elemental structures from which standard CN 12 diameters are derived!

(2) It shows that an invariant parameter in a uniaxial crystal structure may couple the variable parameters of the structure, so as to produce apparent dependences of the cell edges on the diameter of a component atom, which could not possibly arise intrinsically through the direct contacts of the atom with its neighbours.

(3) The observed variations of the cell dimensions of phases with the $AlCr_2C$ structure lead to some interesting observations on the nature of the electronic interactions in true interstitial phases. Hopefully, these may stimulate band-structure calculations to confirm or reject the reliability of such analyses.

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SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.*

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Weissenberg photographs: triclinic-cell parameters from one crystal setting.

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Abstract

A simple method is given for deriving the triclinic-cell parameters from three general reflections by vertical or horizontal measurements on upper-level films.

Introduction

The problem of obtaining, with a Weissenberg camera, all unit-cell parameters from one crystal setting has been studied by different authors. In most methods, measurements on an upper-level photograph serve to determine the origin shift of this level relative to the true origin. Then, two direct angles can be calculated and the cell is completely defined, the four other cell parameters being provided by the oscillation and the zero-level photographs. Buerger (1942, pp. 377ff.) proposed a method of angular lag which requires the recording of both the zero and an upper level on the same film, and involves measurements of low-angle axial reflections. Hulme's (1966) method, easier and more accurate, enables one to determine the unknown angles from measurements of the angular distance between general reflections, either graphically or by iteration. More recently, Hebert (1978) has described a procedure for deriving these angles from measurements of distances between festoons representing either axes or r.l. lines parallel to the axes. A method, quite different from these, has also been reported by Alcock & Sheldrick (1967) where all reciprocal-cell parameters are calculated from the separations of the α_1–α_2 doublets and refined by least squares. In the following method, vertical or horizontal measurements involving three general reflections lead to an easy determination of the remaining reciprocal-cell parameters. These are accurate enough for preliminary work and may be refined later with a diffractometer.

Vertical measurements

Let us consider (Fig. 1) a crystal rotated about c. The r.l. point P has two components, ζ parallel to the rotation axis

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and ξ in the a^*b^* plane. Then,

$$d^{*2}_{hkl} = \xi^2 + \zeta^2. \tag{1}$$

ζ is measured on the oscillation photograph and is related to c by

$$\zeta = lc^{-1}. \tag{2}$$

ξ is obtained from the nth-level film by measuring, in mm, x, the perpendicular distance from a reflection to the central line. One way of indicating the central line is to let the direct beam fall for a few seconds on both extremities of the film and join the centers of the spots with a fine pen. Alternatively, one can record the direct beam by manually moving the cassette throughout its range, but this tends to give a thicker trace. A relation between ξ and x can be derived from two expressions given by Buerger (1942, pp. 223, 254ff.) for the reflection angle Ψ,

$$2 \sin^{-1}(\xi/2 \cos \mu) = \Psi = C_1 x,$$

where C_1 is an instrumental constant equal to 2°/mm for a camera diameter of 57·3 mm and μ is the equi-inclination angle. For such a camera, ξ, expressed in Å^{-1}, is given by

$$\xi = 2\lambda^{-1} \sin x \cos \mu. \tag{3}$$

The general expression for d^*_{hkl} can be written in the form

$$l^2c^{*2} + 2hla^* c^* \cos \beta^* + 2klb^* c^* \cos \alpha^* = d^{*2}_{hkl} - d^{*2}_{hk0}, \tag{4}$$

where

$$d^{*2}_{hk0} = h^2 a^{*2} + k^2 b^{*2} + 2hka^* b^* \cos \gamma^*. \tag{5}$$

Equation (4) can be rewritten in terms of the observable quantities ξ^2_{hk0} $(=d^{*2}_{hk0})$ and ξ^2_{hkl} $(=d^{*2}_{hkl} - l^2/c^2)$:

$$l^2(c^{*2} - c^{-2}) + 2hla^* c^* \cos \beta^* + 2klb^* c^* \cos \alpha^* = \xi^2_{hkl} - \xi^2_{hk0}. \tag{6}$$

Three reflections give rise to three equations of type (6) which may be solved by inverting the matrix of the indices

$$\begin{pmatrix} c^{*2} - c^{-2} \\ 2a^* c^* \cos \beta^* \\ 2b^* c^* \cos \alpha^* \end{pmatrix} = \begin{pmatrix} l_1^2 & h_1 l_1 & k_1 l_1 \\ l_2^2 & h_2 l_2 & k_2 l_2 \\ l_3^2 & h_3 l_3 & k_3 l_3 \end{pmatrix}^{-1} \begin{pmatrix} \xi^2_{h_1k_1l_1} - \xi^2_{h_1k_10} \\ \xi^2_{h_2k_2l_2} - \xi^2_{h_2k_20} \\ \xi^2_{h_3k_3l_3} - \xi^2_{h_3k_30} \end{pmatrix}. \tag{7}$$

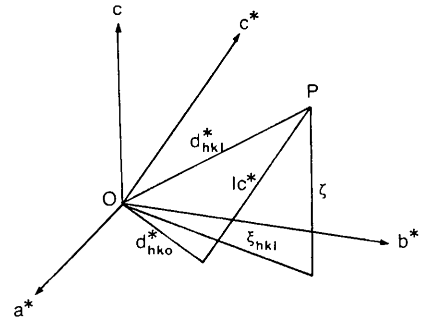

Fig. 1. The components ζ and ξ of d^*_{hkl}.

Frequently, the first level ($l = 1$) will be the only upper level recorded in which case the inverse matrix simplifies to

$$\frac{1}{D}\begin{pmatrix} h_2 k_3 - h_3 k_2 & h_3 k_1 - h_1 k_3 & h_1 k_2 - h_2 k_1 \\ k_2 - k_3 & k_3 - k_1 & k_1 - k_2 \\ h_3 - h_2 & h_1 - h_3 & h_2 - h_1 \end{pmatrix},$$

where the determinant $D = h_1(k_2 - k_3) + h_2(k_3 - k_1) + h_3(k_1 - k_2)$. Since a^* and b^* are known from the zero level, and c^{-1} from the oscillation photograph, (7) yields the remaining r.l. parameters.

Horizontal measurements

When vertical measurements cannot be made because the central line cannot be marked on the film, the ξ's can be calculated from horizontal measurements as shown below.

In Fig. 2, P_1 and P_2 are r.l. points in the nth layer and O is the point of intersection of the oscillation axis with this layer. The angle, φ_{12}, subtended by P_1 and P_2 at O is measured on the film by the horizontal distance in mm (1 mm = 2° in φ) between lines of slope 2 passing through the corresponding reflections. Hulme (1966) has described two methods of using these measurements to determine the shift of the nth-level origin relative to O and, thence, two angles of the direct cell: one method is a least-squares iteration necessitating the availability of a computer, and the other is graphical and, therefore, not very accurate. The following method is simple enough for hand calculation.

In Fig. 2, as the points P_1, P_2 and P_3 are r.l. points in the nth layer, the distances marked d^*_{12}, d^*_{13} and d^*_{23} are deducible from the corresponding distances between r.l. points in the zero layer. Thus, $d^*_{12} = d^*_{(h_2k_2l - h_1k_1l)} = d^*_{h_2 - h_1,\, k_2 - k_1,\, 0)}$ *etc.* These three distances define a triangle $P_1P_2P_3$ and the angle ψ. The values of ξ_1, ξ_2 and ξ_3 can be obtained *via* the angles σ and τ as follows

$$\sigma + \tau = 360 - (\psi + \varphi_{12} + \varphi_{23}) \tag{8}$$

$$\frac{d^*_{12} \sin \sigma}{\sin \varphi_{12}} = OP_2 = \frac{d^*_{23} \sin \tau}{\sin \varphi_{23}},$$

whence,

$$\frac{\sin \sigma - \sin \tau}{\sin \sigma + \sin \tau} = \frac{d^*_{23} \sin \varphi_{12} - d^*_{12} \sin \varphi_{23}}{d^*_{23} \sin \varphi_{12} + d^*_{12} \sin \varphi_{23}}.$$

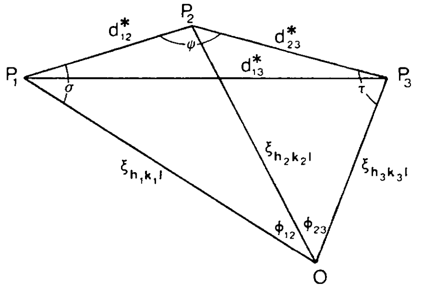

Fig. 2. Quadrilateral used for the determination of the ξ's.

Table 1. *Data for copper*(II) *sulphate pentahydrate*

Cell parameters determined by Brooker & Nuffield (1966)

$a = 6{\cdot}122,\ b = 10{\cdot}695,\ c = 5{\cdot}962$ Å, $\alpha = 97{\cdot}58, \beta = 107{\cdot}17, \gamma = 77{\cdot}55°$
$a^* = 0{\cdot}1740, b^* = 0{\cdot}0960, c^* = 0{\cdot}1760$ Å^{-1}, $\alpha^* = 85{\cdot}80, \beta^* = 74{\cdot}00, \gamma^* = 100{\cdot}75°$

(*a*) Cell parameters obtained from φ measurements using three reflections

Data from Hulme's (1966) Table 2					Results		
h k l	φ_m	*h k l*	φ_m	*h k l*	c^*	α^*	β^*
1 0 1	81·0	0 2 1	83·8	$\bar{1}$ 0 1	0·1760	86·1	73·9
1 0 1	85·5	0 3 1	79·5	$\bar{1}$ 0 1	0·1760	86·2	73·9
$\bar{2}$ $\bar{1}$ 1	103·0	0 3 1	108·0	1 $\bar{1}$ 1	0·1756	85·7	74·5
$\bar{1}$ 1 1	71·0	$\bar{2}$ $\bar{2}$ 1	84·2	$\bar{1}$ 2 1	0·1758	84·7	74·7
1 $\bar{1}$ 1	103·0	0 2 1	83·8	$\bar{1}$ 0 1	0·1762	86·0	73·7
0 3 1	79·5	$\bar{1}$ 0 1	85·0	$\bar{1}$ $\bar{3}$ 1	0·1765	86·2	73·3

(*b*) Cell parameters calculated from ξ derived from Brooker & Nuffield's data

h k l	ξ^2	$\xi^2_{hkl} - \xi^2_{hk0}$	c^*	α^*	β^*
3 8 0	0·7128	0·0732			
3 8 1	0·7860	(0·073)	0·1759	85·81	73·99
2 $\bar{7}$ 0	0·6599	0·0193	(0·1758)	(85·79)	(74·06)
2 $\bar{7}$ 1	0·6792	(0·019)			
$\bar{6}$ $\bar{2}$ 0	1·0520	−0·1034			
$\bar{6}$ $\bar{2}$ 1	0·9486	(−0·103)			

Let

$$\tan \rho = \frac{d^*_{12} \sin \varphi_{23}}{d^*_{23} \sin \varphi_{12}},$$

then,

$$\tan \frac{\sigma - \tau}{2} = \tan (45 - \rho) \tan \frac{\sigma + \tau}{2}. \quad (9)$$

The angles σ and τ can be calculated from their sum (8) and difference (9). Now, with three elements known in both triangles P_2P_1O and P_2P_3O, the ξ's can be calculated and c^*, β^* and α^* derived from (7).

Examples

The parameters c^*, α^* and β^* of copper(II) sulphate pentahydrate have been calculated for both methods described above. The data for this compound given by Brooker & Nuffield (1966) are quoted below. In Table 1(*a*), the values of φ_m have been taken from Hulme's (1966) Table 2 and these lead to estimates that are generally within ±0·3% for c^* and ±0·5° for α^* and β^*. In Table 1(*b*), the values of ξ have been calculated from Brooker & Nuffield data. The values of $(\xi^2_{hkl} - \xi^2_{hk0})$ in parentheses have been rounded and give the parameters in parentheses: evidently, this method of estimation is not unduly sensitive to experimental errors.

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Diffractometric angles for rotation around the diffraction vector.

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Abstract

It is shown that the azimuthal angle ψ of rotation around the diffraction vector and the four angles χ_0, χ, φ' and $90 - \omega$, all belong to one right spherical triangle from which the new relations $\sin \psi = \sin \chi \sin \varphi'$ and $\cos \varphi' = \cos \omega \cos \psi$ are derived. These angles are in fact related by ten trigonometric equations which can also be derived by matrix methods. The setting angles for a full ψ rotation of 360° are easily determined when results of both methods are used together.

Several methods have been proposed to calculate the setting angles ω, χ and φ for a given ψ rotation around the

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