A GRAPHICAL INTERPRETATION OF *X*-RAY DIFFRACTION DATA USING POLAR CO-ORDINATES

WILLIAM C. WHITE,
National Institute of Health, Washington, D. C.

When the symmetry of the crystal studied by the Debye-Scherrer powder diffraction method is unknown or is incompletely known, it is sometimes advantageous to use graphical methods as introduced by Hull and Davey.[1] Even in the simple case when applied to crystals of the cubic system, the preparation of the graph for values of the unit parameter as a function of the angle of diffraction involves considerable labor. The form of Bragg relation, $n\lambda = 2d \sin\theta$, suggests the use of polar co-ordinates.

For crystals of the cubic system,

$$d = \frac{a_0}{\sqrt{h^2 + k^2 + l^2}}$$

and the Bragg equation may be rewritten:

$$\sin\theta = \frac{\frac{\lambda}{2}\sqrt{h^2 + k^2 + l^2}\cdot n}{a_0} \qquad (1)$$

where, in the usual notation λ is the wave length of the incident radiation, h, k, and l are the Miller indices of the reflecting planes, n is the order of reflection, a_0 the unit cell spacing and θ is $\frac{1}{2}$ the angle between the incident and the diffracted beam.

Let a_0 be the polar distance from the origin (radius vector) of a polar co-ordinate system and let θ be the angular co-ordinate. Then $\sin\theta = y/a_0$, where y is the perpendicular distance above the axis $\theta = 0°$ i.e., a_0 is the hypotenuse and y is the side opposite the angle θ in a right triangle. From equation 1,

$$y = \frac{\lambda}{2}\sqrt{h^2 + k^2 + l^2}\cdot n$$

The value of y can therefore be calculated for all possible values of $\sqrt{h^2+k^2+l^2}\cdot n$ at two points and plotted by connecting these points with a straight line. Or in a more simple manner by plotting y at $\theta = 90°$ where from equation 1,

[1] Hull, A. W., and Davey, W. P., *Phys. Rev.*, **17**, 549 (1921).

$$y = \frac{\lambda}{2} \sqrt{h^2 + k^2 + l^2} \cdot n = a_0$$

Then lines can be drawn parallel to the axis $\theta = 0°$ from these points. Table 1 gives the values of y at $\theta = 90°$ for the values of h, k, and l used in the accompanying illustrations and for CuKα, radiation ($\lambda = 1.5374$ Å).

TABLE I

$(h\ k\ l)$	$y=\frac{\lambda}{2}\sqrt{h^2+k^2+l^2}$ $\sqrt{h^2+k^2+l^2}$	y
100	$\sqrt{1}$	0.769
110	$\sqrt{2}$	1.087
111	$\sqrt{3}$	1.331
200	$\sqrt{4}$	1.537
210	$\sqrt{5}$	1.719
211	$\sqrt{6}$	1.883
220	$\sqrt{8}$	2.174
221	$\sqrt{9}$	2.306
310	$\sqrt{10}$	2.431
311	$\sqrt{11}$	2.549
222	$\sqrt{12}$	2.663
320	$\sqrt{13}$	2.771
321	$\sqrt{14}$	2.875
400	$\sqrt{16}$	3.074
410	$\sqrt{17}$	3.169
411	$\sqrt{18}$	3.261
331	$\sqrt{19}$	3.350

In preparing the graph, only one quadrant of the polar co-ordinate system is used. In practice the polar distances (a_0) and the horizontal lines for the values of $\lambda/2\sqrt{h^2+k^2+l^2} \cdot n$ may be drawn on tracing paper. Fig. 1 shows a portion of the graph with the indices of the reflecting planes designated at the right.

For the diffraction pattern studied, the angular values of θ are plotted on a separate sheet of paper. Figure 2 is a plot of the diffraction pattern of NiO from values of θ taken from a pattern obtained with a commercial x-ray diffraction unit, using the "powder wedge" technique. The graph on tracing paper is placed over this sheet and the different values of θ plotted will intersect the values of $\lambda/2\sqrt{h^2+k^2+l^2} \cdot n$ at some constant polar distance a_0 as shown in Fig. 3 where the values of θ in Fig. 2 intersect the horizontal lines of Fig. 1 at a constant value of a_0 at approximately 4.18 which is in close agreement with the accepted value of 4.17.[2]

[2] Wyckoff, R. W. G., The Structure of Crystals, Chemical Catalog Co., Inc., New York, Second Edition (1931), p. 227.

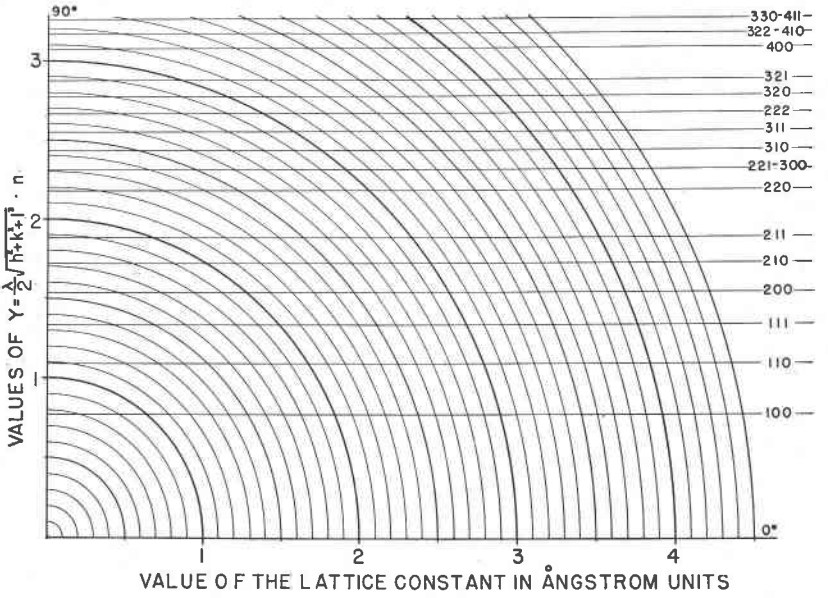

FIG. 1

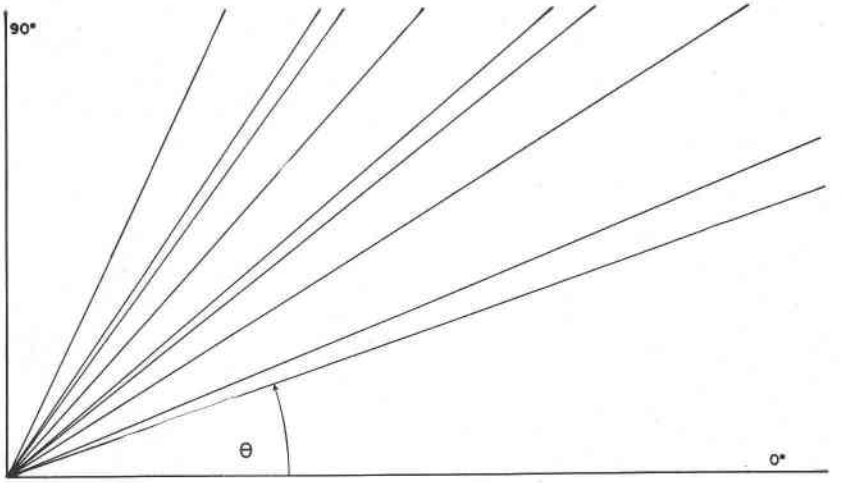

FIG. 2. Graphical representation of the values of θ from the powder diffraction pattern of NiO. The values of θ from a powder camera of 14.32 cm. diameter with $Cu_{K\alpha}$ radiation ($\lambda = 1.537$ Å) are: 18.6°, 21.6°, 31.4°, 37.7°, 39.7°, 47.5°, 53.4°, 55.4° and 64.4°.

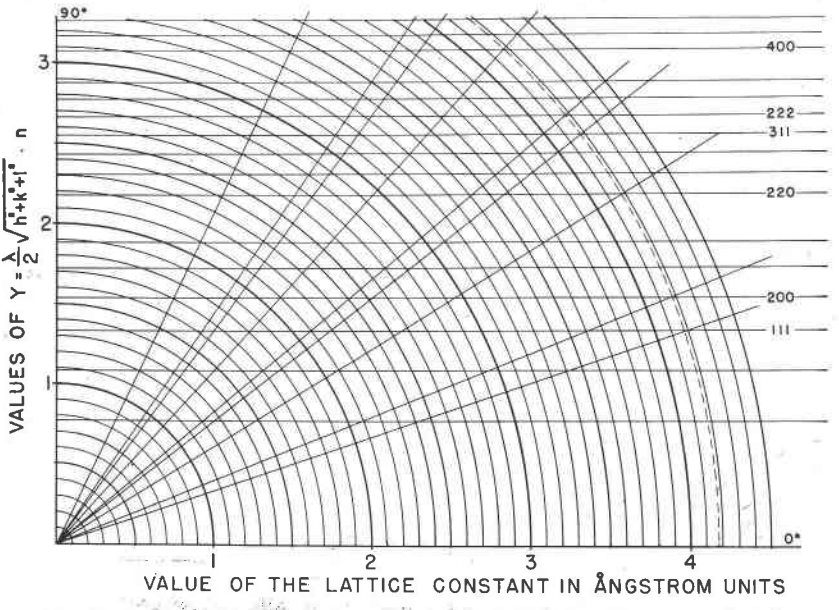

FIG. 3. Solution of the diffraction pattern of NiO giving a value of a_0 at 4.18 Å.

By noting which values of the expression $\lambda/2\sqrt{h^2+k^2+l^2}\cdot n$ must be omitted to give a constant value of a_0, the space lattice may readily be determined. For example, reflections occur for all values (h, k, l) for a simple cube, (h, k, l) all even or all odd for a face-centered cube, $h+k+l$ even for a body-centered cube. In the case of NiO illustrated in Fig. 3, it is seen that reflections occur for values of (h, k, l) equal to (111), (200), (220), (311), (222), (400), (331), etc. This, of course, corresponds to the reflections of a face-centered cubic lattice.

Although this polar co-ordinate method does not directly give a unique solution to other than cubic crystals, it is useful in the interpretation of tetragonal and hexagonal crystals. For instance, it is possible to draw graphs for $(h, k, 0)$ planes and by omitting some values of θ to obtain constant values of a_0. These give an indication of the possible values of the lattice constants which can be checked mathematically. The writer is attempting to extend the usefulness of this method to other crystal systems.