beam, the reflected beam and the Weissenberg axis, respectively, and let $\mathbf{z}$ make an obtuse angle with $\mathbf{s}_0$. It follows that

$$\begin{aligned}\cos\tau &= (\mathbf{s}\times\mathbf{s}_0)\cdot(\mathbf{z}\times\mathbf{s}_0)/\{|\sin(\mathbf{s},\mathbf{s}_0)||\sin(\mathbf{z},\mathbf{s}_0)|\}\\ &= (\mathbf{s}\times\mathbf{s}_0)\cdot(\mathbf{z}\times\mathbf{s}_0)/\{\sin 2\theta_s\cos\mu\}\,.\end{aligned}$$

(Note that μ is the complement of the acute angle between $-\mathbf{z}$ and $\mathbf{s}_0$.) By means of a vector identity, the numerator is transformed to

$$\begin{aligned}(\mathbf{s}\times\mathbf{s}_0)\cdot(\mathbf{z}\times\mathbf{s}_0) &= (\mathbf{s}_0\cdot\mathbf{s}_0)(\mathbf{s}\cdot\mathbf{z})-(\mathbf{s}_0\cdot\mathbf{z})(\mathbf{s}\cdot\mathbf{s}_0)\\ &= \cos(\mathbf{s},\mathbf{z})-\cos(\mathbf{s}_0,\mathbf{z})\cos(\mathbf{s},\mathbf{s}_0)\\ &= \sin\nu-\sin\mu\cos 2\theta_s\,.\end{aligned}$$

Following Buerger (1942), μ and ν are measured in the same direction from the equatorial plane. Hence in the usual experimental arrangements, as in Fig. 1, μ and $\sin\mu$ take negative (or zero) values. Thus the relations

$$\cos\tau=(\sin\nu-\sin\mu\cos 2\theta_s)/(\sin 2\theta_s\cos\mu)$$

and

$$\varrho=\tau+\sigma$$

yield the desired angle ϱ.

The special cases most frequently met in practice are the four combinations of the following two conditions:

a. $\sigma=\pi/2$ or $\sigma=0$.
b. $\mu=-\nu$ (equi-inclination) or $\mu=0$ (normal incidence).

For these, the expressions giving ϱ and P are the following:

$\sigma=\pi/2,\ \mu=-\nu$:

$$\begin{aligned}\sin\varrho &= \tan\nu\operatorname{ctn}\theta_s\\ (1+\cos^2 2\theta_m)P &= \cos^2 2\theta_m(1-\cos^2\nu\sin^2\Upsilon)\\ &\quad +1-\cos^2\nu\sin^2\nu(1+\cos\Upsilon)^2\,.\end{aligned}$$

$\sigma=\pi/2,\ \mu=0$:

$$\begin{aligned}\sin\varrho &= \sin\nu\csc 2\theta_s\\ (1+\cos^2 2\theta_m)P &= \cos^2 2\theta_m+\cos^2\nu(1-\cos^2 2\theta_m\sin^2\Upsilon)\,.\end{aligned}$$

$\sigma=0,\ \mu=-\nu$:

$$\begin{aligned}\cos\varrho &= \tan\nu\operatorname{ctn}\theta_s\,.\\ (1+\cos^2 2\theta_m)P &= \cos^2 2\theta_m[1-\sin^2\nu\cos^2\nu(1+\cos\Upsilon)^2]\\ &\quad +1-\cos^2\nu\sin^2\Upsilon.\end{aligned}$$

$\sigma=0,\ \mu=0$:

$$\begin{aligned}\cos\varrho &= \sin\nu\csc 2\theta_s\,.\\ (1+\cos^2 2\theta_m)P &= 1+\cos^2\nu(\cos^2 2\theta_m-\sin^2\Upsilon)\,.\end{aligned}$$

The relations $\cos\theta=\cos\nu\cos\Upsilon/2$ and $\cos 2\theta=\cos\nu\cos\Upsilon$ for the equi-inclination and normal incidence cases, respectively, have been used in deriving these expressions.

Whittaker (1953) has given an expression for P in generally inclined-beam Weissenberg geometry without stating the orientation of the instrumental axis to which it applies. We find that it is equivalent to our expressions for $\sigma=\pi/2$. Bond (1959) has recently given the reduction of Whittaker's expression for equi-inclination geometry and has combined this factor with the Lorentz correction.

For a given level in the normal incidence method, the angle ϱ takes values in the range σ to $\sigma+(\pi/2)-\nu$ as Υ ranges from 0 to $\pi/2$. A contrary statement by Azaroff (1955) that in this method ϱ is equal to ν, constant for a given level, is evidently in error.

In the case of the precession method, Azaroff (1955) has pointed out that both ϱ and $2\theta_s$ are related in a simple way to co-ordinates on the film. The polarization factor is then easily obtained from his original expression.

References

AZAROFF, L. V. (1955). *Acta Cryst.* **8**, 701.
AZAROFF, L. V. (1956). *Acta Cryst.* **9**, 315.
BOND, W. L. (1959). *Acta Cryst.* **12**, 375.
BUERGER, M. J. (1942). *X-ray Crystallography*, pp. 252 and 300. New York: Wiley.
WHITTAKER, E. J. W. (1953). *Acta Cryst.* **6**, 222.

Acta Cryst. (1960). **13**, 271

A new polymorph of boron.*

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Boron has long been known to exist in several polymorphic modifications, but the systematic study of the structures of these polymorphs was not begun until relatively recently. Hoard *et al.* (1958) have determined the crystal structure of a tetragonal form of boron; the unit-cell dimensions are $a=8{\cdot}75$ and $c=5{\cdot}06$ Å. The cell contains 50 atoms. Sands & Hoard (1957) also reported a rhombohedral form of boron; $a=10{\cdot}12$ Å and $\alpha=65°\ 28'$. The triply primitive hexagonal cell to which this rhombohedral cell may be referred has axial dimensions of $a=10{\cdot}95$ and $c=23{\cdot}73$ Å. The primitive rhombohedral cell contains 108 atoms. More recently, McCarty *et al.* (1958) reported another rhombohedral form of boron; $a=5{\cdot}057$ Å and $\alpha=58°\ 4'$. This cell contains only 12 atoms; the dimensions of the hexagonal cell to which it can be referred are: $a=4{\cdot}908$ and $c=12{\cdot}567$ Å.

All three polymorphs mentioned above have been studied by single crystal methods. Several additional modifications have been reported, based on studies of polycrystalline specimens (Naray-Szabo & Tobias, 1949; Langrenaudie, 1954; Rollier, 1953; Laubengayer *et al.* 1943).

Recently, still another polymorphs was detected in our laboratories.† Specimens were prepared by the

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† The specimen was prepared by one of us (C.T.) at Experiment Inc. Richmond, Va.; X-ray studies were carried out at the Polytechnic Institute of Brooklyn.

Table 1. *Powder diffraction data: Boron*

d (Å)	I/I_0	hkl	d (Å)	I/I_0	hkl	d (Å)	I/I_0	hkl
8·23	4	101	3·46	3	221	2·638	18	313, 115
$7{\cdot}15_5$	8	110	3·358	35	104	2·608	10	322
6·37	30	111	3·266	14	213	2·516	10	224
5·80	5	102	3·191	3	222	2·456	15	410
5·04	100	112				2·442	25	304
		200	3·171	3	114	2·411	18	411, 323
4·76	3	201	3·125	2	311	2·400	16	215
$4{\cdot}52_5$	5	210	3·044	4	302			
4·30	80	211	2·925	5	312	2·386	9	330
4·114	85	202	2·901	4	204	2·380	8	402
3·935	30	113				2·354	5	331
			2·846	3	223	2·263	2	420, 332
3·81	45	212	2·806	3	320	2·233	10	421, 403
			2·746	35	303, 321			

Additional lines not listed.

reduction of BBr_3 by H_2 on incandescent tungsten and rhenium filaments of small diameter. The initial filament diameter was 0·025 mm. and the final diameter of the boron deposit was about 1 mm. The deposition was carried out at atmospheric pressure and at a temperature of about 1540 °K. Contamination by stopcock greases was prevented by using apparatus of Teflon and Pyrex-glass construction without stopcock greases.

Wet chemical analysis of several similarly prepared samples for total boron indicated a boron content exceeding 99% by weight. The main impurity in the boron rods came from the 0·025 mm. diameter metal core and amounted to about 0·7% by weight. From emission spectrographic analysis, aluminum was estimated at 0·005% or less. Traces of other impurities (Ba, Sr, Ca, Cu, Fe, Mg, and Si) were also found which amounted to a total of about 0·02%. The filament method is generally known to produce boron of high purity (Powell *et al.*, 1955). The boron rods appeared black when viewed by reflected light but thin sections appeared red when viewed by transmitted light. Red or reddish crystals of the simple rhombohedral form have been observed (McCarty *et al.* (1958).

The density of the boron was measured by a flotation method and found to be $2{\cdot}364 \pm 0{\cdot}005$ g.cm.$^{-3}$ at 23 °C. Density measurements on rod segments and powdered samples indicated that the effect of rhenium or tungsten, if any, on the above value was less than the estimated experimental uncertainty. The unit cell of this material is tetragonal, pseudo-cubic, with $a = 10{\cdot}12$ and $c = 14{\cdot}14$ Å, both $\pm 0{\cdot}02$ Å. The dimensions of this unit cell are similar to those which have been reported for a tetragonal modification of AlB_1 (Halla & Weil, 1939). However, since spectrographic analyses indicate that only trace amounts of aluminum are present in the tetragonal boron, it is evident that the two phases are distinct in spite of the dimensional similarity of the unit cells. When specimens of tetragonal boron were melted by resistance heating and subsequently cooled, they were found to have transformed to the rhombohedral modification reported by Sands & Hoard (1957).

Powder diffraction data are listed in Table 1; d spacings were computed from patterns recorded on a diffractometer using filtered Co and Cu radiations. Scanning speeds of $\frac{1}{8}$ and $\frac{1}{4}°$ per minute were used in conjunction with fine (0·003″) receiving slits, in order to maximize resolution of closely spaced lines. The pseudo-cubic character of the material was clearly revealed by the splitting or broadening of a number of lines.

The measured density of 2·364 g.cm.$^{-3}$ indicates that the tetragonal unit cell contains approximately 192 atoms (calc. 190·6), possibly grouped in 16 icosahedra. Efforts are being made to grow single crystals for X-ray diffraction study.

References

Halla, F. & Weil, R. (1939). *Z. Kristallogr.* **101**, 435.

Hoard, J. L., Hughes, R. E. & Sands, D. E. (1958). *J. Amer. Chem. Soc.* **80**, 4507.

Langrenaudie, J. (1953). *J. Chim. Phys.* **50**, 629; ca **48**, 4903 (1954).

Laubengayer, A. W., Hurd, D. T., Newkirk, A. E. & Hoard, J. L. (1943). *J. Amer. Chem. Soc.* **65**, 1924.

McCarty, L. V., Kasper, J. S., Horn, F. H., Decker, B. F. & Newkirk, A. E. (1958). *J. Amer. Chem. Soc.* **80**, 2592.

Naray-Szabo, St. v. & Tobias, C. W. (1949). *J. Amer. Chem. Soc.* **71**, 1882.

Powell, C. F., Campbell, I. E. & Gonser, B. W. (1955). *Vapor Plating*, Chapter 5. New York: Wiley.

Rollier, M. A. (1947). *Proc. XI Congr. Pure Applied Chem.* **5**, 935, publ. 1953; Ca **47**, 11941h (1953).

Sands, D. E. & Hoard, J. L. (1957). *J. Amer. Chem. Soc.* **79**, 5582.