near triple intersections of Kikuchi lines*

$\mathbf{H}_1$	$\mathbf{H}_2$	$\mathbf{H}_3$	λ	$\bar{\lambda}$	E
$\bar{8}\ 8\ 4$	$\overline{16}\ \bar{4}\ \bar{4}$	16 0 0	0·03827 Å		
$\bar{8}\ 8\ 4$	$\overline{13}\ \bar{3}\ \bar{3}$	16 0 0	0·03821		
$\bar{8}\ 8\ 4$	$\bar{4}\ \overline{12}\ \bar{8}$	16 0 0	0·03821		
$\bar{8}\ 8\ 4$	$\bar{9}\ \bar{7}\ \bar{5}$	16 0 9	0·03827		
$\overline{10}\ \bar{6}\ \bar{4}$	0 16 8	8 0 0	0·03826	0·03823 Å	94·2 kV
$\overline{10}\ \bar{6}\ \bar{4}$	$13\ \bar{5}\ \bar{3}$	0 16 8	0·03822	±0·00003	
$\overline{10}\ \bar{6}\ \bar{4}$	$\bar{9}\ 3\ 1$	16 0 0	0·03823		
$\overline{10}\ \bar{6}\ \bar{4}$	$\bar{4}\ 8\ 4$	12 0 0	0·03820		

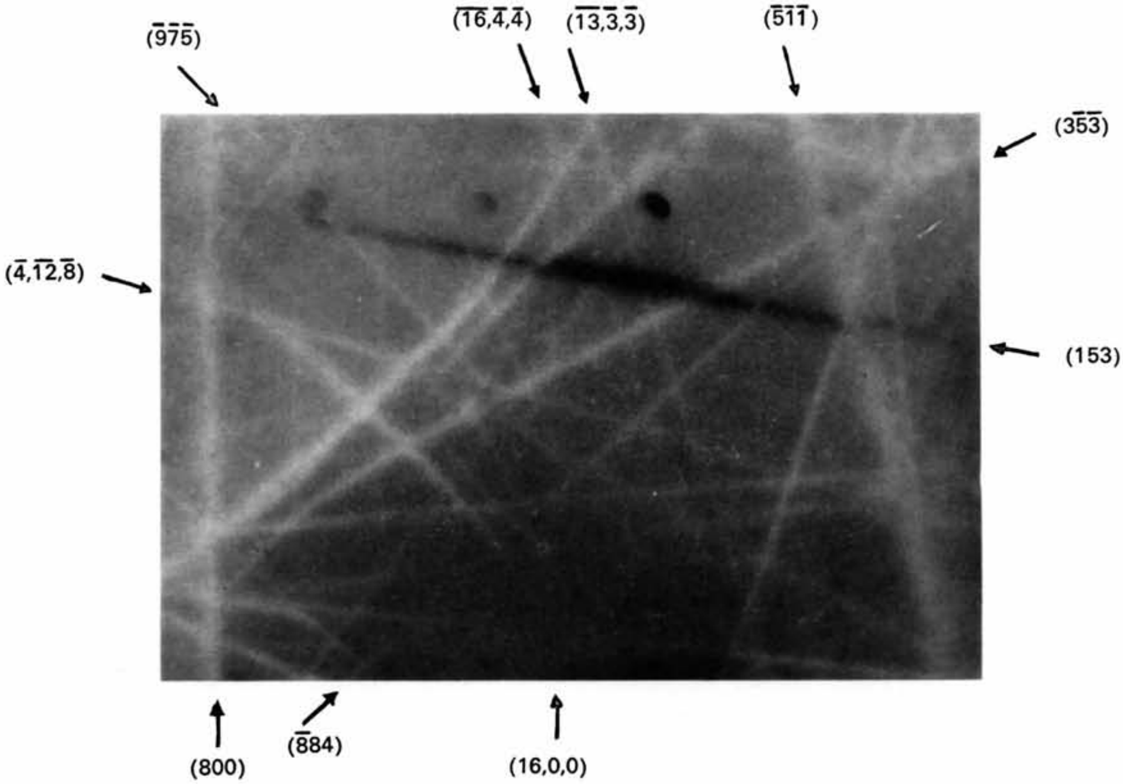

Fig. 2. Transmission Kikuchi pattern from $MgAl_2O_4$ showing some of the deficient line intersections used in the calculations. Voltage: 94·2 kV.

The method has been used on transmission Kikuchi line patterns from the cubic crystal natural spinel, $MgAl_2O_4$. An enlarged section of one of the plates, containing some of the intersections used, is shown in Fig. 2.

From the lattice constant of $a_0(26\,^{\circ}\mathrm{C}) = 8{\cdot}0800$ Å (Wyckoff, 1965), the wavelength associated with the 100 kV switch on a JEM-7 electron microscope is determined. The results are given in Table 1.

The uncertainty in $\Delta R_3/R_3$ can for small ratios be allowed to be as high as 10%. Still the uncertainty in the calculated λ is less than 0·3% which is the estimated *maximum* uncertainty in a single determination. The deviation from the mean value gives a relative uncertainty in this quantity of less than 0·1%.

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Calculation of Absorption Corrections for Photographic Data

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An extension of the methods of Wells (*Acta Cryst.* (1960). **13**, 722) is described for calculating the direction cosines of incident and emergent rays for general camera geometry and for any standard setting of the crystal.

Wells (1960) gives methods for determining the directions of the incident and emergent rays for equi-inclination, normal-beam and precession geometry, with the crystal in a standard setting, with c as the principal axis. This paper generalizes his results (*a*) for any camera geometry, (*b*) for alternative settings of the crystal. Wells's notation is used throughout.

(*a*) *Generalization for any camera geometry*

In particular this covers data recorded by the inclined-beam oscillation technique (Milledge, 1963) and has two aspects

(1) to allow for l taking negative values,

(2) to calculate the sign of the direction cosine ($\cos \angle ZE$), between the principal axis and the emergent ray, when this angle lies in the range $0-\pi$, rather than $0-\pi/2$ (equi-inclination) or $\pi/2-\pi$ (precession).

Wells defines a set of orthogonal axes with $OX \equiv a^*$, OY in the a^*-b^* plane and OZ on the same side of the XY plane as c^* (Fig. 1). Then, considering a reflexion hkl (point P), he examines the lth layer of the reciprocal lattice [Fig. 2(*a*)] and derives the lengths and angles:

$$L_1 = lc^* \sin \omega_2\,,$$

L_2 and ω_3 (determined by the cell constants), ω_4 (a function of h and k only) and

$$\omega_5 = \pi + \omega_4 - \omega_3\,,$$

which determine

$$L_3 = (L_1^2 + L_2^2 - 2L_1L_2 \cos \omega_5)^{1/2}\,.$$

He does not consider the consequence of L_2 being zero, *i.e.* P lying on c^*. In this case ω_4 and ω_5 are both indeterminate, but $L_3 = L_1$ and $\omega_6 = \omega_2$, $\omega_7 = \omega_3$. If L_3 is also zero the reciprocal lattice point lies on OZ, and can never be recorded properly by photographic means.

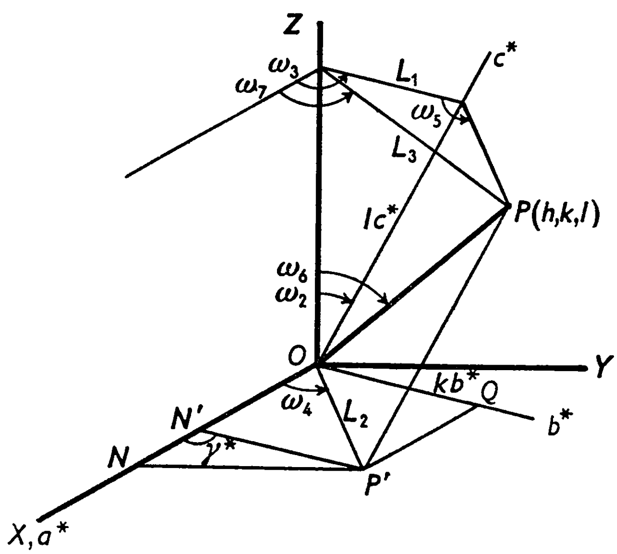

Fig. 1. The reciprocal lattice showing the angles and lengths referred to in the text (from Wells, 1960).