A note on the structure of tungsten carbide.

By JANUSZ LECIEJEWICZ, *Institute of Nuclear Research, Warszawa* 9, *Poland*

The B_h(WC) type of structure was hitherto described in two ways:

either D_{6h}^1, $P6/mmm$, with

| 1 W: | in 1 a | 0, 0, 0 . |

1 C at random in 2 d $\frac{1}{3}, \frac{2}{3}, \frac{1}{2}$; $\frac{2}{3}, \frac{1}{3}, \frac{1}{2}$.

or D_{3h}^1, $P\bar{6}m2$, with

1 W:	in 1 a	0, 0, 0 .
1 C:	in 1 d	$\frac{1}{3}, \frac{2}{3}, \frac{1}{2}$.
	or 1 f	$\frac{2}{3}, \frac{1}{3}, \frac{1}{2}$.

The lattice constants are:

$$a = 2\cdot9065, \quad c = 2\cdot8366 \text{ Å} \quad \text{(Pearson, 1958)} .$$

These alternatives can be readily distinguished using the neutron-diffraction method since the coherent scattering amplitude of thermal neutrons for carbon is $0\cdot66.10^{-12}$ cm. as compared with $0\cdot47.10^{-12}$ cm. for tungsten.

The experiment was carried out using $1\cdot41$ Å neutrons reflected from an Al monochromator cut along the (100) plane. The proportion of second-order radiation in the monochromatic beam was found to be 5%. Measurements up to $2\theta = 80°$ were made automatically on the neutron spectrometer designed by Blinowski (1958).
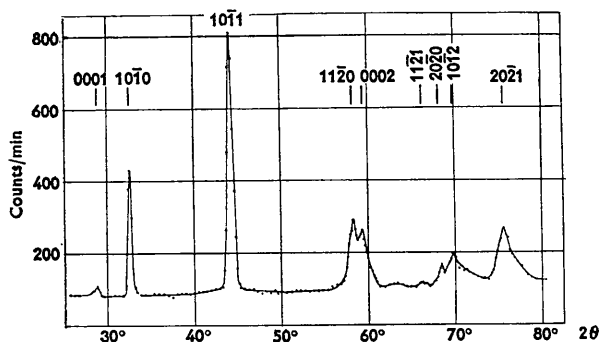

Fig. 1. Neutron diffraction pattern of WC.

Counts were taken each 10′ of scattering angle in the peak area and each 30′ on the background line. The counting time was 14 min. WC powder, 2μ grain size, analytically and X-ray controlled, contained in an aluminium tube 15 mm. in diameter was used. A neutron-diffraction pattern of WC is shown on Fig. 1. A summary of observed and calculated intensities is given in Table 1.

Table 1. *Neutron diffraction data for* WC

$hkil$	I_o	I_c for space group $P6/mmm$	I_c for space group $P\bar{6}m2$
0001	1	0·67	0·67
10$\bar{1}$0	13	0·79	13·10
10$\bar{1}$1	43	28·90	42·70
11$\bar{2}$0	16	18·40	18·40
0002	8	5·88	5·88
11$\bar{2}$1	1	0·95	0·95
20$\bar{2}$0	4	0·23	3·57
10$\bar{1}$2	8	0·45	7·36
20$\bar{2}$1	18	13·60	19·25

The R factor defined as:

$$R = \Sigma(I_o - I_c)/\Sigma I_o$$

is 0·47 for $P6/mmm$ and 0·07 for $P\bar{6}m2$. This leaves no doubt that the space group for B_h type of structure should be $P\bar{6}m2$ with:

| 1 W: | in 1 a | 0, 0, 0 . |
| 1 C: | in 1 f | $\frac{2}{3}, \frac{1}{3}, \frac{1}{2}$. |

For this case the temperature factor as determined from the slope of the plot of logarithm of I_o/I_c versus $\sin^2 \theta/\lambda^2$ is $2B = 2\cdot22$ Å².

References

BLINOWSKI, K. (1958). (Unpublished.)

PEARSON, W. B. (1958). *A Handbook of Lattice Spacings and Structures of Metals and Alloys.* London: Pergamon Press.

On the setting of crystals for X-ray diffraction work.

ISABEL GARAYCOCHEA and HILDA CID-DRESDNER, *Centro de Investigaciones de Cristalografía, Instituto de Física y Matemáticas, Universidad de Chile, Casilla* 2777, *Santiago, Chile*

While attempting to orientate a cleavage fragment of a highly absorbing mineral in an X-ray diffraction camera, we were faced with a small difficulty which led to an improvement in the technique used for setting a given zone axis parallel to the rotation axis of the camera.

The technique usually employed when the zero layer line is recognizable on the film and the misorientation is small is that of Weisz & Cole (1948) which makes use

of a double oscillation (or a double Laue) photograph in combination with the formulae of Hendershot (1937):

$$d_\perp = R \sin 2\theta \sin i_\perp$$
$$d_\parallel = R(1 - \cos 2\theta) \sin i_\parallel, \tag{1}$$

where R is the radius of the cylindrical camera; $i_\perp$ and $i_\parallel$ are the angular errors on the two arcs of the goniometer

head, respectively transverse and parallel to the X-ray beam; $d_\perp$ and $d_\parallel$ are the corresponding displacements of a given diffraction spot from the equator of the film due to $i_\perp$ and $i_\parallel$; and θ is the corresponding Bragg angle.

On one side of the film the two displacements add and on the other side they subtract, and the usual procedure is based on measurements made with two pairs of spots having approximately the same Bragg angle θ, one pair on each side of the film.

It happened that in our oscillation photographs it was impossible to obtain such pairs of spots with approximately the same Bragg angle and, furthermore, in some cases useful spots would only appear on one side of the film, that is, on one half of the equatorial layer line. The following procedure proved quite successful in overcoming this difficulty.

For any given spot in the approximately orientated zero layer line one can write:

$$(1-\cos 2\theta)\sin i_\parallel \pm \sin 2\theta \sin i_\perp = D/R , \qquad (2)$$

where D is one half of the distance between two diffraction spots due to the two successive oscillation (or Laue) photographs at 180° to each other; these two spots would coincide if there were no setting errors. The other symbols have the same meaning as in (1).

As for every such pair of spots D and θ can be measured, and R is an instrumental constant, one can write as many equations (2) as pairs of spots are considered, whatever their value of θ and even in the extreme case where they are all on one of the two halves of the zero layer line.

Two pairs of spots can be used to determine the values of $\sin i_\parallel$ and $\sin i_\perp$, but if more than two pairs are measured a more accurate result can be obtained by a least-squares calculation.

However, in practice, it is best to solve the problem graphically. Each equation (2) represents a straight line if $\sin i_\parallel$ and $\sin i_\perp$ are taken as variables along the coordinate axes. These straight lines can be drawn very quickly as their intersections with the coordinate axes can be determined quite easily. All these straight lines —three or four should be sufficient—intersect or nearly intersect at a point that gives the values of $\sin i_\parallel$ and $\sin i_\perp$.

To avoid mistakes, one should be very strict in observing certain sign conventions. These could be for instance:

(a) $i_\perp$ is positive when, looking against the direction of travel of the incident X-ray beam, the zone axis appears to be tilted in a positive angle relative to the rotation axis of the camera, within the plane perpendicular to the X-ray beam.

(b) $i_\parallel$ is positive when an observer, placed in such a way as to have the X-ray beam passing in front of him from right to left, sees the zone axis tilted in a positive angle relative to the camera axis, within the plane determined by the latter and the X-ray beam.

(c) D is positive or negative when the stronger of the two spots that form a pair (corresponding to the longer exposure time) is above or below the equator of the film, respectively.

(d) It follows from (c) that the conventions adopted in (a) and (b) apply when the goniometer of the camera is in the position corresponding to the longer exposure, and that the corrections have to be referred to this position.

(e) If the film is observed looking against the direction of the incident beam, and the centre of the film corresponds to $\theta = 0°$, the positive sign must be used for the second term of equation (2) for spots on the right, and the negative sign for spots on the left.
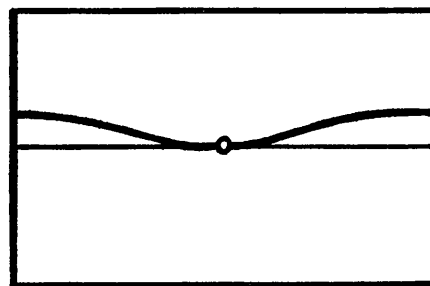
(a)
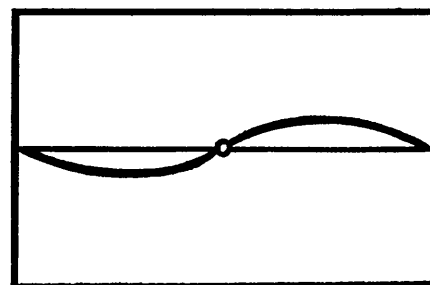
(b)

Fig. 1. (a) Zero layer line for $i_\perp = 0$, $i_\parallel > 0$;
(b) Zero layer line for $i_\perp > 0$, $i_\parallel = 0$.

With these rules in mind, Fig. 1 shows the separate effects of a positive $i_\parallel$ and a positive $i_\perp$. All other cases follow easily. In order to help newcomers to X-ray crystallography in finding the most suitable technique for setting their crystals for their particular case, we give below a list of references on this subject.

We are indebted to Dr N. Joel for suggesting the method outlined in this note.

References

BAIRSTO, A. (1948). *J. Sci. Instrum.* **25**, 215.
BUNN, C. W. (1946). *Chemical Crystallography*, p. 173. Oxford: University Press.
DRAGSDORF, R. D. (1953). *Acta Cryst.* **6**, 220.
HENDERSHOT, O. P. (1937). *Rev. Sci. Instrum.* **8**, 436.
JEFFERY, J. W. (1949). *Acta Cryst.* **2**, 15.
JEFFERY, J. W. (1949). *J. Sci. Instrum.* **26**, 42.
JERSLEV, B. (1951). *Acta Cryst.* **4**, 472.
KRATKY, O. & KREBS, G. (1936). *Z. Kristallogr.* **95**, 253.
LONSDALE, K. (1947). *J. Sci. Instrum.* **24**, 108.
MACKAY, A. L. (1952). *Acta Cryst.* **5**, 691.
ROOF, R. B. (1955). *Acta Cryst.* **8**, 431.
SMITH, J. V. (1952). *Acta Cryst.* **5**, 723.
WEISZ, O. & COLE, W. F. (1948). *J. Sci. Instrum.* **25**, 213.
WINCHELL, H. (1950). *Acta Cryst.* **3**, 396.