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The polarization correction for upper level geometry using crystal monochromatized radiation.

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The polarization correction for crystal monochromatized radiation, when expressed in terms of angles referred to an instrumental polar axis, depends on the orientation of this axis with respect to the plane of incidence at the monochromator. In several presentations of the form of this correction (Whittaker, 1953; Azaroff, 1955, 1956; Bond, 1959), no specification of this variable was made, although a particular value was evidently assumed. We present here an evaluation of the polarization factor taking explicit account of the variable in question.

Azaroff (1955) has given the following general expression for the polarization correction P when crystal-monochromatized radiation is employed:

$$(1+\cos^2 2\theta_m)P = (\cos^2 2\theta_m \cos^2 \varrho + \sin^2 \varrho) \cos^2 2\theta_s + \cos^2 2\theta_m \sin^2 \varrho + \cos^2 \varrho \ .$$

Here θ_m is the Bragg angle of the monochromator, θ_s that of the sample reflection, and ϱ the angle between the normals to the planes of incidence at the monochromator and sample.

Application of this expression to a given technique of measurement is essentially the task of evaluating the angle ϱ in terms of convenient variables. For inclined-axis Weissenberg measurements (see Fig. 1), these may be taken as the latitude angles μ and ν of the primary monochromatic beam and the reflected beam with respect to the equatorial plane of the measuring instrument, together with the Bragg angle of the sample reflection θ_s. The latter can be re-expressed, if desired, in terms of the latitude ν and azimuth Υ of the reflected beam. The orientation of the instrumental axis may be specified by means of a single angle σ, which we here define as the angle between the normals to the monochromator plane of incidence and the plane generated by the Weissenberg axis and the primary monochromatic beam. Fig. 1 shows the geometric elements in an isometric view and projected onto the plane normal to the primary monochromatic beam. The sign of σ is to be taken in such a way that the relation $\varrho = \sigma + \tau$ holds for the acute angles τ and σ as shown in the figure. Here τ is the angle between normals to the planes generated by the primary monochromatic beam with the Weissenberg axis and with the reflected beam.

The evaluation of τ will thus yield the desired angle ϱ. For this purpose, we define unit vectors $\mathbf{s}_0$, $\mathbf{s}$, and $\mathbf{z}$ along the directions of the primary monochromatic

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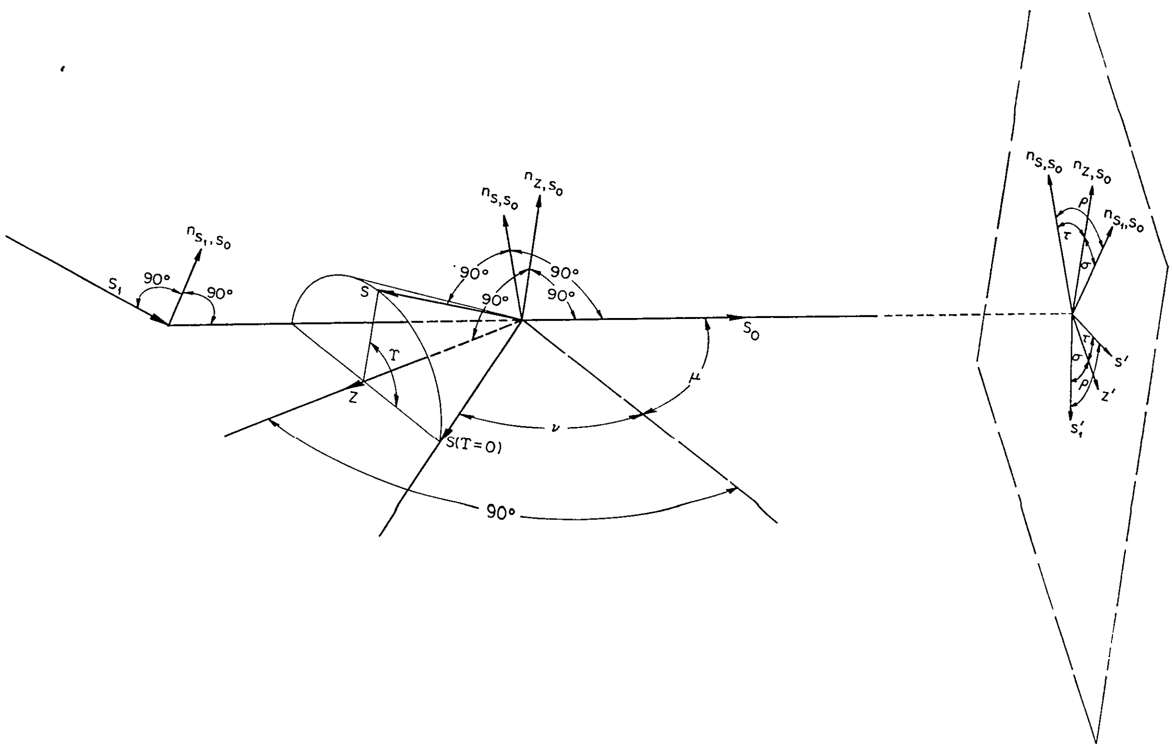

Fig. 1. Isometric drawing of the geometric elements of inclined-axis Weissenberg measurements, and a projection onto the plane normal to $\mathbf{s}_0$. Shown here is the equi-inclination case, so that the angle μ is negative. $\mathbf{s}'$, $\mathbf{z}'$, and $\mathbf{s}_1'$ are the projections of $\mathbf{s}$, $\mathbf{z}$, and $\mathbf{s}_1$, while $\mathbf{n}_{s_1,s_0}$ $\mathbf{n}_{s,s_0}$, and $\mathbf{n}_{z,s_0}$ are the common normals to $\mathbf{s}_1$ and $\mathbf{s}_0$, $\mathbf{s}$ and $\mathbf{s}_0$, and $\mathbf{z}$ and $\mathbf{s}_0$, respectively.

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

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

(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

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

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:

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

$$\sin\varrho=\tan\nu\,\mathrm{ctn}\,\theta_s$$
$$(1+\cos^2 2\theta_m)P=\cos^2 2\theta_m(1-\cos^2\nu\sin^2\Upsilon)+1-\cos^2\nu\sin^2\nu(1+\cos\Upsilon)^2\,.$$

$\underline{\sigma=\pi/2,\ \mu=0:}$

$$\sin\varrho=\sin\nu\csc 2\theta_s$$
$$(1+\cos^2 2\theta_m)P=\cos^2 2\theta_m+\cos^2\nu(1-\cos^2 2\theta_m\sin^2\Upsilon)\,.$$

$\underline{\sigma=0,\ \mu=-\nu:}$

$$\cos\varrho=\tan\nu\,\mathrm{ctn}\,\theta_s\,.$$
$$(1+\cos^2 2\theta_m)P=\cos^2 2\theta_m[1-\sin^2\nu\cos^2\nu(1+\cos\Upsilon)^2]+1-\cos^2\nu\sin^2\Upsilon.$$

$\underline{\sigma=0,\ \mu=0:}$

$$\cos\varrho=\sin\nu\csc 2\theta_s\,.$$
$$(1+\cos^2 2\theta_m)P=1+\cos^2\nu(\cos^2 2\theta_m-\sin^2\Upsilon)\,.$$

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.

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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 $a=8\cdot75$ and $c=5\cdot06$ Å. The cell contains 50 atoms. Sands & Hoard (1957) also reported a rhombohedral form of boron; $a=10\cdot12$ Å and $\alpha=65°\ 28'$. The triply primitive hexagonal cell to which this rhombohedral cell may be referred has axial dimensions of $a=10\cdot95$ and $c=23\cdot73$ Å. The primitive rhombohedral cell contains 108 atoms. More recently, McCarty *et al.* (1958) reported another rhombohedral form of boron; $a=5\cdot057$ Å and $\alpha=58°\ 4'$. This cell contains only 12 atoms; the dimensions of the hexagonal cell to which it can be referred are: $a=4\cdot908$ and $c=12\cdot567$ 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.